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QUANTITATIVE ASSESSMENT OF NON-METALLIC INCLUSIONS  
IN STEEL USING AN IMAGE ANALYSING COMPUTER

by

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## A B S T R A C T

Although the last few years have seen a rapid increase in instruments available for inclusions assessment, little has been done to show the technologist how to use these new and important tools in routine work, such as quality control in steelworks. The researches described in this thesis partly meets that need and discusses how one of these instruments ( the Quantimet Image Analysing Computer ) may be used effectively.

Inclusions assessment under the light microscope is subject to various errors including instrumental, sampling and sample preparation. Effective use of any microscopic technique for the determination of non-metallic inclusion contents of bulk materials is, therefore, inevitably bound up with these problems and it is the responsibility of the technologist to ensure that the above errors are controlled within the required limits. However, this demands a knowledge of not only the magnitude of errors likely to be encountered in practice, their causes and effects but also of the occurrence and distribution of non-metallic inclusions in bulk materials, such as a "micro section", a billet or an ingot of steel.

The first few sections of this thesis are devoted to the origin and classification of non-metallic inclusions commonly found in forging steels, their significance in relation to steel properties, an outline of methods for their assessment, the problems, review of previous work, and an outline of the proposed research. These are followed by sections concerned with studies undertaken to determine the magnitude of errors arising from the use of the Quantimet Image Analysing Computer, sampling and sample preparation procedures. A standard operating procedure for this instrument is suggested to minimise instrumental errors, and a technique developed for routine work described. This technique involves the use of the new Quantimet and a digital computer. A standard sample is used for calibrating the instrument and for adjusting the threshold level. Sampling and sample preparation errors are treated from a practical aspect and variations in inclusion estimates between fields, sets of fields, surfaces and adjacent samples selected from whole billets are discussed. What could actually be achieved in practice in terms of overall speed and accuracy of inclusion estimates are considered.

The next section deals with two investigations: a detailed study of distribution of oxide inclusions in two adjacent ingots of low-alloy steel and a running programme in a steelworks, the objects of which were (a) to obtain additional data on distribution of oxide inclusions in low-alloy steel in order to establish whether or not fields with high inclusion content are preferentially distributed in certain portions of the ingot, and (b) to test whether the technique developed is capable of yielding the necessary information accurately, rapidly and economically. This section also includes work carried out to determine the degree of correlation attainable between the Quantimet and other laboratory procedures, e.g. total oxygen determined by the neutron activation technique, JK count, step-test, etc.

Next, a method developed in the course of this research for improving the contrast between metallic and non-metallic phases in metallographic specimens, notably steels, is described. In this method, a thin, optically transparent, film of an intermetallic compound ( $\text{ZnSe}$ ) is deposited, in vacuo, on a diamond polished and ultrasonically cleaned surface. The conditions for maximum contrast are described and an explanation put forward for the mechanism of enhancement. Typical results, illustrated by colour photomicrographs, show the applicability of the new method in both manual and automatic inclusions assessment.

The final sections are devoted to a general discussion of results obtained in this research and the main conclusions drawn.

I certify that the work presented in this thesis has not been submitted to any other institute for the purpose of obtaining a degree.

.....  
THOMAS REGINALD ALLMAND.

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## 1.00 INTRODUCTION

### 1.10 Origin and classification of non-metallic inclusions

Non-metallic inclusions are present in varying amounts in all commercial steels. They occur in a variety of forms, sizes and shapes, and are the inevitable consequence of the methods and equipment used for the manufacture of this material. Benedicks and Loquist<sup>1</sup> were amongst the first to undertake a systematic study of inclusions in steel. They distinguished between two main types of inclusions, native and foreign. Following the same lines of reasoning, Sims<sup>2</sup> and Baeyertz<sup>3</sup> divided inclusions into two main classes, those that form in the molten or solid steel as a result of separation of oxides, sulphides, nitrides and other non-metallic compounds as a consequence of reactions occurring in molten steel or because of a change in temperature; and those inclusions that are inadvertently trapped in the steel and consist of particles of slag, refractory or other material with which the steel is in contact during melting, refining and casting. Inclusions of the first group are referred to as indigenous, endogenous or natural, and those of the second group as exogenous, adventitious or accidental. The main types of inclusions encountered in commercial steels have been discussed by Allmand<sup>4</sup> and also by Kiessling and Lange.<sup>5</sup>

One helpful factor in the identification and classification of inclusions is that the possible types of inclusions are limited by the composition and history of the steel. Thus, even though our observations of the inclusions under the microscope may not give a positive identification, a knowledge of the chemical compositions and history of how the steel was made and subsequently treated, will allow a narrowing of the possibilities so that a reasonably certain identification of inclusions is possible.

The most important characteristics used for identifying inclusions are shape, optical reflectivity, size and distribution. The shapes of inclusions

in a metallographic section can be seen under an optical microscope. If the sample was from cast steel the shape can give a clue as to whether the inclusion was precipitated from a liquid or solid steel. For example, if the shape is euhedral (regular) or skeletal, it is almost certain the inclusion phase precipitated from the liquid. The shapes of inclusions in steel after mechanical working can assist in identifying whether they are alumina, silicate or sulphide inclusions. Very few of the common inclusions found in commercial fully deoxidised steels are sufficiently plastic at hot-working temperatures to be elongated to the extent shown by the manganese sulphide and some silicate inclusions. Most inclusions are of a more refractory nature and have little tendency to deform upon hot-working.

After shape, probably the next most prominent characteristic of an inclusion is its reflectivity. In general, sulphides are more reflective than oxide and silicate inclusions. Differences in reflectivity of the inclusions arise as follows: when a beam of light travelling through air strikes the surface of another medium, such as an inclusion, part of the beam is reflected back from the surface, part of the beam enters the second medium where some light is absorbed and some is refracted because of a difference in velocity of light in the two media. However, the portion of light reflected back from the surface can vary, depending upon the wave-length of the light. An important factor to be taken into account is the reflectivity of the steel matrix, and since inclusions are frequently examined in the unetched condition, one is comparing the reflectivity of a particular inclusion with that of the highly reflecting steel matrix.

Equally important is the size of inclusions and their distribution. In general, exogenous inclusions are much larger than indigenous inclusions and, therefore, are easily identified by this quantity alone. However, the true identification of inclusions can only be made by a combination of chemical analysis and X-ray examination. The development of the electron microprobe



technique in recent years has made possible the determination of the composition of the inclusion of interest in situ, and this method is now widely used for analysing inclusions.

#### 1.20 Significance of inclusions in relation to steel properties

Much work<sup>6-40</sup> has been carried out to determine the influence of inclusions on steel properties. Our knowledge of this vast subject may be summarised as follows:

In forging and similar operations, sulphide and oxide inclusions (including silicates), when present in gross forms, can give rise to "bursting", leading to a defective product. In machining operations, manganese sulphide inclusions are beneficial, especially if they are rounded, as they act as chip breakers and allow faster machining speeds and longer tool lives to be attained. Oxide, silicate, nitride and other abrasive inclusions can be harmful, as they cause increased tool wear and sometimes breakage of the cutting edge, leading to hold-ups and thus to loss of production. Inclusions can also be an acute problem in stainless steel fabrication giving rise to poor ductility, surface finish and corrosion resistance of the finished products, especially sheets. Inclusions in highly stressed parts, such as crankshafts, ball-bearings, etc., do contribute toward fatigue failure. In welding, sulphide, silicate and aluminous inclusions in the parent material can affect weld soundness indirectly, when absorbed into the pool, and directly by promoting laminations in the unmelted heat affected zone. Sulphide inclusions have been known to contribute heavily to "hot-shortness" and also to poor ductility of cast steel. A summary of inclusion types and their effects has been made by Bridge<sup>6</sup> and is reproduced in Table 1.

When inclusion defects make it necessary to reject forged and machined components, the manufacturer can suffer severe penalties in cost and time. Obviously, detection of harmful inclusions at this late stage in the manufacturing process means loss of the complete part which has increased greatly in

value by virtue of the various forging, machining or other treatments that may have been performed on it. Failure of the part in service due to inclusions can be even worse, since it can result in the loss of the entire structure, and possibly in the loss of human life.

### 1.30 Inclusion assessment in relation to quality control

In the past 20 years, much effort has been expended in attempts to eliminate or control the quantity and type of inclusions present and thus to enhance the quality of the steel. The activity in this field has been world wide, as evidenced by the number of papers published and conferences held on this subject. The diverse conclusions and opinions expressed, however, indicate that the subject is far from well understood. This is due partly to the complex nature of the problem, and partly due to the fact that the steelmaker has been working under somewhat of a handicap in the matter of testing the quality of his products with respect to non-metallic inclusion contents.

Clearly, new steelmaking practices can only be reliably evaluated, and the finished or semifinished products satisfactorily tested for the presence of harmful inclusions, if an objective and reproducible method for assessing the inclusion content of the steel is available. In the absence of such a method, the steelmaker can tumble into many pitfalls. For example, he could draw the wrong conclusions about the effect of a particular change in steel-making practice on the amount of inclusions present; he could classify material as suitable for a particular application when actually it is not, or the reverse, and thus incur unnecessary losses. There could also be the stipulation of unnecessary specifications and the use of unrealistic testing procedures.

In the past, too much emphasis appears to have been placed on developing techniques for rapid analysis of oxygen, the common element of most oxide and silicate inclusions, and not enough work has been done to develop other

methods of determining inclusion content. However, it must be accepted that these techniques have helped to throw some light on certain aspects of the problem of production of steel free from harmful inclusions. But it has become increasingly clear in recent years that total oxygen estimates, however reliable, are not necessarily the best criteria of steel quality. To take a typical example, two steels could have the same low oxygen content of, say, 0.005% oxygen, but one of them could have a significant proportion of the oxide inclusions in a form and size that are acceptable for a particular application, then a total oxygen level of 0.005% would represent a satisfactory steel. The other steel could have a significant proportion of the oxide inclusions in a form and size that are unacceptable, so that a total oxygen content of 0.005% now represents an unsatisfactory steel.

The microscopic methods of examination of steel are the only methods that are capable of providing information about the type, size, shape and distribution of inclusions present in the material. During the last 40 years many methods for assessing inclusions have been designed and used but only a few of them have been adopted for use in comparative work, such as quality control in steelworks. These methods are the chart counts, particularly the JK count<sup>41-43</sup> which takes its name from its sponsors, Jernkontoret of Sweden.

Sewell and Wilcock<sup>44</sup> consider that at least four quantities about inclusions must be evaluated, viz. number or frequency, size, position and type. In addition, they stress that the results of any test must be reasonably reproducible so that the steelmaker can make reliable comparisons. It is generally accepted that a satisfactory quality control method should provide information about the total quantity of inclusions present, the relative quantities of the various types of inclusions, and information on their frequency and size distribution. It should also be rapid and simple in operation, and most important, capable of providing a high level of reproducibility between different operators in order to arrive at the desired

standards of cleanliness. In discussing inclusion assessment in relation to quality control, it is important to realise that the practical problem usually takes the form of determining, within a field of about  $0.5 \text{ mm}^2$  in area, whether the inclusion content exceeds some specified limit.

#### 1.40 Outline of methods for inclusion assessment

#### 1.41 Methods other than microscopic counting methods

1. Chemical methods Three general methods may be categorised according to the mode of removal of the metallic component, the acid method,<sup>45-47</sup> the halogen method<sup>48-51</sup> and the electrolytic method.<sup>52-64</sup> The acid methods of extraction depend on the solubility of the oxides in the dilute mineral acids. The methods are generally not complicated but they are slow and appear to give results only for  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$ . Furthermore, there can be appreciable loss in the non-metallic inclusions. The halogen method of extraction is based on the attack and removal of the metallic constituents through the action of halogen elements. Various procedures have been used with varying degrees of success. Probably the most widely used methods have been those in which the metallic matrix is dissolved in solutions of iodine or bromine in alcohol.<sup>65</sup> Electrolytic extraction is often preferred as a means of separating inclusions because the characteristics of anodic dissolution enable extraction to proceed under more controlled conditions favourable to the retention of inclusions than do chemical extraction. However, with electrolytic separation there must be further purification of the residue in order to obtain a complete isolation of the oxides. The main problem with all these methods is to dissolve the metallic matrix selectively without attacking the inclusions. These methods are also slow, and, therefore, are quite unsuitable for use as quality control methods. Perhaps another reason why they have not been used in routine work is because they do not give information about the size, shape or distribution of the oxides present; only their total amount and the chemical form in which they are present.

2. Ultrasonic method The ultrasonic method<sup>66-68</sup> has been used in the steel industry for detecting the presence of large inclusions (several mm. in size) or similar defects for a number of years. The method uses the penetrating power of very high frequency sound waves to detect discontinuities within the solid steel. A short pulse of high frequency electrical energy is converted by a transducer into short bursts of sound waves which travel through a coupling medium, usually water or oil, into the material under inspection. Non-metallic inclusions or similar discontinuities in the material will reflect the sound waves back to the transducer which reconverts the waves to electrical signals. Hitherto, the method has been used with only limited success for rating steel cleanliness. This approach is very attractive because (a) it is not destructive and enables large volumes of material to be assessed, for example whole billets, and (b) it is rapid. This method is currently receiving a great deal of attention both in this country and in others, but more work remains to be done before it can be developed for measuring the smaller inclusions present in steels.

3. Macro-etch method This test<sup>42</sup> is used to determine the presence of large inclusions and also their location. Tests are prepared by cutting and machining a section through the billet and etching with a suitable reagent. The etched surface is then examined visually or microscopically at a low magnification for inclusions. The method can be used to preferentially etch out certain types of inclusions.

4. Fracture test This test<sup>42</sup> is used to assess inclusions in relatively hard materials. A disc is cut from the billet or bar, nicked along its cross-section, hardened if necessary, and broken by holding one half in a vice and hitting the other half with a hammer. The fractured surfaces are then examined either visually or microscopically at low magnifications for inclusions. It is desirable that fractures be in the longitudinal direction approximately across the centre of the disc. Inclusions less than one

millimetre in length can be detected and measured by this method under favourable conditions.

5. Sulphur printing This is one of the most frequently used tests for determining the distribution of sulphide inclusions. The method also gives an indication of the presence of large oxide and silicate inclusions. A disc is cut from billet or bar, carefully machined or ground and tested by placing the prepared surface on a piece of bromide paper previously soaked in a solution of sulphuric acid in water. A contact time of 2 to 3 minutes is generally sufficient. Sulphides in the steel are attacked by the acid in the paper and results in an evolution of hydrogen sulphide gas which stains the bromide paper giving an indication of the distribution of sulphides on the surface of the sample.

6. Step-down test This test<sup>42</sup> is used to determine the presence of inclusions on the machined surface of rolled or forged steel. The test-piece is machined to specified dimensions below the surface and the surface examined for inclusions. Only those inclusions breaking the surface of the test-piece can be detected. This test gives no indication of the type of inclusions present. Under low magnifications, inclusions smaller than one millimetre in length can be detected and measured. Reproducibility of the test is generally good.

7. Magnetic method This test<sup>42</sup> is used for revealing inclusions on the surface or sub-surface of actual components or test-pieces, such as crankshafts, step-down test-pieces, etc. The general distribution of the larger inclusions only can be revealed but not the presence of inclusions within the bulk of the steel. The method is applicable only to steels which are ferro-magnetic. Inclusions down to 0.5 mm. in length may be revealed. The test must be standardised and performed by skilled personnel since the interpretation of the results requires a considerable degree of skill. The test has the advantage of speed, and large specimens can be examined, but it does not enable the type and exact nature of the inclusions to be ascertained.

8. Radioactive tracer method The radioactive tracer method<sup>69,74</sup> has been used to determine the origin of inclusions. Specific sources, such as slag or ladle refractory, are made radioactive by the introduction of a suitable radioisotope into them. The level of radioactivity in the solid steel or in the individual inclusions, if these are large enough, indicates the amount of pick-up from such sources. Whilst invaluable as a research method, the technique is not suitable as a quality control test because of the elaborate preparations generally required to operate this method.

9. Neutron activation method This technique<sup>75</sup> is comparatively new for the determination of the total oxygen content of the steel. It is reliable and very rapid and, furthermore, has the facility of examining large specimens (1 in. x 0.5 in.). Basically, this testing method depends on a system consisting of three components: a neutron generator, a gamma-ray scintillation detector and a pneumatic transfer system for the placing and removing of specimens remotely. In the neutron generator, deuterons are accelerated to a potential of 150 KeV and are permitted to strike a tritium target, thus forming a compound nucleus, as shown in Fig.1. This nucleus decays into an alpha particle and a high-energy neutron.

The high-energy neutron, on striking an oxygen-16 atom in the sample of steel, forms a second compound nucleus. This second nucleus instantly decays to a proton and a radioactive nitrogen-16 atom. The latter decays with a half-life of 7.4 seconds, emitting a beta particle and gamma rays. Measurement of this induced gamma radioactivity in the sample of steel enables the oxygen content to be determined.

The accuracy and reproducibility of this method is dependent on the oxygen content of the sample. If the oxygen content is only 10 ppm, an accuracy of  $\pm 25\%$  and reproducibility of  $\pm 20\%$  can be obtained. At 500 ppm concentration, an accuracy of  $\pm 5\%$  and reproducibility of  $\pm 1\%$  can be obtained.

The method does not distinguish between the various types of oxides that

may be present in the sample.

10. Vacuum fusion gas analysis Like the neutron activation method this technique is reliable and reasonably fast and, therefore, ideally suited for quality control work but it does not give information on size or distribution of inclusions.

11. Radiographic method In this method either gamma- or X-rays can be used to indicate the presence and approximate location of relatively large inclusions, inclusion segregates or cavities. No indication of the nature of the inclusion, or indeed, whether the defect is an inclusion, can be obtained. The method is more frequently used in detecting defects in castings than in forged material.

12. Electron micro-beam analysis Electron micro-beam analysis is a relatively new method which has been used a great deal in recent years for analysing individual inclusions. With this instrument, which was invented by Castaing,<sup>76,77</sup> a fine beam of electrons is focused at an inclusion and the characteristic X-rays emitted from that spot are analysed with an X-ray spectrometer. By using calibrated standards, the small area of inclusions under examination can be analysed, element by element, often with an accuracy of better than 2%, providing the elements in question are not too low. This method is essentially a research tool since it is slow and does not give information on the amount of inclusions present in the steel. However, an interesting instrument employing the electron micro-beam technique but with additional circuits for sizing and counting inclusions has been developed by the Tube Investments Research Laboratories. Details of this instrument have been reported by Melford.<sup>79</sup>

13. Electron microscopy Electron microscope methods are mainly used in research. The method necessitates using extraction replicas. In general, considerable difficulty is experienced when trying to extract inclusions larger than 10 microns in the replica. However, by using special extraction



techniques and equipment it has been possible to examine inclusions as large as 50 microns in replicas.

The recently developed scanning electron microscope<sup>80</sup> offers a further means of examining inclusions.

14. X-ray methods X-ray diffraction and X-ray micro-radiographic techniques can be used to give useful information about the approximate analysis of inclusions and positive identification of the phases present. Again, these methods are not suitable as routine tests because they do not give information on the amount, size or distribution of inclusions present in the steel.

#### 1.42 Microscopic counting methods

1. Chart methods The most important standard chart methods for inclusion assessment are the Fox count,<sup>81</sup> the JK count,<sup>41-43</sup> the Diergarten count,<sup>82,83</sup> and the less well known Chevrolet Motors (CM) or Walker count.<sup>84,85</sup> All these methods depend upon the examining of a certain number of fields of view on a polished specimen and comparing them with standard charts, shown in Figs. 2-5, which are arbitrarily numbered according to the amounts or severity of the inclusions represented, thus enabling an inclusion number to be derived. In the Fox count, the specimen is examined at a magnification of 130 diameters whilst in the other methods a magnification of 100 diameters is employed.

In the Fox count, the field is given a grade number from 0 to 4 depending on its severity of its inclusion content. Fields estimated as containing less than half the quantity of inclusions indicative of Grade 1 are designated 0, whilst those estimated as containing between  $\frac{1}{2}$  and  $1\frac{1}{2}$  are designated 1, and so on. The final result is presented as an inclusion number obtained by multiplying the number of fields in each grade by the grade number and adding the products. The JK, Diergarten and CM counts are similarly performed except that the inclusion number is entered under

one of several headings, according to the type and form of inclusions present. The counts are generally presented as frequency of fields for each grade, but sometimes an average inclusion number is calculated by proceeding as in the Fox count and dividing the sum of the products by the number of fields examined.

2. Direct methods Various methods have been used and different quantities of the inclusions measured. Kjerrman<sup>86</sup> used a micrometer ocular in the microscope and measured all inclusions down to 0.002 mm. using a magnification of 200 diameters. The estimate was presented as number of inclusions per square millimetre. Herty et al.<sup>87</sup> examining cast steel samples used a magnification of 250 diameters and with the aid of an ocular micrometer measured the diameters of all inclusions that were visible. From this result, the weight per cent of inclusions in the sample was calculated. Epstein,<sup>88</sup> Zieler<sup>89</sup> and Perrin<sup>90</sup> used similar methods to Herty et al. except their measurements were weighted by coefficients.

The above procedures for estimating the inclusion content of steels ignore the width of the inclusion. Hunter, realising this, instituted a method in which the total quantity, the total inclusion length and width of inclusions were all taken into consideration. Hunter's method<sup>91</sup> consisted of projecting the field of view on to a ground-glass screen containing a series of lines 1/10 in. apart over an area of 5 in. long by 3 in. wide. The magnification was standardised at 100 diameters so that the distance between each line of the grid was 0.001 in. on the specimen. Three Veeder counters were used to record the following information: number of fields examined, total lengths of inclusions in 1/10 in. and number of inclusions whose lengths were measured. Inclusions less than 1/10 in. were disregarded. From this data, the total length of inclusions over 0.001 in. long per in.<sup>2</sup> and the average length of inclusions present were calculated. To obtain some idea of the width of the largest inclusion present, the specimen was re-examined

at a magnification of 50 diameters. Inclusions whose widths were less than 0.0001 in. were denoted by the letter A, those between 0.0001 and 0.0005 in. by the letter B, and those greater than 0.0005 in. by the letter C.

In another method advocated by Hunter<sup>92,93</sup> a short piece of billet or bar was halved to provide a section parallel to the rolling direction of the material and extending from edge to edge. This section was then ground on a linisher to give a reasonably flat surface and anodically etched in a syrupy phosphoric acid for about 15 minutes, with a current density of approximately  $2\frac{1}{2}$  amps per in<sup>2</sup>.

The effect of this treatment was to polish the surface to some extent and also to attack the inclusions, leaving clearly recognisable indications of their positions and forms. The etched surface was then examined at a magnification of 5 diameters by projecting on a ground-glass screen ruled with parallel lines  $\frac{1}{2}$  in. apart. This interval, which represented 1/10 in. on the specimen, was taken as the unit of counting. Inclusions smaller than this unit were disregarded. The count was expressed as the sum of the squares of the lengths of the inclusions divided by a factor representing the area of the specimen examined.

Hardy and Allsop<sup>94</sup> used a method based on the lineal intercept principle. In their method, the specimen is examined at a magnification of 90 to 100 diameters using a microscope provided with a mechanical stage and cross-wires in the eyepiece. The specimen is traversed at right angles to the rolling direction of the billet and all inclusions intercepted by the intersection of the cross-wires counted, as illustrated in Fig.6. A measured distance is traversed, made up of several runs to cover the area of the specimen. The result is presented as number of inclusions per centimetre. For most steels a traverse length of 10 to 20 cm. was found to be adequate. The method could be used to estimate different types of inclusions, and was subsequently modified by a BISRA Working Group<sup>95</sup> who recommended the use of two cross-

wires instead of one, to count only inclusions greater than 0.025 mm. long, as illustrated in Fig.7. A procedure for sizing inclusions by this technique was also suggested.

Baynes<sup>96</sup> used a similar method to Hardy and Allsop to estimate the inclusion content in samples of cast steel. In this method a ground-glass screen, upon which is superimposed a set of three lines within a circular field of view, is used. The three lines are at a distance of 0.5 in. apart and the length of the centre cross-wire is 3 in. on the screen. The specimen is traversed at right angles to the cross-wires, each traverse being 0.5 in. All inclusions intercepted by the centre cross-wire are counted, as illustrated in Fig.8. The parallel cross-wire on either side of the centre cross-wire indicate and assure the next set of inclusions to be counted.

The SAE count<sup>97</sup> involves actually counting individual inclusions and measuring their lengths, and comparing the background inclusions with a standard chart. The count provides information on the quantity, average size and type of inclusions by means of a compound expression.

Area analysis, lineal analysis or point counting may also be used for measuring the volume fraction of a particular type of inclusion, e.g. sulphides or oxides. Of these three methods for obtaining volume fractions, that based on area ratios is the oldest. It was Delesse<sup>99</sup> in 1848 who first showed the equality of area ratios and volume ratios. This equality states simply that the fractional area occupied by a phase (in this case an inclusion) on a random plane section equals the fractional volume occupied by that phase or

$$\frac{A_i}{A_t} = \frac{V_i}{V_t} = F_v \quad (\text{Eq.1})$$

where A represents areas, V represents volumes,  $F_v$  is the volume fraction, and i and t refer to the inclusion area and the total area under examination respectively.

In practice, the determinations are made from photomicrographs or

tracings of inclusions by means of a planimeter, provided the inclusions areas are not too small or irregularly shaped.

The development of lineal analysis was due to Rosiwal,<sup>100</sup> who in 1898 showed that the length ratios on a plane section equals the volume fraction of a phase in the specimen. For one phase, the equation is

$$\frac{L_i}{L_t} = \frac{V_i}{V_t} = F_v \quad (\text{Eq.2})$$

The procedure followed in obtaining volume fractions by lineal analysis is to draw or superimpose a straight line on the photomicrograph or image several times at random. The portions of the line falling over the inclusions of interest (indicated by "i" in Fig.9) are measured with the aid of an ocular micrometer or a grid. The fractional lengths are then summed and divided by the total lengths of the lines, as illustrated in Fig.9.

Point counting for obtaining the volume fractions of a phase was originally developed by Thompson<sup>101</sup> who in 1930 showed that by superimposing a grid over the image, counting the intersections falling on the phase of interest and comparing this to the total number of intersections in the grid, gave a measure of the volume fractions of that phase in the specimen. In effect, this procedure can be thought of as giving a ratio of areas, since the finer the spacings on the grid the closer the approach to a coverage by area. The relevant equation in this case is

$$\frac{N_i}{N_t} = F_v \quad (\text{Eq.3})$$

where  $N_i$  is the number of grid intersections falling over the inclusion i, and  $N_t$  represents the total number of grid intersections. The grids used are illustrated in Fig.10.

Bergh<sup>102</sup> has used a variation of this method to estimate the inclusion content of steels in terms of volume fractions and size distribution functions. A grid was used over the image visible through the microscope, and the intersections falling over the inclusions of interest were counted

and compared to the total number of intersections in the grid. The size distribution function was determined in terms of grid spacings. Bergh's method is illustrated in Fig.11.

In a recent publication from Jernkontoret in Sweden a method has been suggested in which the image of successive fields of view are projected on to a screen or chart, such as shown in Fig.12, and drawing on it the inclusions. After the required number of fields have been recorded, the chart is visually assessed by counting and measuring all the inclusions that have been recorded. Three charts are made each at a different magnification to enable inclusions of different sizes to be properly assessed.

3. Automatic methods Melford<sup>79</sup> has discussed some of the automatic methods developed for inclusion assessment. Twelve instruments have been developed and used so far for this purpose. Brief details of these instruments are given in Table 2.

The Cintel flying spot microscope developed for particle size analysis was used by Allmand and Widdowson<sup>104</sup> for carrying out inclusion counts. The method was found to be unsatisfactory as the instrument could use only 35 mm. negatives. This instrument was modified and used by Belk<sup>105</sup> for counting and sizing inclusions in metallographic specimens. It was the first serious attempt made to automate the task of counting inclusions.

In this method, illustrated in Fig.13, a special cathode-ray tube with a finely focused light source is used. As each field of view on the specimen is viewed it is scanned line by line. A photomultiplier cell is used to detect changes in the intensity of light reflected from the specimen surface as it passes over the brightly polished matrix of the steel and the relatively dark inclusions. The optical reflectivity differences are converted to electrical voltage differences. These signals are then processed by special electronic circuits to give information on the number, size and area of inclusions intercepted by the spot in the cathode-ray tube. A one-

line delay memory is used to ensure that the same inclusion is not counted twice. The specimen movement was controlled manually.

In the Quantimet,<sup>106,107</sup> Integramat and Micro-Videomat systems, a television camera is used to assess the sample field by field. The Quantimet system is illustrated in Fig.14. The electrical output from the camera passes into a closed circuit television monitor to provide a television picture, and also into a detector unit where signals emanating from the inclusions are discriminated and selected from the rest of the signal. This is done on the basis of optical reflectivity similar to the flying-spot system. The output from the detector can be fed into the monitor to give a visual display of the inclusions being measured. As in the flying-spot system, reflectivity differences are converted into electrical voltage differences and processed by special electronic circuits to give information on number, size and area of inclusions present in each field of view. A one-line memory system is used to ensure that each inclusion is counted once only. The whole sequence of operation, including specimen movement, is controlled automatically.

In the Scanning Microscope, the TI-Hilger Inclusion Counter<sup>79,108</sup> and the AMEDA instrument, a photomultiplier views the image through a small aperture. As the image of the inclusion traverses this pinhole, variations in the photomultiplier output occur and the resulting signal is electronically processed in a manner similar to the television camera signal in the Quantimet to give information on area of inclusions.

Wojcik et al.<sup>109</sup> have used the Coulter Counter to study oxide inclusions in seamless ingots and their effect on defect occurrence in the finished product. In this technique, illustrated in Fig.15, the oxide inclusions are first obtained in the form of a residue by chemical extraction. The inclusions in the residue are then dispersed in an electrolyte into which dips a glass tube having an immersed electrode on either side. The suspension is made to flow through a small orifice in the glass tube, and as each particle of

inclusion passes through the orifice, it displaces its own volume of electrolyte, thus changing the resistance across the orifice. This change in resistance is converted into a voltage pulse with an amplitude proportional to the particle volume. The pulses are fed to an electronic counter with an adjustable threshold level such that all pulses above the threshold are counted. The latest instrument counts simultaneously at 12 threshold levels. The results of this particle counting represent numbers of particles for each of the 12 selected size groups.

Although the Coulter Counter method provided useful information as to inclusion size distribution, Wojcik et al. found the scope of its utility difficult to evaluate. The technique is applicable to spherical or, at most, to symmetrical inclusions. Furthermore, it is totally dependent on the effectiveness of inclusion isolation procedures.

The TI Automatic Inclusion Counter, Sizer and Identifier<sup>79</sup> employs an electron optical system with X-ray detection. The instrument is essentially a special purpose computer which operates on signals produced by a Scanning Electron Microprobe Analyser in such a manner as to count, size, identify and measure the volume fraction of the inclusions in each field. Counting and sizing is accomplished by means of the electron backscatter signal. Identification is achieved by making various logical comparisons between the outputs of two or three X-ray spectrometers in such a way that inclusions are recognised by the fact that they contain either (a) one element, (b) one element and another, or (c) one element but not another. One field of view,  $\frac{1}{2}$  mm. square, can be assessed in 15 seconds. In this time inclusions down to 1 micron in size may be counted and sized. Inclusions down to 12 microns in diameter can be simultaneously recognised. If it is necessary to recognise inclusions down to 1 micron in size then this must be done at a higher magnification in which case the field of view examined is only 50 microns. The speed of the instrument is too slow for quality control work although this



approach gives all the information generally required by the steelmaker.

The Vickers Automatic Inclusion Classifier<sup>79,110</sup> is an electron optical system. It is illustrated in Fig.16. The instrument is based on the principle that the number of electrons backscattered from a target is a function of the mean atomic number of oxide inclusions commonly occurring in steels; it is possible to distinguish between them and the sulphides. A pulse height analyser is used to separate the pulses emanating from these two types of inclusions. These pulses are fed into a computer which also distinguishes between unbroken oxides, such as a silicate inclusion, and broken oxides, such as an alumina. However, the instrument cannot count the number of inclusions, since it has no memory to distinguish between intercepts from the same inclusions and those from other inclusions, but it can measure the volume fraction of these different types of inclusions, i.e. oxides and sulphides.

The area of each field examined is  $2.50 \text{ mm}^2$ . The system is capable of measuring inclusions down to 3.5 microns in size. It is fast but expensive when compared with the Quantimet and the TI-Hilger Automatic Inclusion Counter

#### 1.50 Decision to use the 'Quantimet' image analysing computer

Apart from the Vickers Automatic Inclusion Classifier and the TI-Hilger Automatic Inclusion Counter, the only other instrument available was the Quantimet. Both the Vickers and TI-Hilger instruments were still under development and were not available commercially in 1967 when this research started. Furthermore, both these instruments do not enable the total number, the total length or size distribution of inclusions in a sample of steel to be ascertained. In the absence of reliable information on which measurements are the best pointers for determining the inclusion content of a steel it was considered desirable to use an instrument which measured as many inclusion quantities as possible. As stated by Melford<sup>79</sup> "Experience may show that only part of this information is required in quality-control work but such a simplification can only follow extensive operating experience".

It was decided, therefore, to restrict part of this project to consideration of this instrumental technique, although it was realised that the two other instruments may be useful, and have certain advantages over the Quantimet.

2.00 REVIEW OF PREVIOUS WORK

- 2.10 Manual inclusion counting methods.
- 2.20 Automatic inclusion counting methods.
- 2.30 Correlation between different methods.
- 2.40 Sampling, including sample preparation.

## 2.00 REVIEW OF PREVIOUS WORK

### 2.10 Manual inclusion counting methods

The first major study in this field appears to be that by the Inclusion Count Sub-Committee of the Iron and Steel Institute.<sup>111</sup> They examined the Fox and Firth-Brown inclusion count methods to determine whether they were capable of giving reproducible results when performed by different observers. Fifteen steels were assessed and each observer used the same samples. There were considerable differences between the counts obtained by each observer, and it was concluded that these methods were too dependent on observer judgment to be useful as standard tests. Further work by Swindon and Hatfield<sup>112</sup> drew attention to the fact that appreciably different results for the Fox Count could be obtained by one observer over the same fields of view when different microscopes are used. For example, using a Leitz instrument a Fox Count of 20 was obtained, whereas with a Zeiss instrument the count was 28. The latter count was obtained at a magnification of 145 diameters, a true field size of 0.9 mm diameter, and an apparent field size of 130 mm diameter in the microscope. The former was obtained with X132, 0.82 and 108 mm respectively. Calculations performed by Swindon and Hatfield showed that the observer who assesses the 'dirtiness' of the specimen in terms of the apparent area of inclusions appearing on the screen; in terms of the number of inclusions times their apparent lengths; or number of inclusions times their relative lengths would tend to report a higher value with the Zeiss than with the Leitz microscope, but the observer who assesses the 'dirtiness' by the relative area of the inclusions to the exposed area would get the correct result irrespective of the size of the field and/or the magnification.

Hatfield and Giles<sup>130</sup> performed inclusion counts on the same samples of steel using magnifications of 132, 520, 1,200 and 2,000 diameters but the same field diameter. They found that the count decreased continuously with an increase in magnification. Spies<sup>113</sup> performed a similar investigation using

the Diergarten Inclusion Count. However, in this case, the tests were performed at a constant magnification of 100 diameters and various field sizes. He found that the counts decreased with an increase in field size.

This problem has also been investigated, but in a different way, by Pelazzi.<sup>114</sup> Using magnifications of 100 and 700 diameters and field areas of 0.25 and 0.03 mm<sup>2</sup> respectively, he showed that the occurrence of fields with 0, 1, 2, 3 ... n inclusions in each case followed Poisson's Law.

In an investigation of the Fox, JK and SAE inclusion counts, Hardy and Allsop<sup>94</sup> showed that none of these methods was capable of giving reproducible results when used by different operators. They found the SAE Count to show the largest scatter of results, and it was stressed that not even the length and nature of the longest inclusion could be decided. With the JK Count, not only was there lack of agreement between observers with respect to total inclusions but also with respect to the relative proportions of different types of inclusions. The Fox Count showed that it was little better than the JK or SAE Counts. The reasons given by Hardy and Allsop for this lack of agreement between observers were mental fatigue associated with performing an uninteresting and repetitive task, and the high degree of personal judgement required in determining the inclusion content of a steel by these methods. To overcome these difficulties, they developed the Lineal Traverse Inclusion Count<sup>94</sup> which was shown to be capable of giving reproducible results when used by different observers.

Before adopting the new Lineal Traverse (LT) Count as a standard test for use in cooperative investigations, the Rolling Ingots Sub-Committee of BISRA studied the level of reproducibility attainable by this method. They found that the level of reproducibility attainable by different observers was the same as that attained by Hardy and Allsop.<sup>94</sup> They also found that the odd high or low values obtained by some observers were due to some of them counting the very small inclusions and others not counting them at all. To overcome this problem

the Committee recommended the use of two crosswires in the eyepiece instead of one, and although this method was shown to be capable of giving more reproducible results than the original method, a later investigation by Allmand<sup>116,117</sup> showed that in the case of low alloy forging steels, the agreement between observers was only slightly better than that attained with the JK Count, and that a recommendation to change to the LT Count was not justified. But in the case of high sulphur free-cutting steels, a change to the LT Count from other methods was justified as it provided a more reliable assessment of the harmful oxide and silicate inclusions present in the material.

In discussing the results of the above researches, particularly the work of Hardy and Allsop,<sup>94</sup> it was argued by some leading workers in this field that part of the variations in inclusion counts reported by different observers might have been due to sampling error since no attempt had been made in these investigations to examine the same fields of view. Accordingly, the Inclusion Assessment Group of BISRA repeated Hardy and Allsop's work but under more stringent experimental conditions using the Fox, JK, LT and Bergh inclusion counts. They found<sup>115</sup> that the agreement between observers was still poor, although each observer had examined the same fields. In this case, however, the largest scatter of results was found to be associated with the Fox Count. With the JK Count, better agreement between observers was obtained for alumina inclusions than for manganese sulphide, silicate or globular oxides. The agreement between observers for the LT and Bergh methods was better, but large differences between observers were still present.

Spies<sup>113</sup> has also found poor agreement between observers when using the Diergarten Count. However, he has attributed this variation to differences in the inclusion concentration rather than to observer judgement. He found that fields with low concentrations of inclusions gave more variable results than did those in which the concentration of inclusions was higher. In fact, when the concentration of inclusions was really high, the agreement between observers was found to be extremely good.

In each counting method, in addition to the more arithmetic aspects of the test, each observer has to exercise some degree of judgement. For example, in the case of the JK Count, the observer must compare the microscopic fields of view with standard photomicrographs on the chart, which are somewhat idealised. This difficulty is increased by the fact that real fields of view may contain two or more types of inclusion, and in order to compare each type with the corresponding series of micrographs on the chart, the observer must attempt to visualise the inclusions of each type as they would appear alone. Similarly, with the Fox Count, the pattern and severity of the inclusion observed in each field of view must be compared with the standard chart. It is considered that variations of personal judgement in performing the basic task of repeated comparison of microscopical fields of view with standard charts, and selecting the type and grade most closely in agreement with each field will give rise to some degree of variation in the inclusion counts recorded by different observers.

With the LT and Bergh counts, the difficulty is not so much the inability of observers to count the same inclusions as the difficulty of seeing the image clearly, since the crosswires or grid intersections in the eyepiece of the microscope are generally seen with greater clarity than the inclusions, and to count or not to count some small inclusions in special situations becomes a real problem. If the SAE Count is used, the observer has to decide the length of the longest inclusion. The longest inclusion in this context does not mean simply the longest unbroken inclusion; strings or groups of particles may also be assessed as 'single' inclusions. Obviously, therefore, it is difficult for the observer to decide the limits of each inclusion and then select the longest. In these circumstances it seems inevitable that the conclusion reached by one observer will frequently differ from that of another.

From the foregoing review of previous work it would appear that none of the manual methods for assessing the inclusion content of the steel, i.e. the

Fox, JK, SAE, Diergarten, LT or Bergh is satisfactory, and the type of count most likely to give reproducible results is one that requires little or no personal judgement on the part of the observer. The solution is to use instrumental inclusion counting methods, which should eliminate the personal error, leaving only the sampling problem to be overcome.

## 2.20 Automatic inclusion counting methods

The possibility of using the flying-spot microscope for inclusion counting was suggested by Allmand<sup>85</sup> in 1958. This possibility also appears to have been investigated independently by Hardy, Allsop and Bridge<sup>118</sup> in 1959. However, it was Belk<sup>105</sup> who, in 1962, demonstrated the feasibility of using this system for assessing the inclusion content of steels. Using a modified flying-spot system he and Lake<sup>119</sup> assessed the inclusion content of steels. They found that each field of view can be assessed in 10 seconds and that a further 10 seconds was required to record the results and adjust the controls or move to a new field. Data acquisition rate was, therefore, about three fields per minute. Results obtained on five samples of steel showed that the method was capable of giving reasonably reproducible results. Specimens for examination on this instrument had to be prepared with a high metallographic finish. Despite the objectivity of this instrument, none was manufactured on a commercial basis. The reasons for this are not wholly clear but it is believed that the high cost of the instrument was a decisive factor.

Because of this, Allmand and Widdowson<sup>104</sup> examined the suitability of the highly successful Rank-Cintel Flying Spot Particle Resolver developed for particle counting and measurement in the medical and powder fields. They showed that the method was capable of counting and sizing inclusions, but it was found to be too slow. To count one field by this method took  $1\frac{1}{2}$  minutes, plus the time taken to record the required number of fields on 35 mm films and produce the negatives. However, instrument standardisation was found to be simpler than with the reflected light version since a 'standard field'



negative could be used before each new run.

A major breakthrough in this field was made in 1963 with the development of the first version of the image analysing computer, known then as the Quantitative Television Microscope (QTM). It was very advanced in design and used metallographic specimens, and was capable of assessing the inclusion content of each field of view in less than a second. Widdowson<sup>120</sup> has examined this instrument for assessing inclusions in steel. He found that it was able to measure the percentage area of sulphide and oxide inclusions separately and count the number of intercepts of the television scanning line with these two types of inclusions. By examining specimens with widely varying oxide content, Widdowson was able to show that the instrument was sensitive to small changes in oxide content. He also showed that there was a strong correlation between the instrument's percentage area oxide measurements and the oxygen contents as determined by vacuum fusion or the carrier gas methods (Ströhlein).

Widdowson<sup>120</sup> also investigated the reproducibility of this technique when used by different operators. Standard settings were adopted for threshold level, and in order to minimise sampling error, each operator carried out the measurements adjacent to a scribed line on each specimen. A batch of ten specimens was examined. He found that the instrument when used under these conditions gave reproducible results.

Belk<sup>105</sup> has criticised the QTM technique on the grounds that analysis of the image is more difficult at the fast scan rate involved, 50 frames per second. However, in 1964, a modified QTM, known as the quantitative image analysing computer ('Quantimet') was developed and put on the market by Metals Research Limited. This instrument would count and size areas of contrast on a microscopic specimen in addition to making percentage area and projection measurements. One of the first evaluation studies to be made of this instrument was that by Allmand and Blank<sup>121</sup>. They found the instrument suitable for

counting and sizing inclusions such as oxide and/or sulphide inclusions, but stressed that the reproducibility attainable was dependent upon the threshold setting. This setting, which has to be made by the operator before using the instrument, was found to be very critical and only small variations in this setting were found to cause large variations in percentage area or projection measurements. Figures reported by Allmand and Blank showed standard deviations on a set of ten replicate measurements to be within 10 per cent of the mean estimate, but differences between different operators' results on the same fields to be nearly as high as 30 per cent of the mean count.

Allmand and Blank<sup>121</sup> also showed that the detection of oxide inclusions separately from sulphides was possible when the oxides were darker than the sulphides. When they were not, for example, in the case of glassy silicates, then accurate detection of oxide inclusions alone was not possible. According to them, this error arises because as the threshold is turned down so that sulphides are not detected, marked under-detection of the lighter coloured oxides occurs. They also found that for satisfactory detection, the inclusions must be at least 3 mm in size on the television screen monitor of the Quantimet.

Another characteristic error has been found to be present with this method. Around each dark oxide inclusion there is a diffused area or zone caused by the finite resolution of the optical system. The reflectivity of this zone is very similar to that of the sulphides and, consequently, is treated by the instrument as sulphides. Franklin<sup>124</sup> has dealt with this problem. He showed that if two  $3\text{ }\mu\text{m}$  inclusions were to be present in the same field of view, and one of them was a sulphide and one was oxide, then the oxide inclusion would be assessed as  $4.5\text{ }\mu\text{m}^2$  and the sulphide as  $6.2\text{ }\mu\text{m}^2$ . In addition, a further  $1.7\text{ }\mu\text{m}^2$  round the edge of the oxide would be counted as sulphide, giving totals of  $4.5\text{ }\mu\text{m}^2$  oxide and  $7.9\text{ }\mu\text{m}^2$  sulphide, instead

of the true values of  $7.1 \mu\text{m}^2$  each. He showed that the error on larger inclusions increases with the diameter of the inclusion, but becomes a smaller proportion of the percentage area measurement itself. For example, it was shown that an  $11 \mu\text{m}$  diameter inclusion of true area  $95 \mu\text{m}^2$  would appear as  $86 \mu\text{m}^2$  if it was an oxide, and  $91 \mu\text{m}^2$  if it was a sulphide. Franklin concluded from the results of his work that the image analysing computer should slightly overestimate the sulphide area measurement and considerably underestimate oxide area, and the error should be proportionally larger for smaller inclusions. He also pointed out that the characteristics of the electronic treatment of the pulses are such that they are inverted and shaped and may not produce errors of the same magnitude as indicated in his mathematical treatment of the problem. Furthermore, he thought that different operators set the threshold level differently which may have the effect of partially off-setting this error.

With respect to counting and sizing inclusions, Allmand and Blank<sup>121</sup> have shown that this is possible provided the instrument (the Quantimet) is correctly adjusted. Count measurements were found to be less sensitive to small variations in threshold than area measurements. However, they stressed that trouble may be experienced with multiple counting when the acceptance angle is incorrectly adjusted for the shape and distribution of inclusions being counted. In general, they found round and oval inclusions to be correctly counted and sized but stringer sulphides or silicates could not always be properly counted, especially when their major axes were parallel to the scanning line of the instrument. They found that such inclusions could often be more easily counted when they were oriented 90 degrees to the scanning line, but this did not enable the inclusion lengths to be assessed.

More recently other workers, notably Roche,<sup>131</sup> Lindon,<sup>132</sup> Franklin<sup>124</sup> and Grethen and Philippe<sup>123</sup> have reported on the suitability of the manually

operated Quantimet for assessing the inclusion content of steels. These investigators have found the instrument satisfactory for this application, and have reported errors which are of the same magnitude as those reported by Allmand and Blank.<sup>121</sup>

Most workers in this field of study, however, have expressed the opinion that despite the speed at which measurements may be made with the Quantimet, it is still too slow for use in routine work, such as quality control in steelworks. To meet this need, an improved version of the Quantimet, fully automatic in operation, was developed. This instrument came out as a prototype in 1966 and was studied by Allmand<sup>126</sup> who used it in a steelworks as a quality control test. He found that it was capable of working for sustained periods measuring fields at the rate of 100 per minute; this time including the printing of the results and moving on to new fields. By examining the samples first by the conventional JK method using skilled operators and then on the instrument, Allmand was able to show that nearly twice the number of specimens could be examined by one operator against two for the manual JK method. He also showed that the instrument was sensitive to small differences in oxide content, but found that large inclusions could be omitted from the count if an adequate number of fields were not examined.

During 1966-67 several alternative instrument to the Quantimet became available as prototypes. These instruments are the Japanese Automatic Scanning Microscope (Model ASM-1), the TI-Hilger Automatic Inclusion Counter, the two German instruments, Micro-Videomat and Interegramat, and the British Vickers Automatic Inclusion Classifier. As far as the author is aware these instruments are still under development and are not, as yet, available commercially in 1970.

### 2.30 Correlation between different methods

Correlation between different methods is of interest for two main reasons: (a) in the early adoption of new methods it is useful to know

how the inclusion quantities they measure compare with those measured by other more established methods, and (b) in the search for a simpler test which could provide a number indicative of the inclusion content of the steel for quality control purposes.

Pickering<sup>128</sup> showed reasonably good agreement between chemically determined oxygen analyses and oxide volume fraction measurements determined metallographically. Similarly good correlations between these two methods have also been reported by Saunders, Forgeng and Farrel<sup>129</sup> and by Bergh and Lindberg,<sup>103</sup> while Knagg<sup>133</sup> has shown a good correlation to exist between chemically determined sulphur content of the steel and Fox Count numbers.

As automatic instruments became available, several investigators compared the instrument's inclusion area measurements and inclusion content determined either chemically or metallographically. One of the first such correlations to be attempted was that by Lake<sup>119</sup> who compared Fox Count numbers with inclusion area measurements determined by the flying spot method. He found good agreement between the two values. A similar correlation was also attempted by Widdowson<sup>120</sup> who compared the Quantimet's oxide area measurements with oxygen contents determined by the vacuum fusion or Ströhlein carrier gas methods. Widdowson also compared the Quantimet's results with those obtained by the Bergh Count. In both these cases he found a strong correlation to exist between the methods. In fact, application of Student's 't' test by Widdowson to these results showed that the correlations were significant at the 0.1 per cent level.

While both Lake and Widdowson found good correlations between Fox Count numbers and the instrument's inclusion area measurements, it is interesting to note that the regression line for their results is not the same within the same range of measurements. Lake's line is not only more displaced but is also steeper. This discrepancy between the two investi-

gators is probably due to variations in the operator's judgement of what constitutes a correct Fox number rather than to gross variations between the two instruments' area measurement.

Roche,<sup>131</sup> Blank,<sup>122</sup> Lindon,<sup>132</sup> Allmand,<sup>125</sup> Franklin,<sup>124</sup> Cottingham et al.<sup>134</sup> and Grethen and Philippe<sup>123</sup> have all attempted such correlations and found the agreement between methods to be statistically significant. All these investigators have used a Quantimet for measuring the inclusion area, except in the case of Cottingham et al. who used the TI-Hilger instrument.

Of these correlations perhaps that by Grethen and Philippe is most interesting because they have compared the inclusion area measurements as determined by the Quantimet, and oxygen content as determined by chemical and the neutron activation methods. They have also compared the inclusion area measurements determined by the Quantimet method and those determined by the method of point counting. Grethen and Philippe showed that although there was a good correlation between the neutron activation oxygen analyses and the area measurements determined with the Quantimet, the latter method consistently measured less oxides than was measured by the reference method. They found the measurements to have a standard error of  $16.12 \times 10^{-3}$  per cent area which was shown to correspond to an analytical error of  $2.5 \times 10^{-3}$  per cent oxygen. Grethen and Philippe attributed this difference between the two measurements to the following possible causes: (a) inadequate resolution of the Quantimet to detect and measure the very small oxide inclusions present in the samples, (b) under-detection of the oxides, and (c) use of too low a value for converting the area measurements to weight per cent oxygen. However, in the case of sulphide inclusions, they found an excellent correlation between the instrument's sulphide area measurements and total sulphur content determined chemically. The agreement between inclusion area measurements determined by the

Quantimet and by point counting was also good but, in this case, better agreement was obtained for oxide inclusions than for sulphides, with the Quantimet slightly overestimating the sulphide area count.

Allmand<sup>121,125</sup> has also shown that a reasonably good correlation exists between the Quantimet's oxide area measurements and the JK Ratings.

Melford<sup>79</sup> criticises correlations between chart assessments and those obtained with automatic instruments on the grounds that they are unreliable as the former are subject to personal judgement. It is certainly unwise to pursue this comparison too far, as only the best and most laborious of the manual methods will achieve the objectivity of automatic instruments. Furthermore, the result of such correlations is to perpetuate the use of arbitrary chart methods in preference to more precise and meaningful numbers obtained by instruments. Nevertheless, it must be acknowledged that chart methods can be only slowly replaced by automatic measurements.

Given a significant and adequate set of measured numbers, the proper procedure would be to publish these new numbers for all of the existing charts, allowing the specification writers to change from chart specifications to equivalent machine numbers at their convenience, or for those without an automatic instrument to convert the machine numbers to equivalent chart ratings or vice versa.

#### 2.40 Sampling, including sample preparation

A search of the literature showed that although much has been written on the subject of inclusion counting, sample preparation and procedures for selection of samples, no work has been reported on errors associated with various sampling procedures currently in use. Comparatively little has been reported on the subject of errors in inclusion counts arising from use of different size billets, and no work at all has been reported on the effect of using sections that are not perfectly parallel to the longitudinal axis of the billet.

Hardy and Allsop<sup>94</sup> showed that for plastic inclusions, such as manganese sulphide and silicates, the inclusion count, as determined by the LT method, varied inversely as the reduction ratio, and a plot of inclusion count (total number of inclusions per centimetre) against reduction ratio gave a hyperbolic curve. For less plastic inclusions, this curve was less hyperbolic, being practically a straight line in the case of the hard undeformable titanium nitride inclusions.

A number of procedures for preparing specimens have been suggested by Vilella,<sup>135</sup> Beregekoff,<sup>136</sup> Forgeng<sup>137</sup> and Allmand.<sup>138,58</sup> Some investigators prefer using dry papers and avoid contact of the polished surface with water on the grounds that in some steels, especially plain carbon and low alloy, it often attacks the metal-inclusion interfaces, resulting in erroneous appearances. Others prefer to use wet and dry papers and have found that contact with water does not affect the appearance of the inclusions.

Satisfactory surfaces can only be produced from experience. It is difficult to give exact details of the sequence of polishing operations, since this is generally found to be dependent upon a number of factors. As far as automatic instruments are concerned, a number of investigators<sup>79,105,120,121</sup> have stated that a higher standard of polish is required than for normal manual methods. The surface of the specimens should be flat, free from scratches, stains or other blemishes. The edges of inclusions should be clearly defined and show no relief effects, and none of the inclusions should be dragged out during polishing. From the survey of literature on automatic inclusion assessment, it would appear that adequate methods are available for producing satisfactory specimens for automatic examination, but more work is needed on possible ways in which contrast between the inclusions and the steel matrix or between the inclusions themselves can be improved. In this connection it is interesting to note that Grethen and Philippe<sup>123</sup> have reported better accuracy for sulphide area measurements after etching the samples in chromic acid.



Widdowson<sup>120</sup> has also found it beneficial to etch the specimen to  
lighten or darken the sulphides.

3.00 PROBLEMS ENCOUNTERED IN AUTOMATIC INCLUSIONS ASSESSMENT  
USING AN IMAGE ANALYSING COMPUTER

- /
- 3.10 Reduction from ingot to billet.
- 3.20 Examination of a plane section.
- 3.30 Sampling procedures.
- 3.40 Sample preparation.
- 3.50 Development of a satisfactory operating procedure for the Quantimet Image Analysing Computer.
- 3.60 Comparison of Quantimet parameters with those of JK, Diergarten and CM ( SAE-ASTM ) methods.
- 3.70 Development of digital computer programmes for rapid processing of Quantimet data.
- 3.80 Outline of present work.

### 3.00 PROBLEMS ENCOUNTERED IN AUTOMATIC ASSESSMENT USING THE IMAGE ANALYSING COMPUTER

#### 3.10 Reduction from ingot to billet

The inclusion content of a steel changes after rolling or forging, due to the way in which inclusions are broken up or drawn out into thin threads by the working process. The results of inclusion counts based on examination with the microscope may therefore be expected to be affected by reduction in area. This point is of great importance in any attempt at correlation of results since the steelmaker frequently takes samples not only from billets of varying sizes, but also when the material has a considerably larger cross section than when sampled by forger or eventual customer.

#### 3.20 Examination of a plane section

Estimation of inclusion content can be expressed in several ways: as volume or area fractions; as total number of inclusions per unit area of steel; as an inclusion size distribution; and as frequency of fields in which the inclusion content as measured by volume fraction, area fraction, total number or size distribution lies between specified ranges. It is generally customary to separate the inclusions counted into types, e.g. sulphides, oxides, silicates, etc.

Such estimations are always based on observations on a plane section of polished material. The inclusion areas observed on such a plane will always appear to consist of a range of sizes because a random plane cuts the inclusions at random positions anywhere from an edge to the largest cross section.

- (a) Consider a random distribution of inclusions in the form of uniform spheres of radius 'a'. Any plane section through this distribution would indicate a range of sizes from zero to  $2a$  since, in general, the equatorial planes of the inclusions will not be intersected. Clearly, the 'true' size of these inclusions is the largest size, i.e.  $2a$ , and

if 'm' inclusions per unit area are seen, then the total cross sectional area of the inclusions present is  $m \cdot \pi a^2$ .

(b) If we now consider a random distribution of inclusions in which most are spheres of radius  $2a$ , but some are larger spheres, then, when we encounter an inclusion of apparent size  $2a$ , we have no means of knowing whether what we observe is the equatorial plane of the spheres of size  $2a$ , or a chordal plane of one of the larger spheres. This argument also applies to distributions containing spheres of size smaller than  $2a$  and thus to inclusions that are randomly distributed with respect to size.

(c) If we consider a 1 cm cube of steel having a volume fraction  $V$  of inclusions, each inclusion being a uniform sphere of radius ' $a$ ' cm., then, if the cube is cut into slices, each of thickness  $2a$  cm. then every inclusion will be cut through once and once only. The number of slices in a 1 cm cube is  $1/2a$  and one face of each slice has an area of  $1 \text{ cm}^2$ . All the inclusions will therefore be seen in an area of:

$$\frac{1}{2a} \times 1 = \frac{1}{2a} \text{ cm}^2$$

The volume of one inclusion is  $\frac{4}{3} \pi a^3 \text{ cm}^3$ .

The total volume of inclusions is  $V \text{ cm}^3$ . Therefore, number of inclusions per unit volume is  $\frac{3V}{4\pi a^3}$ ; and the number seen in unit area =

$$\frac{3V}{4\pi a^3} / \frac{1}{2a} = \frac{3V}{2\pi a^2}$$

(d) Now, by the arguments above, the average observed area of each inclusion will be less than the true (equatorial) area. We may treat this problem by considering the sphere of radius ' $a$ ' sectioned into equal layers of thickness,  $dx$ , so that the total number of layers in the hemisphere is  $a/dx$ . The average 'observed' radius is then the sum of the radii of all layers divided by the total number of layers, i.e.

$$\bar{A} = \frac{\sum_0^a \pi x^2}{a/dx} = \frac{\pi}{a} \cdot \int_0^a (a^2 - x^2) dx$$

$$= \frac{\pi}{a} \cdot \frac{2}{3} \cdot a^3 = \frac{2\pi a^2}{3}$$

Since the true area is  $\pi a^2$  we see that the observed area is  $\frac{2}{3}$  of the true area, so that areas computed by the image analysing computer must be multiplied by  $\frac{3}{2}$  to obtain the true area. The total area of inclusions in unit area of observed surface is the product of the number of particles of inclusions and the average area, i.e.

$$\frac{3V}{2\pi a^2} \cdot \frac{2\pi a^2}{3} = V$$

which confirms the observed proportion of area occupied by inclusions is in direct proportion to the volume fraction of inclusions.

Summarising: if  $\bar{A}$  = observed area as estimated by the image analysing

computer then:  $\bar{A} = \frac{2}{3}$  (true area)

$\bar{A}$  = volume fraction.

The determination of total number of inclusions per unit area or volume of steel is easier and also a more meaningful measurement in the sense that it does not matter where the plane section cuts the inclusions. On the other hand, the determination of size distribution analysis is much more difficult. The basic difficulty in performing this measurement on a two-dimensional plane is that the observed distribution function is likely to be subject to very large errors resulting from the sectioning procedure.

To derive the true distribution function from an observed distribution, it is necessary to assume some form for the sectioning function. In the simplest case, that of spherical inclusions, the probability,  $P_d$ , of an inclusion of diameter  $D$  being sectioned as a circle of diameter less than  $d$  is given by:

$$(1 - P_d)^2 = 1 - (d/D)^2$$

The frequency with which sections of diameter in the range  $d_1$  to  $d_2$  are observed is then just proportional to  $(Pd_2 - Pd_1)$ . A histogram of observed sections sizes can be analysed by making  $(d_2 - d_1)$  equal to the sizing interval on the histogram and noting that the last group on it must all come from the largest inclusions. By making  $d_1/d_2 = d_2/d_3 = \dots = d_n/d_{n+1}$  for the original sizing, a disproportionate number of particles in the smaller size groups can be avoided. The choice of the ratio  $d_n/d_{n+1}$  obviously depends on the range of sizes being analysed. It is usually equal to  $1/\sqrt{2}$ .

For non-spherical inclusions, no feasible mathematically sound procedure exists. But this error should remain constant provided the plane examined is representative, i.e. a sufficient number of inclusions are counted. In comparative work, such as quality control in steelworks, a constant error may be assumed provided the conditions under which the test is performed are standardised.

Variations in inclusion counts could also occur because of variations in the plane of polish used. Variations in the longitudinal plane of polish are particularly serious because the inclusions observed on such planes have no chance at all of being intersected at their largest cross section.

### 3.30 Sampling procedures

As the Quantimet method is concerned with inclusions in individual microscopical fields of view, sampling consists here in assessing a sufficient number of fields so that the sample examined is representative of the whole. The term 'representative sample' is a matter of degree since no sample can be absolutely representative. The usual criterion of a representative sample is that it must be of such size that if a series of such samples is taken from the whole, sample to sample variations are not significant compared with variations to be detected. In fact, for

determinations of inclusion content to be meaningful, it is necessary that the determination on each specimen or sample must reach some level of accuracy which may be agreed upon in advance.

While in theory it is possible to attain any desired level of accuracy, in practice, resources, equipment, personnel and time available for inspection often set limits which must not be passed. A survey carried out by the author of sampling procedures used in this country and others, notably in Sweden, and in the United States of America, showed that the number of fields examined on each specimen varied between 25 and 300.

The automatic Quantimet is capable of examining steel at the rate of one field of view per second, provided only one or two parameters are measured. This means that in a normal period of seven to eight hours, it is possible to examine between 15,000 and 17,000 fields. Now it can be shown that a cube of steel 2 mm on edge, with a volume of  $7.4 \text{ mm}^3$ , weighing only 0.06 gramme to be completely examined by a microscopic method would require the examination of over 25,000 fields of 0.5 mm diameter. Clearly, it will not be possible to examine each specimen selected in great detail. The problem the sampler is confronted with is a decision about how the sample should be selected and the individual specimens examined so that the measurement obtained is a reliable estimate of the inclusion content of the whole. Obviously, he can examine a few specimens, i.e. a small sample, in great detail or a lot of specimens in less detail. Similarly, he could select the samples in a random manner from different parts of the ingot and cast or from one or two positions in the ingot and cast. Unfortunately, we do not yet know the sampling errors associated with such practices and consequently which method is best suited for quality-control work.

#### 3.40 Sample preparation

At present the operators of visual methods (JK, Diergarten, Fox, Bergh, etc.) can subjectively reject scratches, polishing pits and other defects on

the plane of polish, and count only inclusions. The automatic instruments, such as the Quantimet, however, cannot make this differentiation between scratches, polishing pits and inclusions of similar optical reflectivity. Separation of inclusions into different types is possible only if they differ sufficiently in optical reflectivity. However, in practice it is normally only possible to differentiate between oxides and sulphides. Differentiation between different types of oxides, such as silicates and alumina, are not possible because the reflectivities of these two types of inclusion are too close to each other.

Polishing pits, dragged out sulphides and other blemishes on the section which have the same optical reflectivities as oxides, will be counted as oxides. Some inclusions, such as manganese sulphide-telluride inclusions in high sulphur free-cutting steel, will not be assessed correctly due to the fact that the optical reflectivity of the telluride part of the inclusion is too close to that of the polished steel matrix. Similarly the counting of oxide inclusions in cast irons can present a problem because oxide inclusions cannot be separated from the graphite phase.

Another problem is the provision of a large number of specimens polished to a higher degree of polish than required for normal metallographic work.

### 3.50 Development of a satisfactory operating procedure for the Quantimet Image Analysing Computer

The automatic Quantimet is a comparatively new instrument and, consequently, little is known about its performance. Both Allmand<sup>125</sup> and Franklin<sup>124</sup> have carried out some tests on the automatic Quantimet but only problems concerned with threshold setting and resolution of the instrument were studied. Other factors concerned with both short- and long-term stability, calibration and operating procedure of the instrument could also influence the accuracy of the results, but these factors have received no attention.

The errors that might arise with this instrument may be classified into two main categories:



- (i) errors inherent in the design of the instrument,  
and (ii) errors arising due to the manner in which the instrument is calibrated and used by the operator.

The inherent errors may be due to the following problems: variations in blank frame area, variations in camera sensitivity, lack of reproducibility of the computer and data handling systems, lack of resolution, inability of the automatic stage unit to keep the specimen image critically focused over the whole of the traverse chosen, error due to counting and sizing inclusions of different shape, error due to optical intensity variations near the edge of inclusions caused by the finite resolution of the optical system, and edge errors caused by some of the inclusions being intersected by the blank frame.

Bias errors may be due to the following problems: bias in adjusting the focus of the microscope, bias in performing calibrations, and bias in adjusting the threshold setting.

### 3.60 Comparison of Quantimet parameters with those of JK, Diergarten and Fox methods

When a new method is to be adopted, it is important to compare the data obtained by this method with those obtained by other methods, particularly if such methods are fairly well established. The problem here is to convert all chart gradings to equivalent machine numbers to facilitate comparisons between methods.

### 3.70 Development of digital computer programmes for rapid processing of Quantimet data

The instrument is capable of working very fast while taking measurements on thirty different inclusion quantities. Typically, these would be volume fractions for sulphides and oxides separately, their size distribution and projected chord size distribution. Thus, in a relatively short time a vast amount of data can be accumulated on each specimen. For quality-control work, it is imperative that this data should be condensed and presented to the

quality-control department in a form which enables comparisons between samples to be made quickly and reliably.

Inclusion count results are best presented as histograms with averages and standard deviations. The histograms can be plots of the frequency of occurrence of a particular inclusion quantity, such as volume fraction or number in specified size ranges. However, the presentation of results in this form would involve hours of work since the calculations of averages, standard deviations and histograms can be a laborious task. A rapid digital computer method would obviously be a great help, since it is very much faster than manual methods and is also much more accurate and detailed with respect to the mathematics involved. The problem here is to develop various programmes to meet different situations likely to be encountered in practice.

### 3.80 Outline of present work

The proposed stages and main aims of the present work are summarised as follows:

#### Stage 1: Evaluation of the Quantimet

A thorough evaluation of the automatic image analysing computer known as the 'Quantimet'. In this work it was intended to determine the errors associated with this method, their causes and effect on the measured values so that a satisfactory operating procedure could be developed. Particular attention was given to the unresolved problem of making an objective threshold setting.

#### Stage 2: Relation of inclusion chart methods to Quantimet assessment

The JK and Diergarten charts were quantified by examining each of the gradings depicted on these charts on the Quantimet using the special epidiascope in an attempt to determine the inclusion quantities best suited to characterize the standard chart fields.

#### Stage 3: Computer processing of data

Attention was given to the problem of developing suitable digital

computer programmes for rapid processing of Quantimet data. In this work attention was also given to the possibility of the instrument, with the aid of the digital computer method, making complex judgements comparable to, though more precise than, the visual JK or Diergarten chart methods.

#### Stage 4: Phase differentiation

This stage was concerned with an investigation of the thin film interference technique to study the possibility of using it to enhance the contrast of certain types of inclusions so that a better differentiation between inclusions and other phases present can be obtained.

#### Stage 5: Inclusions and their relation to working of steels

A study was made of the effect of reduction in area from ingot to billet on inclusion count results. This work also included a study of the effect of variations in planar sections on the results of inclusion counts with the object of specifying the size of billets to be used and the tolerance limits to which the plane sections must be prepared.

#### Stage 6: Sampling and relation to inclusion results

This stage was concerned with an investigation of the sampling problems. As a first step, an experiment was carried out on three steels of varying oxide inclusion content to determine the variations between determinations made on the same polished surface using different sampling procedures for selecting the fields. This work was followed by an examination of three further specimens of widely varying oxide inclusion content after repeated polishing to determine the variations between different planar sections of the same specimen. Finally, two fifteen-foot long billets, one with low oxide content and the other with a higher oxide content, were examined along their entire lengths to determine the variations between specimens. This involved the examination of about 400 specimens, 200 from each billet.

#### Stage 7: Development of quality control procedures

This stage was concerned with a general programme of inclusion assessment. It was performed in two parts: the first was a detailed laboratory

examination of billet samples from different positions of two adjacent ingots to determine the distribution of oxide content with respect to volume fraction and size distribution, with the object of locating the 'dirtiest' part of the ingot. The second part of the investigation was a 'running' programme on low alloy steel billets performed at a steelworks. The objects of this latter work were to obtain statistically valid results concerning the distribution of oxide inclusions within ingots produced by a standard practice so that a sampling procedure can be decided, and also, to enable a satisfactory automatic inclusion counting method for use in quality-control work to be developed and tested.

#### Stage 8: Quantimet assessment and relation to other methods

This was the final stage of the work and was concerned with comparison of Quantimet assessment with standard laboratory procedures, such as neutron activation, step down test and sulphur printing. The aim of this work was to determine whether a correlation exists between the Quantimet inclusion assessments and those obtained by other established methods. This work was carried out in conjunction with the work on distribution of oxide inclusions in two adjacent ingots (see Stage 7 above).

4.00 AN EVALUATION OF THE AUTOMATIC QUANTIMET FOR  
INCLUSION ASSESSMENT

- 4.10 Principle and mode of operation of instrument
- 4.20 Possible sources of error
- 4.30 Results of tests to determine errors inherent in design of instrument
  - 1. Variations in blank frame
  - 2. Variations in camera sensitivity
  - 3. Overall reproducibility of method
  - 4. Automatic stage unit
  - 5. Resolution
- 4.40 Results of tests to determine errors due to operator bias
  - 1. Focusing of image
  - 2. Setting of threshold level
- 4.50 Relationship between threshold setting and camera sensitivity setting
- 4.60 Further work on setting threshold level
- 4.70 Comparison of visual and Quantimet inclusion assessments
- 4.80 Discussion
- 4.90 Conclusions and recommendations for operation of Quantimet

#### 4.00 AN EVALUATION OF THE AUTOMATIC QUANTIMET FOR INCLUSION ASSESSMENT

##### 4.10 Principle and mode of operation of instrument

A general view of the instrument is shown in Fig.17 and a simplified block diagram of it in Fig.14. The specimen can be imaged by an ordinary optical microscope or an epidiascope. This image is then projected into a television camera which scans it at a speed of 50 frames per second. The output from the camera varies as the electron beam (spot) traverses features of different optical reflectivity. The optical reflectivity differences are changed to voltage differences with an amplitude proportional to the optical contrast and width proportional to the intercept length of the spot with the feature, as shown in Fig.18. This electrical output is passed into a detector unit where the pulses are discriminated, shaped and then passed into an analogue computer which derives the required information.

A delay line memory is used which enables individual features, such as inclusions in steel, to be counted separately. Sizing is accomplished by a system which allows a count to be recorded only if the line intercept is greater than a pre-selected length. Six size controls are provided and by setting these to different intercept lengths, the size distribution function of the inclusions of interest in that field of view can be obtained. The projected length of inclusions of interest in the vertical direction of the scan is obtained by counting the total number of intersections of television line with the inclusions. By calibration, this intercept count can be expressed in inches or millimetres. To measure the area of inclusions, the intercept pulses are integrated to give a measurement directly proportional to the time spent by the spot in the camera traversing the inclusions of interest.

The counting logic, in addition to using a delay line memory

system, uses an anti-coincidence circuit which enables the computer to accept successive line scans from inclusions that are not vertical as coming from the same inclusion. This anti-coincidence circuit can be pre-set to match the type and distribution of inclusions being examined.

Various displays are provided which enable the operator to see at a glance whether or not the instrument has been properly adjusted. When the instrument is computing area measurements, for example, the whole of the inclusion of interest is "filled in" and appears white; when it is computing projection measurement a white spot appears on the trailing edge of each inclusion where it is intercepted by the spot; and when it is computing counts and size distribution on a number basis, a white marker tag is shown against those inclusions which are being counted.

The derived information is passed on to the data multiplexer unit which directs the operation of the particular automatic data handling system chosen and the operation of the stage.

A specimen can be arranged under the microscope, so that a programmed range of fields can be scanned automatically. The length of step to each field in say the X direction can be altered, as well as the length of step in the Y direction, and also the number of steps; so that a range of fields can be chosen according to the optical magnification of the microscope and its resolution, to suit the type of specimen. Therefore a number of measurements may be carried out on one field and the process repeated at a very rapid rate with a predetermined automatic programme of scanning. Speeds of 60 fields per minute can be achieved, the wealth of rapidly accumulated data necessitate the processing of the data on tapes by computer.

#### 4.20 Possible sources of error

The errors that might arise with this instrument may be classified into two main categories: (i) errors inherent in the design of the instrument, and (ii) errors arising due to operator bias in how the instrument is calibrated and used. Under (i) may be listed errors arising due to the following possible causes:

- (a) Variations in blank frame size due to instability.
- (b) Variations in camera sensitivity also due to instability.
- (c) Lack of reproducibility of the computer and data handling systems.
- (d) Inability of the stage unit to keep the specimen image critically focused over the whole of the chosen traverses in a programmed scan.
- (e) Variations in optical intensity near the edge of inclusions.

The main causes of errors arising under (ii) are:

- (a) Operator bias in adjusting the focus of the microscope,
- and (b) Operator bias in adjusting the threshold setting.

#### 4.30 Results of tests to determine errors inherent in design of instrument

4.31 Variations in blank frame. The blank frame is the area of the specimen within which all measurements are made. It is adjustable for size and is set by the operator by suitable calibration with a stage micrometer. A particular feature of the automatic instrument, as opposed to the manual version, is that all measurements are fixed absolutely. Thus, the projection measurement is obtained as number of intercepts of the scanning line with the inclusion of interest. In order to make this measurement meaningful it must be reduced to a length in millimetres or microns. This is done very simply by



measuring the number of lines in the blank frame on saturation and then relating the projection measurement to the height of the blank frame, previously determined by calibration.

In the same way the area measurement is fixed and it must be reduced to a fraction of the blank frame area. This is done by dividing the actual area measurement by the area of the blank frame, or alternatively, setting the blank frame to read 100% on saturation in which case the observed area measurements are obtained directly as a percentage of the blank frame area. Clearly, the accuracy with which any measurement can be related to the actual area examined on the specimen depends upon the accuracy with which the blank frame size is known and also on how free the blank frame setting is from drift.

To determine whether or not variations in blank frame size can be a serious source of error, tests were conducted at fortnightly intervals over a period of eight months in a steelworks environment. Each test was performed under controlled conditions and consisted of switching on the instrument, setting the blank frame to read 100%, and taking an area measurement on saturation at 5-minute intervals over a period of 145 minutes.

The results of four of these tests, typical of the rest, and carried out at two-monthly intervals, are shown in Fig.19. They show that the blank frame area decreases gradually with time dropping 5% on average over the first 80 minutes. But after 80 minutes, it varies less than 1% of the mean setting. Furthermore, all four of the curves are very similar, indicating that the stabilisation characteristics of the instrument have not changed over a period of eight months.

4.32 Variations in camera sensitivity. Two controls of camera sensitivity are provided, manual and automatic. The latter

control of sensitivity is used when the operator is certain that the amount of dark phase (oxide inclusion) will not be more than 20%. For fields with more than 20% dark phase, the manual control of sensitivity is selected. This arrangement is claimed by the manufacturer to give less drift and better isolation from mains interference.

The camera sensitivity is optimised by suitable adjustment to beam current, focus and sensitivity controls. These adjustments must be made by the operator at suitable intervals and present no real difficulties. However, after having made the necessary adjustments, this setting must be free from drift as any variations in camera sensitivity would be expected to affect the detection characteristics of the instrument and, consequently, the accuracy of the measurements.

In order to obtain a realistic idea of the variations, tests were conducted on unetched polished steel specimens containing inclusions. Both manual and automatic controls of sensitivity were investigated. As in the case of the experiments on blank frame, the tests were performed at fortnightly intervals in a steel-works environment. Each test was commenced on a separate day and lasted 145 minutes. The experimental procedure consisted of switching on the instrument, selecting and adjusting the control of sensitivity to be examined, and noting the camera sensitivity reading every 5 minutes as indicated on the meter.

Two typical results obtained at the beginning and end of the eight-month period are shown in Fig.20, from which it can be seen that automatic control of sensitivity is more stable than manual control of sensitivity. In fact, in every test that was performed, the manual control of sensitivity gradually increases with time

rising usually from a setting of 0.7 to over 0.9. Although the automatic control of sensitivity is stable over the whole of the 145-minute period, it is more stable after the first 60 minutes.

4.33 Overall reproducibility of method. The accuracy of the measurements obtained by this method of assessing the inclusion content of steels is partly dependent upon the overall reproducibility with which the computer and data handling systems operate over both short and long periods. In practice a determination is made only once and the error due to instability in the system is not known.

To determine the short-term reproducibility of the method, tests were made on the same field over  $4\frac{1}{2}$  hours, during which time no adjustments at all were made to the instrument. Long-term reproducibility was determined by making thirty replicate determinations at one month intervals over a period of eight months. The tests were made under standardised conditions, and using the automatic control of sensitivity.

A typical result of the short-term reproducibility attained by this method when measuring percentage area and total number of oxide inclusions in a field of view is shown in Table 3. It will be seen that both area and number counts remain virtually constant over a period of  $4\frac{1}{2}$  hours and that the variability (standard deviation) for both these measurements, is less than 2%. The long-term reproducibility attained over the eight-month period is shown by two sets of results given in Tables 4 and 5, which show that the reproducibility attained at the beginning of the eight-month period is the same as that attained at the end of this period.

4.34 Automatic stage unit. With the manual instrument, the operator repeatedly adjusts the focus of the image to keep it

sharp focus, a procedure which is impossible with the automatic instrument. The automatic stage has a maximum movement of 50 mm in the X and 25 mm in the Y direction. Any defocusing of image during counting will affect the apparent size of inclusions, and, consequently, the measurements on them.

To test the precision of the stage unit in the X and Y traverse, a carefully polished and etched steel specimen, revealing a pearlite-ferrite structure, was carefully mounted in the inverted specimen holder. This structure was chosen because of its more uniform distribution over the traverse distance required. The stage was moved to the beginning of the X traverse and the focus of the microscope adjusted until the image was in sharp focus. Using the automatic field selector, the field was moved one field at a time and the focus of the microscope assessed visually. This procedure was repeated until the whole of the X and Y traverses of the stage had been covered. The following objectives were used: X1.25, X2.75, X5, X10, X20 and X40. The oil-immersion objective was not used.

The results showed that the focus of the microscope was maintained over the entire X and Y traverses of the stage when using all except the X40 and X20 objectives. With the X20 objective a traverse distance of 35 mm in the X direction and 15 mm in the Y direction was attained, but in the case of the X40 objective, it was found necessary to refocus the image every 4.8 mm in the X direction and about the same distance in the Y direction.

4.35 Resolution. As stated previously, inclusions are detected by their contrast. Two detection controls are available to the operator so that he can set levels corresponding to oxides in one case and both oxides and sulphides in the other. Subtraction allows the two types of inclusion to be separated. Unfortunately, with this

type of system of detection a characteristic type of error is found due to the finite resolution of the system and to the fact that the distribution of light over the inclusions is such that their edges are rather diffused in appearance. Since these edges of inclusions have the same optical contrast as the sulphide inclusions, they are detected as sulphides and not as oxides. The same problem arises when measuring inclusions that are not entirely black or grey over the whole section, as in the case of semi-transparent inclusions, such as some silicates.

Table 6 shows some typical results obtained with the two detection circuits in operation. Detection 1 was set up to measure oxide inclusions only, and Detection 2 both oxides and sulphides, but only oxide inclusions were presented to the instrument. In this way it was possible to obtain a measure of the error on each field by subtracting the measurement obtained on Detection 1 from that obtained on Detection 2 and expressing the difference as a percentage of the oxide estimate.

It will be seen that over-detection occurs on Detection 2 resulting in the recording of a sulphide measurement even though no sulphides were measured, giving rise to errors in sulphide counts varying between 8.6 to 100% of the oxide estimate depending upon the size of the oxides measured.

From a sampling point of view, the lower the magnification the better the sample, and, consequently, it is desirable in quality-control work to use the X5 objective rather than the X10 or X20 objectives. The use of the lower power objectives, however, presents a problem since the very small inclusions are not detected. This results in fields with relatively severe inclusion aggregates to be graded lower than they would have been had all

of the individual inclusions been properly detected.

Each objective would be expected to perform best when used within a particular range of light wavelength. Unfortunately, this information was not available and therefore it was decided to examine the possibility of increasing the resolution of the system by the use of appropriate filters and illumination. Accordingly, tests were made of volume fractions of inclusions using different lamp settings and filters. The standard 50 W tungsten filament light source and the automatic control of camera sensitivity were used. A typical result of one of these tests is shown in Fig.21. It will be seen that maximum resolution is obtained with the yellow and orange filters and with a lamp setting of 8. With no filter, or in the presence of smoke, purple, and green filters, the resolution, as measured by the area count, is lower by a factor of nearly 2.

#### 4.40 Results of tests to determine errors due to operator bias

4.41 Focusing of image. The operator can choose to adjust the focus of the image by viewing it through the eyepieces of the microscope, or on the television monitor. The latter method is more convenient but has the disadvantage that the image is not seen with the same degree of clarity as when viewed through the microscope. Variations in focus affect the apparent size of inclusions and, consequently, would be expected to influence the accuracy of the measurements. To determine which method of focusing is capable of giving more reproducible results several typical fields, containing various sizes of sulphide and oxide inclusions, were examined repeatedly by two operators after refocusing the image. The tests were made one hour forty minutes after the instrument was switched on, and after the usual calibrations had been performed.

A summary of the results obtained is given in Table 7, from

which it will be seen that on average the variability, expressed as a percentage of the mean estimate, is 4.55% for the monitor method compared with 2.04% for the microscope method of focusing. It will be seen also that the variation (standard deviations and coefficients of variation) decreases when measuring relatively large inclusions such as manganese sulphides in high sulphur free-cutting steels (Field 4), and that differences between operators are not very significant for both methods of focusing.

4.42 Setting of threshold level. This is another task the operator must perform when using the instrument. Three steps are involved: (1) setting the threshold level about which detection is to be made, (2) setting the polarity of the detection, i.e. lighter or darker than the threshold, and (3), setting the resolution of the detection process in the scanning direction.

The operator has a choice of four monitoring displays to help him adjust the threshold level about which detection is to be made. These are 'image', 'meter', 'superimposed direct' and 'superimposed banded'. The 'image' is a TV picture of the field of view of the specimen which is to be analysed. The 'meter' display shows the input pulses to the integrator circuit which is displayed in the form of white pulses on a uniformly dark background. The 'superimposed direct' display shows the meter display just described and a brightened rectangle of the area in the field of view over which the instrument will be taking measurements (i.e. the blank frame). The 'superimposed banded' display is similar to direct display but with some of the scanning lines suppressed.

In practice, the operator selects the polarity and resolution of the detection process by turning the appropriate controls

to the desired positions. With display set on 'superimposed direct' or 'banded', he then selects the threshold level by adjusting the relevant detection control until the inclusion of interest is detected. The correct setting is that which shows the 'white' detected pulses (now superimposed on the image) to just coincide with the boundaries of the inclusions, as illustrated in Fig.22.

The threshold level may be adjusted in one of four ways referred to for convenience as Method A, Method B, Method C and Method D. In Method A, the operator proceeds as previously described using 'superimposed direct' display. The 'white' area is gradually increased and stopped when it coincides with the edge of the inclusion of interest. In this method care is taken not to over-detect the inclusion, i.e. to turn the detection control to a position where noise is detected. In Method B, the operator proceeds as in Method A but carries out the final adjustment while flicking the display switch between 'image' and 'meter' positions. Method C is similar to Method B but final adjustment is carried out with the polarity of the detection process reversed. In Method D, the operator quickly turns the detection control until noise is detected and turns it back until noise is just eliminated.

In order to determine which of these methods of setting threshold level is most reproducible, thirty replicate volume fraction determinations were made on oxide inclusions in a sample of low alloy steel by three operators using, in turn, Methods A, B, and C. Method D was not investigated since it was felt that the noise characteristics of different instruments would not be the same. The particular field used in this test contained a



large number of very small oxide inclusions, typically JK grading 4 (see Fig.3). This type of field was chosen because it was considered that there was less of a chance with this type of field of operators fixing their attention on one or two inclusions while adjusting the threshold level, and thus obtaining more reproducible results. Operators 1 and 2 were experienced while operator 3 had no experience at all in the technique.

A summary of the results obtained is given in Table 8 and is also shown in Figs. 23 and 24. They show that the overall reproducibility of the three methods of setting threshold level is not as good as might be expected, since each method is subject to a relatively large variation (standard deviation). On average, Methods B and C, i.e. the Flicker Methods, are less variable than Method A, both in an absolute sense and when the results are expressed as a percentage of the mean. A statistical examination of these results indicated that at 95% confidence level (two sigma) the variation between different operators was no greater than the variation between repeated determinations made by one operator.

#### 4.50 Relationship between threshold setting and camera sensitivity setting

In order to gain a better understanding of the relationship between camera sensitivity and threshold settings, inclusions in the same fields of view were examined at different threshold and camera sensitivity settings. The experimental procedure consisted of setting camera sensitivity to a definite value (using the manual control of sensitivity) and adjusting the threshold level until the previously determined area measurement for the inclusions in the field of view was recorded.

A typical result obtained on inclusions in one of these fields is shown in Fig.25, from which it will be seen that a perfect correlation exists between the two settings, and that a variation in camera sensitivity of only 0.075 division, as indicated on the meter, is equivalent to a variation in threshold setting of 20 divisions.

To relate this variation in camera sensitivity or threshold settings to actual volume fraction measurements of inclusions in individual fields of view, tests were made on both sulphide and oxide inclusions at different threshold settings using the automatic control of camera sensitivity. A typical result is shown in Fig.26, from which it will be seen that a variation in threshold setting of 20 divisions (or a variation in camera sensitivity of 0.075 divisions on the meter) is equivalent to a variation in volume fraction (area) measurement of 10% for oxide inclusions, and about 30% for manganese sulphide inclusions. Because of the flatter curve of the oxides, variations in threshold setting do not have as much effect on oxide measurements as they have on sulphide measurements.

#### 4.60 Further work on setting threshold level

The poor reproducibility of the three methods, and their subjective nature, make it appear unlikely that any of them would be suitable when high accuracy in the results is required. Methods B and C, although capable of giving slightly less variable results, had also given a relatively large scatter. It was considered moreover that this variability in the measurements would increase as more operators used the instrument. Obviously, a more objective method of setting threshold level is necessary. Accordingly, attention was directed upon alternative methods.

To make the task of adjusting threshold level entirely objective, the possibility of using artificial standards was first considered. Several attempts were made to produce a satisfactory

specimen. These attempts included using very fine wires, casting a lead alloy round them and sectioning the specimen to expose a transverse section through the wires, and using the standard specimen supplied with the instrument, but none produced adequately the contrast, size and distribution of real inclusions.

It is considered that standard specimens will be useful only if they truly represent the variations in contrast, size and distribution of real inclusions present in steel. This approach was, therefore, abandoned and the possibility of using real inclusions in steel samples was next considered.

It was thought that if a range of fields with different severities of inclusions of the relevant types could be selected and their area be assessed by the point counting method to determine the absolute area of inclusions present, then such fields could be used for setting the threshold level. It was appreciated that some quick means of identifying these fields would be required, and it was found that this could be done with micro-hardness indentations. The method for preparing standard specimens is described in Appendix I, Volume 2 of this thesis.

To compare the degree of reproducibility attainable with this new method of adjusting threshold level and that attainable with Method C described in Section 4.42, three operators determined the area of oxide inclusions in the same 100 fields. The procedure consisted of first making a determination after adjusting the threshold level using the standard field method and then repeating the determination after adjusting threshold level, using Method C. Because of the time consuming nature of this test, two specimens only were examined. The test was carried out with the X10 objective and with camera set on automatic control of sensitivity.

A summary of the results is given in Table 9, from which it will be seen that better agreement is obtained between operators when the new standard field method is used than when Method C is used for adjusting threshold level.

#### 4.70 Comparison of visual and Quantimet inclusion assessments

The Quantimet can be used to measure any of the following inclusion quantities:

1. Total number of inclusions.
2. Total projected inclusion length.
3. Total area of inclusions.
4. Inclusion size distribution.

To determine the accuracy with which the instrument could make the above measurements, a series of tests was made. The experimental method consisted of preparing standard photomicrographs with known number, total length and area of inclusions and examining each of these in the instrument. These standards were counted with the major axes of the inclusions 90 degrees to the scanning line. The epidiascope was used to project the image to be analysed on to the TV camera in the instrument. The conditions of the test were such that 0.8 mm square fields were used instead of 0.8 mm diameter fields as depicted on these charts. To determine the effect of counting inclusions with their long axes parallel to the scanning line, some of the standards were re-examined in this orientation, and in order to determine whether small orientation changes of inclusions greatly affected measurements, standard photomicrographs with different shapes of inclusions were also examined at rotations of 2, 4, 6, 8, 10 and 12 degrees from the direction of the line scan.

The results obtained from this work are presented in Figs. 27 to 32, from which it will be seen that there is good agreement

between the visual and automatic assessments for stringer oxide area and total length (projection) measurements, but not for the total count measurements. Better agreement is obtained for uniformly distributed inclusions. It will also be seen that long thin inclusions, if not perfectly parallel to the scanning line, are sized and counted differently from when they are strictly parallel to the scanning line. For example, in the case of an inclusion  $41\mu\text{m}$  long, an 8 degree rotation of the long axis of the inclusion from the scanning line results in an error of nearly 50% in sizing.

#### 4.80 Discussion

The results from this study of the automatic image analysing computer have shown that the blank frame size decreases by about 5% during the first 80 minutes of switching on of the instrument. After this time, the variations are generally less than 1%. This means that the blank frame need not be calibrated frequently, for example before examining each specimen. The manual control of camera sensitivity varied continuously throughout the 145 minute test period, rising from 0.7 to over 0.9 on the meter with an average variation within any one 10 minute period of 0.03 divisions. Better control of camera sensitivity was obtained on 'Automatic' with no detectable variation over the whole of the 145 minute test period.

A variation in camera sensitivity setting of 0.05 - 0.07 divisions is equivalent to a variation of 17-20 divisions on the threshold control, which, in turn, can be shown to be equivalent to a variation in percentage area measurements of about 30% for manganese sulphides and 10% for oxides. These results clearly indicate the need for constant monitoring of camera sensitivity and compensating for any drift by readjusting the threshold

setting. The reason why manganese sulphides are more sensitive to variations in camera sensitivity than oxides is because of the shape of the time-voltage curve and also because of the relatively steep curve obtained when percentage area measurement for manganese sulphide is plotted against threshold setting (see Fig.26).

The reproducibility of the computer and automatic data handling system was variable. High degree of reproducibility was obtained when the inclusions examined were large (5 mm or larger on the screen); smaller inclusions tended to be measured with less accuracy, and on some parameters, standard deviations of up to 5% of the mean measurement were frequently obtained. This variation in reproducibility of the instrument is due to some inclusions being near the limits of resolution. Although an inherent fault in the design of the instrument, this problem is not peculiar to the image analysing computer but to all microscopic methods which use an optical microscope system. When considering results from this instrument, or for that matter from any optical method, it is wise, therefore, to place less emphasis on the smaller inclusions, and, if possible, to set a limit for the size of inclusions to be included in the measurement at any given magnification. As far as the image analysing computer is concerned, this limit is about 3-5 mm on the screen depending on the contrast of the inclusions.

Results show that some improvement in the resolution of the system can be obtained by using the correct distribution of wavelengths of light. With the X5 objective, for example, maximum resolution was obtained with orange and yellow filters. With these filters a 20-50% improvement was obtained in percentage area measurements of small oxide inclusions (see Fig.21)

due to better resolution of the smaller particles.

In general, the precision of the automatic stage unit was considered to be adequate. Results obtained showed that the focus of the image was maintained over the entire X and Y traverses of the stage when using all except the X40 and higher objectives; with the X40 objective, the focus of the microscope needed readjusting every 4.16 mm approximately. It is considered that this is not a serious limitation but it means that fields for evaluation must be selected in blocks such that the X traverse does not exceed 4.16 mm.

When counting and sizing features, serious errors could arise. 'Rounded' inclusions can be accurately counted but inclusions with large re-entrant angles or preferred orientation are either over or under counted, depending upon their size and distribution. The counting and sizing of long thin inclusions such as sulphides and silicate inclusions parallel to the scanning line of the instrument is subject to large errors. In theory it is possible to count such inclusions correctly by adjusting the 'Acceptance Width' control of the instrument, but in practice, this is inconvenient with the automatic instrument.

A further complication arises when measuring oxides and sulphides simultaneously at two detection levels. Due to the edges of oxides appearing diffused, these areas are measured by the instrument as sulphides. The size distribution measurements of sulphides do not appear to be affected by this error but the area measurements are affected. The results obtained in this study show that sulphide area counts may be higher by 10-20% of the oxide area measurement. However, because of lower contrast and needle-like shape of manganese sulphide inclusions in longitudinal sections, they are not all properly detected and their

apparent over-estimation may, therefore, compensate for their under-detection in the first place.

The simultaneous counting of oxide and sulphide inclusions is undesirable for two other reasons:

1. The distribution of oxide and sulphide inclusions is different and, therefore, necessitates the use of different magnifications and sampling procedure.

2. The sulphides in wrought longitudinal sections are so thin that high magnifications must be used to pick them up, but this often results in a significant proportion of them extending more than halfway across the width of the screen, which can result in large errors in size distribution determinations. In view of these difficulties, it is doubtful whether simultaneous measurements of oxide and sulphide inclusions within a field of view is a practical proposition when using longitudinal sections from forged or rolled material, such as bars or small sectioned billets.

Examination of microscope and monitor methods of focusing showed that the former method is more accurate by a factor of nearly 2. This is due to the fact that the image is seen with greater clarity through the microscope than on the screen of the TV monitor. A variation of up to 5% can result due to differences in focusing on the screen. However, when using the microscope method it is essential to adjust the focus of the eyepieces so that the image is in focus on the monitor as well as when viewed through the microscope.

Errors due to variations in adjusting threshold can be quite large. It was found that none of the three methods advocated by users of this instrument for adjusting threshold level is accurate enough. The average coefficient of variation for the three



methods were 1.72% for the filling in method, 1.17% for the flicker method with polarity set on black, and 1.07% for the flicker method with polarity reversed, i.e. set on white. But it should be noted that these variations are for 1 standard deviation only and, therefore, approximately 30% of the determinations made would be expected to have values outside the stated variations. At a lower risk level, say 5%, the above variations would be doubled, but this means that only 5 out of 100 determinations would have values outside the stated values. It should also be noted that these variations are for threshold settings only and that the corresponding mean coefficients of variation for oxide volume fraction measurements were 13.19, 9.01 and 8.32%, respectively. This error, of course, is likely to increase as more operators use the instrument and, clearly, none of the methods used for setting threshold is sensitive enough for accurate quantitative applications in which it is necessary to detect small volume fraction differences between samples, i.e. differences of the order of 2-3%.

The use of standard specimens for adjusting threshold level results in better reproducibility. The use of standards, however, will not guarantee accurate results since this depends on the calibration of the standard, but it will reduce scatter. In many applications, reproducibility between operators or determinations is more important than absolute accuracy.

In general, the automating of the Quantimet has not presented any serious problems. However, some difficulty was encountered in getting the ICT 1905 to accept the tapes from the image analyzing computer. The difficulty was traced to misalignment of the tape in the punch. Two adjusting screws are available and these

must be adjusted so that the tape is correctly adjusted with the same spacing between the characters at the ends. It is worthy of note that this problem did not arise when a slower computer was used, such as an Elliot 903.

Although the punch supplied with the instrument performed satisfactorily, it would have been more convenient to have used a Teletype printer instead of an output writer and punch tape. The possibility of having a direct link up with a main computer is also worth considering, particularly if the instrument is to be used in process control applications.

#### 4.90 Conclusions

1. The automatic Quantimet at Loughborough University requires a period of 80 minutes approximately for it to reach stable operating conditions.
2. During the first 80 minutes of switching on of the instrument, the blank frame decreases by about 5% of the calibrated setting. After this period, however, it decreases by less than 1% over several hours.
3. The manual control of sensitivity is less stable than the automatic control of sensitivity. The variations in the former are too large for accurate measurements to be obtained.
4. The overall reproducibility of the instrument was found to depend upon the type and size of inclusions measured. Practically 100% reproducibility was obtained when measuring relatively dark, large inclusions (larger than 5 mm on the screen), but variations of up to 5% were frequently encountered when measuring inclusions smaller than 5mm on the screen.
5. The automatic stage was found to be capable of maintaining the focus of the specimen over the whole of its traverses in the

X and Y directions for all except the X40 objective; with this objective, focus had to be re-adjusted every 4.0 mm approximately.

6. The resolution of the optical system with the X5 objective was found to improve markedly with orange and yellow filters and with a lamp setting of 8 on the rheostat control.

7. The monitor method of focusing is less accurate than the microscope method by a factor of nearly 2.

8. The two 'flicker' methods of adjusting the threshold level were more accurate than the original 'filling in' method. The coefficients of variation associated with the former methods were 5 to 9% of the mean estimate compared with 12 to 15% for the latter method.

9. The use of a field containing known area fraction of oxide or sulphide inclusions for adjusting the threshold level was found to reduce the coefficients of variation between operators to 2 to 3% of the mean estimate.

10. The setting of the threshold level was found to be very critical and only small variations in threshold setting caused large variations in measurements, particularly area. A 1.7% variation in threshold setting caused a 10% variation in detected oxide area and a 30% variation in detected sulphide area.

11. Area and length (projection) measurements were found to be very accurate but total count and size distribution measurements were less accurate, and the accuracy of the measurements was found to depend upon the shape and distribution of the inclusions within the field of view.

12. The use of two detection levels for measuring oxide and sulphide inclusions simultaneously caused errors in sulphide measurements of between 10 and 20% of the oxide estimates, due

to the 'edge effect' of oxides. The use of two detection levels also caused a sulphide area measurement to be recorded when no sulphide inclusion was present.

#### RECOMMENDED OPERATING PROCEDURE

As a result of the above work, the following procedure was adopted for the operation of the instrument:

1. Switch on instrument including decode modules and ancillary equipment and allow 80 minutes to warm up.
2. Decide on magnification and set instrument on automatic control of camera sensitivity.
3. Using this magnification and specimen to be evaluated adjust step sizes in X and Y direction to cover the region of interest. Test stage to ensure it is correctly adjusted.
4. Set number of fields to be measured.
5. Calibrate blank frame size measuring its area and projection (no. of lines) on saturation as well as its actual height and width on specimen using a stage micrometer.
6. Select a 'standard field' and place it under the microscope.
7. Focus the microscope on this 'standard' image and check camera sensitivity. Adjust if necessary (see Manual, p.47).
8. Set polarity and resolution, the latter to maximum and Display switch to Superimpose Direct or Banded.
9. Make connections on patch board to measure area.
10. Adjust threshold using standard specimen until the known area measurement is recorded. Take this as the correct setting for the first detector unit. Repeat for second detector unit using the appropriate standard field if

second detection level is required. Replace standard specimen with specimen to be assessed, focus to give a sharp image.

11. Adjust minimum chord intervals if size distribution function of features is required. Convenient to adjust these controls one at a time with the actual specimen in view. Starting with Control No.1 at the top and working down. Ensure that the Display control is switched on.
12. Programme the computer making sure that all pins are pushed right into their respective sockets in the patch board.
13. Press all 'Reset' buttons.
14. Switch on 'Standby' on modules.
15. Set instrument to 'Run'.

To examine subsequent specimens, repeat operations 3, 10, 13, 14 and 15. Reset blank frame area every 30 minutes approximately.

5.00 CORRELATION OF QUANTIMET PARAMETERS WITH  
THOSE OF CM (SAE-ASTM), JK AND DIERGARTEN  
METHODS

5.10 Experimental procedure.

5.20 Results.

5.30 Discussion and conclusions.

## 5.00 CORRELATION OF QUANTIMET PARAMETERS WITH THOSE OF CM (SAE-ASTM), JK

### 5.10 Experimental procedure

### AND DIERGARTEN METHODS

As a first step in relating Quantimet measurements to chart measurements or ratings, it was decided to examine on the Quantimet each field shown on the SAE, JK and Diergarten charts (see Figs. 3, 4 and 5). For this examination, the microscope in the instrument was removed and the epidiascope used instead. The three charts examined were reduced in size so that when each standard field was projected on to the TV camera in the instrument, it was possible to accommodate the whole of the original field within the blank frame area of the instrument. It was found that this could be done with the  $1\frac{1}{4}$  inch (f 1.9) Dallmeyer objective when the standard 80 mm diameter fields were reduced to 30 mm. In order to keep the actual area of the fields the same as those of the chart, the blank frame size was adjusted to 80 mm height and 62.5 width which gave an area of  $0.5 \text{ mm}^2$  on the specimen. The procedure consisted of setting the threshold level using a standard field of oxide inclusions of known area and total length, replacing the standard with the chart to be analysed, focusing and making a determination of the area, total length and total number of features in the chart. All the determinations were made with the long axes of the inclusions 90 degrees to the scanning line. In view of the poor accuracy obtained when counting and sizing inclusions with their long axes parallel to the scanning line of the instrument, the charts were not examined in this orientation.

### 5.20 Results

The results obtained on examining the simulated inclusions on the SAE-ASTM chart (Fig.5) are given in Table 10. The results show percentage area, total number and total length of inclusions for each of the eight standard fields. Because of the error associ-

ated with number counts, the actual number of inclusions present on the chart is given in brackets. It will be seen that number counts are accurate only up to grading 6. It will also be seen that the percentage area measurements for silicate inclusions more or less doubles itself for successively higher gradings. The measurements for oxide inclusions, however, does not show the same regular pattern between fields. The counts and the total length measurements increase with an increase in grading number, but not in a systematic manner.

The results obtained on examining the JK-ASTM chart (Fig.3) are given in Table II. It will be noted that although the area measurements for silicate and alumina inclusions are similar for grading 1, the values for successively higher alumina gradings are greater, but the measurements for silicates and globular type oxides are very similar. The total length measurements show the same pattern between gradings. For total number measurements a marked difference exists between alumina and silicate fields. The former show consistently higher values than the latter but, again, accurate counts are not determined for higher gradings, viz. 4 and 5. On the other hand, the counts for globular type oxides are accurately determined at all grading levels and, obviously, are significantly different from either alumina or silicate counts.

The results for the Diergarten chart (Fig.4) are given in Table 12. It will be seen that the area measurements for each type of inclusion roughly doubles itself from grading to grading. The total length measurements also increase in a regular manner but the differences between types of inclusions are not sufficiently marked to enable each type to be separated on just area measurements alone. Total length measurements show the same differences between types and



gradings as area measurements. Total number measurements show marked differences between some types of inclusions but not for other types.

### 5.30 Discussion and conclusions

A detailed analysis of the machine numbers for the charts shows that the only two parameters could be relied upon: percentage area and total length measurements. The main problem in fully automating chart inclusion counting is in characterising the different types of inclusion fields in such a way that these can be separated on the basis of numbers. Unfortunately, the differences in area and total length measurements between the different types of oxide inclusions, as depicted on the charts, are not sufficiently large to enable a clear distinction to be made between them. A further complication arises in that actual fields containing more than one type of oxide cannot be separated as if they would appear alone. Conversion from machine numbers to chart ratings will, therefore, be possible only when the steels contain one type of oxide inclusion, or when it is not necessary to distinguish between different types of oxide inclusions. Any attempt to perform an automatic machine classification of inclusions in individual fields in a similar manner to a human operator would require a major increase in both computer ability and memory capacity. In any case, it is doubtful whether such a sophisticated instrument is needed in quality control work where the primary concern is to determine the presence, or absence, of any field in which the concentration of inclusions, whether oxides, silicates or sulphides, exceeds some specified limit. It is considered that the inability of the instrument to discriminate between alumina, silicate or other types of oxide inclusions is not a serious limitation in routine

work, since the instrument can be arranged to stop on 'over-value' fields which could then be inspected visually by an experienced operator to decide whether the high value is due to alumina, silicate or some other type of inclusion.

6.00    DEVELOPMENT OF DIGITAL COMPUTER PROGRAMMES  
         FOR PROCESSING OF QUANTIMET DATA

6.10    Details of Quantimet output.

6.20    Summarisation and presentation of Quantimet data.

6.30    Details of programmes.

1. Programme QTM 101/H086.
2. Programme QTM 201/H087.
3. Programme QTM 301/H088.
4. Programme QTM 401/H089.
5. Programme QTM 102/H090, QTM 202/H091.
6. Programme QTM 501.

6.40    Basic method of programmes.

6.50    Operating instructions.

## 6.00 DEVELOPMENT OF DIGITAL COMPUTER PROGRAMMES FOR PROCESSING OF QUANTIMET DATA

### 6.10 Details of Quantimet output

Data is recorded on paper tape using a system of coding shown in Fig.33. The equipment is designed to measure the oxide inclusions and then oxide plus sulphides. To facilitate this, two detection units are provided. Sulphide measurements are obtained by subtraction. The measurements on oxide inclusions are printed out first, followed by the measurements on oxide plus sulphides. The measurements are recorded as four-digit numbers. When fully programmed, the Quantimet measures fifteen parameters on oxide inclusions present in the field of view and a further fifteen parameters on oxides plus sulphides. The measurements are made in the following sequence:

Oxides:  $C_0 P_0 C_1 P_1 C_2 P_2 C_3 P_3 C_4 P_4 C_5 P_5 C_6 P_6 A$

Oxides + Sulphides:  $C_0 P_0 C_1 P_1 C_2 P_2 C_3 P_3 C_4 P_4 C_5 P_5 C_6 P_6 A$

where  $C_0$  = total counts

$P_0$  = total projection

$C_1 P_1 \dots C_6 P_6$  = measurements greater than a set intercept  
length in the scanning direction

$A$  = area measurement

The count measurements are for a certain size of field determined by the optical magnification of the microscope. It is customary to relate these measurements to one square millimetre on the sample. Thus, if the area of the field of view examined is  $0.25 \text{ mm}^2$ , then, the results are multiplied by 4. The projection measurements are fixed absolutely. For example, a measurement of 25 means that the scanning line had intercepted the inclusions twenty-five times during one complete scan of the field. By itself this measurement

has little value, but it becomes meaningful if it is reduced to a total length in millimetres or microns. This is done by saturating the blank frame and measuring the total number of lines in it, and noting what these are in terms of millimetres or microns by measuring the blank frame height with a stage micrometer. The actual measurement in length is obtained by dividing the actual measurement by the number of lines, and multiplying the quotient by the actual height of the blank frame measured in millimetres or microns. Similarly, the area measurement must be reduced to a percentage of the blank frame area by dividing the actual area by the area of the blank frame on saturation.

This brief discussion of the Quantimet output illustrates the problem of sorting out the Quantimet data before it can be condensed into a form where it is possible to see it as a whole rather than as measurements on individual fields.

#### 6.20 Summarisation and presentation of Quantimet data

Since quantitative metallographic values of inclusion counts are based on statistically small samples representing a large volume of frequently variable material, they may in no case be regarded as exact values. Usefulness of the values determined depends upon the reliable estimation of their accuracy and precision with reference to the actual mean values for the material samples. Actual variability of the material is itself an important factor in quality control, hence the reliability in the determinations for individual fields and specimens should be sufficient to permit separation of actual variability of the material from uncertainties of measurement.

The first stage in the process of summarisation of the data is to get the results into some sort of order. This is done by

going through the individual measurements and forming a Frequency Table, such as that shown in Table 13. This table summarises the oxide area measurements obtained on 100 separate fields on the same specimen. It tells at a glance the number of times (frequency) a variable quantity, called a variate (in this case oxide area measurements) takes a specified value in a given total number of occasions (the total frequency, here 100). Such a presentation of data may be adequate for quality control, but graphic portrayal of data becomes difficult with large numbers of different parameters. In such cases it is more convenient to express the results in terms of statistical constants which describe the shape of frequency distributions.

The average or mean is almost always the best measure of location of a frequency distribution, and, since it is the most 'representative' value, the average is the most valuable statistic for condensing data. The average is calculated, as is well known,

by:

$$\bar{X} = \frac{\sum_{I=1}^N X_I}{N}$$

where  $\bar{X}$  = the average

$X_I$  = the individual measurements

$\sum_{I=1}^N X_I$  = the sum of the individual measurements from the 1st to the Nth

$N$  = the number of determinations

Although this is an important statistical constant, its exclusive use carries the process of summarisation to the point of suppressing important detail. For example, it tells us nothing of the spread or variability in measurements.

The degree of variation can be measured by a number of statistical constants, of which the variance, the standard deviation

and coefficient of variation are the most important. These constants are calculated as follows:

$$\text{variance, } V = \frac{\sum_{i=1}^N (X_i - \bar{X})^2}{N - 1}$$

$$\text{standard deviation, } = V = \sqrt{\frac{\sum_{i=1}^N (X_i - \bar{X})^2}{N - 1}}$$

$$\text{coefficient of variation, } CV = \frac{V}{\bar{X}}$$

$$\text{or as a percentage of the mean} = \frac{V}{\bar{X}} \times 100$$

The use of (N-1) as the divisor in this formula may be explained by considering the standard deviation to be a root mean square of the independent difference within a measurement set. There is always one less difference in a set than the number of measurements (e.g. there is but one difference between two values).

An average oxide estimate of 0.20% area with a standard deviation of 0.02% means that 68.3% of the measurements determined will be within  $0.20 \pm 0.02\%$ ; 95.5% will be within  $0.20 \pm 0.04\%$ ; and 99.8% will be within  $0.20 \pm 0.06\%$ .

A knowledge of the standard deviation of a set of measurements thus enables sound inferences to be made about the results between samples. In general, if the difference between two means is greater than two standard deviations, the difference is considered to be real rather than <sup>due</sup> to pure chance.

### 6.30 Details of programmes

Seven programmes written in ALGOL have been developed for processing of Quantimet data. These programmes were developed with the assistance of the staff of Guest, Keen and Nettlefold Group Technological Centre, Wolverhampton, and the Computer Centre of

Loughborough University. The programmes developed are as follows:

QTM 101  
QTM 201  
QTM 301           For use with Elliot 903 computer  
QTM 401  
QTM 102  
QTM 202  
QTM 501

QTM H086  
QTM H087  
QTM H088  
QTM H089           For use with ICT 1905 computer  
QTM H090  
QTM H091  
QTM H115

6.3.1 Programme QTM 101/H086. This programme calculates the mean and standard deviation of counts, projected length and percentage area of one detection level (oxides in this case) using one data tape produced by the Quantimet.

Worked example: (typical read-out from computer)

QTM101

DATE 221168

FIELD WIDTH 0.30 MM

FIELD HEIGHT 0.40 MM

EXAMINED AREA 0.5 MM<sup>2</sup>

ONE DETECTION LEVEL ONLY

221168 0.4 0.3 0.5 5 0 1 5 10 40 2 0.5 1

"QTM 101 TEST DATA"

COUNT (NO GREATER THAN STATED SIZE IN MICRONS)

<u>SIZE</u>	<u>MEAN</u>	<u>STD.DEV.</u>
0.0 (total)	9.4000	4.5056
1.0	5.0000	4.0000
5.0	1.0000	1.4142
10.0	0.6000	0.8944
40.0	0.0000	0.0000



PROJECTED LENGTH MICRONS

<u>SIZE</u>	<u>MEAN</u>	<u>STD.DEV.</u>
0.0 (total)	30.8000	20.9929
5.0	3.8000	6.0992

PER CENT AREA

<u>MEAN</u>	<u>STD.DEV.</u>
0.0925	0.0789

NUMBER OF FIELDS SCANNED 20

NUMBER OF FIELDS REJECTED 0

FIELDS WERE USED IN 5 BLOCKS OF 4

The flow diagram and details of this programme are given in Appendix 2, Volume 2.

6.3.2 Programme QTM 201/H087 is similar to QTM 101/H086 but calculates on two detection levels - one for oxide inclusions and the other for sulphides. Details of this programme are given in Appendix 3, Volume 2. The flow diagram is similar to QTM 101.

Worked example (typical read-out from computer):

QTM 201

051268 0.20 0.17 0.5 5 0 15 10 40 2 0 5 1

"QTM 201 TEST DATA"

QTM 201

DATE 51268

FIELD WIDTH 0.17 MM

FIELD HEIGHT 0.20 MM

EXAMINED AREA 0.50 MM<sup>2</sup>

FIRST DETECTION LEVEL OXIDES

COUNT (NUMBER GREATER THAN STATED SIZE MICRONS)

<u>SIZE</u>	<u>MEAN</u>	<u>STD.DEV.</u>
0.0	18.0000	17.0000
1.0	5.0000	4.0000
5.0	0.0000	0.0000
10.0	0.0000	0.0000
40.0	0.0000	0.0000

PROJECTED LENGTH MICRONS

<u>SIZE</u>	<u>MEAN</u>	<u>STD.DEV.</u>
0.0	57.0000	50.0000
5.0	1.0000	1.0000

PER CENT AREA

<u>MEAN</u>	<u>STD.DEV.</u>
0.0313	0.0300

SECOND DETECTION LEVEL SULPHIDES

COUNT (NUMBER GREATER THAN STATED SIZE IN MICRONS)

<u>SIZE</u>	<u>MEAN</u>	<u>STD.DEV.</u>
0.0	24.0000	20.0000
1.0	24.0000	20.0000
5.0	20.0000	18.0000
10.0	5.0000	3.0000
40.0	0.0000	0.0000

PROJECTED LENGTH MICRONS

<u>SIZE</u>	<u>MEAN</u>	<u>STD.DEV.</u>
0.0	157.0000	141.0000
5.0	71.0000	66.0000

PER CENT AREA

<u>MEAN</u>	<u>STD.DEV.</u>
0.1980	0.1781

NUMBER OF FIELDS SCANNED 18

NUMBER OF FIELDS REJECTED 6

FIELDS WERE USED IN 1 BLOCK OF 15

6.3.3 Programme QTM 301/H087 is similar to QTM 101/H086 but can process more than one data tape. Also calculates the means and standard deviations on the complete set of data tapes.

The flow diagram of this programme is similar to QTM 101. Details of the programme are given in Appendix 4, Volume 2.

Worked example (typical read-out from the computer):

091268 0.4 0.3 0.5 5 0 1 5 10 40

2 0 5 1 3

QTM 301 TEST DATA

QTM 301

DATE 91268

FIELD WIDTH 0.30 MM

FIELD HEIGHT 0.40 MM

EXAMINED AREA 0.50 MM<sup>2</sup>

ONE DETECTION LEVEL ONLY

COUNT (NUMBER GREATER THAN STATED SIZE IN MICRONS)

<u>SIZE</u>	<u>MEAN</u>	<u>STD.DEV.</u>
0.0	9.4000	4.5056
1.0	5.0000	4.0000
5.0	1.0000	1.4142
10.0	0.6000	0.8944
40.0	0.0000	0.0000

PROJECTED LENGTH MICRONS

<u>SIZE</u>	<u>MEAN</u>	<u>STD.DEV.</u>
0.0	30.8000	20.9929
5.0	3.8000	6.0992

PER CENT AREA

MEAN

STD.DEV.

0.0929

0.0789

NUMBER OF FIELDS SCANNED 20

NUMBER OF FIELDS REJECTED 0

FIELDS WERE USED IN 5 BLOCKS OF 4

6.3.4 Programme QTM 401/H089 is similar to QTM 301/H088 but

calculates on two detection levels corresponding to oxides in one case and oxides + sulphides in the other. Also calculates means and standard deviations.

The flow diagram and details of this programme are given in Appendix 5, Volume 2.

Worked example (typical read-out from computer):

QTM 401

030169 0.20 0.37 0.5 5 0 1 5 10 40

2 0 5 1 3

QTM 401 TEST DATA

QTM 401

DATE 30169

FIELD WIDTH 0.37 MM

FIELD HEIGHT 0.20 MM

EXAMINED AREA 0.5 MM<sup>2</sup>

FIRST DETECTION LEVEL OXIDES

COUNT (NUMBER GREATER THAN STATED SIZE MICRONS)

<u>SIZE</u>	<u>MEAN</u>	<u>STD.DEV.</u>
0.0	9.0000	2.8284
1.0	2.5000	0.7071
5.0	0.0000	0.0000
10.0	0.0000	0.0000
40.0	0.0000	0.0000

PROJECTED LENGTH MICRONS

<u>SIZE</u>	<u>MEAN</u>	<u>STD.DEV.</u>
0.0	28.5000	9.1924
5.0	0.5000	0.7071

PER CENT AREA

<u>MEAN</u>	<u>STD.DEV.</u>
0.0336	0.0071

SECOND DETECTION LEVEL SULPHIDES

COUNT (NUMBER GREATER THAN STATED SIZE IN MICRONS)

<u>SIZE</u>	<u>MEAN</u>	<u>STD.DEV.</u>
0.0	10.0000	2.8284
1.0	11.5000	2.1213
5.0	9.5000	0.7071
10.0	2.5000	0.7071
40.0	0.0000	0.0000

PROJECTED LENGTH MICRONS

<u>SIZE</u>	<u>MEAN</u>	<u>STD.DEV.</u>
0.0	73.5000	4.9497
5.0	35.5000	3.5355

PER CENT AREA

<u>MEAN</u>	<u>STD.DEV.</u>
0.2043	0.1946

NUMBER OF FIELDS SCANNED 18

NUMBER OF FIELDS REJECTED 6

FIELDS USED IN BLOCKS OF 7

6.3.5. Programmes QIM 102/H090 and QIM 202/H091: both these programmes are similar to QIM 101/H086 and QIM 201/H087, but in addition give the frequency of fields with specified percentage area measurements. If the specified limits, which are variable, are taken to correspond to those of the visual chart gradings, the measurements are obtained directly in terms of the relevant chart gradings. QIM 102/H090 is for one detection level and QIM 202/H091 for two detection levels. Details of these two programmes are given in Appendices 6 and 7, Volume 2.

The flow diagrams are similar to programmes QIM 101 and 401 respectively.

Worked example (typical read-out for computer):

QIM 102

221168 0.4 0.3 0.5 5 0 1 5 10

40 2 0 5 1

QIM 102 TEST DATA

QIM 102

FIELD WIDTH 0.8 MM

FIELD HEIGHT 0.6 MM

EXAMINED AREA 0.48 MM<sup>2</sup>

ONE DETECTION LEVEL ONLY

COUNT (NUMBER GREATER THAN STATED SIZE IN MICRONS)

<u>SIZE</u>	<u>MEAN</u>	<u>STD.DEV.</u>
0.0	9.4000	4.5056
1.0	5.0000	4.0000
5.0	1.0000	1.4142
10.0	0.6000	0.8944
40.0	0.0000	0.0000

PROJECTED LENGTH MICRONS

<u>SIZE</u>	<u>MEAN</u>	<u>STD.DEV.</u>
0.0	30.8000	20.9929
5.0	3.8000	6.0992

PER CENT AREA

<u>MEAN</u>	<u>STD.DEV.</u>
0.0925	0.0789

FREQUENCY OF FIELDS

<u>PER CENT AREA SUB-RANGE</u>	<u>EQUIV. JK GRADING</u>	<u>PER CENT FREQUENCY OF FIELDS</u>
0.03	0	55
0.03 - 0.20	1	25
0.20 - 0.55	2	20
0.55 - 1.20	3	0
1.20 - 2.70	4	0
2.70	5	0

NUMBER OF FIELDS EXAMINED 100

NUMBER OF FIELDS REJECTED 0

FIELDS WERE USED IN BLOCKS OF 1

QTM 202/H091 presents the oxide measurements first and then the sulphide measurements using the format shown above. Details of this programme are given in Appendix 7, Volume 2.

6.3.6 Programme QTM 501; this programme is different from the programmes so far <sup>described</sup> in that it expresses the count and percentage area measurements as frequencies per 1000 fields, as shown below by a typical read-out from the computer.

6314 A

FIELD  $0.370 * 0.280 = 0.1036$  SQ MM

1 PARTICLE PER FIELD = 9.65 PER SQ MM

400 FIELDS

SERIES 1

FREQUENCIES PER 1000 FIELDS

PARTICLES PER SQ MM	NO. > THAN					
	0.0 $\mu$	3.0 $\mu$	10.0 $\mu$	20.0 $\mu$	50.0 $\mu$	100.0 $\mu$
0.0 to 7.0	443	923	993	1000	1000	1000
7.0 " 10	328	53	5			
10 " 15	0	0	0			
15 " 20	158	18	3			
20 " 30	48	5				
30 " 45	15	0				
45 " 70	10	3				

% AREA			FREQ
0	TO	0.02	870
0.02	"	0.03	48
0.03	"	0.05	40
0.05	"	0.07	20
0.07	"	0.10	3
0.10	"	0.15	10
0.15	"	0.20	5
0.20	"	0.30	3
0.30	"	0.45	3

FINISH



#### 6.40 Basic method of programmes

The first part of each programme reads a preliminary data tape specifying the Quantimet operating parameters, including the count and projected length sizes. It then forms, in an array BIN, a binary key to the Quantimet tapes, placing a 1 for each count size and a 0 for each projected length size, in the order of their appearance within each field. An array VAL contains the actual size levels and taken together with BIN, completes the 'picture' of the Quantimet tape. This part of the programme also decides the corrections to be made to the measurements and on how many fields shall be combined into each basic data block, if the "Examined Area" is less than the field area. This means that the statistical results of the distribution of inclusions are taken over several fields equivalent to the area over which it is desirable to assess the results and not over a field area which is dependent upon the optical magnification being used in the Quantimet. This approach is sometimes necessary as the Quantimet "sees" only part of the normal field of view.

The LININ procedure reads in a field of the Quantimet tape and partially checks its validity. A field will be rejected if it contains the incorrect number of words (i.e. the number of counts, projected lengths and area). A field will also be rejected if any word contains more than five digits, if there is a parity error or if there is an illegal character on the tape. The remainder of each of the first six programmes listed earlier carries out the size counting, calculates the means and standard deviation, and prints out the results. The seventh programme forms frequency histogram only.

It should be noted that on occasions when the sum of the 1st and 2nd detection levels is less than the 1st detection level,

which is obviously a Quantimet error, the programme will reject measurements on that field of view. Similarly, if the operator were to enter a value in "Examined Area" which is less than the area of the true field, then, the whole tape will be rejected.

#### 6.50 Operating instructions

Two data tapes are required for each programme. The first data tape includes the preliminary data tape and any titles when dealing with sets, and the grading limits when frequency histogram and/or chart grading are required. The second data tape contains the title of the Quantimet data tape which, for example, might be the sample number, ingot number, or cast number. It is only necessary to enter a second data tape with each subsequent use of the programme unless the preliminary data in the first data requires revising. A typical Quantimet Data Form for use in conjunction with an Elliot 903 computer is shown in Appendix 8, Volume 2.

7.00 INCLUSION ASSESSMENT IN SPECIFIC LOCATIONS

7.10 Introduction

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7.50 Macro-sampling errors

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7.60 Discussion and conclusions

1. Effect of section type

2. Effect of section size

3. Effect of number of fields examined

4. Effect of area of field examined

5. Effect of sampling conditions

## 7.00 INCLUSION ASSESSMENT IN SPECIFIC LOCATIONS

### 7.10 Introduction

Although new techniques for assessing the inclusion content of the steel are being actively pursued, the sampling and sample preparation fields still retain "art" characteristics, and have not been seriously studied.

In quality control work, sampling of a cast of steel usually consists of cutting a disc from the ends of a few billets representing different portions of the ingot and cast. Procedures for selecting and examining samples vary from steelworks to steelworks. In general, it is not convenient to take more than twelve specimens from each cast of steel on a routine basis, since the preparation and examination of more than twelve specimens impedes progress. However, with instruments such as the automatic Quantimet, more specimens, i.e. a larger sample, may be taken, but even with an instrument of this type, which is much faster than human operators, there is a limit to the amount of testing that can be carried out. What the steelmaker would like to know is the errors associated with procedures in which just one or two samples, i.e. specimens, are examined, and taken to represent billets which are several metres long. He would also like to know what improvement he could expect by doubling or trebling the sample size, and the variations in inclusion content within the same samples. Additionally, he would like to know how many fields should be examined on each specimen and how these should be selected, since it may not be possible to cover the entire polished surface of the specimen in the time available for inspection.

Although adequate techniques are available for metallographic preparation of specimens, two problems connected with sample

preparation can be additional sources of error. These are the use of different size billets and variations in planar sections used for inclusion determinations. In this part of the thesis, work carried out to investigate the above problems is described.

## 7.20 The effect of reduction in area on inclusion count determinations

7.2.1 Experimental details. To examine the effect of reduction in area of steel on inclusion count determinations, two 36 cwt (1828.88 kg) ingots were selected. These were rolled in the usual manner into 6 inch (15.2 cm) square blooms and a piece of material of suitable length selected from each. After selecting a  $2\frac{1}{2}$  cm thick slice, the samples were step-forged to provide smaller sections as follows: 15 cm x 15 cm, 10 cm x 10 cm, 7.5 cm x 7.5 cm, 5 cm x 5 cm and 2.5 cm x 2.5 cm diameter. Each of these sections was then cut in half through the longitudinal axis of the material parallel to the rolling direction of the billet, and one of the cut surfaces polished and examined on the Quantimet. One hundred fields, each  $0.5 \text{ mm}^2$  in area, were examined and the following parameters measured on each field: percent area, total number of inclusions, numbers greater than 10  $\mu\text{m}$ , 15  $\mu\text{m}$ , 20  $\mu\text{m}$  and 25  $\mu\text{m}$  and the total length of inclusions. The determinations were made with the X5 objective with which the system had a resolution of about 3.5 microns. The determinations were made on oxide inclusions only, and with their major axes parallel to the TV scanning line.

7.2.2 Results The results obtained are summarised in Tables 15-17. They show that percentage area and total length measurements decrease with a decrease in section size. In general, there is also a decrease in the number of inclusions greater than 20  $\mu\text{m}$  as the section size decreases. Between 5 cm x 5 cm and 10 cm x 10 cm sections, the effects of reduction in area are not very

significant.

### 7.30 Effect of variations in planar sections on inclusion determinations

7.3.1 Experimental details. A high sulphur free-cutting steel and a low alloy forging steel hot rolled into 5.71 cm square ( $2\frac{1}{4}$  in<sup>2</sup>) sections were used in this investigation. The reason for selecting these steels in the form stated was because they provided a wide range of inclusion quantities of interest for examination. The free-cutting steel contained predominantly manganese sulphide inclusions of widely varying sizes, while the low alloy steel contained needle-shaped manganese sulphides and alumina stringers of varying sizes.

Planar sections illustrated in Figs. 34 and 35 were carefully prepared using conventional metallographic techniques. Planar sections A and C were prepared from the same sample after it had been cut in half to provide two sections, while section B was prepared from an adjacent sample. Section A was taken parallel to the rolling direction and through the longitudinal axis of the billet; section B sloped from edge to centre of the billet making a 6 degree angle from the longitudinal axis of the billet; while section C sloped in the rolling direction of the billet and also made a 6 degree angle from the longitudinal axis of the billet.

Each of these sections was carefully polished and subjected to a detailed examination on the Quantimet. The examination was carried out with the X10 objective and the following measurements were determined: percentage area, total count and number of inclusions greater than 10, 20, 50 and 100  $\mu$ m. An assessment was made of both oxide and sulphide inclusions in each field of view, and one thousand fields were examined on each specimen. The

examination was carried out on unetched specimens, and with the long axes of the inclusions oriented parallel to the TV scanning line.

7.3.2 Results A summary of the results obtained in this investigation is presented graphically in Figs. 36 to 40 and in Tables 18 and 19. They show that a variation in sectioning of 6 degrees from the longitudinal axis of the billet, and in the rolling direction, can double the total number of inclusions counted, and, what is more important, it leads to serious under counting of larger inclusions. Mean percent area measurements are more or less the same for all three planar sections, but results shown in Figs. 38 and 39 show that there is a reduction in the count of number of fields with high values (severity ratings) for Section C.

#### 7.40 Micro-sampling errors

7.4.1 Experimental details By micro-sampling errors is meant the errors due to examination of too few fields of view. Due to the time-consuming nature of methods used for assessing the inclusion content of steels, it is not always possible to examine too many fields on each specimen, particularly when a detailed estimate of various parameters is required. Thus, it is of interest to examine the errors or variations resulting between determinations based on different numbers of fields, i.e. sample sizes.

Three samples of steel, labelled 1, 2 and 3, were used in this investigation. They were selected from low alloy forging steels made in an electric furnace by the double slag process. The samples were all selected from 5.71 x 5.71 cm square billets. They were selected to provide varying oxide contents. Sample 1 was graded as "dirty", i.e. as having a number of gross oxide inclusions, sample 2 was graded as "average", and sample 3 as "clean". These

gradings were purely arbitrary and were based on JK counts on fields selected from edge to centre of the specimens.

Longitudinal sections of these samples were prepared, as previously described, and the specimens examined on the Quantimet. The examination was carried out with the X10 objective, which gave a magnification to the TV monitor screen of 537 diameters. With this arrangement, the area of each field of view examined on the specimen was  $0.25 \text{ mm}^2$ , and the resolution of the system  $1.8 \mu\text{m}$ .

The fields were selected in a systematic manner from edge to centre of the billet sample so that it was possible to relate any measurement to a particular position on the specimen surface. One thousand fields were examined on each specimen, and on each the following measurements recorded: total number of oxide inclusions (i.e. numbers greater than  $1.8 \mu\text{m}$ ), numbers greater than 3, 10 and  $20 \mu\text{m}$ , and the area of each field occupied by oxide inclusions. It should be noted that the oxides were alumina, which was present as both "stringers" and as discrete uniformly distributed particles, similar to types B and D shown on the JK charts (Fig.3). A standard sample was counted before and after each determination to ensure that the instrumental error was kept less than 3% of the mean estimate.

7.4.2 Results The results are given in Appendices 9 to 11, Volume 2 of this thesis. Three sets of data are given: percentage area counts showing the mean estimates and the number of times the determination takes a value within certain specified limits in a given total number of occasions, and size distribution results. It should be noted that the limits chosen for the distribution table correspond to the six gradings of the JK count (Fig.3). Thus, if preferred, the first part of the tables may be read



directly as JK counts. It should also be noted that the results are expressed as frequencies per 100 fields for convenience even though only 25 fields were used to calculate the mean estimate. Grand summaries of these results are given in Tables 20 and 21, from which it will be seen that specimens 2 and 3 have very similar mean percentage area counts, with specimen 2 having slightly larger inclusions. Specimen 1 has not only a high percentage area count but also larger inclusions. The results given in the Appendices show that it is not possible to distinguish reliably between the three specimens on the basis of one determination only, i.e. the mean of 25 fields, because of the large variations between determinations.

In many laboratories, inclusion estimates are based on 100 fields. It was of interest, therefore, to examine the variations between each set of 100 fields chosen from edge to centre of the sample. The counts for each set of 100 fields were calculated and are shown in Figs. 41 to 44, from which it will be seen that large variations still exist between determinations for all parameters measured. It should be pointed out that only the first six sets of results, i.e. for 600 fields, are shown. However, these results clearly show that despite a variation of 100 per cent or more between determinations in some cases, it is possible to distinguish between the "dirty" specimen (No.1) and the two "cleaner" ones (Nos. 2 and 3), but not between the two "cleaner" ones.

As a matter of interest, it was decided to pursue this work further and consider the variation between the means of samples of varying sizes (i.e. number of fields). The results obtained for percentage area measurements are shown in Fig.47,

from which it will be seen that at least 200 fields, but preferably 300 fields, should be examined in order to distinguish reliably between inclusion contents of the three specimens.

Examination of the results obtained on different parts of the specimens (from edge to centre of the specimens) shows (Fig.46) that the sample graded as "dirty" (specimen 1) has a high concentration of oxide inclusions toward the centre, and it is interesting to note that if this part of the "dirty" specimen had not been examined, it might easily have been graded the same as the two "cleaner" ones (specimens 2 and 3).

In quality control, the estimation of the inclusion content of the steel usually takes the form of determining the absence or presence of fields with values greater than a specified limit. Fig.45 shows that the occurrence of fields with percentage area values greater than 0.20 and 0.55% (equivalent to JK gradings greater than 2 and 3 respectively), from which it will be seen that the curve for the "dirty" specimen (No.1) is markedly different from those for the two "cleaner" ones. For the "dirty" specimen, fields with values greater than 0.20% oxides occur at a frequency of two every 100 fields, whereas in the case of the two "cleaner" specimens (Nos. 2 and 3) they occur at the rate of less than 1 per 100 fields. For fields with percentage area values greater than 0.55%, the frequencies were 1 per 200 fields for the "dirty" sample, and none at all for the two "cleaner" ones.

## 7.50 Macro-sampling errors

7.5.1 Experimental details By macro-sampling errors is meant the errors due to examination of too small a sample, i.e. too few specimens. As stated earlier, the usual practice is to select one or two samples from the ends of billets several metres long.

It is tacitly assumed that this sample is representative of the whole billet.

Samples can be taken only from the ends of billets, and, therefore, it is desirable to investigate the variation in inclusion content along the whole billet so that sound inferences may be made. In the interest of economy it is also desirable to investigate whether or not the variation resulting from repeated determinations made on the same sample after repolishing is more or less than that resulting between samples taken along the length of the billet.

For this investigation two billets of low alloy forging steel were used. The billets were from the bottom of the ingot. Other details of the cast from which the two billets were selected are given in Table 22.

The billet marked P587 was graded as "clean", i.e. as being reasonably free from gross oxide inclusions, typically, fields similar to grading 3 on the JK chart. The billet marked T369 was graded "dirty". These gradings were made on the basis of sulphur printing results, JK counts and magnetic crack detection test results.

Each billet was cut up along its entire length every 25 mm approximately and provided 180 adjacent samples. Sample 1 was from the top end of the billet and sample 180 next to the bottom discard. Each of these samples was carefully identified and sectioned, as illustrated in Fig.34, polished, and examined on the Quantimet.

The examination was carried out with the X5 objective and yellow filter. With this arrangement, the resolution of the system was about  $3.6 \mu\text{m}$  and the area of each field  $0.5 \text{ mm}^2$ . All the samples were examined in the unetched condition, and on each sample one hundred fields of view were counted. It should be noted that

this is equivalent to examining two hundred fields with the X10 objective. On each field, which was selected automatically and in an unbiased manner by the instrument, the following measurements were determined: total number of inclusions, i.e. number of oxides greater than  $3.6\mu\text{m}$  (the limit of resolution of the system), their numbers greater than 5, 10, 15, 20 and  $25\mu\text{m}$ , total length of oxides (projection count), and the area of each field occupied by oxide inclusions. It should be noted that the oxides present in the samples were alumina which was present as both "stringers" and as discrete, uniformly distributed particles, similar to Types B and D on the JK chart (see Fig.3). A standard sample was counted before and after each determination, and the results accepted if the results of the two determinations were within 3%.

Three samples from billet P587 (Nos. 1, 53 and 126) were also examined after thirty repolishes in order to determine the variation in oxide content within samples. These three samples were selected on the basis of the first count which showed them to contain widely varying inclusion content. In either this work or the work on the entire billet, the sulphide inclusions were not counted. The inclusions were counted with the long axes of the alumina "stringers" lying 90 degrees to the TV scanning line.

## 7.52 Results

The results of the investigation on billets P587 and T369, to determine the variations between samples, are given in Tables 23 and 24, and Appendices 12 to 15, volume 2 of this thesis. The percentage area results are given in the Tables and the projection and size distribution results in the Appendices.

An inspection of the results given in Tables 23 and 24 shows that the variation in percentage area counts between samples for both billets is considerable. The between sample variation for the

"dirty" billet is greater, showing a variation ranging from 0.02 to 0.23% compared with 0.01 to 0.16% for the "cleaner" billet.

Examination of the summary results (Fig. 48) shows that the mean oxide content and standard deviations for the "dirty" and "clean" billets are  $0.061 \pm 0.0031\%$  and  $0.035 \pm 0.0018\%$  respectively. While this difference between the mean oxide content of the two billets is statistically significant, it is clear that a prohibitive number of samples from each billet will be needed in order to establish this difference reliably, since a large number of samples from the two billets give the same, or very nearly the same, mean percentage area values. Careful inspection of the results given in Tables 23 and 24 show that two adjacent samples, displaced by only 20-25 mm approximately along the billet vary by as much as 800 and 500% for the "dirty" and "clean" billets respectively. However, in general, the variations between samples for both billets are less than this, but still too large to enable the inclusion content of the two billets to be estimated reliably on the basis of one sample only.

Considering fields with percentage area values greater than 0.55% (equivalent to JK grading 3), it will be seen that these results also show considerable variation between samples. Taking a steel billet to be "dirty" when the sample representing it contains one or more fields with percentage area value exceeding a value of 0.55%, it will be seen that the "dirty" billet could have been classified as "clean" on 40 occasions out of 180; conversely, the "clean" billet could have been classified as "dirty" on 33 occasions out of 180. In order to distinguish reliably between the two billets on this basis counts on at least 20 samples are needed.

These results also show that samples with fields with high percentage area values (i.e. 0.55% and greater) tend to be localised on a macro-scale, ranging over several samples and that such

regions are not uniformly distributed along the billet.

The results of the repeated examinations of three specimens from billet P587 (Nos. 1, 53 and 126) are given in Tables 25 to 27 and Appendices 16 to 18. It will be seen that the variation between repeated determinations is still large, but not as large as that found between different samples taken along the billet length. It will be noted that sample No. 53 originally graded as having a high oxide content is rated a number of times the same as sample No. 126 originally graded as having a low oxide content. It will be seen also that the oxide content of sample 53 diminishes after the 16th repolish. This effect is probably connected with the segregation of oxide inclusions from edge to centre of the billet previously noted, since the effect of repolishing is to move the polished surface towards the edge of the billet. The original count would be through the longitudinal axis of the billet, whereas the count determined after the 30th repolish would be in a region 3-4 mm from the edge.

#### 7.60 Discussion and conclusions

1. Effect of Section Type. Inclusion counting methods based on the microscope rely heavily on the type and size of section used for accuracy. Since inclusions become deformed and oriented in the rolling direction of the billet, variations in sectioning the billet sample would be expected to influence the results, since the inclusions will be intersected in different positions. A detailed study of three different sections, as shown in Fig. 35, has been made using samples taken from low alloy forging and high sulphur free-cutting steels. Figs. 36 to 40 show various estimates obtained on such sections, which show that an error of only 6 degrees in sectioning the billet sample can double the total number of sulphide and oxide inclusions counted; and what is particularly important, it can

lead to serious under estimation of fields with high percentage area values and the number of large inclusions. The interesting point to note, however, is that mean percentage area counts (Fig.40) for both sulphides and oxides are unaffected by sectioning errors. The reason for the high total number counts for Section C (Fig.35) is probably due to the fact that in this section, the chances of intercepting inclusions are much better than in sections that are perfectly parallel to the longitudinal axis of the billet, such as Section A (Fig.34). The ideal section for total number counts would obviously be a transverse section. These results suggest that the determination of different parameters requires different sections.

2. Effect of Section Size. The effect of section size (in the range 15 to 2.5 cm) on oxide inclusion counts is shown in Tables 15-17. These results show that mean percentage area and size distribution counts decrease as the section size is decreased. This effect is considered to be due to both fragmentation and subsequent dispersion of the larger alumina particles during rolling.

Sulphide inclusions were not assessed, partly because they could not be properly resolved and partly because they were not considered detrimental to steel properties and machinability. But, had these inclusions been counted, a marked variation in the size distribution results would have been observed, since visual examination of the samples showed that as the section size became smaller, the lengths of the inclusions became greater.

The effects of reduction in area on inclusion counts noted are of great importance in any attempt at correlation of results, as the steelmaker frequently takes samples when the material has a considerably larger cross section than when sampled by forger or roller or eventual customer.

The results of this work show the importance of using the same size billets and sections for determination of inclusion counts, and if this is not possible to make due allowances for possible errors. It also shows that the same planar section need not necessarily be the best section for the determination of all inclusion parameters.

3. Effect of Number of Fields Examined. The results of the work on micro-sampling errors, given in Appendices 9-11, Tables 20 and 21, and Figs. 41 to 47, show that on low alloy forging steels with inclusion contents similar to those of samples used in this investigation, at least 300 fields should be examined in order to distinguish reliably between an acceptably "clean" steel and one which is just acceptable. A clean steel being defined here as one with a mean oxide content of less than 0.01% and containing no fields with percentage area values greater than 0.55%. The work shows that it would be better to look at 300 fields on the half of the specimen surface nearer the centre than over the whole surface extending from edge to centre of the billet sample. However, it is considered that more work is needed in this field before this practice can be firmly established.

4. Effect of Area of Field Examined. It is considered that it is not only the number of fields examined that is important but their area also. With the X10 objective, the Quantimet has an effective field area of  $0.25 \text{ mm}^2$  approximately. Under these conditions at least 300 fields should be examined, but with the X5 objective, which has an effective field area of about  $0.5 \text{ mm}^2$  (incidentally the same as the JK count), it is considered that the number of fields that should be examined need be only 100 to 150. Assuming the specimen area available for inspection to be  $600 \text{ mm}^2$ , 300 fields each  $0.25 \text{ mm}^2$ , or 150 fields each  $0.5 \text{ mm}^2$ , would give a 12.5% coverage, 100 fields 4%, and 25 fields 1% coverage. In many areas



of studies a 4% coverage will be considered a large sample.

5. Effect of Sampling Conditions. The results of the work on macro-sampling errors, i.e. the examination of too few samples, given in Appendices 12 to 15, Tables 23 and 24, and Fig.1, show that on low alloy forging steel with inclusion content similar to those of billets P587 and T369 used in this investigation, at least 20 samples should be examined in order to distinguish reliably between a "clean" billet and one which is not. The work shows that two adjacent samples, only 20 mm apart, can in one case be rated as "clean" and acceptable, and in the other case as "dirty" and unacceptable. The work also shows that a sample rated as "clean" on the first count can be rated as "dirty" after repolishing, i.e. after removal of only 0.5-0.75 mm of material. However, it is also clear from the results recorded that the chances of a "dirty" sample being rated "dirty" again after repolishing are high. At least three determinations on the same sample, made after repolishing, are needed to rate the sample reliably. When only one sample is used to determine the oxide inclusion content of a billet several metres long, a variation of between 500-800% in mean area counts has been observed. Strictly speaking each type of steel has its own sampling requirements and standards should be agreed between supplier and user. The work shows that it would be better to look at 100 fields on three different surfaces than 300 from the same surface.

## 8.00 GENERAL PROGRAMME OF INCLUSIONS ASSESSMENT

### 8.10 DISTRIBUTION OF OXIDE INCLUSIONS IN TWO ADJACENT INGOTS

1. Introduction
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### 8.20 RUNNING PROGRAMME AT A STEELWORKS INVESTIGATING LOW ALLOY STEEL BILLETS

1. Introduction
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3. Experimental details
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5. Discussion and conclusions

## 8.00 GENERAL PROGRAMME OF INCLUSIONS ASSESSMENT

### 8.10 Distribution of oxide inclusions in two adjacent ingots of low alloy steel

8.11 Introduction In routine work, such as quality control in steelworks, the general practice is to select the samples for inclusion assessment from billets representing different portions of the original ingot. Thus, samples may be taken from the top, middle and bottom billets of several ingots within a cast of steel.

This method of sampling, commonly known as representative sampling, is used in preference to random sampling, because it is suspected that variations in inclusion content exist between different portions of an ingot and cast of steel. By employing representative sampling, it is hoped to minimise the chances of some parts of the ingot supplying a disproportionately large portion of the samples. However, it has not been clearly established whether these variations are systematic, and which portion of the ingot has the largest inclusion aggregates. In fact, no detailed information has been published to show the variation in amount, total number and size distribution of inclusions between the different portions of an ingot and cast of steel.

Clearly, successful sampling for inclusion assessment demands a knowledge of the inclusion distribution, since on practical grounds it is possible to examine a limited number of samples only. Obviously, such a sample will in all probability be strongly biased, and the presence of any systematic variation and its magnitude should be determined and taken into account in assessing the results.

As part of a programme of work concerned with the development of an automatic inclusion counting method, it was decided to use the new automatic Quantimet (shown in Fig. 17) to determine whether or not systematic variations in amount, type, total number and size of

oxide inclusions exist between samples drawn from various portions of two adjacent ingots of low alloy steel. It was also decided to determine whether a correlation exists between the Quantimet method and other laboratory techniques used for assessing inclusion content.

It was appreciated that ingots are individual to particular steelworks and steelmaking processes, and that the results obtained in this work may not be representative of those obtained on ingots produced by other steelworks and steelmaking processes. Nevertheless, it was believed that, by confining our investigation to ingots of one quality at a single works (in order to develop our techniques of sampling and our studies of correlations with other techniques of examination), it would be possible to establish the suitability, or unsuitability, of the Quantimet as a quality control instrument. This would be of value in attempts now being made to set up International Standards of cleanness assessment.

In this study, particular attention has been given to the selection of ingots, since it was felt that any work of this kind would be useful only if it were performed on ingots which have "troublesome" inclusions. In the past, ingots for detailed study have been selected at random. In this approach, it is left to chance as to whether or not they contained the type and size of inclusions that are generally considered to be harmful. It is possible to select an ingot in which the incidence of such inclusions is so low that it is virtually impossible to obtain any useful quantitative information about them.

The two ingots chosen for this study were selected on the results of step-down tests and sulphur print tests which had indicated that they may contain a slightly higher incidence of harmful inclusions than normally present in this quality of steel. The ingots selected

were considered to be typical of those which, on limited sampling, may be graded as having inclusions larger than 1 mm long.

8.12 Experimental details Details of the two ingots are given in Table 28. The ingots were labelled 1 and 2. They formed the last group in a series of five produced by bottom pouring. It should be noted that a piece, 55 kg (10 cwt.), was left over in the ladle after teeming. After rolling, each ingot produced 10 billets, 73 mm (2.7/8") in section and 394 to 398 cm (12'11" to 13'1") long.

Adjacent samples (for the different tests) were cut from the ends of each billet. The exact position of these samples in the original ingot is given in Table 29 and Fig.49. The samples for sulphur printing and examination on the Quantimet were in the form of discs 12 mm (0.5") thick. Those for the step-down and magnetic crack detection tests were 25 cm long, while the samples for oxygen determination were 15 cm long. These samples were subsequently hot forged into round bars, 32 mm in diameter.

The samples for sulphur printing required no preparation. Those for the Quantimet were cut up to provide two specimens, A and B, as illustrated in Fig.49. As can be seen, specimen A represents material from edge to mid-centre position of billet, while specimen B represents material from mid-centre to centre plus 5 mm. All specimens were ground on silicon carbide emery papers and diamond polished to a 0.25  $\mu$  finish. Two samples for oxygen determination were cut from the forged bars and machined all over to give specimens 25 mm (1") dia. and 12 mm (0.5") thick. The samples for step-down and magnetic crack detection tests were first rough machined and then finished to dimensions shown in Fig.50. The final machining was carried out under the following conditions:

Depth of cut	0.2 mm
Feed	0.2 mm
Cutting speed	150 m/min
Nose radius of tool	0.8 - 1.2 mm
Side angle	75 degrees approx.

After testing, the test pieces were cut in half along their axes and the two halves of each test piece re-examined as described later.

The specimens for examination on the Quantimet were initially subjected to a careful visual examination using both low (X100) and high (X600 and X1000) magnifications to determine the type of inclusions present in the specimens, and also to ensure that the surface to be subjected to Quantimet assessments was satisfactorily polished.

After this preliminary examination, the samples were assessed for oxide inclusions content on the Quantimet using a standard operating procedure. The test was carried out with the X10 objective (X537 magnification to TV monitor screen) and a field area of  $0.25 \text{ mm}^2$ . The entire surface of the specimen 2 cm x 1.5 cm approximately, was examined, and the following measurements determined on each field: total number of oxides down to the resolution of the instrument ( $1.8 \mu$ ), number of oxides greater than 3, 10, 20, 50 and  $100 \mu$ , and percentage of each field ( $0.25 \text{ mm}^2$ ) occupied by oxides. The test was performed using unetched specimens, and with the long axes of the inclusions lying parallel to the TV scanning line. A standard sample was used to adjust the threshold level of the instrument before examining each specimen, and 1000 fields were assessed on each specimen.

Total oxygen was determined by irradiating each specimen for ~~30 minutes~~ <sup>5-6 CS.</sup> with high energy neutrons, produced by the Analox equipment, and measuring the resulting gamma radiation.

The step-down test pieces were examined visually in good light, and the numbers and aggregate lengths of inclusion noted. The test pieces were then cleaned and tested on a magnetic crack detecting machine, using a current of 1000 amps. As in the step-down test, the numbers and aggregate lengths of inclusions were noted. The examination of the step test pieces cut in half followed the same testing procedure as described above. Several of the inclusions detected were examined under the microscope and on the electron probe microanalyser.

Sulphur printing was performed in the usual way using a 3%  $\text{H}_2\text{SO}_4$  solution in distilled water, followed by visual inspection of the prints for dark brown stains. If one or more stains were visible, the sample was classified as "spotty", indicating possible presence of "large" inclusions, or inclusion segregates in the sample.

### 8.13 Results

Metallographic examination Manganese sulphide inclusions predominated in all specimens; these were thin and needle-like in shape. At high magnification (X1000), a number of these sulphides had small angular oxides embedded in them. All specimens also contained oxide inclusions, which took three forms: discrete particles, stringers with no second phase, and stringers with second phase. Examination of inclusions revealed by the step test showed that the appearance of these was similar to the stringers with second phase found in the Quantimet specimens. The composition of these different oxides, as determined by the electron probe microanalyser, is given in Table 30. It will be seen that the discrete particles of oxides and stringers with no second phase are alumina ( $\text{Al}_2\text{O}_3$ ), while those with a second phase are manganese-alumino-silicates. It is worthy of note that all three types of oxide described above contain between

2-4% CaO. The oxides embedded in some of the sulphide inclusions were too small for accurate probe analysis, but traces taken across these inclusions indicated that they were rich in aluminium.

Quantimet assessments A detailed summary of the Quantimet percentage area and size distribution results obtained on 66 specimens, representing different portions of the two original ingots, are given in Tables 31 to 38. It will be seen that relatively high mean percentage area values occur in the axial middle and bottom positions (sample B, Fig. 49). These positions of the ingot also have more larger inclusions. As the inclusion size range and percentage area range increase there is an increase in the variability of the measurements. Over 85% of the fields examined have percentage area values in the range 0 - 0.03%. Of the remainder, the majority have values in the range 0.03 - 0.20%. Fields with percentage area values corresponding to JK grading 3 or greater (i.e. 0.55%) occur at a frequency of less than 1%, i.e. 1 per 100 fields. Such fields occur randomly anywhere in the ingot, but they appear to occur more frequently in the axial regions of the ingot, particularly in the middle and bottom positions, i.e. 44-63% and 90 to 99% from the top of the ingot. It will also be seen that the majority of oxides are less than 10 $\mu$ m, and that very few, and then only in some specimens, are larger than 100 $\mu$ m. The mean percentage area measurements do not always reflect these variations in oxide content.

Oxygen determinations Oxygen determinations made on billet samples representing different portions in ingots 1 and 2 are given in Table 39. These results show the mean of two determinations each made on a separate sample.

The results show considerable scatter. As in the case of the Quantimet assessments, the bottom and middle portions of the two



ingots are associated with relatively high oxygen values compared with other portions of the ingot. The mean oxygen contents of both ingots are the same (0.0053% for ingot 1 and 0.0050% for ingot 2). As shown in Appendix 19, a good correlation exists between oxygen contents determined by neutron activation and Quantimet methods.

Sulphur printing These results are given in Table 40, from which it will be seen that samples representing the middle and bottom portions of the two ingots are classified as "spotty", indicating the presence of "large" inclusions in the samples, while those from other portions in the ingot are classified as "clear".

Step-down tests (visual and magnetic crack detection) The results of these two tests are given separately for ingots 1 and 2 in Tables 41 and 42 respectively. The tables show the number of inclusions counted and their aggregate length for steps 1, 2 and 3. In these tests, only inclusions 1 mm or greater in length were counted. The results obtained after cutting the test pieces in half are given in Appendix 21.

It will be seen that more inclusions are counted by magnetic crack detection than by visual inspection. Some of the highest estimates are recorded against samples representing the middle and bottom portions of the two ingots. In a number of instances steps 2 and 3, i.e. material nearer the centre of the billets, are associated with higher counts than step 1. This is shown more clearly in Appendix 21 which shows (on a 1:1 scale) the size and position of the actual inclusions found in the step test pieces after they had been cut in half along their longitudinal axes, i.e. in 66 halves. For convenience, the inclusions occurring in the two halves are shown as if occurring on the same plane.

#### 8.14 Discussion and conclusions

The results obtained in this investigation show that, although high oxide estimates can occur at any position from top to bottom, and from edge to centre, of the original ingot, there is a higher probability of them occurring in the middle and bottom portions, particularly in the central regions of the ingot. What is particularly interesting is that this trend is indicated in results obtained by the different methods employed to assess the inclusion content of the samples.

A statistical treatment of the Quantimet percentage area measurements and total oxygen contents determined by neutron activation, given in Appendix 19, shows that the correlation between determinations made by these two methods is highly significant.

While there is also an obvious correlation between the Quantimet, sulphur printing and step-down assessments, the results obtained in this work clearly show that the Quantimet is not a particularly useful tool for detecting and measuring inclusions larger than 1 mm, simply because of the manner in which these "larger" inclusions are distributed in steel. This is brought out clearly in Appendix 21, from which it will be seen that the distance between inclusions is often considerably larger than the size of sample normally used in microscopic examination.

The stringer inclusions, with a second phase, are believed to belong to a different inclusion population from those with no second phase. The source of the latter is in the deoxidation of the steel, but the former is a matter for further study.

This work has yielded useful basic information on the type, size, amount and distribution of oxide inclusions in two adjacent ingots of low alloy steel, and has shown that significant systematic

variations in inclusion content exist between samples selected from different portions of the two ingots. However, it is obvious that more data is needed before it can be established whether or not these variations in oxide content follow the same pattern in the majority of ingots produced by a given steelmaking process.

#### 8.20 Running programme at a steelworks on low alloy steel billets

8.21 Introduction The investigation of distribution of oxide inclusion contents in different portions of two adjacent ingots had shown that fields with high counts were more numerous in the central, middle and bottom portions of the two ingots.

Knowledge such as this is of great value in designing an economical and successful sampling procedure for inclusion assessment; since the steelmaker can concentrate on selecting and examining samples from those portions of the ingot where the chances of finding fields with high inclusion values are greatest.

This of course is biased sampling, but as the practical problem usually takes the form of determining the presence or absence of any field in which the concentration of inclusions exceeds some specified limits, there is little point in wasting money and valuable time examining samples drawn from the cleaner portions of an ingot or cast.

However, it is still to be shown that systematic variations in oxide inclusion content exist between samples drawn from different portions of an ingot and cast. Although the previous investigation of distribution of inclusions in two adjacent ingots had provided useful information, the work had been undertaken on two ingots specially selected for their relatively high inclusion content, and it was felt that whilst they may be typical of "dirty" ingots, they may not be representative of the majority of ingots manufactured by this process over a range of casts.

This was investigated during work performed at a steelworks over eight months during which period more than 1000 samples and several thousands of determinations were made. The work was limited to the investigation of low alloy steels (mainly specifications SAE 8620 and En 16). This was necessary in order to minimise the influence of steelmaking variables.

8.22 Cast details Details of the casts of steel used in this investigation are given in Tables 43 to 45. With a few exceptions, they were all to specification SAE 8620 or En 16. They were normal production casts selected at random, and were made in an electric arc furnace using different finishing processes. Some of the casts were made by the single slag process; some by the double slag process; some by single slag vacuum degassed process, and some of the casts were "grain refined", which was achieved by making controlled additions of aluminium to the "trumpet" during teeming. Deoxidation was effected by SiMn to the melt in the furnace and finishing with Al to the ladle. Thus, the steels examined may be classified into five groups depending upon the finishing process employed, as follows:

- A - Single slag
- B - Single slag, grain refined
- C - Double slag, grain refined
- D - Single slag, vacuum degassed, grain refined
- E - Single slag, vacuum degassed

All casts were rolled in the normal manner into billets whose identity in the ingot and cast were carefully observed.

8.23 Experimental details In view of the time consumed in preparing and determining total number, size distribution, total projection (aggregate length) and volume fraction (percentage area)

measurements on a statistically valid sample of the polished surfaces of the specimens, it was considered that the number of samples taken from each cast of steel should be reduced to a reasonable number. It was decided to select one ingot from each group that was cast, and from each ingot to select four samples along it corresponding to positions 6%, 53%, 90% and 99% respectively from the top of the ingot including the discard. It was found that this could be achieved by taking samples from the ends of the bottom billet, from the bottom end of the middle billet and from the extreme end of the top billet.

Longitudinal sections were selected and polished in the conventional manner using diamond lapping compound. All specimens were finished to a  $1\mu$  finish which was found to be adequate. During the sectioning and polishing operations, care was taken to ensure that a true longitudinal plane through the centre of the billet, and exactly parallel to its rolling direction was being prepared for examination, as a previous investigation (described in this thesis) had shown that variations in sectioning of the sample could result in relatively large errors in measurements.

The examination of the samples on the Quantimet followed the same lines as previously described. Details under which this examination was made are given in Table 46. It should be pointed out that the selection of fields was done automatically by the instrument, and that all samples were examined unetched after a preliminary visual inspection of the polished surface to ensure that the standard of polish was satisfactory. Oxide inclusions only were assessed.

In one investigation fifty-six samples were examined. These were drawn from Casts P200, 2542 and 2387. Each sample was examined in four areas, referred to here as Zones 1, 2, 3 and 4. Zone 1 covered the area edge to 4 mm; Zone 2, 4 to 8 mm; Zone 3, 8 to 12 mm;

Zone 4, 12 to 16 mm, i.e. to the centre plus 3 or 4 mm beyond. These samples were examined on the Quantimet under conditions A (Table 46).

In another investigation 52 adjacent samples from one of the bottom billets of Cast P587 were examined in two areas only, which corresponded to Zones 1 + 2 and 3 + 4 above. These samples were examined on the Quantimet under conditions B (Table 46).

The samples from the remainder of the casts including Casts P200, 2542 and 2387, were examined in one area only, extending from mid-centre to centre position of the billet plus 3-4 mm beyond, i.e. Zones 3 + 4. These samples were examined on the Quantimet also under conditions B (Table 46). It should be stressed that in this and in the other examinations a standard sample was used to adjust the threshold level of the instrument before examining each sample. This was necessary in order to minimise the influence of operator bias.

#### 8.24 Results

##### Variation in oxide content from edge to centre of billet

A summary of the results obtained on the 56 samples from Casts P200, 2542 and 2387 is given in Table 47 and Figs. 51 and 52, while a summary of the results on the 52 adjacent samples from a bottom billet of Cast P587 is given in Table 48 and Figs. 53 and 54. The tables also give the standard deviation of the mean percentage area and projection counts, and show the degree of variation that exists between fields, i.e. between 5500 in one case (Table 47) and 5100 in the other (Table 48) for determinations made within each zone. Figs. 51 and 53 show the distribution of fields with percentage area counts greater than 0.2%, i.e. equivalent to a JK grading of 2, whilst Figs. 52 and 54 show the distribution of mean percentage area determinations. The standard deviations shown in these instances

are not the variations between fields but the variations between the means of 100 fields examined on each zone.

From an inspection of the mean percentage area results (Figs. 52 and 54), it is obvious that there is no difference between the estimates determined on different portions of the sample. However, from an inspection of the measurements obtained on individual fields (Tables 47 and 48, Figs. 51 and 53), it is clear that there is a tendency for the number of fields with percentage area values in the ranges 0.55 - 1.20%, 1.20 - 2.70% and  $> 2.70\%$  to increase as the sample is traversed from edge to centre of the billet. It is interesting to note that neither the <sup>mean</sup> size distribution nor the <sup>mean</sup> projection counts show these variations to exist in oxide inclusion content between the different portions of the sample extending from edge to centre of the billet.

Variation in oxide content between billet samples drawn from four positions in the original ingot

Complete results were available for fourteen casts of steel (53 ingots) only, and these are given in Appendices 22 to 29. They show for each sample examined, the frequency of fields within six specified percentage area ranges, the mean area and projection estimates and their standard deviations, and size distribution counts. Summaries of these results are shown in Figs. 55 to 58, from which it will be seen that samples drawn from the 90 and 99% positions from the top of the ingot contain the highest oxide content. The billet samples drawn from these portions of the ingot have not only higher mean percentage area and projection counts (Fig. 55) but also more fields with percentage area values greater than 0.55%, i.e. fields with inclusion content equivalent to JK grading of 3 or greater (Fig. 56); they also have slightly more larger inclusions (Fig. 57).

Fig.58 shows the percentage of samples from each of the four positions along the ingot in 53 ingots that were graded worst with respect to frequency of fields with percentage area values of 0.2% or greater. It will be seen that 80% of these samples are positions from the bottom of the ingot and one billet length away from the bottom; 15% are from the middle of the ingot; and only 5% are from the top of the ingot.

Variations in oxide content between billet samples drawn from different groups in the cast

In order to determine whether or nor systematic variations in oxide inclusion content exist between ingots drawn from various groups in the cast of steel, the results obtained on 45 ingots from different casts of steel were arranged into groups. Group 1 consisted of results obtained on those ingots selected from the first group in the cast; Group 2, those selected from the second group, and so on. The results for four such groups are given in Table 49 and Figs. 59 and 60, from which it will be seen that no systematic variation in the mean percentage area, projection or size distribution measurements exists between <sup>centre</sup> samples drawn from different groups. The results when expressed as number of fields within six specified percentage area ranges, however, show (Table 49 and Fig.59 ) that in the range greater than 2.7% one field for Group 3 and two fields for Group 4 ingots are encountered, whereas none are recorded for ingots from Groups 1 and 2. It is unwise to read too much into these differences between samples from different groups, but this is worth bearing in mind, since there is a slight indication that ingots teemed at the end of the cast may contain more fields with higher percentage area values than those teemed at the beginning of the cast.



Variations in oxide inclusion content between samples drawn  
from casts made by the same and different finishing processes

In order to determine whether variations in oxide inclusion content exist between casts of steel made by the same and different process, the mean percentage area and projection estimates for each cast of steel were calculated from the results given in Appendices 22 to 29. A summary of these mean estimates is given in Tables 50 to 52, and a statistical examination of the percentage area data in Table 53. It will be seen that the variation in oxide content between samples drawn from different casts of steel and made by the same finishing processes is small compared with variations between samples drawn from casts of steel made by different processes.

On the average, casts made by the single slag process show the highest oxide content. When aluminium is added to these single slag casts, there is a tendency for the steels to become cleaner. Vacuum degassing of single slag casts results in lower oxide content, and subsequent treatment of these casts <sup>with Aluminium</sup> does not result in steels with higher oxide content. But none of these finishing processes produces steels as low in oxide inclusion content as casts made by the double slag process. It is interesting to note that although the mean percentage area estimate of single slag vacuum degassed and grain refined casts is lower than that of single slag grain refined casts, the standard deviations of the two groups of casts are very similar, indicating that the vacuum degassed casts must have contained a significant proportion of fields with high percentage area values.

8.25 Discussion and conclusions The results obtained in this investigation have shown that on the average "clean" steels do not show such marked systematic variations in oxide inclusion content from edge to centre of the billet as do "dirty" steels. Nevertheless,

the central axial portions of the "clean" billets were found to contain slightly more fields with percentage area values greater than 0.55% than portions nearer the edge of the billet.

On the other hand, marked systematic variations in oxide inclusion content exist between samples drawn from the four positions along the ingot, the samples from the extreme bottom end (next to the discard) and one billet length away containing the highest oxide content. A careful analysis of results of samples from each ingot rated as being the "dirtiest" with respect to the number of fields with percentage area values of 0.2% or greater showed (Fig.58) that 80% of these samples were drawn from these positions, i.e. from the bottom portion of the ingot; compared with 15% from the middle position and only 5% from the position in the top of the ingot.

Previous investigations by Dickenson (139), the Iron and Steel Institute (140) and Salmon Cox and Charles (141) have shown that silicate and oxide inclusions tend to segregate to the bottom of the ingot. This was found to occur in both small and large commercial ingots. However, it has also been reported that the "dirtiest" portion could be in other regions of the ingot. In the Iron and Steel Institute's work (140) on heterogeneity of steel ingots published in 1932 for example, it was shown that ingots could be classified into three categories depending upon where the greatest concentration of silicates occurred. As a general rule, the highest concentration occurred in the central lower part just above the radius of the bottom. But in some ingots this maximum was three quarters of the way up the ingot, while in still others it occurred at about 55% from the top, i.e. very near the middle of the ingot. More recently Sewell and Wilcock (44) have shown that billet samples representing the bottom of the ingot have the highest inclusion

content as determined by the Fox inclusion count method. Clearly, the results obtained in this work are in very good agreement with the above qualitative results.

The results obtained in this work have also shown that no systematic variation in oxide inclusion content exists between samples drawn from different groups. As in the edge to centre study there was, however, a slight indication that the samples from the last two groups may have contained slightly more fields with high percentage area values than those from the first two groups, but this could not be firmly established.

As is to be expected, the variations (standard deviations) between the mean percentage area estimates on a set of fields are less than those between individual fields. It has also been observed that the variation between casts made by the same finishing processes is less than that between casts made by different processes. The fact that the Quantimet was able to detect these relatively small differences between casts made by different finishing processes is very encouraging indeed, because it shows that the instrument can be usefully employed for checking and controlling the oxide inclusion contents of steels.

Of the three inclusion measurements determined, percentage area, projection and size distribution, percentage area counts were found to be most sensitive to small variations in oxide content, particularly when the results were expressed as a frequency distribution showing the number of times values within different ranges were counted in a given number of fields.

## 9.00 SAMPLE PREPARATION - THIN FILM INTERFERENCE METHOD

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## 9.00 SAMPLE PREPARATION - THIN FILM INTERFERENCE METHOD

### 9.10 INTRODUCTION

Assessment of non-metallic inclusions on the Quantimet is dependent upon differences in optical reflectivity between inclusions and matrix material, and between the constituent phases of the non-metallic material. In turn the sensitivity will depend on the intensity and wavelength-distribution of the incident illumination.

In the case of manganese sulphide and oxide inclusions these differences in reflectivity are sufficiently great to allow identification to be made directly under normal white light, but sometimes this is not so. For example in the examination of forging steels, silicate and alumina inclusions could exist side by side in the same field of view. As the reflectivity of these two types of inclusions is very similar they cannot be distinguished from each other on reflectivity alone. Another example is in the examination of sulphide and silicate inclusions. Large inclusions are easily distinguished and measured separately, but small inclusions are not. A further example is in the examination of sulphide inclusions in high-sulphur free machining steels; the manganese sulphide phase of the inclusion is easily discernible, but the lead telluride phase is not. These situations can lead to serious errors of assessment when using automatic counting procedures such as the Quantimet, since an instrument of this type is less sensitive to small differences in reflectivity of the inclusions than is the human eye.

As part of a programme in which the Quantimet was being used in the automatic assessment of non-metallic inclusions in steel, it was decided to investigate the extent to which the contrast of inclusions could be improved by the deposition of thin optically

transparent films of intermetallic compounds on the surface of a polished metallographic specimen. A wide range of evaporation sources is now available, as a result of developments in the optical and electronic industries. In the work described here the source used was zinc selenide (ZnSe).

The technique which follows was originally developed by Pepperhoff et al. for revealing retained austenite in a martensitic matrix (see references 142 to 158).

#### 9.20 Apparatus

A vacuum coating unit of the type normally used for preparing electron microscopy specimens was used, where the following additional considerations arise:

1. The thickness of the deposited film is determined visually by observing the interference colours forming on the surface of the specimen, so that at all times a good view of the polished surface of the specimen is necessary.
2. The interference colour (and, hence, the optical contrast) is a function of film thickness, so that the apparatus must be provided with a remotely-operated screen for controlling the coating operation.

The apparatus used in this investigation was the MICRO BA-3 Miniature Coating Unit (Fig.61) manufactured by Balzers High Vacuum Ltd. This equipment is capable of an ultimate vacuum of better than  $2 \times 10^{-6}$  torr. In this work, it was possible to pump down the chamber loaded with an unmounted specimen to the working vacuum of  $5 \times 10^{-5}$  torr in about 8-10 minutes.

Conditions 1 and 2 above were met by adaptations of the vacuum chamber, which is a Pyrex glass cylinder 8 cm in diameter and 25 cm long. The two ends of the chamber are closed by easily changed

quick-release flanges, one of which carries the specimen holder whilst the other flange carries the screen used for controlling the film thickness as well as the electrodes, crucible and evaporation source. The operator's view of the specimen surface is ensured by surrounding the source by a replaceable glass cylinder which restricts the area of deposition on the outer walls of the chamber; a pin-hole diaphragm of stainless steel sits on this cylinder in a position such that there is an unobstructed path for the evaporated material to reach the specimen surface.

#### 9.30 Source material

Zinc selenide of 99.99% purity was used in the form of particles 2-3 mm diameter. These were packed into a molybdenum boat and were capable of giving uniform evaporation, with complete absence of spitting in the temperature range 400-800°C.

#### 9.40 Experimental procedure

Specimens were prepared by standard metallographic polishing techniques (down to 1  $\mu$ m diamond) followed by ultrasonic cleaning. The optimum specimen-source distance in the coating unit was determined by a "trial-and-error" method and found to be about 12 cm, but is not very critical because of the geometry used.

With the remotely controlled screen in position, the current was slowly increased until the source material began to vaporize. At this point the screen was sharply removed from the path of the vapour and the specimen surface, illuminated by a small point-source of light, was carefully watched until the interference colours began to appear. When the correct interference colour (in the present work it was second-order blue) appeared, the screen was brought sharply in front of the source and the power turned off.

These conditions were found to be reproducibly, in terms of a given current setting, size of boat and quantity of source material,

and gave specimens that could be subsequently examined under manual or automatic optical microscopes, such as the Quantimet.

It is interesting to note that specimens prepared two years previously and stored under normal dry metallographic conditions showed, on re-examination, the same colour contrast and delineation of structure, as on the day they were coated. Thus, the technique may be of importance as a means for the preservation of polished specimens.

#### 9.50 Theory of interference colours

If we consider the idealised case of reflection, from a plane parallel film of thickness  $d$ , of light having a wavelength and initial intensity  $I_0$  it is possible to write an expression for the intensity  $I$ , after the light has travelled a distance  $x$  through the film in the form:

$$I = I_0 \exp(-4 \pi \mu kx/\lambda)$$

where  $\mu$  and  $k$  are characteristic of the film material and are known respectively as refractive index and absorption index.

That part of the light which is reflected at the first surface will give rise to a reflected intensity  $r_1$ , and that part which is refracted in the medium, reflected at the second surface and ultimately emitted again will have intensity  $r_2$ , and so on. Interference between  $r_1$ ,  $r_2$  ... will occur whenever the effective paths travelled by the light reflected at the two surfaces differs by an odd number of half wavelengths. In a certain thickness range, usually between 100 and 400 Å, depending upon the material, visible light reflected at the outer surface will be out of phase with that reflected at the inner surface and partial extinction will occur.

For incident white light, when the film thickness is sufficient



for interference of the yellow light, this will be subtracted from the wavelength spectrum to give the predominantly blue reflection which was used in producing most of the films used in this work.

But this alone will not explain the contrast that arises between the various areas of the specimen, and which makes the method valuable. Two factors are believed to operate. The smaller effect is that due to differences in length of geometrical paths between the harder (inclusions) and softer (metallic) areas which arise in metallographic polishing. Of greater significance is the effect due to variations in absorption index ( $k$  in the equation above). For metallic coatings  $k$  is large so that small variations in the structure of the coated layer will have a relatively large influence on  $I$ , and thus on the effective length of the optical path in the film.

Variations in the structure of the coated layer can arise from the manner in which the film is deposited on the different areas of the specimen. Electron diffraction studies have shown (152) that deposits on amorphous or inactive crystalline substrates tend to grow in a preferred orientation such that the close-packed plane is parallel to the surface of the substrate; whereas on active crystalline substrates the orientation tends to be epitaxial to the substrate, due allowance being made for "misfit" due to differing ionic radius.

Similarly, Boettcher and Haas (153) have shown that thin metallic films deposited on substrates at normal temperatures are highly continuous on oxide surfaces, but much less so on clean metal surfaces.

This evidence, applied to the present situation, suggests that the zinc selenide film is capable of exhibiting significant

differences in effective absorption index in the different areas above the different features of the specimen, and that it is these variations which cause the marked differences of optical contrast that are illustrated in the next section.

## 9.60 Results

9.6.1 Inclusions in high-sulphur steels. Some examples of the interference-film technique are illustrated in the photomicrographs. Fig.62 shows a longitudinal section cut from a billet of high-sulphur free machining steel containing lead and tellurium. Diamond polishing followed by light etching in 1% nital solution clearly reveals the manganese sulphide phase but the lead telluride phase is barely visible; in fact, if it were not for the contrast at the boundary between this latter phase and the steel matrix, (resulting from etching) it would be almost impossible to detect it at all with this standard preparation technique.

The same section was repolished, coated with zinc selenide and examined in bright field illumination using white light. Fig.63 shows the result. The steel matrix is coloured blue, the manganese sulphide orange, the lead telluride yellow and a further phase, believed to be a manganese-lead-telluride complex, pale blue, is also revealed. It should be noted that positive in situ identification of composition of the individual phases is best made by electron probe microanalysis, and this is the method that was adopted. The coating is so thin that excellent probe data for the substrate is usually obtained.

In some high-sulphur free machining steels, usually those with higher silicon or aluminium contents, the sulphides appear long and needle-like, sometimes with poor contrast between them and the steel matrix, as shown in Fig.64. After coating with zinc selenide, the result shown in Fig.65 was obtained, which

clearly shows a distinct improvement in the contrast between these features.

Apart from sulphides, high-sulphur free machining steels contain composite oxide-manganese sulphide-silicate inclusions, such as that shown in Fig.66. Fig.67 shows the same inclusion after coating to the blue interference film stage. The increase in contrast between the various phases is apparent even though the photomicrograph has been produced in black and white. Under the blue zinc film, the steel matrix is coloured blue, the manganese sulphide orange, the silicate light orange to yellow, and the oxide phase purple.

Another type of inclusion sometimes found in cast high-sulphur steels is iron sulphide. Under bright field illumination, these inclusions are pale yellow to khaki in colour and because of their relatively high reflectivity are not easily discernible. But when samples containing these inclusions are coated with ZnSe into the blue film range and then examined, the inclusions assume a brilliant pale blue colour, quite distinct from the blue of the steel matrix.

9.6.2 Inclusions in forging steels Forging steels may contain alumina, silicate and manganese sulphide inclusions. The alumina and silicate inclusions are similar with respect to optical reflectivity. This is also true of small manganese sulphide inclusions which are often indistinguishable from silicates. Also, examination of an inclusion aggregate, such as an alumina one, using bright field illumination, shows the reflectivity of the individual particles to be very similar, even though careful optical and electron probe microanalysis will show that some of these aggregates are composed of quite different phases, including manganese sulphides. The above technique greatly enhances the differences between phases in this type of material, as shown in Figs. 68 and 69. In Fig.69, the

inclusion is shown as seen through a blue-green interference film under bright field illumination. It will be seen that three distinct types of inclusions can now be identified.

Fig.70 shows small manganese sulphide and silicate inclusions in a longitudinal section of a low alloy steel, as seen through a blue interference film in bright field illumination using white light. As before, the steel matrix is coloured blue, the silicates yellow and the manganese sulphides orange. Before coating with ZnSe these inclusions could not be positively identified under a low magnification, but are now clearly discernible.

9.6.3 Inclusions in cast irons Metallurgical materials sometimes exhibit porosity as well as solid non-metallic inclusions. A particularly complex material is grey cast iron which may contain oxide, iron and manganese sulphides, and other inclusions, flake graphite and pores; the last two are particularly difficult to distinguish from one another, both visually and when examined on the Quantimet. Fig.71 is a photomicrograph of a grey cast-iron sample showing grey graphite flakes, dark pores, light grey manganese sulphide and pink titanium cyano-nitride; while Fig.72 shows the same specimen after coating with ZnSe. Here the iron matrix is coloured blue, the graphite flakes yellow, the titanium cyano-nitride inclusions pale blue, the manganese sulphides orange, and the porosity is unchanged.

9.6.4 Effect of film thickness on contrast between inclusions and steel matrix In the determination of the best film for inclusion work, examples of the above inclusions were examined after successive repolishing and coating, thinner (red), and very thin (yellow) films were produced. These are illustrated in Figs. 73 to 75, from which it will be seen that neither coating produces as good a contrast as the blue or blue-green films used in the examples discussed above.

9.7.0 Discussion and conclusions The thin film interference technique discussed in this section of the thesis was used to improve the contrast between non-metallic inclusions and the steel matrix, and between the constituent phases of the non-metallic material.

The work has shown that this technique, hitherto used mainly for improving the contrast of other metallurgical features, is useful in non-metallic inclusion identification, in providing greater contrast for proper interpretation of structure, particularly when the inclusions are to be quantitatively assessed on the Quantimet, and as a preservative of polished surfaces.

The method relies on the deposition of an optically transparent metallic film. Variations in absorption coefficient in this type of film are important in providing the improved contrast observed. It will be appreciated that the condition of the specimen surface at the time of deposition is important and this is why it is recommended that the additional step of ultrasonic cleaning after diamond polishing is included. The importance of reproducible specimen preparation techniques in any form of automatic inclusions assessment cannot be over-emphasised.

Though, in all the samples examined, improved contrast was observed, it was found that this improvement in contrast was not always sufficient for satisfactory examination of the phases on the Quantimet, even with white light and filters. An example is in the assessment of very small inclusions of manganese sulphide in the presence of silicates. These cannot be positively differentiated in conventional bright field illumination, but may be identified after deposition of a blue ZnSe film which colours the sulphides orange and the silicates yellow. These colours are, however, too close together to provide adequate contrast for the

Quantimet. It was also found that the colours of silicate and alumina inclusions are too close together to provide adequate contrast for separate analysis on the Quantimet.

On the other hand adequate contrast between the steel matrix and manganese sulphide-lead-telluride inclusions in high-sulphur steels was achieved for automatic analysis by using this technique. The method also enabled the various phases in grey cast iron to be detected and measured on the Quantimet.

Finally, it is considered that the interference film technique is important also as an aid to clearer interpretation of the phases present, particularly as a preliminary test for selecting the phases for electron probe microanalysis.

## 10.00 DISCUSSION OF RESULTS

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10.60 SAMPLE PREPARATION - THIN FILM INTERFERENCE TECHNIQUE

## 10.00 DISCUSSION OF RESULTS

### 10.10 Instrumental technique: errors, their causes and effects

Blank frame Fig.19 shows that the blank frame area after calibration decreases by about 5% during the first 80 minutes of switching on of the instrument. After this time, the variations are less than 1%. This means that in practice the blank frame need not be calibrated frequently, for example, before examining each specimen. What is particularly significant is that the same results are obtained from each test conducted at regular intervals over a period of 8 months, which indicates that the long-term warming-up characteristics of the blank frame circuit had not altered during this period.

Camera sensitivity Fig.20 shows the results of a similar series of tests carried out to determine the variations in camera sensitivity setting. The manual control of sensitivity varies continuously throughout the 145-minute test periods, rising from 0.7 to over 0.9 divisions on the meter. On the other hand, extremely good stability of camera sensitivity setting is obtained using the automatic control.

Camera sensitivity and threshold level Fig.25 shows that a variation in camera sensitivity setting as small as 0.075 divisions on the meter is equivalent to a variation in threshold control setting of 17-20 divisions which, in turn, is shown in Fig.26 to be equivalent to a variation in percentage area measurements of about 30% for manganese sulphides and about 10% for oxides.

The reason why manganese sulphides are more sensitive to variations in threshold setting is due probably to the "filling-in" characteristics of the instrument. The oxides are "filled-in" or detected first, and then the sulphides. It has been observed that for the same number of divisions on the threshold control, a



larger area of the sulphides is filled than oxides. Hitherto, the "filling-in" errors for both these types of inclusions have been assumed to be the same. Fig.26 shows they are not. This may be the reason why Grethen et al.(123) found they could obtain more accurate per cent area results for sulphides when they etched the samples to darken them.

Clearly, the manual control of camera sensitivity is not stable for long enough periods for it to be really useful, at least, not on the instrument tested.

Automatic control of sensitivity Tables 3 to 5 show that the standard deviations of measurements per cent area and counts performed on oxide inclusions (using automatic control of camera sensitivity) over a period of  $4\frac{1}{2}$  hours are less than 2% of the mean estimates, and that the variability of measurements performed at the beginning and end of the eight-month period is the same. Obviously, the degree of reproducibility attainable would be dependent upon feature size and optical intensity. Large, dark contrast features would be expected to give better reproducibility than small, light contrast features.

Magnification selection Experience with the Quantimet suggests that for good reproducibility an optical magnification should be selected such that the majority of particles on the TV monitor screen are at least 5 mm in diameter. The above tests were conducted on oxide inclusions, typically JK grading 2B (Fig.3) with some inclusions less than 5 mm in diameter.

Resolution and filters Results obtained in this work (Fig.21) show that significant improvement in resolution of the system can be obtained by using the correct wavelength of light. With the X5 objective, for example, maximum resolution is obtained with orange

and yellow filters. With these filters a 20-30% improvement is obtained in percent area counts of oxide (alumina) stringers, composed of different sizes of particles, some near the limits of resolution (1-2 mm on the screen), due to better resolution of the smaller particles.

Microscope stage In general, the stage of the microscope is capable of keeping the image of the specimen in focus over the whole of its programmed traverses in the X and Y directions, when using all except the X40 and higher objectives. With the X40 objective, the focus of the microscope needed readjusting every 4 mm approximately. This is not a serious limitation in routine work as it is unlikely that this objective will be used regularly. In any case, the programme may be varied so that the instrument selects fields in blocks such that the X traverse does not exceed 4 mm. On the whole, the automatic stage performed extremely well even when it was used continuously for long periods of up to 16 hours a day over several months.

Effect of inclusion shape Figs. 27 to 32 show that when sizing and counting inclusions variable results are obtained. Rounded inclusions are accurately counted but inclusions with large re-entrant angles or preferred orientations, such as alumina stringers, are either over or under counted for a given acceptance angle setting. The counting and sizing of long, thin inclusions, such as manganese sulphides in longitudinal billet or bar sections, with their long axes lying parallel to the TV scanning line, is generally subject to relatively large errors. In theory it is possible to count such inclusions correctly, but in practice, this is inconvenient if not impossible to achieve with the automatic Quantimet.

Two detection levels A further complication arises when measuring oxide and sulphide inclusions simultaneously using the two detection circuits provided. Due to the edges of oxides appearing diffused,

these areas are measured by the instrument as sulphides. Table 6 shows that sulphide counts may be higher by 10 to 20% of the oxide percentage area measurements. However, because of poor detection of sulphides due to their needle-like shape and higher reflectivity, estimates of sulphides are likely to be lower than the actual values, and their over-estimation due to the "edge effect" may off-set the error due to poor detection. But what is particularly disturbing is the fact that sulphide counts may be recorded even when there are no sulphides present!

Furthermore, different magnifications and instrument operating conditions are needed to measure properly each of these two types of inclusions, since oxide inclusions are distributed in steel in a different manner from sulphides. Consequently, each of these inclusions requires a different sampling method. It has also been observed that the size distribution limits chosen for one type of inclusion are generally not suitable for the other type. For these various reasons it is questionable whether these two types of inclusions should be measured simultaneously, particularly in the case of forging steels.

Focusing method In contradiction to the conclusions of some operators of this instrument, tests on various types of inclusions in steel have confirmed what was found in a previous research (121) namely, that the method used to adjust the focus of the microscope is important. Table 7 shows that focusing the image through the microscope gives more reproducible results than when the image is focused whilst observing it on the screen of the TV monitor. In fact, these results show that variations of up to 5% in the per cent area measurements of oxides can occur when using the monitor method.

Threshold level However, by far the most serious error is that due to bias on the part of the operator in adjusting the detection control (threshold level). Three commonly used methods for adjusting the threshold level were tested and each found to give poor reproducibility. Figs. 23 and 24 show that at 2 sigma level, the coefficients of variation of threshold control settings for the three popular methods are: 3.44% for the original filling-in method; 2.34% for the "Flicker" method with polarity set to detect black; and 2.14% for the "Flicker" method with polarity reversed. In terms of percent area variations, the above values correspond to 26.38%, 18.02% and 16.64% respectively. Thus, contrary to the current view that the "Flicker" method gives greatly improved accuracy, these results show that whilst the "Flicker" method is certainly the most objective of the three methods, it is still likely to give large errors.

Use of a standard Table 9 shows that much better reproducibility in adjusting the threshold level is obtained by using some sort of a standard. The use of standards, however, will not guarantee accurate results since this obviously depends upon how it was calibrated in the first instance, but it will reduce scatter in the results. In the assessment of non-metallic inclusion contents of steels, for routine work, good reproducibility is considered to be far more important than absolute accuracy.

In deciding on the form the standard sample should take, it is worth remembering that it is likely to be needed frequently and that time is a very important factor. The method, therefore, should be simple and quick. In contradiction to the views of some operators, tests carried out on routine basis suggest that the examination of several, or even one standard sample over a large area involving

hundreds of fields would be unworkable however sound in theory it may seem. The method most likely to be found useful will be one in which carefully identified and calibrated single fields are used.

The use of such a standard for routine work has four obvious advantages:

(i) The performance of any particular component part of the instrument could be quickly tested, e.g. rate of drifting of camera sensitivity and the sensitivity control re-set; (ii) because of its simplicity and quickness it can be used to adjust the threshold level of the instrument before and after examining each specimen, thus ensuring that the measurements obtained are reliable; (iii) considerable time can be saved than when a standard sample with several hundreds of fields is used; (iv) new standards can be easily prepared by comparing the new field to be used as a standard against the old one under the same conditions, i.e. threshold setting, etc.; this task takes only a matter of seconds.

Automation of Quantimet In general, the automating of the Quantimet has not presented any serious problems. Some difficulty was encountered in getting an ICT 1905 computer to accept the tapes from the Quantimet. This trouble was traced to a fault in the design of the tape punch used. It is worthy of note that with the slower Elliot 903 computer this problem did not arise; and, in fact, the rejected tapes were subsequently run on this machine.

With the present method of punch tape system, the computer staff have to prepare preliminary data tapes denoting specimen number and other relevant details. Also, if the tape is to be decoded it has to be done using a special decoder and printer which is often situated some distance away from the Quantimet. The output writer is relatively slow. It is considered that a teletype

printer, instead of the output writer and punch tape supplied at present, would have overcome the above difficulties. It is interesting to note that the new automatic Quantimet now being manufactured are supplied with this arrangement.

Conclusions From long periods of use of the Quantimet during this research and with the knowledge of the above results, four major conclusions can be drawn about its operation:

- (i) The more obvious one is the importance of using some sort of a standard sample to adjust the threshold level.
- (ii) The manual control of sensitivity should not be used, at least on the instrument tested, and at least 80 minutes should be allowed for the instrument to reach stable operating conditions.
- (iii) The count measurements in certain situations may not be as reliable as the percent area and projection measurements.
- (iv) Provided the above precautions are taken, the instrument will give accurate and useful information on non-metallic inclusions in steel.

#### 10.20 Correlation of Quantimet parameters with those of SAE, JK and Diergarten methods

Results given in Tables 10 to 12 show that only percentage area and projection measurements give good correlation. Total count measurements also give good correlation but only for the less densely populated fields. These results also show that Quantimet percent area and projection measurements cannot be relied upon to give separate counts for oxide and silicate inclusions, since the differences between these are too small. However, with the additional count parameter it is possible, at least in theory, to distinguish between the continuous silicate type of inclusions and the broken alumina type of inclusions, but in view of the uncertainty of the

accuracy of this measurement, it cannot be used with absolute confidence.

A further complication arises in trying to obtain a machine chart rating assessment of the inclusion content of a sample and, in characterising fields with more than one type of oxide inclusion in such a way that the instrument gives separate counts for each one.

Despite these difficulties there is evidence to suggest that conversion from machine numbers to chart ratings is useful, particularly in the early stages in the adoption of automatic methods. Such conversions should be attempted only when the steel contains predominantly one type of oxide inclusion, e.g. silicate or alumina only. Some steelmaking practices employed produce predominantly one type of oxide inclusion, and it is considered that in these situations conversion from machine numbers to JK, Diergarten or SAE-ASTM chart methods is justified; but not in cases where more than one type of oxide inclusion is produced and when it is necessary to obtain separate estimates for each of them.

Any attempt to perform an automatic machine classification of inclusions in each field of view in a similar manner to a human operator would require a much more sophisticated instrument than the Quantimet. It is debatable, therefore, if such an instrument is really needed in quality control work where the primary concern is generally to determine the presence or absence of any field in which the concentration of inclusions, whether oxides, silicates or sulphides exceeds some specified limit.

It is considered that the ability of an instrument to distinguish between alumina and silicate or some other type of oxide inclusion is not a serious limitation in routine work. For quality control in steelworks, the instrument could be programmed to stop on "over-

value" fields which could then be inspected visually by an experienced operator. He could then decide whether the high value is due to alumina, silicate or some other type of inclusion. Provided the over-value is fixed at a reasonable level, none, or very few, over-value fields will be encountered in clean steels.

#### 10.30 Development of digital computer programmes for summarising Quantimet measurements

This part of the work was quite straightforward. Computer processing of digitalised information about inclusions offers great advantages in rapid handling of large amounts of data and in calculating the means and standard deviations for proper interpretation of results obtained by the Quantimet. However, it should be pointed out that no amount of processing can restore precision which has been lost in making the measurements. Specifically, no set of results computed by methods, however elegant, can be expected to be valid to a precision greater than that inherent in the basic measurement initially determined.

All six programmes developed were found to be of great use. The means and standard deviations of mean estimates of 500 fields, for example, can be obtained in less than 10 minutes, which includes the time taken to prepare the computer for a particular programme. Additionally, it has been possible to get the computer to form a histogram table of area percent measurements showing the number of fields counted in different percent area ranges. In simple cases, i.e. when the steel contained only one type of oxide inclusions, it has been possible to instruct the computer to convert the machine numbers to JK, SAE-ASTM or Diergarten ratings. Probably the greatest convenience may be found in the fact that the data is produced in a standard printed form which could be passed to managers concerned



with the manufacture and processing of steel. A copy of the results may also be sent to the customer for information. Once inclusion count measurements are determined and recorded in this manner for every cast of steel produced, the possibility is opened for performing a variety of statistical calculations to determine which steelmaking practice gives the cleanest steels and determining other operating trends.

#### 10.40 Inclusions assessment in specific locations

10.4.1 Effect of reduction Results in Tables 15-17 show that percent area count of oxides (alumina stringers) decrease with a decrease in section size. Also, the number count for inclusions greater than 20  $\mu$ m decreases with a decrease in section. This is believed to be due either to the breaking up of larger alumina particles into smaller ones during rolling, or to a change in the distribution of these inclusions due to their becoming strung out and dispersed during rolling.

Theoretical considerations indicate that for inclusions which are perfectly plastic at the temperature of rolling or forging, that is, which deform at the same rate as the steel, the inverse relationship would be followed exactly, whereas for hard inclusions, the inclusion count would remain constant. Departures from this ideal state may be thought of as being the result of real inclusions being on the one hand neither perfectly plastic nor perfectly hard; or on the other hand, being comprised of mixtures of plastic and hard inclusions.

Changes in size and shape of inclusions with reduction were more noticeable for manganese sulphides, but, unfortunately, these could not be measured on the Quantimet due to reasons previously discussed. But it is considered that if it had been possible to

obtain an assessment of them, a profound effect with decrease in reduction would have been noted. Hardy and Allsop (94) using the LT count method, found that when the inclusions are hard, the variation of inclusion count with the reduction is small; but when the inclusions are soft the inclusion count varies inversely with the reduction ratio.

10.4.2 Sectioning errors Figs. 36 to 40 and Tables 18 and 19 show that a variation in sectioning of  $6^\circ$  from the longitudinal axis of the billet when preparing the billet sample for examination can double the total number of inclusions counted. What is more important, it leads to serious under-estimation of the larger inclusions; percentage area measurements are unaffected.

It is believed that the importance of reproducibility in sample preparation is not widely appreciated, particularly in laboratories concerned with routine work. Much greater attention should be paid in the future to this aspect of the work, if reliable inclusion assessments are to be obtained. In fact, there seems little point in installing expensive sophisticated instruments when the sample preparation methods are such that they can lead to serious errors.

Turning now to the effects observed, it is believed that the reason for the higher total number counts in the sample slightly inclined in the rolling direction of the billet is that there is a greater chance of cutting through inclusions in this section than in an ideal longitudinal one. Theoretical considerations suggest that an even better sample would be one which is  $90^\circ$  to the rolling direction, i.e. a transverse sample, since in such a sample the chances of cutting through a stringer inclusion would be even greater because of the preferred orientation of inclusions.

10.4.3 Micro-sampling The work on micro-sampling, i.e. selection of fields of view on a polished surface of a specimen for inclusions assessment (see results in Appendices 9 to 11, Tables 20 and 21 and Figs. 41 to 47) shows that for steels, similar to those used in this work, it is necessary to examine at least 300 fields, but preferably more, in order to obtain a reliable mean percentage area measurement, or an estimate of the number of fields in which the concentration of oxide inclusions is 0.55% or greater, i.e. fields equivalent to a JK grading 3. Estimates of inclusion content based on 25, or even 100 fields are associated with very large errors.

On a typical sample with an area of  $600 \text{ mm}^2$ , 300 fields (each  $0.5 \text{ mm}^2$ ) is equivalent to a 12.5% coverage which is considered to be a reasonable sample from a statistical point of view.

It should be stressed, however, that the above procedure (300 fields), whilst giving reasonable precision in separating a "dirty" steel from a "clean" one, will not separate two "clean" steels with inclusion contents similar to those of Specimen Nos. 2 and 3 used in this work (see section 7.4.1). To distinguish between steels with inclusion content similar to these two specimens at least 600 fields must be assessed, which with the automatic Quantimet would take about 10 minutes, if percentage area counts only are determined. Compared with visual manual JK counting this is about three times faster. In routine work, it is desirable to assess each sample in 2 to 3 minutes.

10.4.4 Macro-sampling Tables 23 and 24 and Fig. 48 show that very large variations exist between samples taken from the same length of billet. In practice, a sample is usually taken from one end of a billet. These results show that a billet considered to be "dirty" on such a rating system was, in fact, rated as "clean" on

40 occasions out of 180. Conversely, the "clean" billet was rated as "dirty" on 33 occasions out of 180. This suggests that perfectly good steel can be rejected, or a dirty steel accepted.

These results also show that fields with high inclusion values tend to be segregated on a macro-scale all along the billet, giving clean and dirty regions and that the chances of one of these regions being at the end of a billet is roughly 1 in 20, i.e. 20 adjacent samples, each about 2 cm thick, would have to be taken before finding a "dirty" sample, i.e. a sample that has one or more fields per 100 fields in which the percent area count is 0.55% or greater.

Hitherto, it was tacitly assumed that a count determined on one polished surface is representative of that sample. Tables 25 to 27 show that this is not so, but that the variation between surfaces is nearly as large as that between specimens.

10.4.5 Conclusions The results of these investigations lead to the following major conclusions:

- (i) Sample preparation procedure must be standardised.
- (ii) The samples should be from the same size of billets or very nearly the same size.
- (iii) Between 300 to 600 fields need to be examined on each specimen, depending upon the size of inclusions to be assessed.
- (iv) At least 10 samples should be examined from each billet, and each sample examined at least 3 times using three different surfaces.

#### 10.50 Comments on the general programme of inclusions assessment

##### 10.5.1 Distribution of oxide inclusions in two adjacent

ingots Results in Table 30 indicate that low alloy steels of the type used in this investigation contain predominantly two types of oxide inclusions: alumina and manganese-alumino-silicates. A

particularly interesting point connected with these results is the presence of 2 to 4% CaO in these inclusions. Since CaO can come initially from furnace slag only, it is interesting to speculate how both these types of inclusions come to have significant amounts of this oxide.

It is believed that small particles of CaO are present in steel during the stage of melting and refining and that these particles act as nuclei for the precipitation of alumina inclusions, which form as a result of "killing" steel with aluminium. Much of this material rises to the top, but some remains in the steel as inclusions.

The manganese-alumino-silicate inclusions are considered to form after tapping, probably during teeming. The exact mechanism by which they form is not known but it is believed that they result from entrapment of "scum", which is present on the tops of ingots during solidification and which is drawn into the steel by currents set up during cooling. This scum would be expected to contain the deoxidation products mentioned above, and, of course, the CaO. Theoretical considerations suggest that such inclusions should occur wherever the conditions for their entrapment are most favourable, but more frequently in the central portion of the ingot, which is the last part to solidify. The results of inclusion counts determined by various laboratory procedures in the present work confirms this.

Tables 31 to 38 show that although high Quantimet oxide values can occur at any position from top to bottom, and from edge to centre of the original ingot, there is a high probability of them occurring in the middle and bottom portions, particularly in the central regions of the ingot. What is particularly interesting about these results is the fact that this trend in the distribution

of oxides is also indicated in results obtained by step, magnetic crack detection and sulphur printing methods (Tables 39 to 42 and Appendix 21.

Appendix 20 shows that despite the scatter in results there is a good correlation between total oxygen contents determined by the neutron activation and Quantimet methods.

The major conclusion to be drawn from this investigation is that steels, if "dirty", are more likely to have "large" inclusions in the central, middle and bottom portions of the original ingot and, therefore, for purposes of routine inspection of steel for this type of inclusion, samples should be selected from billets corresponding to these positions.

10.5.2 Running programme on low alloy steel casts Tables 47 and 48 and Figs. 51 and 54 show, as expected, that on the average "clean" steels do not show such marked systematic variation in oxide inclusion content from edge to centre of the billet as do "dirty" steels. However, even in these "clean" steels there is a tendency for the central axial portions of the billet samples to contain slightly more fields with percentage area values greater than 0.55% than portions nearer the edge of the billet.

What is of greater interest, however, is the marked systematic variation observed at four different positions along the original ingot. Appendices 22 to 29, and Figures 55 to 58 show that the heaviest concentration of fields with high percentage area values occurs in billet samples corresponding to the extreme bottom and one billet length away, i.e. 10% up the ingot; the middle of the ingot (position 53% from top of ingot) is next highest in oxide content, and the top samples, next to the discard, the cleanest. Fig. 58 shows that there is only a 15% chance of a billet from the

middle portion of the ingot being rated as "dirtest"; only a 5% chance of a billet from the top of the ingot being rated as "dirtest"; but there is a 40% chance of each of the bottom billet samples being rated as "dirtest".

The results of this investigation clearly lead to the conclusion that the best sampling positions in the ingot for this type of steel is the extreme bottom and one billet length away, and that it is better to take the four samples from the bottom billet than one from four different positions along the ingot.

Table 49 and Figs. 59 and 60 show that there is no marked systematic variation in oxide inclusion content between ingots selected from each group. This is in contradiction to the view held by some that ingots teemed at the end of cast are "dirtier" than those teemed earlier. This argument, of course, is based on the fact that the steel teemed at the end of cast does not remain liquid for as long a period as that teemed earlier and, therefore, the chances of large inclusions floating up are less. There is probably some truth in this, but it cannot be accepted that this is the main factor influencing the incidence of large inclusions in ingots.

None of the 14 casts examined in this work was considered really "dirty". If such casts had been examined, it is possible that the ingots from the last group might have contained larger inclusions. Furthermore, of the 14 casts examined, 9 were teemed in two groups which would not result in such a large drop in the temperature of the steel.

Perhaps one of the most encouraging sets of results obtained in this investigation is that shown in Table 53, which shows that casts of steel made by the double slag process have on the average the lowest mean percentage area value; single slag casts have the

highest oxide content (higher by nearly a factor of 3); whilst single slag, grain refined and single slag, vacuum degassed and grain refined casts have inclusion contents between these two levels.

These results, therefore, suggest that the sampling and testing methods employed in the latter part of this investigation are practically sound. A further interesting point that emerges is that the Quantimet is able to pick up these differences in inclusion content resulting from the use of different finishing practices.

#### 10.60 Sample preparation - thin film interference technique

Figs. 62 to 75 show that this new technique is useful in providing contrast for inclusions to be quantitatively assessed. Though in all samples examined greater contrast was observed, it was found that this improvement in contrast was not always sufficient for satisfactory examination of the various phases on the Quantimet, even with a Xenon light source. Unfortunately, alumina and silicate inclusions in steel cannot be differentiated by this method as the resulting colours are too close together to provide adequate contrast for separate analysis on the Quantimet. But the technique is considered to be valuable as an aid to clear interpretation of the phases present in optical microscopical examination, and also, as a preliminary test for selecting phases for electron probe analysis.



## II.00 SUMMARY AND CONCLUSIONS

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OF JK, DIERGARTEN AND CM (SAE-ASTM) METHODS

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## 11.00 SUMMARY AND CONCLUSIONS

The work recorded here has produced results which explain much of the operational characteristics of the automatic Quantimet when it is used to determine the inclusion content of steels. It also shows the magnitude of errors encountered in sampling and sample preparation, how oxide inclusions are distributed within ingots and between different casts of low-alloy steels.

The following points summarise the main findings of this research. For convenience, these points are listed under the main fields of research conducted, as follows:

### 11.10 Work on Quantimet

- (a) Blank frame setting drifted by about 5% from the original calibrated setting during the first 80 minutes of switching on of the instrument, but after this period it varied by less than 1% over approximately 1 hour.
- (b) The camera sensitivity setting, as measured on the meter in front of the instrument, produced variable results. On manual control, it varied continuously, even after 80 minutes, rising from its original setting of 0.7 to over 0.9 over a period of 145 minutes. On automatic control of camera sensitivity, it varied only slightly during the first 30 minutes, and hardly at all after this period.
- (c) With automatic control of camera sensitivity, and after the instrument had attained a stable operating temperature (80 minutes), the overall reproducibility of the instrument (including the automatic data handling systems) was found to be a function of feature size and contrast. Practically 100% reproducibility was attained over a period of  $4\frac{1}{2}$  hours when determining percent area, total projection, total count and

size distribution measurements of relatively dark (typically oxide) inclusions greater than  $5\mu\text{m}$  on the TV monitor screen. With inclusions smaller than this size, particularly those with poor contrast (typically manganese sulphides), variations (standard deviations) of up to  $\pm 5\%$  of the mean estimate were frequently encountered.

(d) The setting of the threshold control was found to be very critical and only small variations in this setting caused large variations in the measured values, particularly percent area. In general, a variation of the order of  $1.7\%$  in threshold setting, i.e. a variation of about 20 divisions on the threshold control, caused a  $10\%$  variation in detected oxide area.

(e) Camera sensitivity and threshold setting of the instrument are directly related. A deliberate variation in camera sensitivity of 0.07 divisions was found to correspond to a variation in threshold setting of 17-20 divisions, which, as shown above, can result in significant errors.

(f) The setting of the threshold level produced significant variations. Three methods were investigated by three operators who produced coefficients of variation in the range  $0.72\text{--}2.01\%$ . The original "filling-in" method produced a mean coefficient of variation in threshold setting of  $1.76\%$ ; the "flicker" method with polarity of detection set to detect black  $1.07\%$ ; and the "flicker" method with polarity of detection set to detect white  $1.17\%$ . These gave corresponding mean coefficients of variation in detected oxide percent area measurements of  $13.2$ ,  $9.0$  and  $8.3\%$  respectively.

(g) Differences between operators in setting threshold were found to be less than differences between methods. For three operators, the mean coefficients of variation of the detected oxide percent area measurements were found to be within the range  $9\text{--}11.5\%$ .

(h) The development and use of a standard sample method for adjusting the threshold level gave practically 100% reproducibility between measurements of a given operator as well as between measurements of different operators.

(i) Standards which use single fields were found to be highly satisfactory for setting of the threshold level. Standards which use an area comprising of a hundred or more fields were found to be unsatisfactory from the point of view of convenience and speed.

(j) The microscope method of focusing the image to be analysed gave better reproducibility than the TV monitor method. The standard deviation of the mean setting of the former method was lower than the latter method by a factor of nearly 2.

(k) The automatic stage was found to be capable of maintaining the image critically focused over the whole of its programmed traverses in the X and Y directions for all except the 20X and 40X objectives. With the 20X objective about  $\frac{3}{4}$  of the whole traverse was attained, but in the case of the 40X objective, the focus needed re-adjusting about every 4 mm in both the X and Y directions.

(l) Extremely good correlation was obtained between the percent area and projection measurements determined by the instrument and visually. The correlations between the instrument's number counts and the visual number counts were found to be variable. On some inclusions, good correlation was obtained between the two sets of measurements but on others, the correlation was relatively poor. Poor correlation was generally obtained when the inclusions were either too long or too closely distributed in the field of view and when this distribution varied from field to field.

(m) In many samples, the manganese sulphide inclusions could not be satisfactorily measured, because they were either too long and

needle-like or the magnification selected was suitable for detection of oxides but not sulphides.

(n) The use of two detection levels (available on the automatic Quantimet) produced errors in the sulphide estimates ranging between 10 to 20% of the detected oxide values due to the "edge effect" of oxides. Due to the same effect, it was found that a sulphide area measurement was recorded even though no sulphides were present.

(o) The long-term reproducibility and general behaviour of the instrument, measured over a period of 8 months, remained the same.

(p) Fluctuation in mains due to operation of steelworks' plants (electric arc furnaces, rolling mills and other ancillary equipment) did not affect the performance of the instrument. However, the shunting of railway engines some 20 yards away caused the focus of the image to be temporarily distorted due to vibrations. During these periods it was found necessary to stop the instrument and repeat the measurement later. If the machine were left unattended, this effect could result in an erroneous result being recorded for that specimen.

(q) The instrument has not suffered any serious breakdown even though during the first 8 months it was operated continuously for 16 hours a day, 4-5 days a week.

(r) The use of yellow or orange filters with the 5X objective improved the resolution of the instrument; in some cases, by as much as 50%.

#### 11.20 Correlation of Quantimet parameters with those of JK,

##### Diergarten and CM (SAE-ASTM) methods

(a) Generally, good correlation was found between the Quantimet's percent area and projection measurements and chart gradings. As the chart grading increased there was a corresponding increase in

the measured values. log-normal plots of Quantimet parameters versus chart grading gave practically a straight line. Quantimet number counts also gave good correlation with chart grading numbers, but generally only in the lower ranges, i.e. 1-3 grading in the case of the JK method (except for silicates) and 1-5 grading in the case of the Diergarten method.

(b) Using the above correlations it has been possible to convert from machine numbers to chart gradings and vice versa, and also from JK to Diergarten rating or vice versa for the same types of inclusions.

(c) Good agreement was obtained between chart ratings of a specimen of steel made visually by two experienced operators and by the machine, when no attempts were made to distinguish between different types of oxides.

(d) Attempts were made to fit a mathematical equation to each set of correlation lines in an attempt to distinguish between the various types of inclusions depicted on these charts, but the differences between them were found to be too small to enable a suitable equation to be developed. Furthermore, fields with more than one type of oxide complicated matters as the instrument could not measure each type separately.

(e) It is concluded, therefore, that conversions from one chart method to another, or from machine to chart methods will be possible only when it is not necessary to distinguish between the various types of oxide inclusions; or when the steel contains predominantly just one type of oxide inclusion.

#### 11.30 Development of digital computer programmes for summarising of Quantimet data

(a) Fields for the same number of measurements were examined nearly

twice as fast when data was recorded by a tape punch than when it was recorded by an output writer.

(b) Several digital computer programmes have been developed for summarising the Quantimet data. As an example, 8,000 measurements on seven different parameters could be processed to give their mean, standard deviation and frequency table showing the number of fields counted with values within six specified ranges, and all these results calculated and printed out in less than 10 minutes. The processing of this amount of data on a single specimen by manual means would have taken over a day.

(c) Digital computer programmes have also been developed which convert Quantimet numbers to chart ratings.

(d) It has also been possible to write programmes which give a frequency distribution of the Poisson type for each specimen showing the number of fields counted with 0, 1, 2, ... n number of inclusions greater than five different sizes (a variable in the programme).

(e) Programmes have also been written which give grand summaries, for example in situations where it is necessary to deal with measurements obtained on several specimens.

(f) The above programmes have been tried out on an Elliot 903 and an ICT 1905 computer and have been found to work satisfactorily.

In general, the processing of paper tapes produced by the Quantimet was found to be easier on the slower Elliot 903 than on the ICT 1905. Separate preliminary data tapes are needed and the processing of these tapes first and then the Quantimet data tape was found to be inconvenient. Also, to get a transcription of the Quantimet data Tape it was necessary to take it to the Computer Centre situated some distance away from the Quantimet. The use of a Teletype printer

similar to that used for computer read-outs, instead of the output writer and tape punch supplied with the Quantimet, would have solved these difficulties in addition to being faster than the output writer.

(g) Several Quantimet data tapes were rejected by the faster ICT 1905 computer due to faulty alignment of tape in the tape punch. Although care was taken to ensure that the tape was set up properly, drifting sometimes occurred which resulted in these tapes being rejected by the computer. On the other hand, the slower computer accepted these tapes and processed them satisfactorily.

(h) The use of this system for recording and processing of Quantimet data was found to do away with the arithmetic involved in obtaining separate measurements for oxide and sulphide inclusions, in converting projection intercept measurements to actual inclusion lengths in millimetres or microns, and in forming frequency tables, all of which are extremely time-consuming and laborious to calculate.

#### 11.40 Inclusions assessment in specific locations

Effect of reduction in area Reduction in area from 6" sq. to 1" dia. was found to affect the results. Oxide percent area measurements decreased from 0.04% to 0.01%, and there were also fewer inclusions greater than 20  $\mu$ m in the smaller section. Between 4" and 2" sq. sections, the counts were not much affected.

#### Effect of variations in sectioning billet samples

(a) A variation of 6° from the axis of the billet in the rolling direction nearly doubled the total number counts for both oxide and sulphide inclusions in high sulphur free-cutting and low-alloy steels. It also decreased the count of number of large inclusions, or fields with high percent area values.

(b) A similar amount of variation from the axis of the billet but 90° to the rolling direction, i.e. with a surface sloping from edge



to centre of the billet, did not affect the results significantly.

(c) Mean percent area measurements were found to be unaffected by either of the above variations in sectioning billet samples.

#### Work on sampling

(a) Variations between fields were very large. As an example, for three steels with varying oxide content, the mean percent area and standard deviation estimates based on 100 fields for 30 such determinations were within the range  $0.02 \pm 0.02$  to  $0.05 \pm 0.26\%$  for the relatively clean sample with an average area count of 0.02%;  $0.01 \pm 0.01$  to  $0.09 \pm 0.33\%$  for a sample with an average area count of 0.03%; and  $0.01 \pm 0.01$  to  $0.17 \pm 0.34\%$  for a sample with an average area count of 0.07%.

(b) The method of basing the inclusion content of a sample on mean estimates of 25, 50 or even 100 fields (current practice in some works) is subject to very large errors and was found to be totally inadequate for distinguishing reliably between samples of varying inclusion content.

(c) Results obtained suggest that in order to attain a reasonably high degree of precision in the results, each specimen must be examined at least 9 times (using a new repolished surface each time) under microscopic conditions that ensure at least a 10%, but preferably a 20% coverage of each surface.

(d) Larger variations than those encountered within a sample (conclusion (a) above) exist between determinations made on adjacent samples selected from entire length of billets.

(e) Results obtained suggest that between 10 to 15 specimens are needed from each billet in order to attain a reasonable degree of precision in the results.

## 11.50 General programme of inclusions assessment

### Work on two adjacent ingots

- (a) Detailed examination of two adjacent ingots of low alloy steel (En 16) after rolling into billets showed that although individual fields with concentrations of oxide inclusions greater than 0.55% (JK Rating 3) could occur anywhere in the ingot, they tended to occur more frequently in the half of the specimen nearer the centre of the billet. Quantimet, step test, magnetic particle test and sulphur printing results supported this finding.
- (b) The highest concentration of oxide inclusions occurred in billets representing the middle 20% and bottom 20% of the ingot, i.e. over two billet lengths approximately in the above positions of the ingot.
- (c) Fields with lower percent area values, as for example values of 0.55% or less, tended to be more uniformly distributed not only from edge to centre of the billet but also from top to bottom of the ingot.
- (d) Microscopic and electron probe examination of oxide stringer inclusions (revealed by microscopic and magnetic particle examinations) showed two types of inclusions to exist. One of these types contained about 94%  $\text{Al}_2\text{O}_3$  and between 2-4% CaO. The other type, usually, but not necessarily the larger ones, contained significant amounts of MnO,  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$  and CaO. This latter type of inclusion also had some practically pure alumina particles embedded in them. Examination of the small oxide inclusions found embedded in sulphides showed these to be high in Al, but due to their small size their exact composition could not be determined.
- (e) Statistical examination of percent area measurements determined by the Quantimet and total oxygen values determined by the neutron activation method showed the correlation between these measurements to be highly significant.

(f) There was also an obvious correlation between Quantimet measurements and those of other tests employed (e.g. sulphur printing, step testing and magnetic particle testing).

(g) Step down test pieces when cut in half through the axis of the billet and examined revealed more inclusions than when step tested in the ordinary manner. This effect was attributed to the concentration of the larger inclusions in the central portion of the billet which was not represented by the step test pieces used.

(h) In general, tests conducted on billet samples selected from both ingots gave the same results and exhibited the same degree of variability between samples.

#### Running programme at a steelworks

(a) Although on individual specimens there was a systematic segregation of fields with high oxide percent area values toward the centre of the billet, on the average, this difference from edge to centre of the billet was not found to be marked, presumably because the majority of steel samples examined were relatively clean.

(b) On the other hand, a marked systematic segregation of oxide percent area values was found to exist between the top, middle, one billet length from the bottom and bottom billets. In general, samples from the latter two positions in the ingot gave the highest counts, those from the middle of the ingot gave the next highest count, while those from the top of the ingot gave the lowest counts.

(c) Analysis of the results obtained on 212 samples selected from 53 ingots showed that 80% of the samples rated worst from each ingot, i.e. as having the highest oxide percent area estimate, 80% of these samples were from the two bottom billets; 15% were from the middle billets, and only 5% were from the top billets.

(d) The majority of the individual particles making up the stringers

or inclusion aggregates were less than  $20\mu\text{m}$  and over 90% of them less than  $10\mu\text{m}$ .

(e) There was a strong indication from the results that on the average the two samples from the bottom of the ingot contained slightly larger particles of inclusions.

(f) Percent area, total projection and total number of oxide inclusions were all found to be useful quantities in rating specimens, but size distribution measurements were found to be of little value for reasons given in conclusion (d) above.

(g) No marked difference in oxide measurements exists between samples selected from four different groups in the cast. But there was a slight indication that samples drawn from the last group may have contained slightly more fields with percent area values greater than 0.55% (equivalent to JK Rating 3).

(h) Differences in oxide content between casts finished by the same process were less than differences between casts finished by different finishing processes.

(i) Double slag process produced steels with the lowest oxide content (average percent area 0.028%) and single slag process steels with the highest oxide content (average percent area 0.070%). In general, single slag vacuum degassing process produced steels with inclusion content between double slag and single slag processes.

(j) Results obtained show that cleaner steels are often produced when aluminium is added for grain refining to casts made by single slag process than when it is not added.

#### 11.60 Sample preparation - thin film interference technique

(a) In all the samples examined, the use of the thin film technique gave an improved contrast, but this improvement was not always adequate for automatic counting procedures, but it was found to be

of benefit for manual counting procedures.

(b) The use of this technique revealed the lead-telluride phase of manganese sulphide-lead telluride inclusions which could not be revealed clearly by conventional etching and microscopic techniques.

(c) The technique also made possible separate automatic measurements to be determined on dark inclusions and for pores and graphite phase in grey cast-iron samples, which could not be accomplished by conventional sample preparation techniques.

(d) The technique was also found to be valuable as a preservative of polished surfaces.

(e) This method could not produce sufficient contrast between different types of oxides, e.g. alumina and silicates for it to be usefully employed in routine automatic inclusion counting work.

(f) When using ZnSe as the coating material, the best contrast was obtained with the blue and blue-green films.

(g) The technique has been found useful for revealing phases to be subsequently analysed on the electron probe microanalyser.

#### 11.70 General conclusions

Automatic inclusion counting in a steelworks for quality control is now a practical possibility, as the above discussion on the performance of the automatic Quantimet shows. The stability and reproducibility of this instrument has been thoroughly checked and found to be excellent providing certain precautions are taken, so that if the cost of the instrument could be justified (around £8000 to £10,000, depending upon the degree of automation) it will yield inclusion counts more quickly and objectively than the visual methods, and with virtually no operator error.

Although the Quantimet will give for each field the area occupied by inclusions, the total number of inclusions present, their

size distribution, their aggregate length and chord size distribution, the results of this research show that total number and size distribution counts cannot always be determined as reliably as percent area and projection measurements.

Furthermore, the work carried out here on sectioning errors shows that total number and size distribution counts are more sensitive to variations in sectioning the billet sample than percent area measurements. Unfortunately, projection counts were not determined, but as percent area measurements are derived from the projection or intercept counts, they too are likely to be insensitive to variations in sectioning.

Despite the efforts of a works' metallurgist to keep the above error under control, it must be assumed that a certain proportion of the samples examined will not have been properly sectioned and, therefore, it is desirable to choose parameters that are not too sensitive to variations in sectioning.

Even if the above two problems could be overcome by more control over the operation of sectioning billet samples, and use of a more sophisticated instrument, it must be acknowledged that size distribution counts of inclusions in longitudinal, metallographic sections, prepared from billets (or bars), is a highly biased estimate of the true size distribution. For spherical particles a sound mathematical formula exists for correcting the apparent distribution to the true distribution function, but for non-spherical particles such corrections are difficult to apply. This problem has been given a fairly complete treatment by Hilliard and others (see References 155-164). In theory, it is possible to make such corrections, but in practice the procedure is unwieldy, and the distribution determined from a two-dimensional structure cannot be easily

related to the bulk material.

Whilst it is acknowledged that particle size distribution is important in some quantitative metallographic work, it is questionable whether this information is really needed in routine inclusion counting work, such as quality control in steelworks, where the primary concern appears to be in determining the presence, or absence, of any field in which the concentration of inclusions of a particular type exceeds some specified limits.

Because of the above difficulties in determining the number and size-distribution measurements of inclusions in longitudinal two-dimensional metallographic sections, particularly the latter measurement, it is suggested that for quality control purposes at least these two parameters should be given low priority, and attention directed at using the more accurately and easily determinable parameters: percent area and projection.

The volume fraction (percent area) of inclusions is generally agreed to be highly significant to low ductility, easy fracture, machinability and other behaviour of material affected by inclusions or similar non-cooperating phases, e.g. graphite in cast-iron. Edelson and Baldwin<sup>154</sup> for example, found volume fraction of all non-cooperating phases to be additive and a prime factor in the behaviour of copper-based compacts. Inclusions could behave in the same way and it is possible that it is the amount of inclusion present in a given microscopic area that matters and not what they are or how they are distributed.

It must also be acknowledged that the Quantimet will not distinguish between different types of oxides, e.g. alumina stringers, silicate stringers, etc. Discrimination between such inclusions is needed mainly because conventionally one large inclusion is

considered to be more harmful than the same volume of inclusion distributed as several small particles, and silicates tend to occur in larger sizes than most other types of inclusions commonly found in steels. Thus, discrimination between silicate and alumina is often needed for reason of size or severity rather than for reason of chemical identity.

This sort of deficiency is inevitable with any automatic machine which is competitively priced. Whilst it must be accepted that a human operator using a manual method could derive a lot more information, one must weigh these limitations of the automatic methods against the numerous obvious advantages they offer. The most important of these are good reproducibility, objectivity, labour saving and speed in not only determining the inclusion count, but also in processing of the data with suitable statistical constants. The latter enable sounder inferences to be made about the inclusion content of that sample of steel than would be possible otherwise. In any case, if it is accepted that it is the fields with high concentrations of oxides that matter, then the differentiation problem can be overcome as previously discussed by programming the instrument so that it stops on these "over-value" fields for visual inspection by the experienced operator to decide whether the "over-value" is due to a silicate, alumina or some other type of inclusion. It is unlikely that this procedure will be unduly time-consuming as it is highly improbable that a "clean" steel will contain a lot of these fields.

The results of this research clearly show that very large sampling errors exist with methods currently being used for sampling steel. But the economics of inclusion counting in a steelworks are such that even with an instrument such as the automatic Quantimet,



with a speed of 1 field per second, the required number of fields and samples needed, to reduce this error to a reasonably low level, cannot still be examined in the time available. Clearly, what is needed is a faster and cheaper instrument, since the solution to this problem is to sample more extensively and use more than one instrument.

In the meantime, sampling errors can be minimised by drawing samples from the correct positions in the ingot and cast. The results from this research show that for the steel and steelmaking practice examined, the correct sampling positions are from the ends of the two billets representing the bottom of the ingot from each group in the cast. The sampling error can be further minimised by examining only one half of the specimen, i.e. the region extending from mid-centre to centre of the billet sample.

Strictly speaking each type of steel made by a particular process and in a particular works has its own sampling requirements which should be determined experimentally in advance before standards are agreed between supplier and user.

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