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**THE DEVELOPMENT OF FRICTION  
MODIFYING BACK COATS  
FOR OPTICAL TAPES**

by

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**A Master's Thesis**

**Submitted in partial fulfilment of the requirements  
for the award of**

**The Institute of Polymer Technology and Materials**

**Engineering**

**of the Loughborough University of Technology**

**January 1995**

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## Abstract

For many years magnetic tapes and discs have been the standard method of storing large amounts of information, and increasingly optical discs are being used for some archive operations where high density storage is required. Optical Tape is the next logical step in the media storage market, offering increasingly higher data storage capacities at lower cost per byte of information than other storage methods.

Information is written onto optical discs and tapes by use of a focused laser. Writing a pattern of pits that are recognised as 1s and 0s by the computer, thus storing information using binary code as does magnetic media.

Optical Tape, unlike magnetic media, has a very smooth sensitive active layer, this is where information is written. The speed at which the tape moves through the tape drive necessitates a back coat to modify the frictional properties of the tape.

This programme of work investigates the development of backcoats for two different optical tapes, from the point where it was realised that a backcoat would be required to the production of a development grade material.

It was discovered that there was a fine balance between the degree of surface roughness required for good handling with appropriate frictional properties and prevention of damage to the sensitive active layers, either during tape handling or when the tapes are archived for long periods.

## Acknowledgements

The Author would like to acknowledge the assistance she has received during this project from the Staff of the Optical Data Storage Team at ICI Imagedata, Brantham, Essex. In particular much of this work could not have been completed without the assistance of Malcolm Deex. She would also like to thank her husband for putting up with her during the writing up.

*Supervisor 1.*

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## 1.0 INTRODUCTION

Most information that is to be archived is recorded onto magnetic tapes which are then stored in temperature and humidity controlled libraries. For most applications the storage capacity available from the various formats of magnetic media is adequate. However, as storage capacity requirements have increased, the amount of data that can be recorded on magnetic tape is becoming insufficient.

The tapes used vary from the type of small cassette we see in telephone answering machines, to 12 inch reels which have many hundred metres of tape. A typical High Density Digital Recording (HDDR) media, consisting of a 35 mm wide tape on a 380 mm reel has a capacity of some 100,000 mega bytes ( $10^{23}$  bytes). (1,2) ?

Capacity remains insufficient for industries that generate large quantities of information, like remote sensing and weather stations.

*Where* Storage capacity is lacking, tapes have to be changed frequently and huge warehouses are required to store the recorded tapes.

During the 1980s optical discs became available, increasing data storage capacity by many times. One optical disc is capable of storing the same amount of information as several magnetic discs of the same size. Optical data storage offers other benefits; speed of data retrieval, longer projected lifetimes and a way of permanently storing information. However, the storage volume offered by discs is still wanting for large data generators, unless expensive robotic jukeboxes are used. These jukeboxes work in a very similar manner to those used for music. The nature of the technology used for optical discs was not transferable to a flexible tape format.

Research in the late 1980s by Abbott, Burgess and McLean et al (3-7), at ICI Electronics, showed that it was possible to coat a dye, which could be activated by a laser and so record information onto flexible surfaces. From this work the idea of a flexible optical data

storage tape was derived, offering the possibility of vastly increased amounts of information on a single tape.

Early work concentrated on factors such as: what thickness of dye layer was required, how data was written and read back, how the laser affected the dye and use of an overcoat without impairing data storage capacity. During this time a partner company, Creo, was designing and building an optical tape drive.

*Development of*  
The first tapes produced had concentrated on the active layer, <sup>and</sup> it soon became clear that friction and wear would have to be addressed by application of a carefully designed and optimised backcoat.

The work reported here details the development of a suitable backcoat for optical tape, including the need for a back coat, properties required from such a coating and the development of theories regarding tape handling and abrasion.

## 1.1 Optical Disc Format

As recently as 15 years ago a new method of data storage came onto the market, in the shape of optical discs, whereby a greater amount of information, Philips claimed approximately 500 <sup>(MB)</sup> mega bytes, could be stored in the same physical area, e.g. a 5 ¼ inch disc. As opposed to 1 - 1.5 MB on a magnetic disc. A 5 ¼ inch magnetic disc could be expected to store between 0.4 MB (early floppy discs) & 50 MB (hard discs).

MB  
or  
MB

### 1.1.1 Disc Manufacture

Discs are produced <sup>from</sup> glass masters, by etching information in a digital form and/or a series of grooves onto a photo resist layer coated onto a glass plate: this is the master disc. For CDs (compact discs) music is digitised and the information formatted onto a master disc, CD ROM's (compact disc read only memory) have only a series of

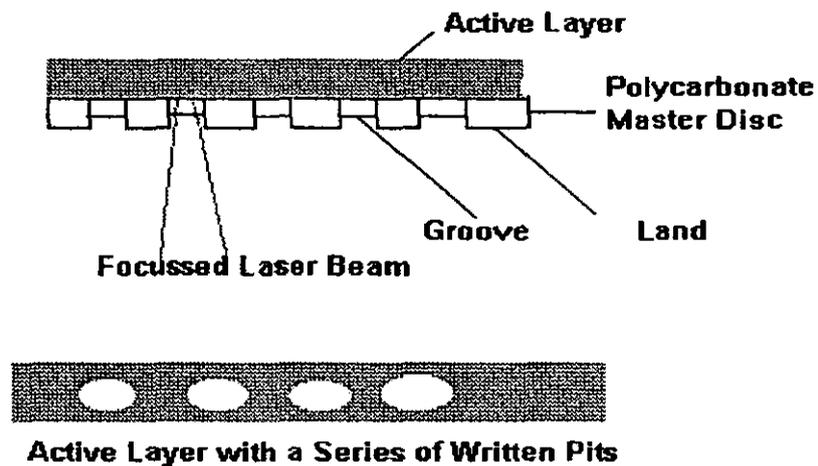
tracks impressed. The tracks are approximately  $1\mu\text{m}$  in width and either spiral or concentric grooves and lands, the width of which are in the order of  $1\mu\text{m}$  (Fig 1). The glass master is then coated with a thin conductive layer and a further, thicker metal layer is deposited on top. The glass and metal masters can then be separated, the metal master being used to  <sup>mould</sup>   ~~make~~  optical discs from a high grade polycarbonate resin. A reflector layer consisting of, for example, aluminium or germanium tellurium alloys, is then either sputtered or evaporated onto the stamped surface. This layer accepts pits allowing information to  <sup>write</sup>  be written and read back. (8) A protective layer, usually an acrylic, is then coated on top of the sensitive active layers, to protect them from dust, scratches and aggressive atmospheres.

## 1.2 Data Storage

Information is written using a laser, the laser emits a coherent beam of light which diverges as it leaves the laser, this is collected using mirrors and formed into a parallel (collimated) beam of light. The light beam then enters a beam splitter and is passed through a quarter wave plate which rotates the light through  $90^\circ$ . The light beam is then passed to the objective lens, which can be moved by the focus servo to direct the beam onto the recording layer. A tracking servo is used to move the beam of light onto the tracks of the disc. Light reflected from the disc surface returns via the same path, however it is now  $180^\circ$  out of phase with the initial light beam. Instead of passing through the beam splitter, it is reflected onto a photo detector where it is amplified to produce both data and feedback to enable the focus and tracking servos to keep the laser in the desired position. (2)

When information is written onto CD ROM's laser light passes through the substrate and is focused  <sup>S</sup>   ~~X~~  on the reflective layer. (Fig 1) Data is recorded using a beam of laser light of sufficient power to cause a change in the state of the recording layer. This can be a

change in crystalline state or a phase change (8), the altered state has a different percentage reflectivity to the original and thus can be read back by the laser. The most common type of disc at present is the WORM (write once read many). Information is recorded with a pattern of pits and lands being recognised as a series of <sup>ones</sup> 1s and <sup>zeros</sup> 0s by the computer. (2)



**Figure 1** Diagram showing pre-formatted polycarbonate disc with active layer and active layer with a series of written pits.

### 1.2.1. Comparison with Magnetic Recording

Magnetic recording heads are in constant contact with the media, thus head crashes, which occur when the spinning disc starts to wobble and the disc comes into contact with the recording head, are a real possibility. As the magnetic media and recording head are in contact, a certain amount of inertia has to be overcome to move the media. This inertia has frictional and temperature/humidity components, termed stiction. In high humidities the stiction can be so high that either the media will not move or layers of the magnetic media are physically removed resulting in loss of information and damaged

recording head and disc. Optical recording heads do not contact the tape or disc, thus the likelihood of head crashes is removed.

### 1.2.2. Optical Tape

From production of rigid optical discs the next logical step was to produce a tape format, thereby increasing data storage capacity even further. It is the intention of this thesis to investigate the development of a back coat for two types of optical tape. The 10 12 format as described below, a 3480 format will be described later.

Firstly, ICI 10 12 Optical paper, which was designed for use with the world's first optical tape drive, the Creo 1003. A 10 12 tape, the name is derived from its storage capacity, can store  $10^{12}$  bytes of information on 833 metres of 35 mm wide tape. The volumetric storage of 10 12 tape has been reported to be the equivalent of 100 high density 12 inch instrumentation tapes and 2,000 of 5 ¼ inch optical discs. (9, 10, 11)

Secondly, 3480, a lower storage capacity format for use in general industry, this format should be capable of storing approximately 3,500 mega bytes per cubic inch, this represents more than an order of magnitude improvement over an equivalent magnetic media format. As with the early optical discs the tape was a WORM format, which is particularly suited to archival data storage. This type of system is presently in use in several remote sensing locations around the world. Magnetic media was considered to be the highest density data storage medium and even with the recent well-publicised archival problems, is still generally the preferred method of data storage. Magnetic media is limited in many ways: storage capacity; archival life; product lifetime; time between recommended rewinds (to prevent creep, etc.); Price per byte of information; data rates; susceptibility to magnetic fields; inadmissibility in court. Thus optical tape could exploit many of these areas by capitalising on its inherent advantages: storage

capacity; cost per byte; lifetime and rewind interval; market advantages could be gained in other areas.

### 1.3 1012 Optical Tape - Properties

In a paper presented to the Tape Head Interface Conference (THIC) Dr J. Duffy showed that the 1012 format optical tape had a proposed lifetime in excess of 30 years, this was assessed using a Battelle ageing test. (11,12,13) <sup>in</sup> a class 2 Battelle test, <sup>115</sup> where 1 day testing <sup>is</sup> deemed to be equivalent to 6 months in a normal Los Angeles office environment. There appears to be some question as to whether this is a rather severe test. Battelle test conditions consist of  $23^{\circ}\text{C} \pm 1^{\circ}\text{C}$  and  $75\% \pm 2\%$  relative humidity,  $10\text{ppb} \pm 2\text{ppb}$   $\text{H}_2\text{S}$ ,  $10\text{ppb} \pm 2\text{ppb}$   $\text{Cl}$ ,  $100\text{ppb}$   $\text{NO}_2 \pm 15$  ppb. The Battelle test is a recognised standard within the magnetic media industry and as such gave the 30 year lifetime claim for 1012 optical media credibility.

The recent publicity regarding stored tapes, has shown that archival life of magnetic tape is suspect, in terms of chemical degradation.

(14) Unlike magnetic media, the active layer of optical tape has no contact with either read head or with bearings in the drive, resulting in low wear over many thousands of rewind cycles. (11,12,13)

Manufacturers of magnetic media for the high quality data storage market normally recommend rewind intervals of between 6 months and 3 years, Jorgensen presents a graph which indicates rewind intervals of  $2\frac{1}{2}$  months at archive conditions of  $17^{\circ}\text{C}$ , humidity is not mentioned. (15)

When tapes with PET as their base material are wound under tension, creep of the base PET takes place with time. The effect of creep is to reduce the tension of the wraps within the tape, as tension decreases due to stretching of the base, so that it becomes more likely that damage will occur within the tape. The rate of creep is not constant throughout the tape, because tensions are different at different

diameters of the tape, thus where tension is higher, creep will be greater, this can lead to folds and 'cinches' in the tape reel. This is prevented by rewinding tapes after pre-determined intervals, termed rewind interval.

Creep measurements using 10 12 tapes (16) has placed rewind intervals for optical tape in the region of 5 years, i.e. the tape would only require rewinding once every 5 years as opposed to once every 3 months for magnetic media. In large archive libraries rewinding tapes can occupy a person full time. Work being carried out at the moment suggests that rewind intervals for optical tape could possibly be increased to as much as 30 years. This of course appeals to the budgets of large companies as increased intervals between rewinds can significantly reduce overheads.

A 10 12 tape has been costed at 0.5 to 1.0 US cents per byte (at present exchange rates that is less than 1p), this is quoted as being a significant saving. (12)

Volumetric density data rates for the 10 12 product have been quoted as up to 1.5 MB per second, this can be increased by use of multiple lasers. ICI claim that the access time for a record of data in a 1 terra byte tape is 37 seconds. (12)

### 1.3.1 3480 Optical tape

At the time of the report the final structure of the 3480 tape format had not been decided. Tape drives are also still in the research stage. Thus, it will not be possible to describe the optics or read/write system. However, because of the much increased data rates being required and, the use of shorter wavelength lasers to increase data density, it seems unlikely that the recording layers will be similar to the 10 12 structure. However, it is intended that the tape will be approximately 180m long and 1/2" wide (the mixture of imperial and metric measurements is entirely of American basis).

## 1.4 Tape Structure

Figure 2 shows the basic structure used for both 1012 and 3480 format tapes. For research purposes the same active layer was used on both tape formats, however, it was intended that the 3480 tape would eventually have an inorganic active layer, rather than the organic dye/binder used for the 1012 tape. The function of the layers will be described in more detail.

All the early work was done using the 1012 format material, as a business decision halted the use of filled backcoats on this format, the back coat formulations were transferred to the 3480 research programme.

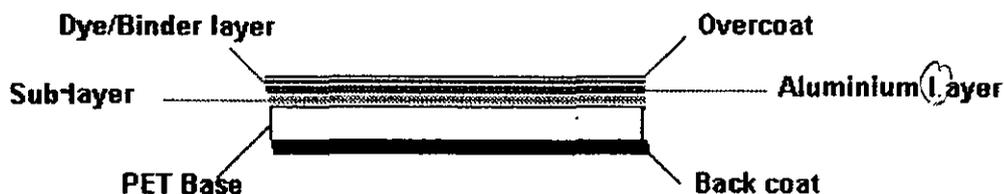


Figure 2 1012 and 3480 Tape Format

### 1.4.1 Base Film

The base films used were:

- a) A 75  $\mu\text{m}$  poly(ethylene terephthalate) (PET) Melinex D612 ex ICI films, (17) for the 1012 product,
- b) A 23  $\mu\text{m}$  PET, Melinex 451 ex ICI films, (18) for the 3480 format.

One side of the polyester was coated 'inter draw', i.e. a slip coating, filled with an amorphous fumed silica, applied to the extruded PET after the first mono-axial stretching process, the film was then biaxially stretched to its final structure. This coating prevented the two smooth faces of the polyester from coming into contact, without which, high pressures exerted in master roll format would lead

to blocking.<sup>1</sup> (19) Filler in the slip coat not only prevented blocking but assisted in the handling of the web material both at the manufacturers and the converters, by modifying the frictional coefficient of the film.

The slip coat itself did not have appropriate frictional properties for optical tape, being too smooth and tended to cause blocking against the active layers. Thus an additional friction modifying back coat was required. The side onto which the active layers which would be coated was smooth.

The PET film was supplied in large rolls 5,000<sup>m</sup> long and 2<sup>m</sup> wide termed master rolls, these had to be slit into smaller rolls 12 inches wide and 2,000<sup>m</sup> length for use on the ICI Imagedata pilot plant. The 12 inch rolls were termed 'jumbos'

Deformation of the base film occurred when: the film was wound over an imperfection in the base, <sup>or</sup> there were thickness variations in the film, or debris on the central core. As the number of wraps increased the effect caused by small pieces of debris or deformations also increased; these were termed base deformations. Base deformation severity increased as base film thickness decreased and was present in several forms:

- a) A diamond pattern which was termed "chicken wiring" was caused by differential pressures across the web mainly due to micron scale differences in film thickness between the edge of the jumbo and the centre
- b) <sup>In</sup> Areas where the polyester was raised, <sup>caused</sup> 'high lines' were caused, i.e. defects at the core of the jumbo a line of different thickness film, or a crease in the film.

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<sup>1</sup> Blocking occurs when two smooth surfaces come into intimate contact, usually physical forces, e.g. hydrogen bonding, but on occasion chemical forces, bind the two layers together such that they are difficult, if not impossible, to separate. This can lead to: transfer of the slip layer from one surface to another: deformation of the base: tearing of the base when the tape was unwound: and in extreme cases can result in a solid block of PET.

### 1.4.2 Back coat

The very first tape samples produced utilised the slip layer as a back coat. It was at this point that the authors' involvement began. However, it became clear that the slip coat would not be acceptable as a back coat, for three reasons:

- a) The slip coat was applied as a continuous coating after the film had been mono-axially stretched <sup>However,</sup> and at subsequent bi-axial stretching, due to differential flexibilities, became discontinuous therefore the frictional coefficient tended to be inconsistent across both the length and width of the film.
- b) Early wear data indicated that the slip coat was too abrasive.
- c) As well as being inconsistent, the friction was too high for handling on the test drive.

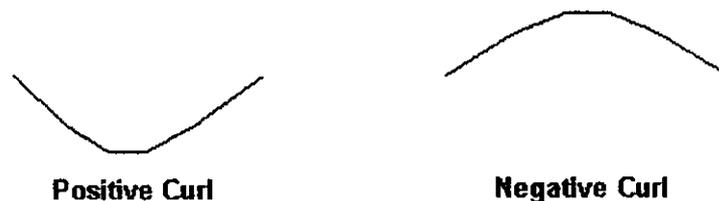
### 1.4.3 Sub coat

The base films used contained a percentage of <sup>re-work</sup> re-cycled polyester from previous batches, also production was not carried out under clean room conditions and thus debris from various sources tended to be incorporated into the web. Debris analysed by the manufacturers was found to be of several forms: metallic (iron, titanium, etc.): organic (skin flakes, sweat droplets, etc.). These defects tended to protrude from the surface of the film and caused many and varied problems.

Organic defects often had different properties e.g. surface tension, to that of the base film, thus the subsequent layers either adhered poorly or retracted from the area resulting in a larger defect in the active layers. If the defects were greater than the coating thickness they protruded through the active layers. This then caused problems in writing and reading information, wear, handling, product lifetime and/or initiated chemical degradation of the active layers. Thus the

active life of the product could be reduced by physical wear or by chemical degradation. If the layers retracted from a defect because the flow of dye coat and overcoat was interrupted, the surrounding active layers exhibited a change in reflectivity, due to a change in film thickness, ~~the~~ sources of read/write errors. If large defects protruded from the surface then information could not be recorded in that area.

Thus, to try and minimise these sorts of problems an unfilled 'subbing' layer was introduced to smooth over and cover defects. This sub layer consisted of a multi functional, acrylate system which was crosslinked by ultra violet irradiation. Because the sub layer was highly cross linked there was a high degree of shrinkage associated with the cured film, this was not of any import on a thick  $75\mu\text{m}$  base. However, when the technology was transferred to thinner  $23\mu\text{m}$  base, sub layer shrinkage induced severe positive curl. (F ig 3) It is proposed that positive curl be denoted as active layer edges curing towards themselves.



**Figure 3 Positive and negative curl. (Active layer uppermost)**

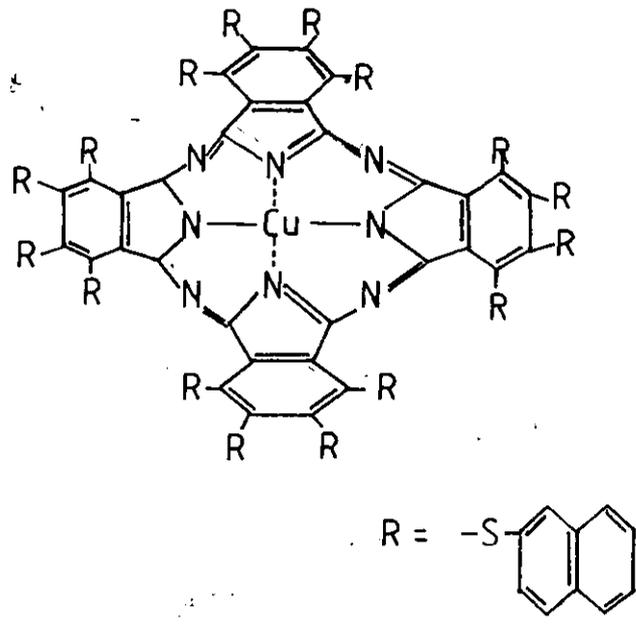
#### **1.4.4 Reflector layer**

The reflector layer was sputtered using a Vac Tec SX2 sputterer and consisted of a 95% pure aluminium material. The layer was coated at 50 nm, at this thickness bulk reflectivity of aluminium was achieved. Calibration of the reflectivity is done using a gold mirror as nominal

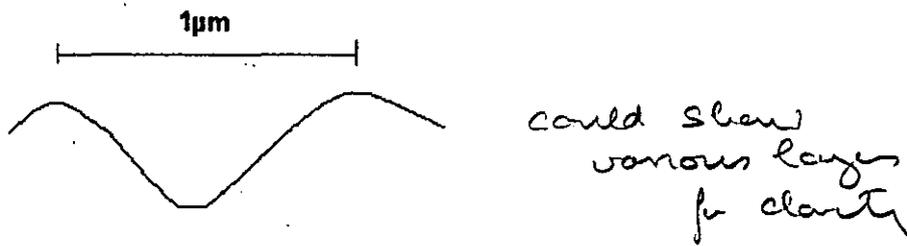
100% reflectivity, on this scale sputtered aluminium exhibited in the region of 90% reflectivity. A sample of sputtered aluminium, <sup>was</sup> then <sup>used</sup> to re-calibrate the spectrophotometer to 100%, this is then used to monitor the reflectivities of the dye and overcoat layers.

**1.4.5 Dye layer**

The dye layer consisted of a 3:1 blend of a copper phthalocyanine material which absorbed strongly in the region of 830 nm, the structure of which is presented below, and a polyester resin.



When a pit is written using a laser, heat generated by the laser melts the dye/binder and deposits the dye/binder from the activated area around the edge forming a pit, as shown.



**Figure 4** Written pit with debris around edges

It was possible to store information in this format, however, the wear properties were extremely poor giving short product lifetimes, therefore it was deemed necessary to add a protective overcoat.

#### 1.4.6 Overcoat

The intention of the overcoat was to protect the active layers from *mechanical damage* physical and chemical attack. In the early days of the project the overcoat was some 240 nm thick, this was calculated to be a quarter wavelength at 830 nm, thus not providing an optical barrier to the read/write laser. However the sensitivity of the tape was significantly reduced by such a thick overcoat. A 240 nm overcoat reduced the depth of the pit thus making the difference in reflectivity of written and unwritten areas smaller and resulting in poor read back of data. To correct this, more power was required from the write laser to deform the rigid overcoat and ensure the required pit depth. However when the media was wound into tape form under pressure, the pits/bubbles formed in the active layers were squashed resulting in loss of data.

To enable the laser power to be reduced a 40nm film was used and provided optimum read/write properties. At this 'thinness' the protection provided was sufficient to obtain the required number of wear cycles whilst maintaining optical sensitivity. The overcoat consisted of a radiation cured material, a blend of a hexa-functional aromatic poly(ester urethane acrylate) and a di-functional aromatic poly(ester urethane acrylate).

#### 1.5 Pit formation in an overcoated dye/polymer layer

Heat generated by action of the laser, in the thin absorbing dye layer was used to write a pit, the laser was active for around 100 nano seconds per pit. The heat generated caused irreversible changes in

this layer and created pits of approximately 1 $\mu$ m diameter (3). Much work has been done by Abbott, Burgess & McLean et al (3-7) in the investigation of pit formation. When the early work was carried out with these systems a 240 nm thick overcoat was being used. Theory indicated that heat generated by a laser in media with a 240 nm overcoat produced a blister or a bubble. Although the actual mechanisms are still not clearly understood, several models have been suggested for pit formation (6): evaporation of dye/binder, bubble formation and surface tension gradients. The latter theory proposed that thermal stresses produced by action of the laser rose sufficiently rapidly to produce failure between the aluminium and dye interfaces and dye and overcoat interfaces resulting in blister formation. (5) This theory discounted the suggestions that gaseous elements produced by heating of the dye/polymer produced sufficient pressure on the heated overcoat to cause blister formation.

It has been experimentally observed (6) that the process of pit formation proceeds in two distinct phases:

- a) A hole is generated in the dye/polymer for a laser power pulse of 15 nano-joules, this phase takes about 10 nano seconds. During this period the temperature generated in the dye binder is sufficient to overcome the energy threshold for hole formation.
- b) A hole growth phase when energy released is converted into kinetic energy which promotes hole growth to produce the final pit. This phase takes approximately 85 nano seconds at the same laser power.

At the focus point the laser provided a light spot of 1 $\mu$ m diameter focused on the dye layer, the overcoat layer being optically invisible. The laser is calibrated such that when activated, a pit of 1 nm depth is written (depth measured by atomic force microscopy). The depth of the pit corresponds to a maximum change in dye reflectivity, from 45% to approximately 12%. To read back information the laser power is reduced and the difference between 45% & 12% reflectivity

recorded as 1 or 0. The power setting for the read back laser is determined for individual tapes, normally being in the region of 2 nanojoules.

Modelling (3-7) has suggested that temperatures up to, and possibly exceeding 1000 °C are reached in the area of the pit.

Early work speculated that in the time scales appropriate to pit formation there would be an absence of thermal degradation. There has as yet been no evidence to support or deny the speculation.

In contrast to a 240 nm overcoat where a bubble or blister is formed in the active layers, when a thin (40nm) overcoat is employed there is sufficient impetus to blast dye/binder and overcoat out of the pit. This has been termed ablative writing. The mechanism of gas formation, ablation and the physical form of the dye/binder during ablation has been the source of much speculation and theorising, which as yet is not formalised. What is apparent is that dye/binder and overcoat return to the tape and various parts of the tape drive as debris. The ablated debris is the source of read/write errors and also of wear and thus must be cleaned from the tape drive. At present this is achieved by vacuum nozzle, located near the read/write head.

## **1.6 Comparison with magnetic media.**

Much of the early back coat development work used the formulations, chemistry and dispersion techniques of magnetic media. However, it became evident that the technology used in the magnetic media industry could not be utilised and that specific techniques and processes would have to be developed. Magnetic media used PET as in optical tapes. A sub layer is not required in magnetic media as defects in the base film do not have disastrous effects as in optical media.

### 1.6.1 Back coat

Some forms of magnetic media used a highly filled PET base, the surface roughness of the PET is sufficient to produce the friction characteristics required, thus a backcoat is not always necessary.

Where a back coat is present, a non magnetic coating, which provides the required handling and wear properties, is used. Backcoats often contain carbon black to prevent static build up and help prevent air entrainment during handling, both on the plant and on the drive. In both optical and magnetic tapes, air entrainment between layers of tape can cause problems with winding tension, lateral movement of the tape and in some cases be the cause of defects in the tape.

Unlike optical tape, the magnetic layer is usually coated first and fully cured before the back coat is applied, to prevent any problems with handling or blocking. (20) Optical tapes have the backcoat applied first, before the active layers. To apply the active layers first results in scratches and other various defects on the optically active surface.

Most magnetic media back coat systems are filled almost to the Critical Pigment Volume Concentration (CPVC). CPVC is the point at which there is insufficient binder to fully coat pigment particles and hold the particles together in a consistent coating. The filler is the cheapest part of the coating solution. Most back coats have two different filler types, a very small particle size material which provides a general background roughness and larger particles to give regular bearing points and so modify the frictional properties. Fillers used in backcoats include: carbon black, fumed silica, calcium carbonate, titanium dioxide, alumina and various metal oxides. (21)

Polyurethanes were the most commonly used back coat binder.

Previously most binders were ~~polyester, urethanes~~ however recent problems with hydrolysis of the polyester (14,22,23) have led to the use of other polyol/binders, such as polyethers, polycaprolactones, polycarbonates, etc. all of which tend to have better resistance to

hydrolysis. In many magnetic tapes polyurethane binders have been replaced by radiation cured materials.

As with optical tape the back coat for a magnetic tape must have good adhesion to the base material, provide optimal friction properties and not introduce curl into the media.

### **1.6.2 Active Layers**

The formulations used for the active side of the tape are very much dependent upon the end use, for high volume low cost audio tapes different magnetic pigments would be used than would for high quality data storage applications, iron oxides and chromium dioxides respectively.

Standard analogue tapes can contain as much as 70% by weight of magnetic pigment (around 50% by volume) this is not as close to the CPVC as backcoats. It has been reported that increasing beyond this loading can have deleterious effects on the physical and chemical properties of the tape without improving the recording characteristics (21). Strong interaction between magnetic particles and binder is essential to ensure: optimisation of mechanical properties within the coating: adhesion to a substrate and particle/binder adhesion. The choice of binder and pigment is determined by several factors: final cost of the product: quality required by the market: chemical interactions, etc.

Bradshaw and Bhushan showed that the chemistry of the binder/magnetic particle interaction does not significantly effect frictional coefficient of the media. More significant with relevance to degradation are the effects of hydrolysis of the media at high humidities. (22,23,24) It is not the intention of this thesis to discuss the merits of various magnetic pigments and binders.

Dispersion of magnetic pigments in binders must be as homogenous as possible to ensure not only good coating properties but optimum

recording characteristics. The binder was usually the same on both over and backcoats, which helped to reduce any differential curl and assist in tape run-ability. (20,21)

As most polyol/isocyanate/polyurethane <sup>based</sup> systems require a longer cure than EB or UV cure the magnetic layer is applied first; this is partially heat cured on the coating machine. A thermal post cure is completed before any further layers are added. Before thermal post curing the particles are aligned by application of a magnetic field.

The final tape (both active and back coat layers) requires the following properties: blocking resistance: low wear: resistance to degradation at elevated temperatures and humidities: high signal output: must not shed coating or filler either during tape slitting or in use: good adhesion of coatings to the substrate: low frictional coefficient: low curl: flexibility: hard wearing whilst not inducing wear of tape drive components, e.g. guide fingers and read/write heads.

### 1.6.3 Lubricants

Lubricants normally used were perfluoro carbon materials applied at between 1 and 4 nm thickness, however, this was very dependent upon surface roughness of the media. It has been established that the use of a lubricant improves the lifetime of magnetic media. (25,26) By reducing the frictional coefficient between the active layers and the read/write head and improving the 'run-ability' of the tape in the drive, both of which reduced the wear on tapes.

Lubricants aid handling, reduce friction and stiction in magnetic tapes. The lubricant is considered to be present in two phases, an adsorbed layer and a mobile phase. (25,26) The thickness of the adsorbed layer is dependent upon the surface roughness profile of the media. It is generally believed that lubricants which are well adhered to the media surface provide optimum properties, however, the precise chemistry

depends upon binder and filler chemistry. Streator et al. examined different types of lubricants within the perfluoropolyether family. They showed that the presence of lubricant reduces the rate of friction build up (except where the lubricant layer is thick enough to introduce stiction forces). Low viscosity lubricants performed best, being the most mobile. Beyond the optimum lubricant layer thickness, friction was found to increase with increasing lubricant thickness. (25)

Lubricant improved friction and handling however if the lubricant layer was too thick the likelihood of magnetic head/media interface stiction increased. (27)

#### **1.6.4 Manufacture of Magnetic Tapes**

The appropriate binders, pigments, fillers, wetting agents, adhesion promoters and crosslinking agents require dispersion to form a stable, homogenous coating. Dispersion was usually carried out in a bead mill, however to ensure that no large agglomerates or gels are coated onto the tape the solution is filtered prior to coating. (21,28,29,30,31) The filtration process involved the use of several filters of decreasing pore size, employed in series, intended to gradually remove any unwanted material. As filtration was usually carried out under pressure this process also re-dispersed any loosely formed gels and agglomerates. (31)

After coating the active layers and back coats were post cured to ensure maximum physical properties. More and more manufacturers are now looking to replace standard polyurethane technology, with electron beam cured acrylate copolymer coatings thus removing the necessity for a post cure stage. (20,32)

#### **1.6.5 Writing Information**

Most common types of magnetic media consist of magnetic oxide pigments held in a binder, which have been aligned by application of a

magnetic field. Bringing the tape into contact with a magnetic source, e.g. the writing head induces the particles to orient out of alignment with the majority of the particles. Thus series of realigned particles can be read as 1s and 0s. This process can be repeated, in theory an unlimited number of times, however in practice there are many factors which can reduce the effectiveness of magnetic media.

### 1.6.6 Degradation of magnetic media

As discussed above the binder in most magnetic media was of polyester-polyol based polyurethane. It is well documented that aromatic polyester-polyurethanes, and to a much lesser extent polyether-polyurethanes are susceptible to hydrolytic degradation. The binder hydrolyses to the extent that when the head and media are brought into contact under reading conditions the head removes some of the binder and magnetic particles, permanently destroying the recordings. (14)

<sup>or just</sup> Polyester-polyurethanes have been shown to interact with magnetic particles such as gamma  $\text{Fe}_2\text{O}_3$ ,  $\text{CrO}_2$  and water vapour, in a deleterious manner. (21) This hydrolytic interaction results in the production of low molecular weight fragments, and in turn in reduction of mechanical properties of the binder, increased friction and increased susceptibility to stiction. (21,26,33)

It is believed that the magnetic pigment itself may play a large part in the hydrolysis of polyester polyurethanes, Bradshaw et al. have shown that gamma- $\text{Fe}_2\text{O}_3$  does not induce hydrolysis to the same extent as  $\text{CrO}_2$ . (22) This degradation is of course dependent upon the binder formulation and conditions used where the tapes are stored.

Individual tape formulations require analysis before any statements can be made.

### 1.6.7 Print-Through

Print-through is a phenomenon in magnetic media whereby the magnetic fields surrounding particles and data sets can be strong enough to induce re-orientation of magnetic particles in adjacent layers of tape. Thus the information on one layer corrupts information on the next and can result in data being imperfectly copied. Flux lines from one layer can reach contacting wraps of tape and with time, temperature and external 'fields' cause the adjacent layer to become weakly recorded. This phenomenon tends to be more prevalent with smaller magnetic particles. Jorgensen has published a mathematical correlation for time for print-through to occur versus particle size. (15) This is a magnetic phenomenon and tends to be associated with thinner base products such as 12  $\mu\text{m}$  media. The term 'print-through' has been adopted for use in optical tape, where it refers to the physical imprint of particles in one layer of tape onto the next layer of tape, resulting in a textured surface.

### 1.6.8 Drop Outs

Drop outs can be compared with the defects that cause read/write errors in optical tape, they can be caused by an imperfection in the coating or debris particles between the tape and tape head which distances the tape from the tape head preventing reading/recording data. (15,20)

### 1.6.9 Stiction

Stiction is the phenomenon which occurs, normally between a magnetic tape and a ceramic recording head, whereby the recording head sticks to the magnetic surface. Magnetic media (floppy discs, hard discs and tapes) is in constant contact with the recording head,

unlike optical tapes. The gap between optical tape and the read/write head is several nanometers, the size of gap differing between optical tape drives, depending upon focus of the laser.

The term stiction has also been used in the optical tape industry, where it describes two layers of optical tape sticking together so strongly that the interfacial tensions are stronger than the bonds between active layers and substrate: active layers may be removed during unwinding of the tape.

In the magnetic media industry it has come to mean the action that causes adhesion between head and media and the force required to unstick the two and overcome friction or stiction forces. When a liquid is present a meniscus tends to be formed, between recording head and media, and energy is required to overcome the forces involved. Stiction is especially pronounced when adsorbed moisture and/or excessive quantities of lubricant are present. (33,34,35)

### **1.7 Aims of the research**

As stated above the aim of the programme was to develop, test and bring to production a friction modifying backcoat for flexible optical data storage media. Preferred properties included the following:

- a) Friction modification
- b) Should not abrade the overcoat during tape handling
- c) Be anti-static
- d) Exhibit good adhesion to Melinex
- e) Be non-shedding
- f) Provide the correct frictional properties without the addition of a silicone slip agent
- g) Have a degree of abrasion resistance to the rollers, guide fingers and capstans in the drive.

These properties will be expanded on in the following sections.

### 1.7.1 Friction Modification

Friction modification was required because the active side of optical tape is very smooth. Therefore, against a smooth back coat the ~~force to~~ <sup>force to overcome</sup> friction would be higher than the adhesive bonds between the dye layer and the aluminised layer. This would lead not only to blocking but result in partial removal of the dye and overcoat layers from the aluminium, and produce adhesion to the backcoat. Thus a backcoat that modifies the coefficient of friction, and allow the tape to move against itself during handling of the tape was required. If the coefficient of friction is too low the tape would move against its self too easily, resulting in tape reel tension and handling problems.

### 1.7.2 Abrasion

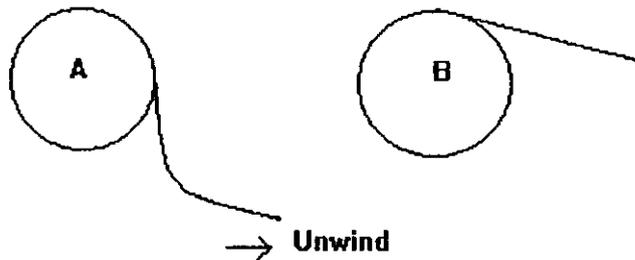
The overcoat on optical tape was initially 250 nm thick, but was later reduced to 40nm, at this film thickness the abrasion resistance was negligible. The drive manufacturers' specification require <sup>that</sup> the tape survive 100,000 cycles at 8m/sec read/write speed and 15m/sec rewind speed. Thus a balance between friction modification and abrasion resistance was required. / why?

### 1.7.3 Static build up

Optical tape with the development grade backcoat was susceptible to static build up, especially during high speed winding. Static charge built up to such a degree that a discharge spark was seen between tape and metal parts of the tape drive. This discharge tended to induce 'edge damage', which occurred when the active coat was vaporised by the effects of the discharge.

Less spectacularly an anti-static tape handled with greater ease, both during manufacture on coating machinery and in use on tape drives

(Fig 5 B). Tapes with high static tended to have poor unwinding profiles (Fig 5 A), in that rather than the tape exiting the spool cleanly the static charge held the tape to the spool beyond the natural unwind point.



**Fig 5 Effect of Static charge on tape unwinding**

If the adhesion of the backcoat to Melinex was poor or it had a tendency to shed particles of filler and/or resin during use, this debris induced wear of the active sides reducing the lifetime of the tape, and also possibly leading to loss of information.

### **1.8 Backcoats employed initially**

The backcoat resin employed at the start of the programme was a di-functional epoxy acrylate, with a low filler loading of pre-dispersed alumina hydrate (pseudo boehmite). The alumina hydrate was received in chip form, as approximately 40% filler in equal parts of cellulose di-acetate and di-iso-octyl phthalate. No Quality Control was performed on the materials either by the suppliers or by ICI upon receipt. The backcoat initially was thought to provide the required frictional properties, however, it was reported to suffer from shedding. Shedding occurred when a back coated jumbo was passed through the coating machine for subsequent coatings. As the PET passed around small diameter rollers in the machine, the flexing of the PET film caused flakes of backcoat to be removed. It was considered that the shedding problem arose from poor adhesion of the hard, brittle resin to PET. A standard formulation was not available as the percentage

filler content had to be changed with each batch, to maintain a constant post filtration product.

## 2.0 Aims

For many years magnetic tape has been the industry standard method of storing data. As commerce, academe and governments have ever increasing data storage requirements, capacities of the different formats have been pushed to the limits of the available technology.

A higher capacity data storage medium is now available in the form of optical data storage tape. Optical tape uses a layer of optically active material where information is written and read by use of a laser.

Information is written by changing the character of the optical layer (each type of tape using a different method), using a laser. The laser writes a spot of  $1\mu\text{m}$  diameter or less, thus the data storage capacity is much greater than any form of magnetic media presently available.

The aims of the programme were to develop, test and bring to pilot stage a friction modifying back coat for flexible optical data storage media.

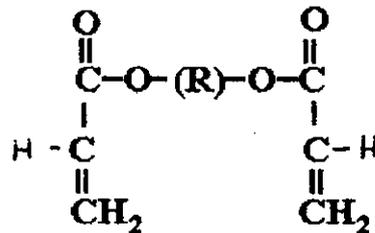
Properties required of the back coat include the following: friction modification: be non-abrasive against the overcoat: be anti-static: exhibit good adhesion to Melinex: be non-shedding and to have a degree of abrasion resistance to the rollers and capstans in the drive.

This thesis will describe the programme to develop a friction modifying back coat for ICI Imagedatas' optical data storage tape.

ICI Imagedata are presently working with optical drive manufacturers to develop new tape drives and optical tapes for several markets. A large data storage tape with the capacity to store  $10^{12}$  bytes of information is now in use in remote sensing. Other formats for smaller users are in the design stage.

### 3.1.2 Ultra Violet Curing

Ultra violet initiators mixed with low molecular weight oligomers (or monomers) containing unsaturated end groups, which when irradiated with ultra violet light, produce dry films within seconds. The oligomers used in the research were multifunctional acrylates, i.e. they had between two and six unsaturated acrylic end groups.



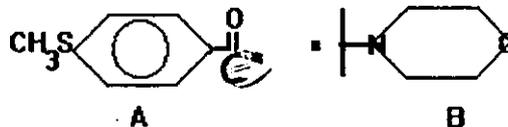
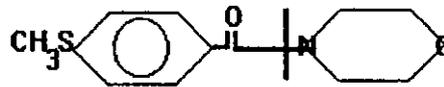
Where R is an oligomeric material, e.g. urethane, epoxy, acrylate, polyester, soya oil, etc. Functionality (f) is the term used to describe the number of reactive end groups available. For film production oligomers with more than two acrylic groups or ( $f \geq 2.2$ ) are preferable as a crosslinked film is then produced. 2 x cross?

When Irgacure 907 (Fig 6) is irradiated by ultra violet light free radicals are formed by the Norrish I process.

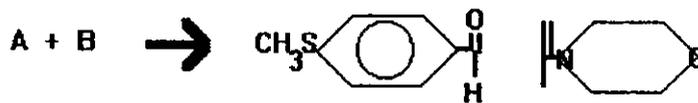
$\beta$  hydrogen abstraction results in a primary radical pair, further reaction with a hydrogen donor, e.g. unsaturated oligomers, produces an oligomer with a free radical site which will react with adjacent unsaturated groups, resulting in either chain extension or crosslinking depending upon the functionality of the polymer used.

(Fig 6)

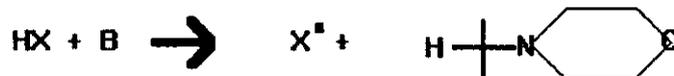
The acrylate group provides the hydrogen for the reaction resulting in a cleaved double bond that subsequently reacts with further acrylic groups to produce crosslinks. The complexity of the network is dependent upon the functionality of the acrylic oligomer.



- Beta Hydrogen abstraction (primary radical pair)



- Reaction with a hydrogen donor



• = free radical

Figure 6 Norrish I reaction path for Irgacure 907

Di-functional materials produce linear structures, whilst oligomers with a functionality of greater than 2.2 produce cross linked networks, the distance between crosslinks being determined by:

molecular weight of the oligomers and the functionality of the oligomer.

Hexa-functional oligomers will produce a system with higher crosslink density than a system of functionality 2.2.

U.V curing technology has many advantages, however, the techniques used to produce thin films by bead coating negated many of the advantages.

- a) Solvent free, for the production of sub-micron films a low solids solution was required.
- b) Low energy consumption, ovens were required to drive off solvent before the film passed through the UV curing head.
- c) Less wastage, use of low solids solutions inevitably wasted large amounts of solvent.

### **3.1.3 Screening of Resins**

Historically the resin used for the back coat was Ebecryl 600, an epoxy di-acrylate, which produced a brittle coating with poor adhesion to PET. As good adhesion of the back coat was a major requirement a programme of screening acrylated resins was evaluated.

All resins were used in a chosen solvent as 6% solutions, each with the same initiator and additives. The initiator as described above was Irgacure 907 ex Ciba Geigy, also added was an oxygen inhibitor Uvecryl P 115 ex Radcure Ltd. The back coat was applied at 0.5  $\mu\text{m}$ . Reduced cure due to oxygen inhibition was a possibility, without the addition of either a passive inhibitor such as wax or an active inhibitor, e.g. Uvecryl P 115. Uvecryl P 115 is a di-functional tertiary amine, with acrylic functional groups, molecular weight 500 - 600.

It has been shown that significantly more energy is required to cure the top 1 μm of films exposed to air, because of oxygen inhibition. Oxygen has a triplet ground state, which although not sufficiently reactive to initiate polymerisation can compete with the initiator molecules and thus prevent polymerisation. The chemical processes involved have not as yet been determined. (38,39) The presence of unpolymerised material on the surface of a film has been shown by Wright and Nunez. They also showed that the effects of oxygen inhibition can be reduced by increasing UV exposure time, light intensity and by increasing photoinitiator concentration.

Oxygen inhibition can be further reduced:

- a) Curing in an inert atmosphere such as nitrogen
- b) Adding a wax that migrates to the coating surface providing a protective layer, through which oxygen does not readily pass.
- c) Adding an amine, which is believed to react preferentially with the peroxy radical RO<sub>2</sub> to give a more reactive radical, thus preventing premature chain termination. (38,38)

The formulation used for screening the resins was as shown in Table 1

**Table 1 Formulation used for screening of Resins**

<b>Material</b>	<b>% by Weight</b>
Resin	6
Solvent Mixture	44
Irgacure 907	0.24
Uvecryl P115	0.30
Solvent Mixture	49.46

Several types of acrylated resins were tried, physical type and results are presented in Table 2. (37)

**Table 2 Trade name and chemical type for range of acrylate resins, ex Radcure Ltd.**

<b>Type</b>	<b>Resin</b>
Acrylic Oligomer in HDDA	Ebecryl 754
Acrylic Oligomer in TPGDA & HDDA	Ebecryl 745
Tetra-functional polyacrylate	Ebecryl 810
Siloxane Polyacrylate	Ebecryl 1360
Di-functional polyacrylate	Ebecryl 600
Acrylated Soya Bean Oil	Ebecryl 860
Di-functional aliphatic urethane acrylate	Ebecryl 4835
Di-functional aliphatic urethane acrylate	Ebecryl 264
Di-functional aromatic urethane acrylate	Ebecryl 210
Di-functional aliphatic urethane acrylate	Ebecryl 4833
Di-functional aliphatic urethane acrylate	Ebecryl 4858
Tri-functional aliphatic urethane acrylate	Ebecryl 5264*
Hexa-functional aromatic urethane acrylate	Ebecryl 220
Hexa-functional aliphatic urethane acrylate	Ebecryl 1290
Hexa-functional aliphatic urethane acrylate	Ebecryl 5129*

\* These resins only became available during the later stages of the research.

It was decided that Ebecryl 264 and Ebecryl 4835 would provide the properties required. However, these materials were found to be of limited availability and therefore alternative resins were selected for further work, these being Ebecryl 1290 and Ebecryl 220. Both Ebecryl 220 and Ebecryl 1290 produced films with good adhesion to Melinex and excellent abrasion resistance.

However, when fillers were added, even at low loadings, adhesion was found to be reduced. Adhesion was improved by the addition of a di-functional aromatic urethane acrylate, Ebecryl 210, which also acted as a flexibilizing agent.

Radcure subsequently introduced a hexa-functional aliphatic urethane acrylate, Ebecryl 5129 which was claimed to be a more flexible oligomer than Ebecryl 220 with good abrasion resistance. Trials showed this to be the case and it was used for the remainder of the research.

FTIR analysis indicated that Ebecryl 5 129 and 220 were very similar, being aliphatic and aromatic homologues of each other. It is generally recognised that aliphatic urethanes are more flexible than aromatic urethanes.

It is believed that Ebecryl 5 129 and Ebecryl 220 are acrylated methyl 4,4 di-cyclohexyl di-isocyanate (or HMDI) and methyl 4,4 diphenyl di-isocyanate (or MDI).

### **3.1.4 Fillers Used**

#### **Alumina hydrate**

It was decided initially to limit fillers examined to calcium carbonate, fumed silica and a new grade of alumina hydrate, using the alumina hydrate chips as a control.

Prior to particle size analysis (Malvern Automeasure method) it was believed that the particles were 1 $\mu$ m or greater. Subsequent testing of solution samples in various solvents showed that particle size varied from 0.1 $\mu$ m to 0.7 $\mu$ m. Solvent type affected the breadth of size distribution as well as average particle size. (Fig 7)

Alumina chips were added to the resin solution to achieve approximately 1.25% alumina w.r.t. resin, previous analysis showed that DIOP, cellulose acetate and alumina hydrate were present in roughly equal quantities in the chips.

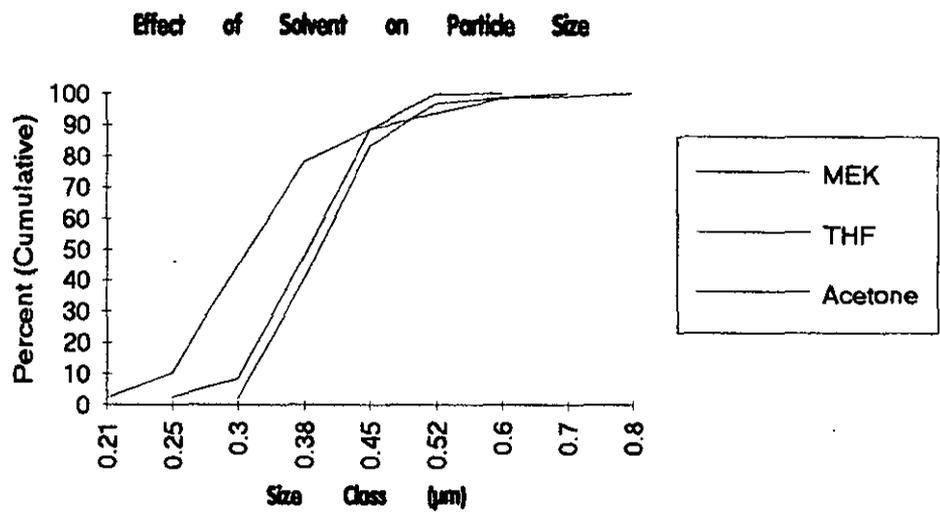
#### **Calcium carbonate**

Two samples of precipitated calcium carbonate were obtained from ICI Chemicals and Polymers;

(i) Winofil SPM was surface treated with calcium stearate, particle size of 0.075 $\mu$ m.

(ii) Winofil SUF with an untreated surface and particle size of 0.04 $\mu$ m.

(40)



**Figure 7**

### 3.1.5 Back coat properties

At the coat thickness used the epoxy di-acrylate (Ebecryl 600) was sufficiently flexible for back coat use, however the adhesion obtained was less than desired. (41) Ideally a pull off of 0 in 8 pulls was required; Ebecryl 600 usually gave 2 or 3 squares pulled off in 8 pulls. A frictional coefficient between 0.2 & 0.3 (BTM 207) had been determined by consultation with the drive manufacturers. (42) The tape generally handled well in the unwritten state on the Ashe test drive, however there was a tendency for static build up in the tape which caused wear and handling problems.

*define  
& describe  
failure!*

## 3.2 Techniques and Equipment

### 3.2.1 Bead Milling

Implementation of the dispersion of filler particles was done with a Dispermat SL horizontal bead mill, which was explosion proof, had variable speed control and the capability for recirculation milling. The mill chamber had a 50 ml capacity, and with a recirculation chamber fitted 150 ml - 1 litre of mill base could be made per batch. The optimum conditions for the mill were determined to be 3000 rpm, which equates to an impeller peripheral tip speed of 5m/second, a recirculation rate of 100 mls/min, 80% of the milling chamber filled with beads, (i.e. 40 mls of beads in the milling chamber), with the milling time dependent upon formulation. In the early stages of the research, glass beads were used, which were later substituted with zirconium oxide beads. These have a higher density and given the same milling conditions, will normally produce a finer particle size mill base than can be achieved with glass beads.

The rear of the milling chamber was separated from the motor by a set of lip seals, these were cooled and lubricated with a solvent, which had to be compatible both with the lip seals and with the mill base.

Bead milling was found to produce more repeatable dispersions than other techniques tried, as monitored by particle size analysis.

### **3.2.2 Particle Size Analysis**

Particle sizing was initially carried out on a Malvern Automeasure 4700 V4, at ICI Materials, Runcorn Heath. The equipment is capable of grading particles in size ranges of between 3 nm and 3000 nm, and measures particle size by quasielastic light scattering spectroscopy, also known as photocalorrelation spectroscopy. (43,44)

The technique required that solutions be colloidal. Brownian motion of the particles scattered the laser beam light and the returning light was used to estimate particle size. The Malvern instrument measured the rate of change of light fluctuations to determine particle size.

Cleanliness of the apparatus and in the preparation of samples was important for reproducibility of results. Later it became possible to perform particle sizing on site using a Fritsch Laser Particle Sizer Analysette 22. Although the particle size range was more restricted at the lower end of the range, easy access to the equipment was felt to be more important. The Fritsch apparatus had a particle size range of 160 nm to 1,160,000 nm (1.16 mm) in classes, the class used most frequently was the smallest range of 160 to 25,000 nm. (44)

The Fritsch also operated on the principle of light scattering, however it measured scatter angles, particles of the same size scatter light at the same angle. This provided information on particle quantity. An integral computer calculated the particle distribution which was quoted in 31 classes based on grain size and mass components.

The Fritsch required that a stream of liquid be passed through the measuring cell, a minimum of 500 ml of solution being required for each measurement. The Fritsch was capable of measuring the actual solutions coated, although dilution was occasionally necessary with the more concentrated solutions. This compared favourably with the

Malvern where very dilute solutions were examined in a  $1\text{cm}^2$  cell the excessive dilution introducing possible errors. (43)

### 3.2.3 Filtration

Filtration proved to be a necessary part of the manufacturing procedure, and several types of filter were tested. Each manufacturer appears to have their method for classifying filters, thus making them difficult to compare. Two types of filter, were extensively tested, Nippon Roki nominal filters and Pall absolute filters.

Filter ratings were defined by the National Power and Fluid Association (NFPA); nominal filters were defined thus: "an arbitrary micron value assigned by the filter manufacturer, based upon removal of some percentage of all particles of a given size or larger." To measure the rating a 'contaminant' of known particle size and size distribution was introduced upstream of the filter element, the flow downstream of the filter element was then analysed microscopically. A given nominal rating usually means that 98% by weight of a contaminant above a given size was removed.

Absolute filters were defined by the NFPA as the diameter of the largest spherical particle that will pass through a filter under specified test conditions. It was an indication of the largest opening in the filter element.

Pall claim this type of rating can only be assigned to an integrally bonded medium. It should also be noted that the test and the definition were proposed by Dr Pall (Chairman of the board, Pall Corp.). (31)

### 3.2.4 K Bar Coating

The K Hand Coater consists of stainless steel K Bars wound with stainless steel wire and an impression bed of synthetic rubber. Each K bar has wire of specific diameter thus by using bars with different diameter wires a range of wet coat thickness films can be obtained

Tense - part 1.

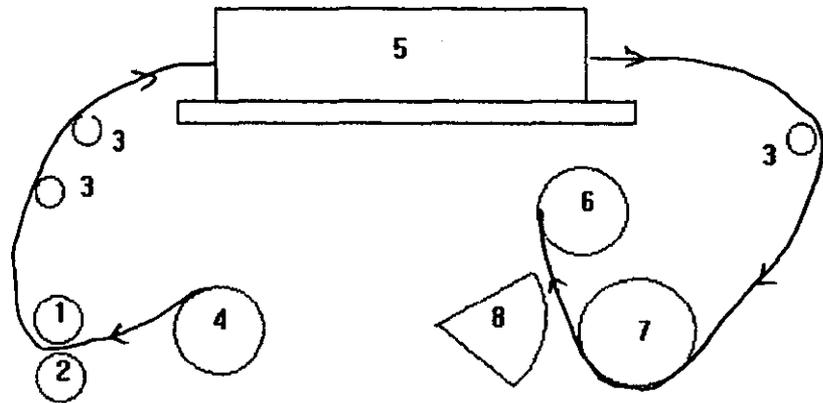
(4  $\mu\text{m}$  to 200  $\mu\text{m}$  or 0.00015 inches to 0.020 inches). A sample of the liquid to be coated <sup>was</sup> placed on a clean, horizontal A4 sheet of Melinex, which <sup>was</sup> affixed to the impression bed, and drawn down at a steady rate using the K bar as a doctor blade, without rolling the bar. The K bar <sup>was</sup> then cleaned. Residual solvent <sup>was</sup> allowed to evaporate from the coated sample before being exposed to UV lights. Tense

### 3.2.5 Bead coating

The solution described is coated onto the web by a process known as bead coating. Melinex web is threaded through the machine as shown. (Fig 8) As applicator roller speed is increased more solution is collected on the roller, thus coating thickness is increased. Film thickness could also be changed to an extent by the speed the web is passed through the coating head, size of the gap between the coating rollers and viscosity of the solution. Solution viscosity and flow behaviour affected the quality of the film achieved. Bead coating has been used for many years on the Brantham site where it has been determined that a Newtonian solution provides the best results. The coated film is then passed through a drying oven where the solvent is driven off, and finally past a UV source where polymerisation is initiated, the cured, coated web is then wound up ready for the next coating.

### 3.2.6 Ultra Violet Curing

UV curing on an A4 size Melinex sheet was performed in the laboratory using a Primarc Minicure. Sheets to be cured are immobilised, by using double sided adhesive tape to attach them to a flat piece of metal (or shuttle). There is also an infra red component in the lamps used and heat is produced, if the web is not securely fastened film crinkling and distortion tends to occur. The shuttle was passed through the Minicure on a drive belt. UV radiation is provided by two high pressure mercury



- 1 = Tension Roller
- 2 = Applicator Roller
- 3 = Idler Rollers
- 4 = Unwind
- 5 = Oven
- 6 = Rewind
- 7 = Chill Rollers
- 8 = U V Source

*How is sheet  
fed?*

Figure 8

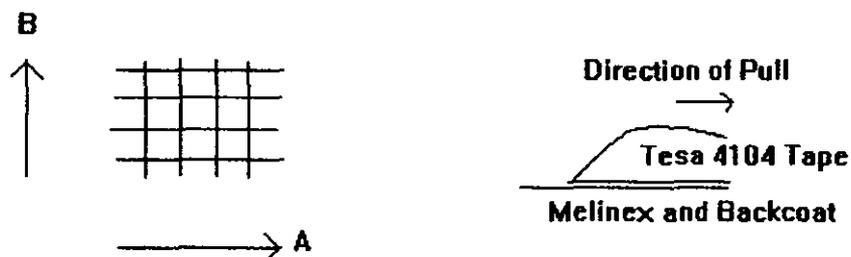
vapour lamps, set in parallel. The dose of radiation, measured in mJ/cm<sup>2</sup> by a 'light bug', is controlled by varying the speed of the drive belt. The light bug measured power output of the lamps and is used to ensure that achieved levels of irradiation are reproducible.

### 3.2.7 Adhesion Testing

Adhesion of a back coat to Melinex is tested with adhesive tape (Tesa Film 4104). The tests took the form of two grids each approximately 2cm<sup>2</sup> they are scored by hand using a scalpel to ensure that the back coat is cut, whilst leaving the PET film intact. The first grid (Fig 9) contains 9 squares, Brantham test method No 207 and the second grid; a laboratory test (Fig 10) contains 100 squares (41).

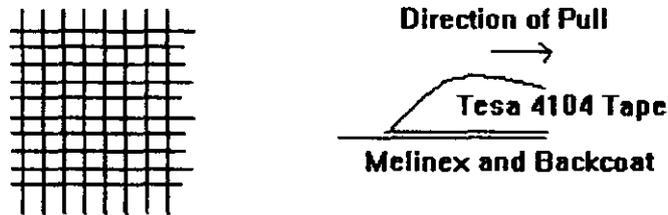
BTM 207 consists of nine squares marked on the coated layer, a piece of Tesa tape is placed over the test area, pressed on with the thumb, then ripped away at 180° with respect to the film (Fig 9). This procedure was repeated a further 7 times, giving a total of 4 pulls in direction (a) and 4 pulls in direction (b). Results are quoted as the number of squares (x) removed in 8 pulls: i.e. x/8. Poorly adhered samples were characterised by the number of pulls (y) to remove all nine squares: i.e. 9/y. Test areas are examined under the optical microscope to assess damage. The test is operator dependent and results are treated as an indication of adhesion rather than as an absolute value.

*reproducibility*



**Figure 9** Nine Pull Adhesion Test (BTM 44)

Using a scalpel, one hundred squares are marked on the back coat (F ig 10). The test is carried out as described above for the 9 square test, with the exception that a single Tesa tape pull is employed.

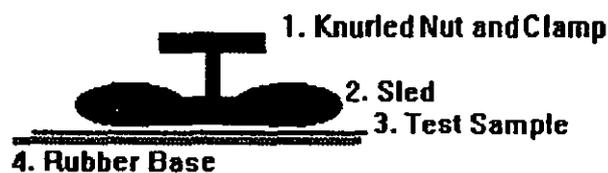


**Figure 10 One Hundred Square Adhesion Test**

### 3.2.8 Friction Testing

Two friction tests have been used in this programme:(42,45)

- a) A rig using a brass sled as in (F ig 11). The sled is pulled at constant speed using an Instron tensometer, with a pulley to convert the normal vertical motion into horizontal motion. (42). Using a 2 inch x 4 inch template for guidance a sample (3) of coated PET is cut and fastened to the sled (2) with the back coat facing away from the sled and secured to the sled using the clamp (1). A 10 inch x 4 inch x 1/8 inch sheet of neoprene rubber (4) is attached to the base plate of the Instron with double sided tape.



**Figure 11 Friction Rig**

The calculation is completed as follows, the weight of the sled, 588 grams:

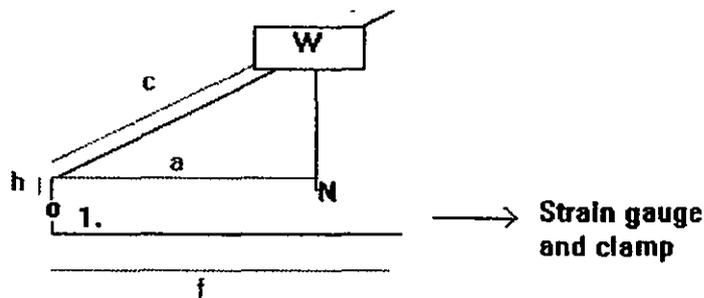
Chart Reading

5.88

The rubber sheet is used to reduce both damage to the test pieces and the effects of accumulated static charge. A sample measuring approximately 3 inches x 6 inches is cut freehand, and is attached, at one end overcoat uppermost, to the rubber with double sided tape. The sled is placed on the sample and the wire attached by a pulley to a load cell on the tensometer. Test speed is 50 mm/min, an X Y plotter records the load; results are calculated as shown and quoted as static and dynamic (or kinetic) friction.

- b) The Dye Diffusion Thermal Transfer (D2T2) (45) rig utilises a motor driven plane and a strain gauge to measure the coefficient of friction. A sample of slit tape (35 mm wide x 50cm long) is attached to the friction plane at one end (1), back coat uppermost with double sided tape, a sample of tape with the overcoat down is attached to the strain gauge using the clamp). The friction plane is driven away from the strain gauge, at constant speed. Load is measured using the strain gauge and recorded on chart paper.

Figure 12 shows a line diagram of the friction rig.



**Figure 12 D2T2 Friction Rig**

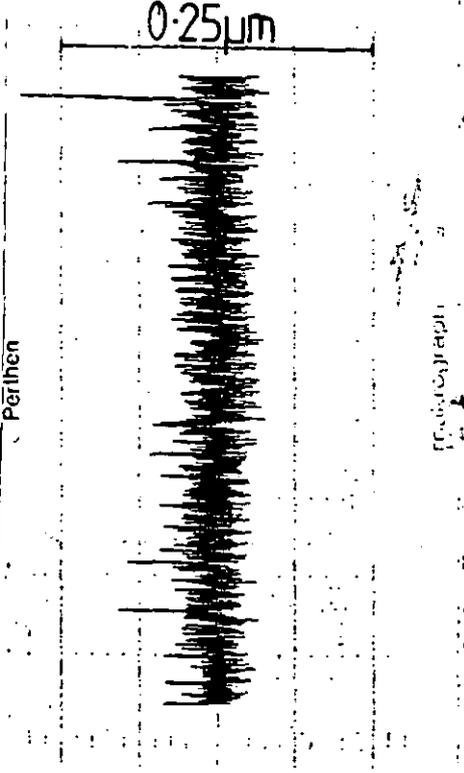
The calculation used to determine the coefficient of friction is detailed below.

Where:  $\mu$  = Coefficient of friction

$F$  = Reading from force gauge. (Kg.f)

$f$  = Horizontal distance between pivot point and rubber roller. (m)

LC M 0.00 MM  
 VER 0.25 YH  
 HOR 500.0 YH



== STATISTICS ==

Feinprüf			milligraph
RA	N=	5	
XS		0.031	YH
X		0.057	YH
MAX		0.095	YH
MIN		0.025	YH
RANG		0.069	YH
RA		0.095	YH
Mehr			
RA		0.086	YH
RA		0.025	YH

Figure 13

a = Horizontal distance between centre of weight and fixed end of cantilever. (m)

c = Distance along cantilever to centre of weight. (m)

h = Vertical distance between pivot point and end of cantilever. (m)

W= Weight of cylinder. (Kg)

N= Force. (N)

### **3.2.9 Surface Profile Measurements**

A Perthen Perthometer SP6 (46) is used to record topographic measurements. (Fig 13) The operational ranges of measurement are 0.1  $\mu\text{m}$  to 1.0  $\mu\text{m}$  and 25  $\mu\text{m}$  to 2.5  $\mu\text{m}$  in the vertical and horizontal planes respectively. Pre-set programmes are used on the Perthometer to obtain the following measurements:

- a) Average peak to trough surface roughness, (RA) is expressed with standard deviation, indicating the degree of protrusion of asperities present in the coating.
- b) Average peak to peak distance (RSM), is indicative of the relative proximity of asperities.
- c) Average magnitude of variance with respect to a machine set base line (RSK). A typical trace annotated with statistics is presented. (Fig 13)

The profile of the trace is always considered alongside the statistical surface roughness.

### **3.2.10 Stiction Testing**

The stiction test was designed to save both time and media. It was necessary to understand how the media would perform under pressure and micro-climatic conditions experienced at various diameters in a full reel. To use a full tape for this type of destructive test is not only time consuming in terms of tape manufacture but also expensive. A

rough costing to produce a single full jumbo, from which 3 tapes can be obtained is more than £2,000.

The stiction test is combined with the friction test to produce figures which will give an indication of several factors regarding the media. Including the unwinding properties after long term storage, print through and any delamination or blocking effects.

The stiction test uses samples of media measuring 10cm x 3.5cm (the tape width). These are placed active side against back coat side, and then inserted at 90° to the reel between wraps of a full tape of raw Melinex (880m x 35mm x 75µm). The tapes are then placed in a humidity cabinet for 3 days at 45°C and 60% RH, the programme used involves a gradual increase in temperature and humidity to and from test conditions, before and after the test, to prevent condensation. Condensation produces artificial defects which would not normally be seen in normal use. The samples are removed and tested on the D2T2 friction rig.

### **3.2.11 Layer to Layer Testing**

The layer to layer test is a standard magnetic media test which was adapted for use with optical tape. The test involves using a mandrel of ½ inch diameter and winding a (1m) length of tape onto it, the tension of the wraps is simulated to equate to the pressures found nearest the reel of a 3480 tape. This is done by hanging a 700g weight on the length of tape. The tape uses a standard overcoat and experimental back coats. When the tape is wound up and secured the mandrels are placed in an environmental cabinet using the same environmental conditions as the stiction test. After being allowed to return to ambient conditions the tape is released and the ease of unwinding from the mandrel noted.

- a) Class 1 pass: the tape comes off the mandrel without any assistance,

- b) Class 2 pass: the mandrel was rotated to aid unwinding of the tape. Further classes involved hanging weights on the tape to assist unwinding, it was deemed that anything worse than a class two pass would not be worth progressing and so the method was not investigated any further.

Tapes were always assessed under the optical microscope for damage.

### **3.2.12 Tape Handling**

Investigation of tape handling and wear was carried out on several drives, 75  $\mu\text{m}$  tape intended for the Creo drive was tested on an Ashe and on a Creo drive;

- a) The Ashe test drive was built to ICI Imagedata specifications, to enable 35mm tape to be handled in a similar manner as on the Creo drive. However, the Ashe could only test handling and wear, and does not possess any read/write capability.
- b) When a Creo drive became available all testing was transferred to it. The Creo drive reads and writes data, which is a more sensitive measure of wear progression than visual examination, (the loss of information by this mechanism is termed data drop out rate). Tapes that appeared to have good wear properties on the Ashe were seen to fail after fewer cycles on the Creo drive.
- c) 23  $\mu\text{m}$  tape for the Laser tape (or LMSI drive) drive was tested on a Lasertape testbed which did not have read/write capability. The Laser tape test bed was a modified magnetic 3480 drive, thus it was possible to examine the performance of a tape on a drive.

### **3.2.13 Image Analysis**

Image analysis was carried out on a Joyce Loebel image analyser attached to an optical microscope, allowing damage to be characterised. Data was quantitatively assessed visible defects could be obtained as: number of defects, defect area, defect breadth, length,

width, geometric error rate and average area of defects. The image analyser was used to monitor increase in wear with handling. The microscope attached to the image analyser could be used for dark or bright field examination. By using either transmitted or reflected light, pinholes in the aluminium layer and debris and surface defects on the dye and overcoat layers were examined separately.

### 3.3 Experimental: 75µm Back Coats

#### 3.3.1 Alumina Hydrate

A 1:1 blend of Ebecryl 220 and Ebecryl 2 10 in 90% solution with sufficient alumina chips added to produce 2% alumina hydrate w.r.t. resin, improved the adhesion of the coating. A 1:1 blend of Ebecryl 220 & 2 10 was used during much of the early filler screening.

Alumina hydrate ex BA Chemicals Ltd was also used, the grade obtained was Bacosol 2A SD which was available as a powder with primary particle size 0.2µm, and was free from cellulose di-acetate and DIOP (47). Again problems were met with suspension, the ICI product Atlox 4991 provided sufficient suspension.

Bacosol was coated at two loadings (30% and 2.5% filler w.r.t. resin); these two loadings were chosen to enable comparison with the high loading Winofil and Aerosil also with the standard alumina chip formulation.

why?

#### 3.3.2 Calcium Carbonate

Advice from ICI Paints, Slough, suggested that the best way to progress would be to make a stable mill base and then formulate serial dilutions to produce the final solutions. A vertical bead mill was available on site and milling was carried out in resin and solvent, using a mixture of 1mm and 2 mm glass beads. Several mill bases containing Winofil SPM ex ICI Chemicals and Polymers were made (40), these were unstable and settled rapidly. Several dispersion aids were tried, but did not improve dispersion or suspension. Winofil SUF gave much better results (Fig 14).

A horizontal bead mill was purchased, and optimum milling conditions were determined for the various mill bases. Few people in ICI had any experience with low solids systems containing fillers, and dilution methods were investigated. For Winofil systems it was found

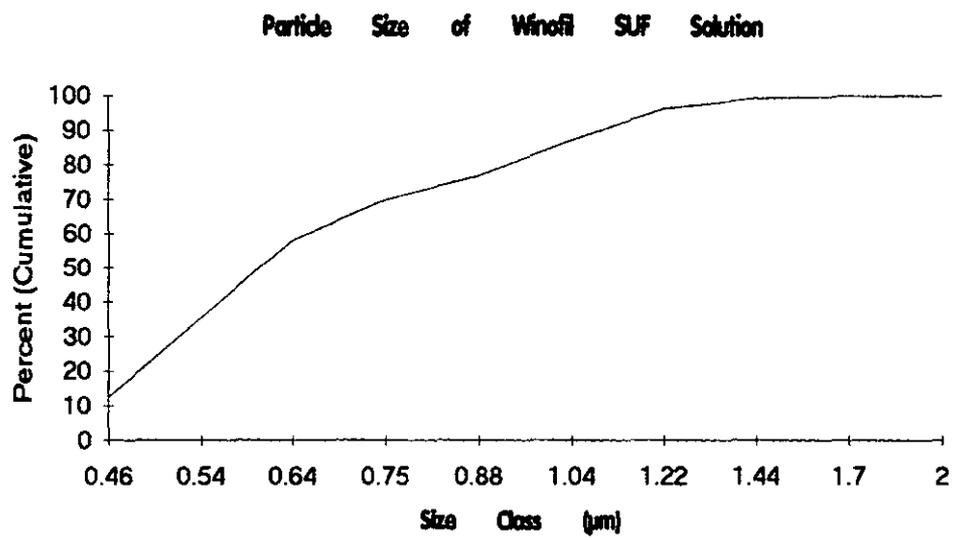


Figure 14

that a serial dilution method was best. The dilution was carried out in several steps. Solutions of resin and solvent were made up 2:1, 1:1 and 1:2 resin : solvent respectively. These were added slowly to the millbase, initially drop wise and gradually in greater quantities, the final dilution used solvent alone. This method of dilution reduced the concentration gradient slowly, preventing the solution agglomerating (or 'throwing out'). The dispersions appeared to remain suspended for around two days, however as they were opaque it was difficult to tell if there was any heavy settlement. To improve the suspension of Winofil SUF dispersion/suspending agents were tried as shown. (Table 3)

**Table 3 Settlement times for Calcium carbonate with and without dispersion aids. (Calculated from % clear liquor.)**

Time (hours)	No <sup>o</sup> dispersion agent	Calgon PT	Fortimax	Cellulose di-acetate
1	1.7	4.5	46	0
2	1.7	5	47	1.7
4	2.6	5	65	2.5
6	2.8	5.5	72	3.5
16	6	7	82	5
24	7	9	86	6
48	10	21	87	7

A second set of experiments was carried out using Lecithin and iso-cetyl stearate as dispersion agents in the milling step, and then adding cellulose di-acetate and poly (vinyl pyrrolidone) (PVP) during the dilution stage. Problems had been come across with cellulose

di-acetate blocking the filter screen on the bead mill; adding the suspending agent after milling solved this. Settlement trials showed the best formulation to be that containing Winofil SUF and cellulose di-acetate alone. Milling trials were carried out to optimise milling time, speed etc. A Magnatek trial with 10%, 20%, and 30% filler w.r.t. resin was carried out using the optimised solution.

### **3.3.3 Aerosil R 972**

Aerosil R972, a hydrophobic fumed silica, primary particle size of 0.014 µm was obtained from Degussa. (48) As manufactured the surface of Aerosil has silanol groups which render the product hydrophilic. Some 75% of the silanol groups are chemically reacted with di-methyl di-chlorosilane, leaving methyl groups on the surface thus making it hydrophobic. Degussa indicated the hydrophobic grade Aerosil R972 may produce a suspension which would give some thixotropic structure aiding suspension.

A sample of hydrophobic fumed silica Aerosil R972, was milled in Ebecryl 5129 and iso-cetyl stearate; the suspension and particle size results were excellent. (Fig 15) Very little development work was required and a formulation was quickly obtained which was used throughout the work programme. A tape was manufactured and identified as JO/114/02, JO indicated the month of manufacture January: 114/02 the run number on the Dixon 2 machine.

### **3.3.4 Alumina hydrate back coat.**

As a control a standard 2% alumina hydrate/Ebecryl 600 back coat was tested; to complete the experiment a high alumina hydrate/Ebecryl 600 tape was manufactured. Alternative forms of alumina hydrate were also tested: these had controllable and repeatable particle sizes, the alternative alumina hydrate was Bacosol 2A SD. (47) It had been intended to carry out a direct comparison of the back coat

Particle Size of Aerosil R972 Solution

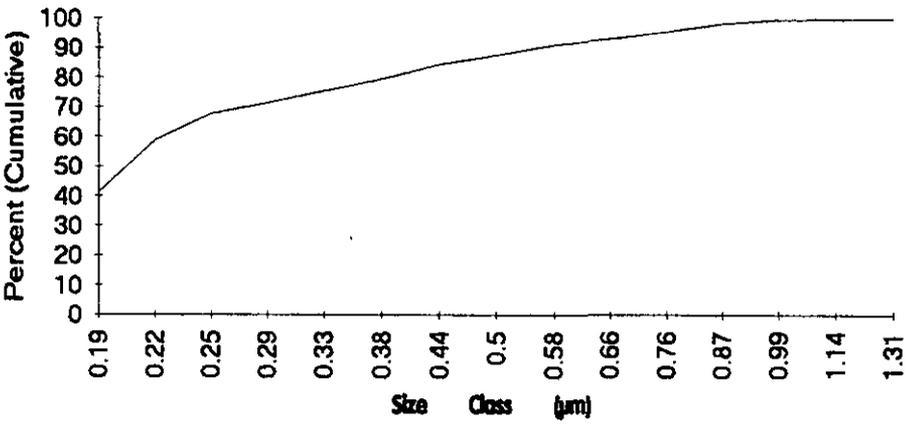


Figure 15

formulations, however, Bacosol 2A SD and Ebecryl 600 proved to be incompatible. The jumbos were produced at the following filler loadings .

Standard alumina hydrate/Ebecryl 600	nominal 2% and 30%
Bacosol 2A SD/ Ebecryl 5 129	2% and 30%

The jumbos were produced during a single trial to minimise any difference between machine runs, humidity, temperature etc. The trial reference was JO/104/A, B, C & D indicating a January production date and run no 104

- A. Control, nominal 2% alumina hydrate
- B. 30% alumina hydrate
- C. 30% Bacosol 2A SD
- D. 2% Bacosol 2A SD

### **3.3.5 Filtration**

The filtration step was in fact more akin to sieving; particles greater than 1µm had to be retained on the filter, whilst passing those smaller through unrestricted. The back coat was approximately 0.5µm thick, therefore the average particle size and size distribution were important. Solvent and filler solutions passed easily through a 1µm PTFE membrane filter. However, when resin was added the filter blocked very quickly. Filtration experts suggested that a depth filter would perform better than a membrane filter.

As an initial trial a Cuno 5µm nominal depth filter was assessed using a calcium carbonate Ebecryl 5 129 dispersion. The largest particles measured in the unfiltered solution were 0.9µm, and after filtration the largest was 0.6µm. Also tested were nominal 1.0µm and 0.8µm Nippon Roki depth filters. These did remove large particles, however, there were concerns about repeatability. Results of challenge tests carried out by Pall showed that a 0.8µm nominal filter was equivalent to one of their 17µm absolute filters. A research project was set up

with Pall Filtration to look at absolute filters, polypropylene 3µm and 5µm filters were assessed. These filters blocked very quickly, it was believed that the particles were too coarse for the filter. However, it was discovered by Pall that the problem was actually caused by the major solvent, (acetone) swelling the filter fibres and thus reducing the size of the pores. The filters were replaced with Nylon 'Profile' filters. A trial was carried with a resin/Aerosil R 972 solution using four grades of absolute filters 20µm, 10µm, 7µm and 5µm (Fig 16), 5µm being the smallest pore size available in the range. From these results it was decided to use the 5µm Pall filters as standard.

### 3.4 Results of high filler loadings on 75µm tape

#### 3.4.1 Resin Recommended

**Table 4. Results of Resin Screening**

Type	Adhesion	Abrasion	Shedding
Ebecryl 754	0	V. poor	Nil
Ebecryl 745	0	Poor	Sheds
Ebecryl 810	0	Poor	Nil
Ebecryl 1360	0	V. poor	Sheds
Ebecryl 600	10	poor	Sheds
Ebecryl 860	0	V. poor	Nil
Ebecryl 4835	0	Good	Nil
Ebecryl 264	15	Poor	Nil
Ebecryl 210	0	Poor	Sheds
Ebecryl 4833	0	OK	Nil
Ebecryl 4858	0	Poor	Nil
Ebecryl 5264	0	Good	Nil
Ebecryl 220	50	Good	Nil
Ebecryl 1290	0	Good	Nil
Ebecryl 5129	0	Good	Nil

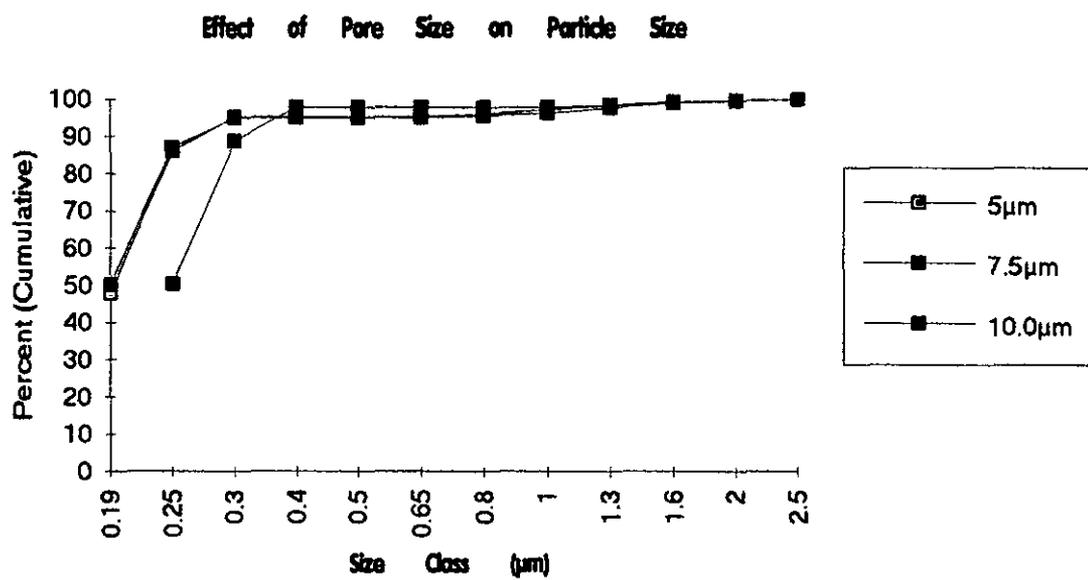


Figure 16

Most of the work was done using Ebecryl 5 129 as the base resin, this material had excellent adhesion to the treated and untreated sides of both 23  $\mu\text{m}$  and 75  $\mu\text{m}$  PET film, good flexibility and did not crack or shed when the web was slit into tape format. Results are shown in Table 4

### 3.4.2 Winofil SUF back coat

**Table 5. Adhesion results for MT 099 A, B and C.**

	<b>MT 099A</b>	<b>MT 099B</b>	<b>MT 099C</b>
<b>9 Square Test</b>	0/9	0/9	0/9
<b>100 Square Test</b>	0% Removed	0% Removed	0% Removed

**Table 6. Friction Results for MT 099 A, B and C, coatings applied using the Magnatek coater.**

	<b>Static Friction vs. Own Overcoat</b>	<b>Dynamic Friction vs. Own Overcoat</b>	<b>Static Friction vs. Standard Overcoat</b>	<b>Dynamic Friction vs. Standard Overcoat</b>
<b>MT 099A</b>	0.206	0.189	0.243	0.19
<b>MT 099 B</b>	0.204	0.162	0.243	0.19
<b>MT 099C</b>	Not determined	Not determined	0.239	0.194

### 3.4.3 Aerosil R 972 back coat.

Table 7. Results for Aerosil R972/Ebecryl 5129 back coat

	JO/114/02
Static Friction vs. standard overcoat	0.249
Dynamic Friction vs. Standard overcoat	0.205
9 square adhesion test	0/9
100 square adhesion test	0% removed

### 3.5 Tape testing

Solutions of Winofil SUF, Bacosol 2A SD, alumina chips and Aerosil were coated on Magnatek 1 (MT 1). The jumbos were then aluminised, dye coated, overcoated and slit into tapes to be tested on the Ashe. Tapes tested on the Ashe were: 10%, 20% and 30% Winofil SUF, 25% Aerosil R 972, 2.5% and 30% Bacosol 2A SD and 2.5% and 30% alumina hydrate chips, w.r.t. resin. All the back coats had friction values in the range 0.15 to 0.4 when tested against their overcoats. Creo had identified this range as being suitable for good winding properties on their drive. The tapes were subjected to 4,000 cycles on the Ashe test drive; friction vs. their overcoats; adhesion, 100 squares and 9 squares and surface roughness, before tape slitting.

#### 3.5.1 Tape Handling and Wear Results: Winofil SUF back coat

Unlike the original back coat, the tapes handled well on the drive at low winding pressures and tensions, without entangling the drive. It was also noted that the tapes did not suffer from static build up during the handling process. Static charge was measured, and found to be

some 10 times lower than the standard Ebecryl 600/ alumina hydrate back-coated tapes.

To complete the test the tapes were wound at low tension, 3.5 N and 100m/min. At these tensions tapes with the old formulation could not be wound without entangling the drive. At 10% and 30% filler loadings no failure was seen. At 20% filler loading failure occurred, this was seen as an anomalous result. Approximately 200m of each tape was available, normally a full reel of 880m would be used.

After 4000 cycles the back coat proved to be sufficiently abrasive to have worn away the overcoat, dye and aluminium layers on MT 099 A, B and C. The damage occurred in patches at the edges of the tape indicating this was where wear was at its most severe. Damage was ranked 10% filler < 20% filler << 30% filler. The results indicated that although the friction values were of the appropriate magnitude to achieve good handling on the Ashe, wear produced during handling was excessive.

### **3.5.2 Tape Handling and Wear Results: Aerosil back coat**

The tape handled well with minimal static build up. Unfortunately the high filler loading caused excessive wear resulting in areas of the active layer being removed. The tape was wound at low tensions, using the same conditions as reported above. The reel did not exhibit failure under any of the wind conditions tested.

### **3.5.3 Tape Handling and Wear Results: Alumina hydrate back coat**

The tapes all handled well, without entangling the drive. High static build up was noted, as had been expected, with the control sample. Static build up did not occur with either of the Bacosol tapes. The high alumina hydrate loading failed when wound at low tensions, 3.5 N and 100m/min.

3.5 is a mixture of sometimes melted to sometimes results

The control, part A gave the best wear performance, with little damage noted. Tapes from parts B & C, as with previous high filler loadings, were badly damaged.

During testing readings of 0.1 KV or less were obtained for Winnofil SUF and Aerosil R972 coatings; standard alumina chips with high and low filler loadings were measured at 0.45 KV and 1.4 KV respectively. Interestingly the high filler loading of Bacosol 2A SD also gave a low static charge reading of 0.1 KV, but unfortunately there was insufficient length of low loading tape to measure the charge build up.

#### 3.5.4. Environmental testing

Tapes, 35 mm wide and 880m long were exposed at 45°C and 60% relative humidity for 3 days, the test cycle included a gradual ramp to achieve these conditions and hence prevent condensation and temperature shocking when the tapes were placed in the chamber, followed by a ramp down to external ambient conditions after the test.

— and

#### 3.5.5 Winnofil SUF

All three tapes exhibited loose reel formation after exposure. There was no large scale blocking as was normally seen with the control formulation. However, all the samples exhibited some degree of microscopic surface damage of the overcoat. The amount of damage was roughly proportional to the filler loading in the back coat, with the highly filled formulations showing the most extensive damage. It was felt that this extensive damage, which has since been termed 'print through', would compromise media performance both for wear resistance and active layer lifetime. It was determined that this microscopic damage did not penetrate the surface but deformed the surface of the tape.

**Table 8 Results from environmental testing MT 099 A, B and C**

MT 099A	MT 099B	MT 099C
Outer wraps of tape loose. Some evidence of particulate transfer. Medium level of microscopic damage.	Very loose reel formation. Fairly high level of microscopic overcoat damage. Unreel characteristics good.	Loose reel formation evidence of print through to layers resulting in high level of microscopic damage.

### 3.5.6. Alumina Hydrate back coat

Bacosol 2A SD was tested, as previous results with the original alumina hydrate showed a tendency for blocking, leading to failure.

#### Part C 30% Bacosol 2A SD

The reel was loose after exposure, however there was no static or blocking. There was a high degree of print through and damage of the active layers.

## 3.6 Discussion: High Filler Loadings on 75µm web

### 3.6.1 Resin

The resin chosen was a hexa-functional urethane acrylate oligomer. Cure measurements indicated that some 75% of the acrylate groups had been converted, as compared with approximately 95% for the di-functional epoxy system. The FTIR technique used, however, was not sufficiently sensitive to determine the degree of crosslinking.

It was believed that this was probably the maximum achievable cure under the conditions used and allowing for the cage. The cage effect tends to occur in multi-functional systems: when a large percentage of

available reaction sites has resulted in crosslinks, producing an increasingly rigid three dimensional network. Within this type of network the free space of chain ends and freedom of chain movement, hence of unreacted double bonds, is decreased. This type of network has unreacted double bonds but initiator is no longer able to move into contact positions thus limiting the degree of polymerisation. The point at which the cage effect prevents any further crosslinking is dependent upon many factors: functionality of the oligomers: equivalent weight per functional group: flexibility of the molecule: viscosity of the system at the start of the reaction and rate of viscosity increase during the reaction. As the film could not be removed using an adhesive tape, it was deemed to be sufficiently cured for the requirements.

however  
hexafun ct -  
→ 12 sites  
do you need  
100% cure  
to produce  
a good  
3D network?

### 3.6.2 Winofil SUF back coat

Calcium carbonate was the first filler examined, obtainable in spherical form with well-characterised particle size and shape. The research group at ICI Runcorn Heath were prepared to enter into a collaborative research programme to investigate requirements for particle size, shape and surface treatment.

Calcium carbonate, however, was not as successful as had been expected. <sup>its</sup> it proved difficult not only to disperse repeatably but also to maintain in suspension. Tape handling showed that the back coats were too abrasive with areas where all the coatings had been removed and a high level of print through. The severity and depth of print-through indicated that the particle size was not as well controlled as had been suggested by the solution particle sizing, or that agglomeration had occurred between particle sizing and coating.

### **3.6.3 Aerosil R 972 back coats**

Production of a mill base with Aerosil R 972 proved repeatable and reliable, with suspensions that were stable for several weeks. Particle size was in the range required.

Highly filled Aerosil R 972 back coats proved to be too abrasive for good handling although the coefficient of friction was that required by the drive manufacturer. Tapes proved to handle well and performed well in environmental tests, with minimal loosening of the reel after testing, indicating that although friction was low enough for handling it was sufficiently high to prevent inter layer slippage.

### **3.6.4 Alumina hydrate back coats**

A loose reel, with good unwind properties, and severe overcoat damage was found with the high Bacosol 2 A SD loading. The high alumina chip loading also gave a loose reel and good unwind properties. The alumina hydrate/cellulose acetate/DIOP filler did not provide the required properties, it was felt that by changing to a filler which was well-characterised (i.e. particle size distribution, particle shape, surface treatment, Moh hardness and was the same from batch to batch) would improve manufacture, handling and wear properties. Bacosol 2A SD was used to determine if DIOP and cellulose acetate, present in the standard filler, were the root cause of dispersion, wear and static problems. Results indicated that to a large extent the problems associated with standard back coats were a result of the substantial quantities of cellulose acetate and DIOP present in the alumina hydrate chips. As neither of these materials have reactive functional groups they are not bound into the polymer and thus free to migrate to the surface of the coating.

It is believed that static build up is a major cause of wear. When static build up discharges against adjacent metal parts of the tape

drive, sparks are observed. When discharges are seen the edges of the tapes are also seen to be badly damaged, and it is believed static discharge is the cause of edge damage, which in turn produces further wear of the active layers.

was surface or not? permanent measured?

Damage caused by discharge tends to be in the form of:

- a) vapourised active layers (aluminium/dye/binder/overcoat) which when cool returns to various parts of the tape and drive as particulate debris.
- b) remains at the edge of the tape as 'chunks' of loosened material which are then free to tumble across the surface of the tape causing further damage.

Visual analysis was carried out on these tapes. Edge damage and bald spots caused by wear were easily seen. — Photos ?

A loose reel, with good unwind properties, and severe overcoat damage was found with the high Bacosol loading. The high alumina chip loading also gave a loose reel and good unwind properties.

When samples used for friction tests were examined it became obvious that the high degree of wear was associated with the back coat. The friction test is a single pull of a back coat sample, under a load of 200 gm, across the overcoat surface, this action can produce deep gouges in the active layers. Tape handling in the drive increases this effect many fold and result in bald spots and severe wear of the active layers.

Results from media with high filler loadings were the first indication that it would not be possible to transfer back coat technology from the magnetic media industry, and that new and innovative techniques would have to be developed for each application. High filler loadings produced an unacceptably high degree of wear and print through, the active layers were worn to such a degree that in places they had been removed to reveal bare PET base.

### 3.7 Low Filler Loadings on 75µm web

#### 3.7.1 Alumina hydrate back coat

As part D from JO/104 had proved to be the best of all the tapes tested it was decided to look at this material in more detail. Particle size of a back coat made using untreated Bacosol 2A SD is shown. (Fig 17)

The filler needed to be easy to disperse and to remain in suspension for a minimum of 36 hours. The coatings were carried out on the Dixon 2 coater. Results are presented below.

**Table 9 Results from Bacosol Trial**

	<b>4% Bacosol 2A SD</b>	<b>6% Bacosol 2A SD</b>
<b>Static Friction</b>	0.38	0.42
<b>9 Square adhesion</b>	0/9	0/9
<b>100 Square adhesion</b>	0% Removed	0% Removed

A decision, based on optical performance, to change from a thick (250nm) overcoat to a thin (40nm) overcoat was made. The required write characteristics required could not be obtained using a thick overcoat. The new overcoat had different properties; friction was considerably higher, wear resistance lower and the new problem of delamination arose: areas where the dye and overcoat had been ripped off the aluminium. It was presumed that uncontrolled particle size and the unnecessary 'junk' introduced with the chips, i.e. DIOP and cellulose acetate were at the root of the problem.

To be able to examine different back coats and the extent of the problem, it was necessary to have a test where these delaminations could be assessed quickly and easily, preferably before the optical layers had been coated. Advice from a magnetic media consultant indicated that the problem was caused by a combination of blocking

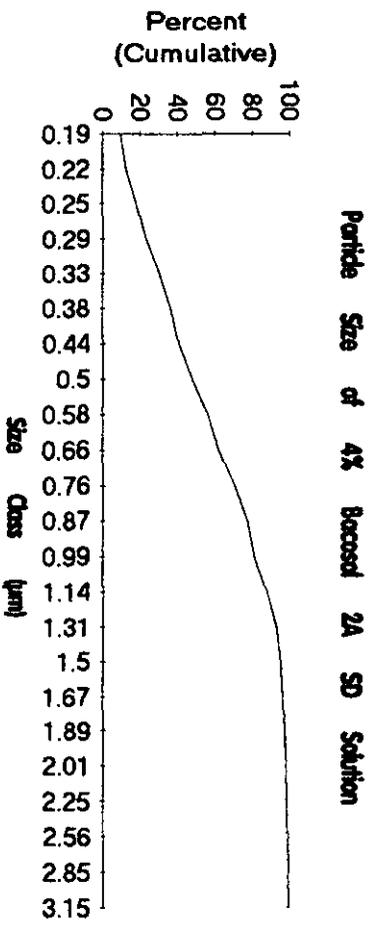


Figure 17

and high static friction, known as 'stiction' (24,25,30,36). Stiction results showed that there were not only vast differences between types of back coat (Fig 18) i.e. Bacosol 2A SD and alumina chips but between different batches of alumina chip back coat.

After the decision to drop high filler loadings, work continued mainly on low loadings of Bacosol 2A SD.

Previous work suggested that 2% filler gave good friction, winding, handling and wear results. Although this was the loading required with thick overcoats an optimised loading needed to be determined for thin overcoats. Four loadings were examined (6.0%, 4.0% and 2.0%, filler w.r.t. resin). These were filtered through a 5µm Pall absolute filter and coated on MT1. Winding handling and stiction trials were done on all the tapes, of which the 4.0% Bacosol 2 A SD gave the best results.

### **3.7.2 Aerosil R972 Back coats**

Milling trials were carried out to try and reduce the particle size of the Aerosil R972 back coat solution. Iso-cetyl stearate was used to aid dispersion. An Ra of 0.029µm +/- 0.02µm was obtained from a 5% filler loading. Although there were still some large particles present as indicated by the occasional blips, (Fig 19) the general background roughness was reduced. It was believed that most of these large particles could be removed by appropriate filtration.

Nylon 'Profile' filters ex Pall were examined. A trial was carried out looking at three different pore sizes, 10µm, 7µm & 5µm. The coating solution used for this trial was a 10% Ebecryl 5 129 solution with Aerosil R972 at 10% w.r.t. resin: a total solids solution of 11%.

Results are presented over:

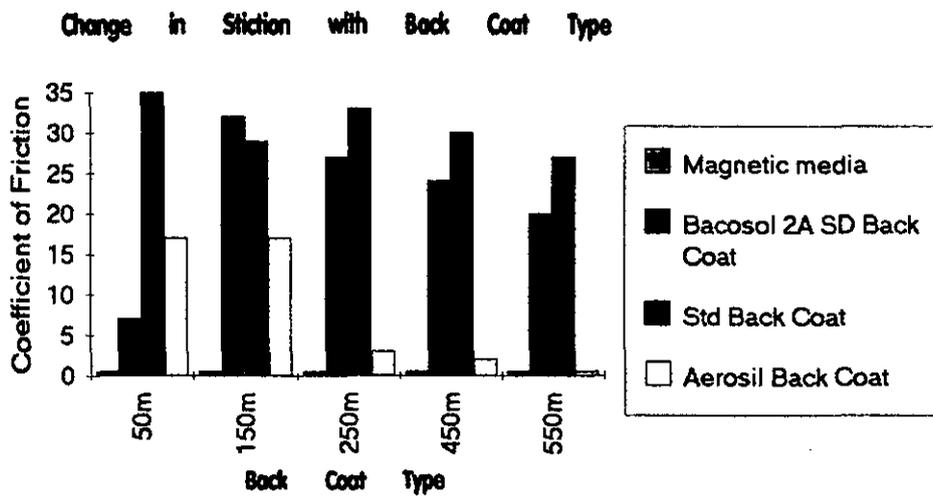


Figure 18

Pertheo  
 LT 0.60 MM  
 LM 4.00 MM  
 LC H 0.80 MM  
 RA 0.029 VM  
 R SM 14 VM  
 R SK 4.38

mikrograph

R PROFIL

LC H 0.80 MM  
 VER 0.25 VM  
 HOR 250.0 VM

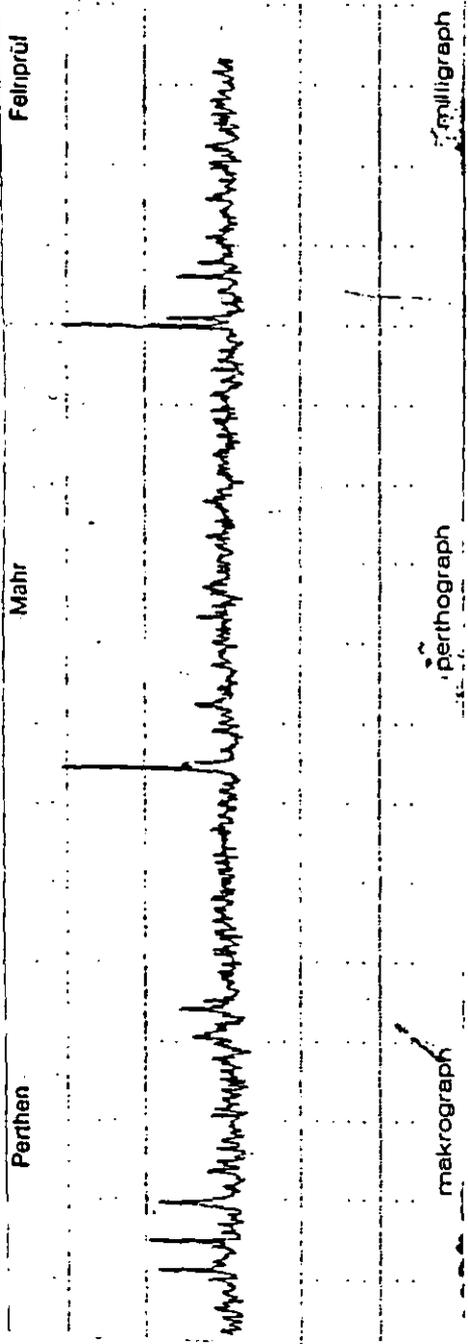


Figure 19

**Table 10 Results for Aerosil R972/Ebecryl 5129 filtration trial.**

	5µm filter	7µm filter	10µm filter
<b>Static Friction</b>	0.283	0.251	0.258
<b>Dynamic Friction</b>	0.22	0.2	0.22
<b>Haze</b>	5.21	4.53	7.64
<b>% Solids * (from ashings)</b>	89	83	85

(\*% Solids of unfiltered material 0.92)

(Fig 20) shows particle size frequencies as measured on a Fritsch particle sizer.

From the results it was decided that we would be able to use the finest filter thus removing most of the larger particles, Pall estimated that their 5µm filter would probably also remove most of the particles larger than 2µm. It was decided to work with this filter and examine finer filters if it became necessary.

### **3.8 Testing: Low loading back coats**

#### **3.8.1 Testing at Creo: Bacosol 2A SD back coats.**

Handling trials were performed at Creo using the worlds first optical tape drive the Creo 1003 Optical Tape Recorder. These trials indicated that although the tape handled far better than the original samples there were still a few problems. A 4% Bacosol 2A SD back coated tape was judged to be suitable to forward to Creo. The coefficient of friction measured was 0.15, discussions with Creo drive engineers had suggested that this was the right order of magnitude. Static build up had been reduced to almost zero, wear tests on the

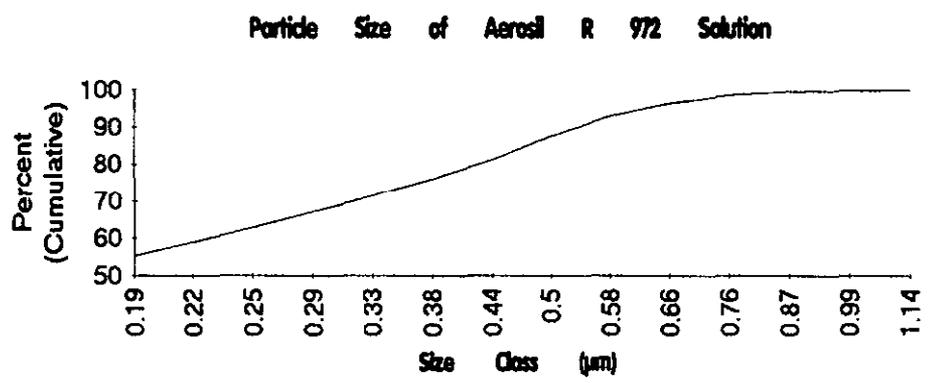


Figure 20

samples and the manufacturing method was well documented and repeatable.

Initial results were very good, the tape wound well and noise previously associated with winding was absent, stiction was not initially apparent. The noise was reported as 'creaking' and was attributed to static charge dissipation and the effects of blocking when the layers separated.

However after 4,000 test cycles, wear began to increase rapidly until failure occurred. It was not initially clear what the mechanisms of wear were and the tape was returned to ICI Imagedata for analysis.

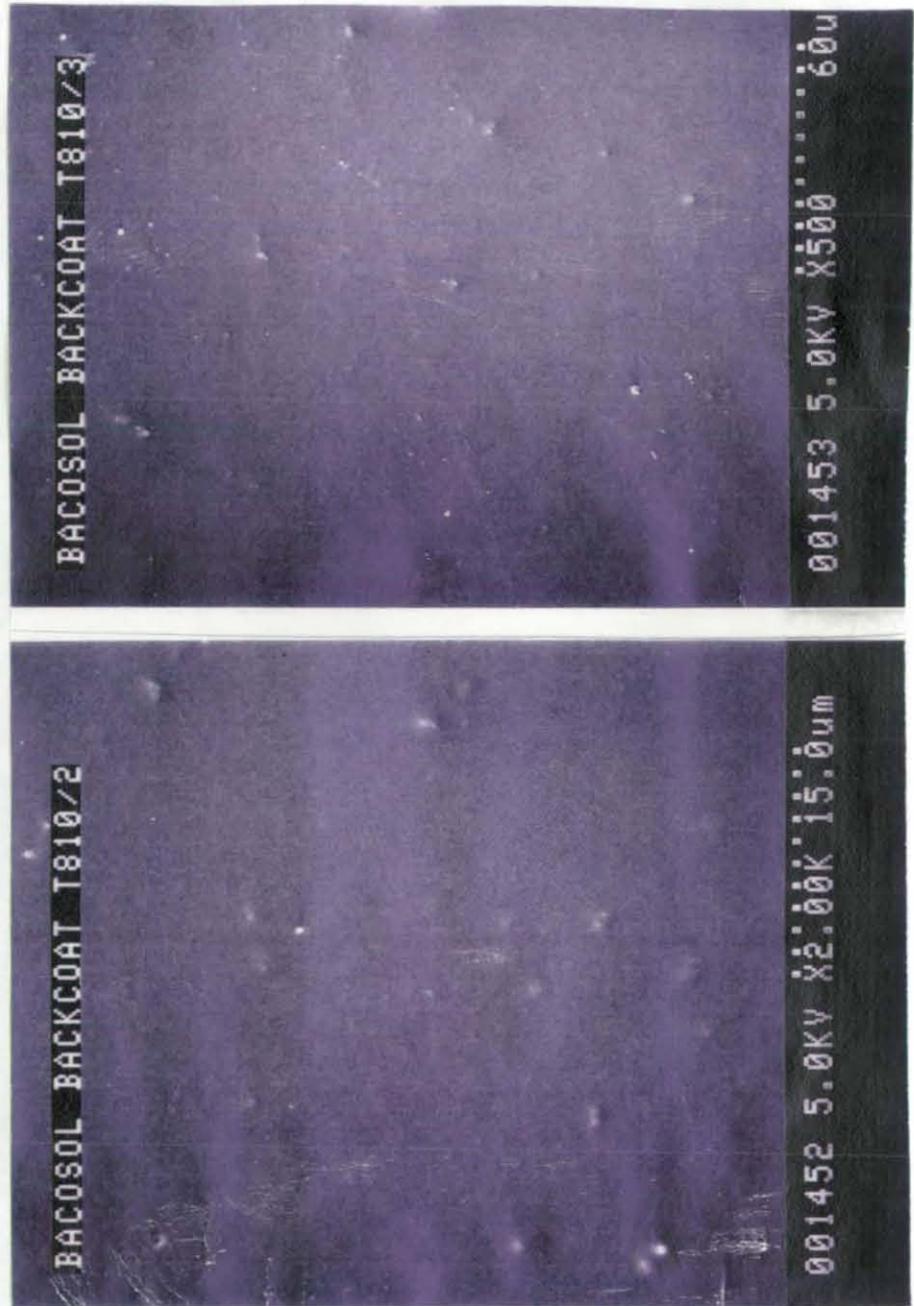
SEMs of tape returned gave an indication what the method of failure was. (Fig 21) As the photomicrograph shows there a few large well spaced 'bearing' points. SEMs of the back coat showed there were areas where filler was absent. Stiction results varied from tape to tape even though the production process was well controlled.

After this failure a business decision was taken to pursue a different back coat route that will not be discussed here. The filled back coat programme was transferred to the Laser tape programme that involved coating onto 23 µm tape.

Comparison of the two low loadings of alumina hydrate showed the Bacosol tape to be the best of the whole group for a secure reel and good unwind. The alumina chip reels showed the normal blocking and static electricity problems. At similar filler loadings as the standard back coats (~2%) a tape with Bacosol 2A SD showed a ten fold reduction in static build up.

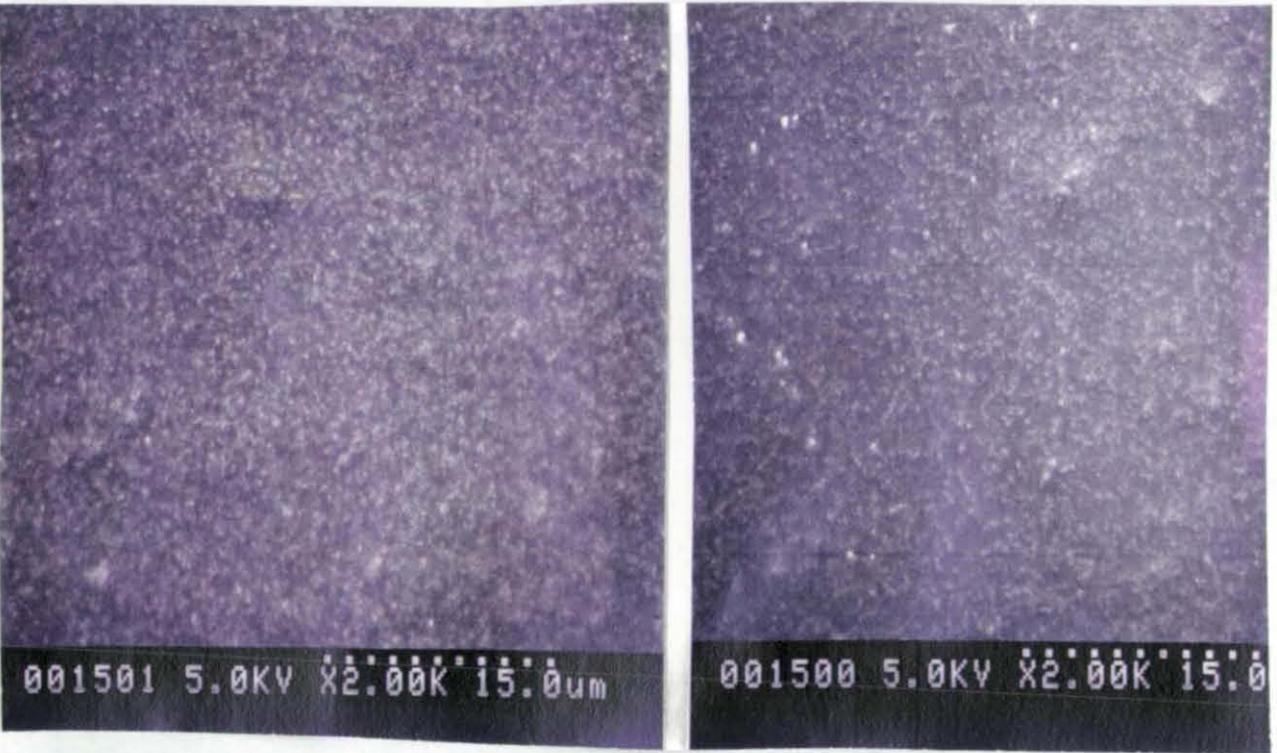
### **3.8.2 Tape Handling and Wear Results: Aerosil R 972 back coats**

Whilst the Bacosol based tapes were being analysed at Creo, tapes with low loadings of fine particulate fumed silica, Aerosil R972 ex Degussa (particle size approximately 14nm) had been produced. As photomicrographs (Fig 22) and Perthometer trace (Fig 19) show, the



SEMs of Bacosol 2A SD back coats

Figure 21



SEMs of Aerosil R 972 back coats

Figure 22

dispersion had a smaller particles that were more closely spaced. Tests to simulate pressures at which micro blocking occurred, without having to produce a full assembly roll, showed that the finer Aerosil back coat was not as susceptible to micro-blocking. Thus for the reasons presented below Aerosil R972 was used in the remainder of the test programme on 75µm web;

- a) Aerosil R 972 coatings, like magnetic media did not appear to suffer from stiction,
- b) SEMs (F ig 22) showed that the Aerosil back coat had a much greater particle frequency in a given area, with few if any 'bald spots' where stiction could occur,
- c) Aerosil R 972 back coats had been shown by particle sizing to be more repeatable than Bacosol 2A SD back coats.
- d) Aerosil R 972 solution stayed in suspension for weeks and was easy to manufacture.

Initial microblocking tests run on back coat samples containing Aerosil R 972 indicated that the back coats were as resistant to micro blocking as was magnetic media, having similar stiction and friction values. (F ig 23)

Further work ~~however~~, suggested that age of the dye/polymer layer affected the microblocking mechanism. Newly coated active layers were more susceptible to microblocking than older samples. (F ig 24)

Two reasons for this were postulated:

- a) Older overcoats had undergone a degree of 'dark cure', i.e. continued to polymerise away from the ultra violet source, leading to an increase in the degree of polymerisation.
- b) Silicone slip agent in the overcoat had time to migrate to the surface, reducing the likelihood of microblocking.

Unfortunately no further work was done, as a business decision halted the filled back coat programme on 75 µm base film and transferred it to 23µm web.

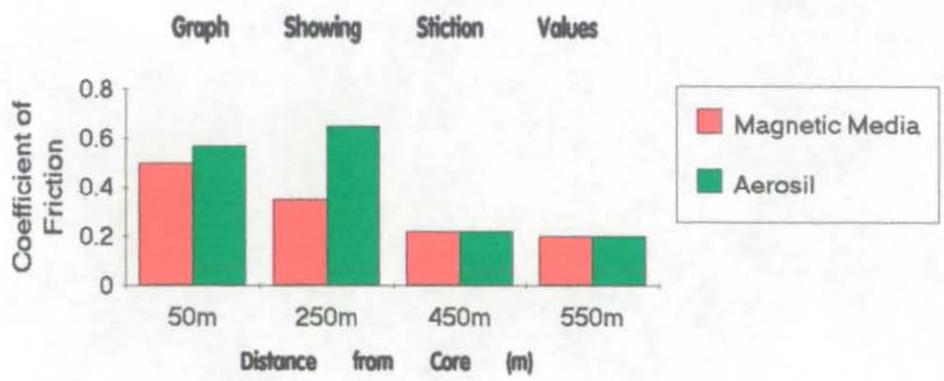


Figure 23

Stiction Values for Aged vs New Back Coats

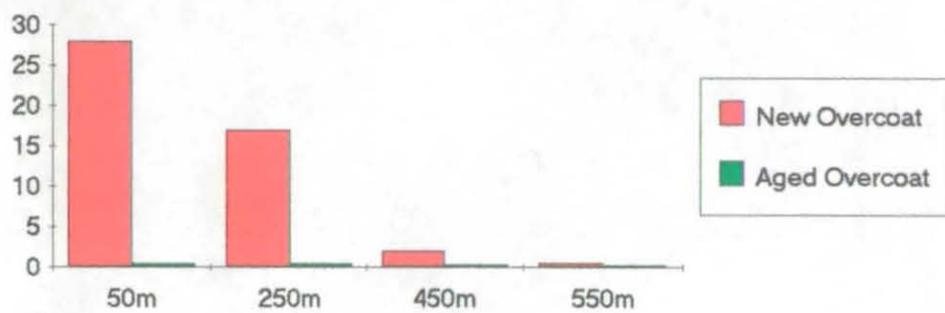


Figure 24

Change of Tape Pressure with Radius

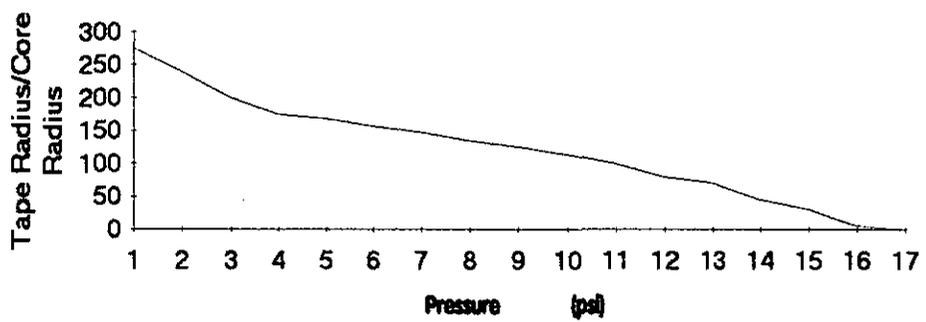
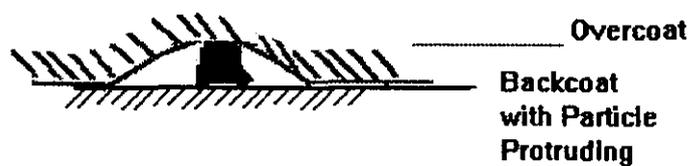


Figure 25

### 3.9 Discussion: Wear Mechanism for Bacosol back coats.

Towards the centre of the tape, pressure exerted by almost 1 kilometre of tape was calculated to be more than 70 psi. (Fig 25)  $5 \text{ bar} = 0.5 \text{ MPa}$

As can be seen from photomicrograph (Fig 21) the Bacosol 2A SD is well dispersed. There are however large unfilled areas around each particle, the photomicrograph shows the particles to be about  $10 \mu\text{m}$  in diameter. These large filler particles acted as 'bearing' points, preventing close contact between overcoat and back coat during tape handling. However, when the tape was in storage, the unfilled areas around the bearing point were able to come into closer contact due to the high pressures the tape was wound. Around any filler particle there was close enough contact between back coat and overcoat for bonds to form. These bonds proved to be stronger than those between overcoat and dye/binder and also between dye/binder and aluminium. Thus when the tape wraps were separated, the active layers remained attached to the back coat and a phenomenon termed microblocking occurred.



**Figure 26 Back coat with protruding particle and overcoat of next layer in intimate contact with back coat around particle**

This was where active layers were transferred to the back coat, two types of failure were seen

- a) Adhesive failure between overcoat and dye/binder, and
- b) Adhesive failure between dye/binder and aluminium.

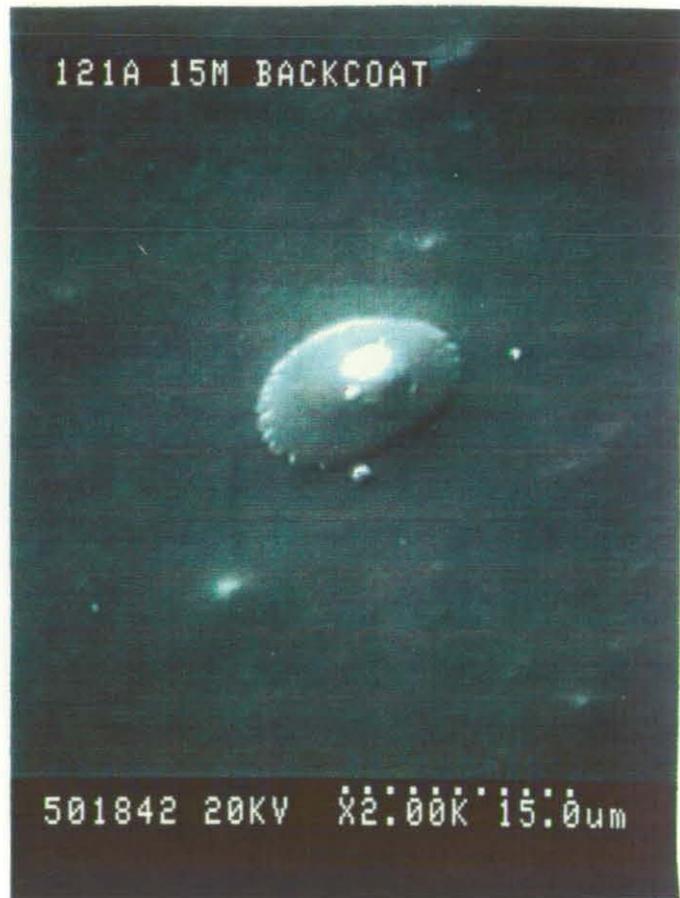
The photomicrograph in figure 27 shows an area of back coat where overcoat has transferred. The bonds between aluminium and PET were stronger than the microblocking forces, having been formed during the high energy sputtering process, failure was not seen between these interfaces.

This mechanism was termed microblocking, because it was of the order of tens of microns, not the large scale blocking seen previously only being visible by microscope analysis.

Examination of photomicrographs and Perthometer traces confirmed that the distance between filler particles in the Bacosol 2A SD back coat was greater than 100 $\mu$ m. Either more filler or a second smaller filler would have to be introduced to provide a background roughness and decrease the distance between particles thus preventing the two surfaces from contacting too closely. Earlier experience showed that highly filled coatings were too abrasive indicated that a smaller particle size filler would be required, either alone or with another larger filler to produce a bimodal coating, such as Bacosol 2A SD with, for example a colloidal silica.

### **3.10 23 $\mu$ m web: Low filler loadings.**

Tapes had been made with the standard alumina hydrate/cellulose acetate/DIOP back coat. However, when cartridge and inserted into the drive for wear assessment they proved impossible to handle. The tape turned over, creased, 'cinched' and stretched, it was thought that these effects were due to the very high coefficient of friction between the back coat and overcoat. The coefficient of friction against a standard overcoat on 23 $\mu$ m tape was found to be more than 1.0. This provided an upper limit for the coefficient of friction. It had been noted that identically formulated coatings tended to produce higher coefficients of friction on thin 23 $\mu$ m base than on thicker 75 $\mu$ m base. Two reasons have been postulated.



SEM showing 'tenting'

Figure 27

- a) 23  $\mu\text{m}$  tape was much more flexible than 75  $\mu\text{m}$  tape as such it was easier for the back coat and overcoat surfaces to come into closer contact over a larger surface area, thus increasing the coefficient of friction.
- b) 23  $\mu\text{m}$  film appeared to be more susceptible to static build up during the coating process, it was thought may increase the coefficient of friction.

Results from trials indicated that fumed silica fillers would produce less aggressive back coats. Photo-micrographs showed that the Aerosil material had a much finer particle size distribution, and that the back coat had a very fine background roughness rather than a few well spaced bearing points. It was decided to put effort into developing a method for manufacturing and coating an appropriate fumed silica back coat.

### **3.10.1 Aerosil back coats.**

Previous results on 75  $\mu\text{m}$  tape proved that high filler loadings were not the route to progress. Thus having chosen Aerosil R 972 formulations were transferred directly from the 75  $\mu\text{m}$  programme to the new 23  $\mu\text{m}$  programme.

The inherent friction of similar coatings was much higher on 23  $\mu\text{m}$  film than on 75  $\mu\text{m}$  film, thus a range of filler loadings had to be investigated. The range chosen was 2%, 5% & 10% filler w.r.t. resin. It became necessary to find another friction rig, as results obtained on the standard rig were greater than the range of the available load cell. A rig described previously as the D2T2 friction rig was used.

A new tape identification system had been introduced and was; A1/013 A, B and C, A1 referring to tapes made in 1991, 013 being the trial request number and A, B & C the individual parts of the trial. A = 10%, B = 5% and C = 2%, Aerosil R 972.

**Table 11** Test results for 2, 5 and 10% Aerosil R 972/  
Ebecryl 5129 coated onto 23µm web.

	A1/013A	A1/013B	A1/013C
<b>9 square adhesion</b>	0/9	0/9	0/9
<b>100 square adhesion</b>	0%	1%	1%
<b>Surface roughness</b> (µm)	0.057±0.031	0.029±0.002	0.032±0.002
<b>D2T2 static friction</b>	0.5	0.46	0.55
<b>D2T2 dynamic friction</b>	0.38	0.34	0.42

Adhesion was not adversely affected by having a large number of small particles dispersed in the resin.

Statistics suggested that surface roughness was low but when the traces were examined the it was seen that there were still several large sharp peaks, presumably corresponding to large poorly dispersed particles. (F ig 22)

### 3.10.2 Carbon black back coats

Carbon black is one of the softest fillers available and could be obtained with primary particles in a size range similar to that of fumed silica. A programme was begun to look at dispersing and suspending carbon black in Ebecryl 5129. It is well known in the paint industry that carbon black is difficult to disperse. The initial formulations were used were ones recommended an MMIS, multi client report (20). However, it was soon discovered that by trying to modify these high pigment volume concentration (PVC) back coats to produce a low PVC back coat, was not going to be successful, for many reasons:

- a) The magnetic media industry, <sup>tend to</sup> use, two pack urethane resin systems thus resin/pigment/dispersion agent interactions are different from acrylate systems.

b) Magnetic back coats are loaded almost to the CPVC to produce as cheap a coating as possible.

c) Previous research has shown that low solids systems were needed. In highly filled systems the quantity of filler and high viscosity serve to suspend filler particles.

d) Magnetic back coats are applied generally by different methods to the ICI bead coating method that requires a low viscosity solution.

The early formulations did not stay in suspension long enough to justify manufacture of tapes. Several experts within ICI were consulted about suspending fillers in very low solids systems and help

was received from the Speciality Chemicals Division at ICI Blakely, *Blakely*

who recommended a combination of their Solsperse 5,000 and Solsperse 24,000 (50) in conjunction with Monarch 1000 ex Cabot.

(49)

A programme of dispersion evaluation resulted in the following formulation, which when carefully diluted produced a back coat that was stable for several days figure 28 shows particle size decrease with milling time.

**Table 12 Formulation for carbon black back coat**

<b>Millbase</b>	<b>%</b>
Ebecryl 5 129	10
Monarch 1000	10
Solsperse 5000	0.5
Solsperse 24000	1.5
Solvent	15
<b>Initiator solution</b>	
Acetone	71.8
Uvecryl P 115	0.6
Irgacure 907	0.6

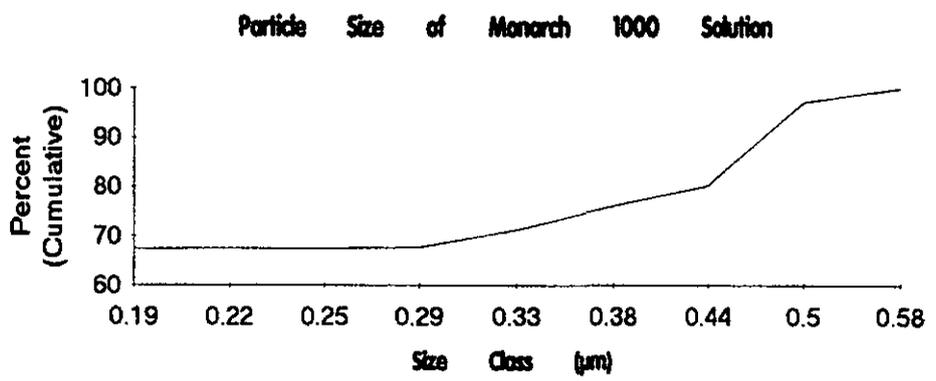


Figure 28

The two Solperse materials were mixed with acetone and resin on a high speed disperser, carbon black was then added and mixed for 20 minutes. The mixture was then transferred to a bead mill where it was milled until the required particle size was achieved. A programme identified milling for 30 minutes at 3000 rpm, which equates to an impeller peripheral tip speed of 5m/second, a recirculation rate of 100 mls/min, 80% of the milling chamber filled with beads, (i.e. 40 mls of beads in the milling chamber), with the milling time being dependent upon formulation. Solution lifetime trials suggested that they would remain stable for several weeks, which was a necessity for production where solutions would be made and stored for some time before use. During the first trial (no 85097) due to uncontrollable circumstances the gap between coating rollers increased. This resulted in a thicker coating than had been intended. A second effect of the thicker coating was a phenomenon termed "needle lines". This was a coating defect caused by fast applicator roller speed. The effect of needle lines can be seen in figure 32.

Carbon  
lines

The next trial (85 126) examined the effect of milling in different solvents, cyclohexanone and toluene. As figure 29 shows there were still needle lines present, although in the thinner coating they were not visible to the eye.

The next stage was to coat a carbon black back coat at a lower filler loading with improved particle size distribution. Trial 85 149 was a two part trial which investigated different milling methods:

- a) Milling in solvent and dispersing agent only then stabilising the millbase with resin.
- b) Milling in resin, solvent and dispersing agent, and again stabilising with further resin.

Both parts of this trial had 5% carbon black w.r.t. resin.

The coating had good visual quality and good adhesion although perthometer traces indicated that there were still a number of larger particles present.

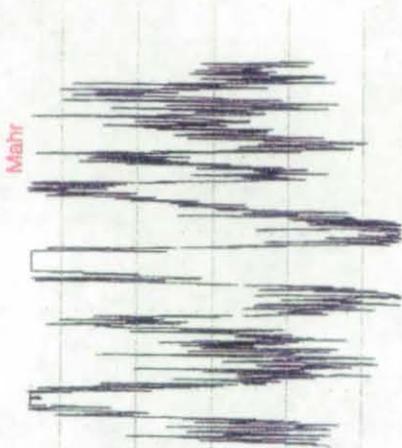
RA 0.651 YM

Feingriff R SK 0.86

R SM 219 YM

milligraph

R PROFIL  
LC M 2.50 MM  
VER 0.50 YM  
HOR 2500.0 YM



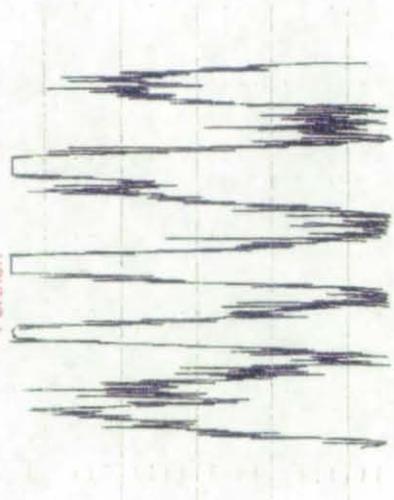
Mahr

perthograph

Mahr

R PROFIL  
LC M 2.50 MM  
VER 0.50 YM  
HOR 2500.0 YM

Perthen



perthogi

makiograph

RA 0.789 YM



Figure 29

### 3.11 Testing: Low filler loading on 23µm tapes

#### 3.11.1 Tape Handling and Wear Results: Aerosil R 972 Tapes.

A standard 3480 magnetic tape drive had been modified by the American company, Laser Tape Inc. to enable optical tape to be handled and assessed. This involved changing the orientation of several of the capstan and idler rollers in the drive to ensure that the active side of the tape was not presented to a roller surface.

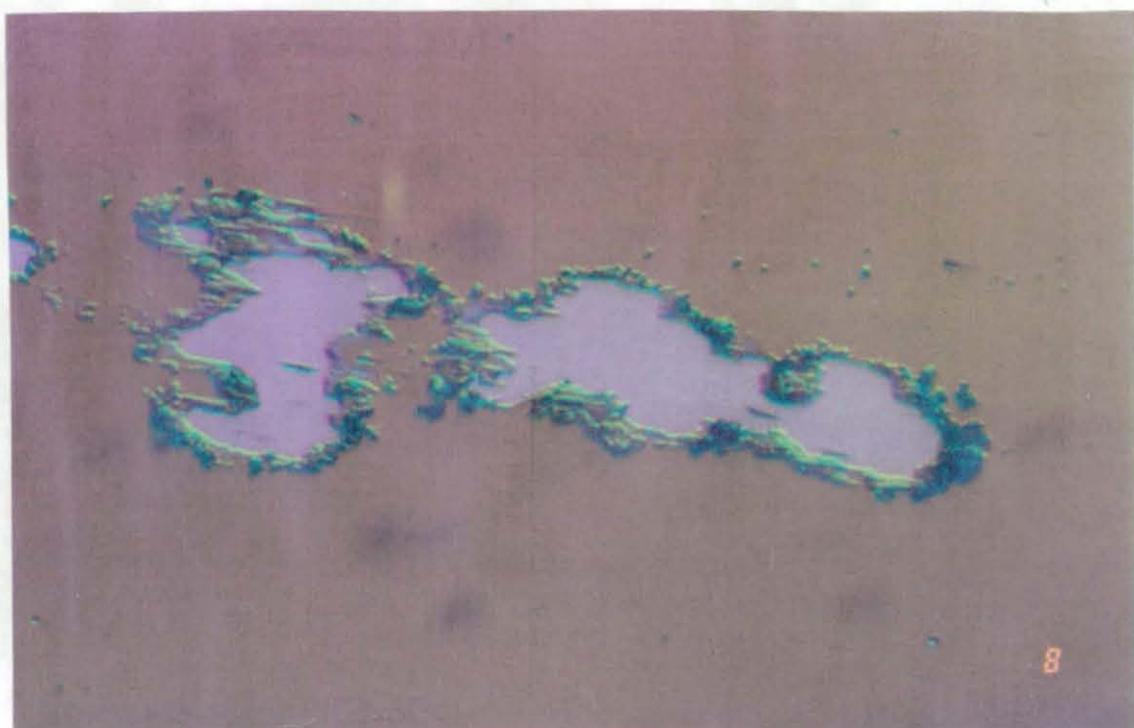
A series of trials was carried out to produce 1/2 inch tapes for testing on the LMSI drive. The trial reference system had been again changed, to comply with BSI requirements, numbers were assigned from 85000. Trial no 85 12 1 investigated 5% & 7% Aerosil R972 w.r.t. resin, trial no 85 130 had an Aerosil loading of 10%.

Results from layer to layer test

- a) Part A 5% Aerosil R972. The test visually gave a class 1 pass, however when inner wraps of tape was examined under the microscope it could be seen that delamination or microblocking had occurred. (Fig 30)

**Table 13 Results from trial 85121 and 85130.**

<b>Aerosil loadings</b>			
	<b>8512A (5%)</b>	<b>85121B (7%)</b>	<b>8513 C (10%)</b>
<b>9 square adhesion</b>	0/9	0/9	0/9
<b>Film thickness</b>	0.6µm	0.6µm	0.5µm
<b>Static friction</b>	0.26	0.29	0.25
<b>Layer to layer test</b>	Class 1	Class 2	Not done
<b>Surface Roughness (Ra) in µm</b>	0.016±0.003	0.011±0.001	0.88±0.22



Micro-blocking

Figure 30

b) Part B 7% Aerosil R972. The outer wraps passed class 1 however the inner wraps where pressure was greater was a class 2 pass. When examined under the microscope microblocking was again evident.

As read/write optics were not available for the tape drive, the only method available for monitoring wear was to visually inspect lengths of tape under a microscope for scratches, defects and debris not present before wearing.

Aerosil R972 tapes were all tested for 4000 cycles (equivalent to 8000 head passes i.e. one unwinding and one rewinding) Tapes produced previously with an alumina hydrate/Ebecryl 600 back coat had failed after 1 cycle.

Visual analysis of types of scratch and damage included:

- a) Areas where overcoat and dye or just overcoat appeared to have been 'ploughed up' and the debris then deposited. (F ig 31b)
- b) Characteristic scratches. (F ig 31b)
- c) Slitting damage tumbling across surface of tape. (F ig 31c)
- d) Edge damage caused by wearing of the cracks induced during the slitting process. (F ig 31a)
- e) Gouges were thought to have been caused by loose particulates, dust, loose filler, edge and general debris, etc.

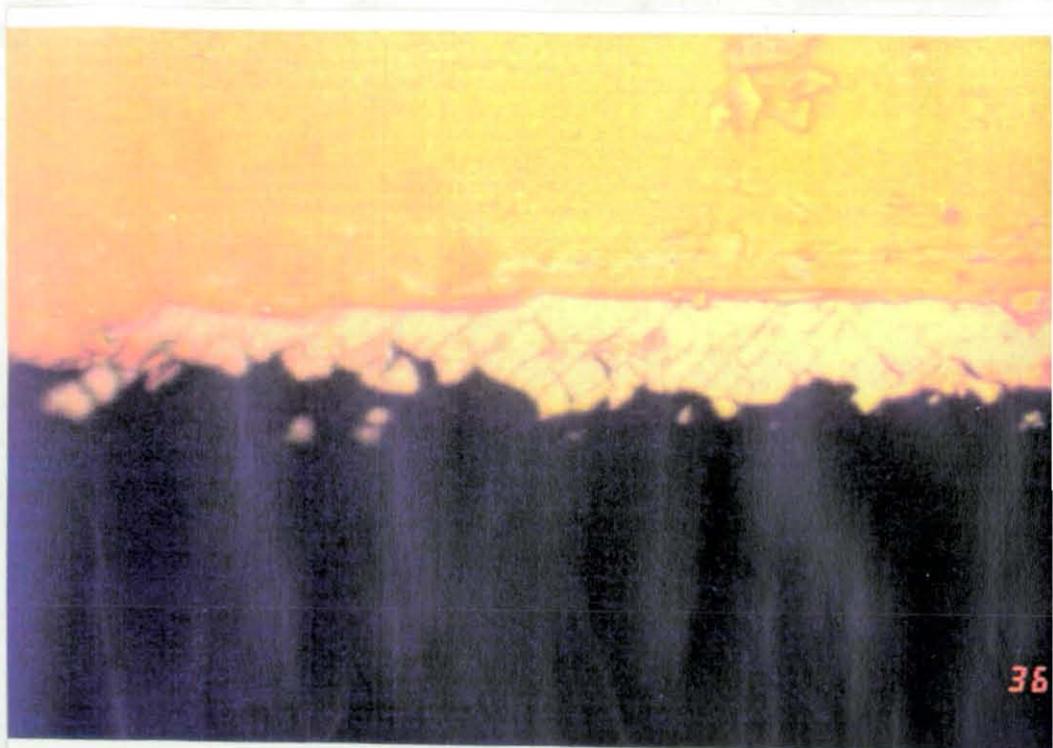
The tape edges were denoted reference and top edged respectively.

The reference edge was the edge of the tape that was downmost in the cartridge, this edge sat against a solid flange. The top edge was presented to a series of flexible guide fingers. When tapes were cartridge'd up it was ensured that the same slit edge was presented to the reference flange each time.

Due to the orientation of the cutting blades on the slitting apparatus one of the tape edges was supported by the bottom slitting blade and one tape edge remained free flying, unsupported. It had been previously noted that the supported edge tended to exhibit less edge damage. This phenomenon was more noticeable in worn tapes the supported edge displaying less edge damage.

'Edge  
damage'

Figure 31 A



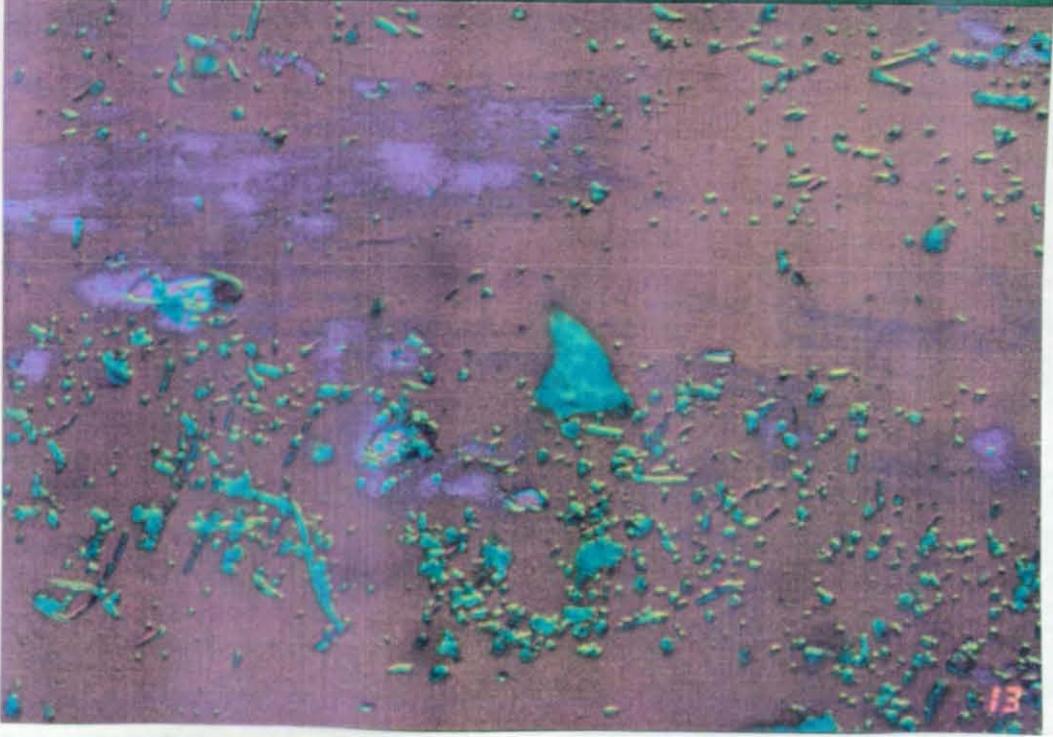
'Ploughed  
areas'

Figure 31 B



Damage  
caused by  
debris

Figure 31 C

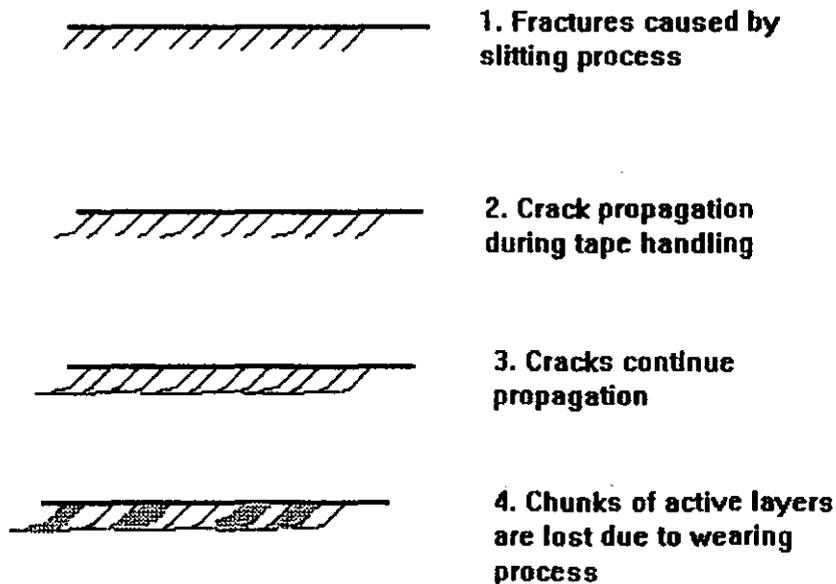


Edge damage was visible as a series of fractures at between 45° and 60° to the edge, which were parallel to each other, the fractures then propagated gradually until in the worst cases they were aligned parallel to the tape edge.

Where fractures had joined up parallel to the tape edge, the inset area of active layer was susceptible to removal during wearing, then being free to tumble across tape surface and cause much of the damage. Slitting optimisation was felt to be one method of improving edge quality (Fig 32). However, it is not the intention of this report to discuss slitting techniques.

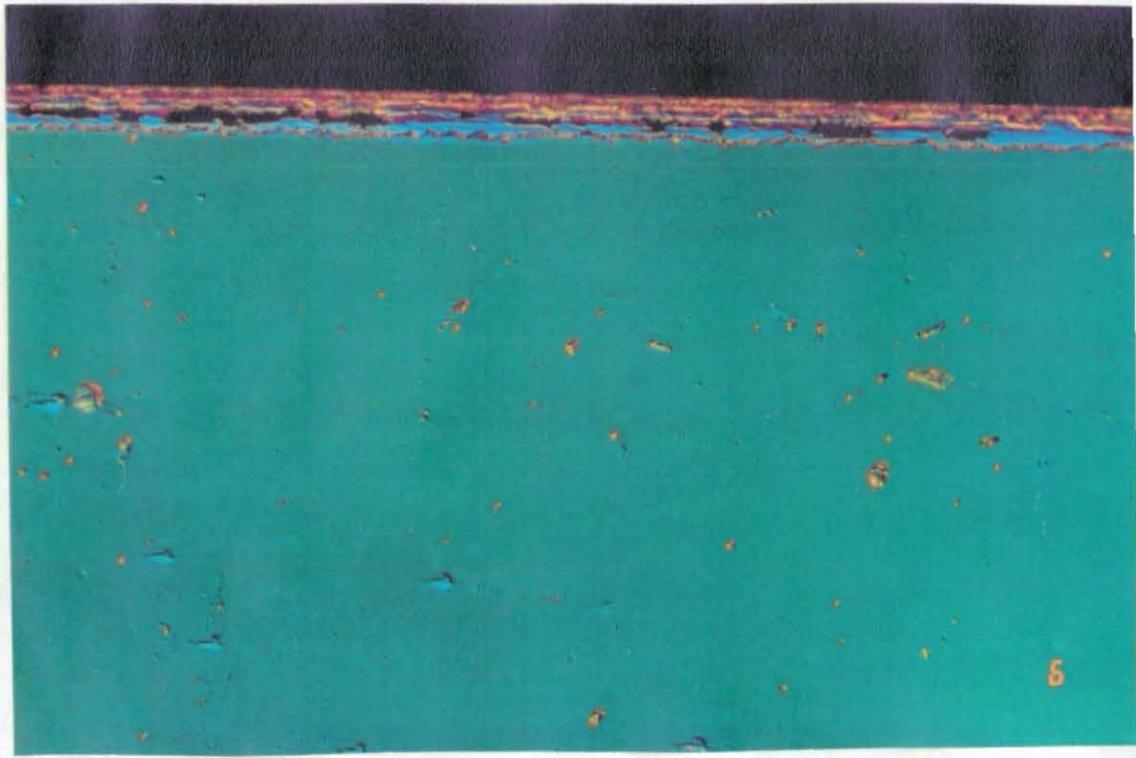
Previous assessment by other workers (52) had concluded that magnetic media, albeit to a lesser extent, suffered from cracks at the edges of the PET base very similar to those seen in optical tape and recorded as edge damage. Most magnetic tapes consist of a single polymer layer, coated on each side of PET base with good adhesion. Tape samples with 7% Aerosil back coat were seen to have the least amount of wear and edge damage. However, the degree of wear on the tape was not acceptable for commercial use, figures 33 & 34 show the damage to the active layers after 4000 cycles.

*what do they show?*



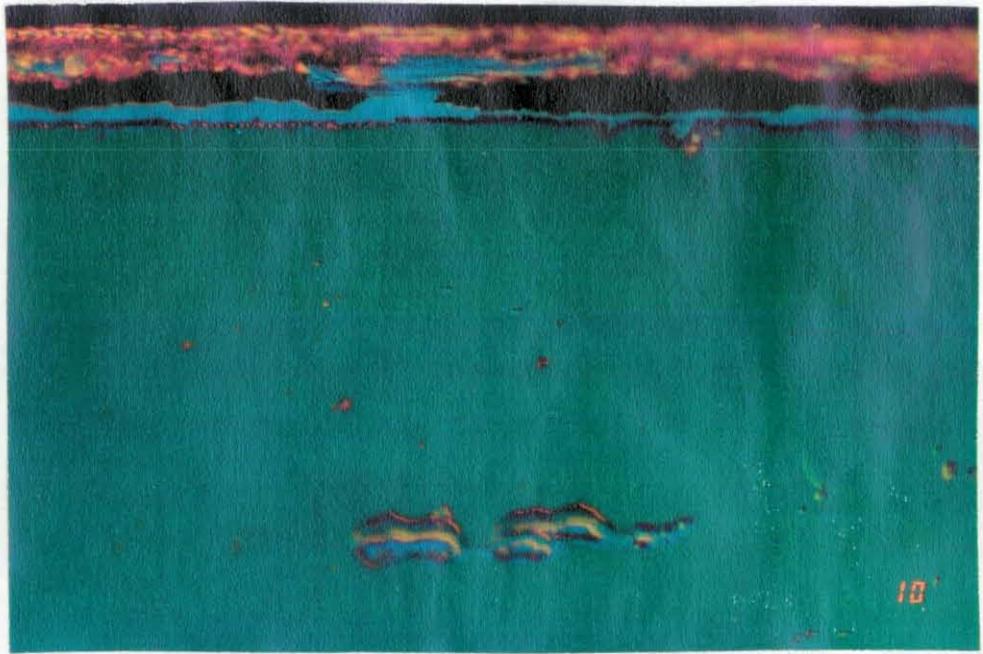
**Figure 32**

**Edge damage**



Damage caused by wearing tape for 4,000 cycles

Figure 33



Damage caused by wearing tape for 4,000 cycles

Figure 34

Loose material from edge fractures was not the sole cause of high levels of wear, filler particles protruding from the back coat caused much of it as shown by gouges produced in the active layers during friction testing. Although in reducing the damage to this level was quite an achievement, the product was required to survive 40,000 cycles, thus the back coat required further re-formulation.

### 3.11.2 Tape Handling and Wear Results: Monarch 1000 back coats

**Table 13 Results from Trial number 85126 A & B**

	85126 A	85126 B
<b>9 square adhesion</b>	0/9	0/9
<b>Film thickness</b>	0.6 µm	0.5 µm
<b>Layer to layer test</b>	Class 1	Class 1
<b>Static friction</b>	0.25	0.27
<b>Surface roughness (Ra) in µm</b>	0.146 ± 0.019	0.082 ± 0.014

Tapes were produced with 10% Monarch 1000 w.r.t. resin. Results before handling were extremely good with nothing removed in the adhesion or the stiction tests. Surface roughness was high, however, it was decided to wear the tapes and determine the effect of high surface roughness with a soft filler.

Handling and winding were excellent. After wearing the tapes were examined, the active layers had black streaks on them, when these marks were removed using iso propyl alcohol, to prevent damage of the active layers, there was no evidence of damage to the tapes.

Elemental analysis of the black smears upon the active side produced no results, carbon not being detected by the elemental analysis process available, it is presumed that the smears were carbon black.

Elemental analysis would be expected to show sulphur from the

*2 so why  
no C. ?  
- XDR  
couldn't  
remove  
to C. !*

initiators if resin was present and normally other elements if the debris was extraneous dust. All wear tests were carried out in a clean room environment, to minimise external contamination.

As the surface damage was minimal it seemed reasonable to suggest that the smears were not accompanied by large chunks of resinous debris. The particle size distribution of trial 85 126 was quite high, with a large percentage of particles in excess of 1µm.

Further carbon black tapes were produced, 85 149 A & B with 5% filler loading w.r.t. resin. Two coatings and were made using the same formulae, the difference being milling conditions. Both solutions had a pre-coating particle size distribution of less than 0.5µm with 70% of that being smaller than 0.3µm. Milling conditions appeared to make little difference to the particle size, indicating that the formulation was robust.

**Table 14 Results for trial 85149 A & B**

	<b>85149 A</b>	<b>85149 B</b>
<b>Adhesion 9 square</b>	0/9	0/9
<b>Film thickness µm</b>	0.5	0.5
<b>Coefficient of friction</b>	0.15	0.13
<b>Layer to Layer test</b>	Class 1	Class 1
<b>Surface roughness (µm)</b>	0.32 ± 0.002	0.029 ± 0.002

Surface roughness and friction figures were similar for both coatings. Layer to layer tests gave a class one pass indicating that micro-blocking should not be a problem. Wear assessment of the tapes showed them to be superior to the Aerosil tapes 85 12 1 A & B, with less visible damage after 4,000 wear cycles. Assessment of the tapes showed that as with the Aerosil tapes edge damage was the major feature with debris causing problems.

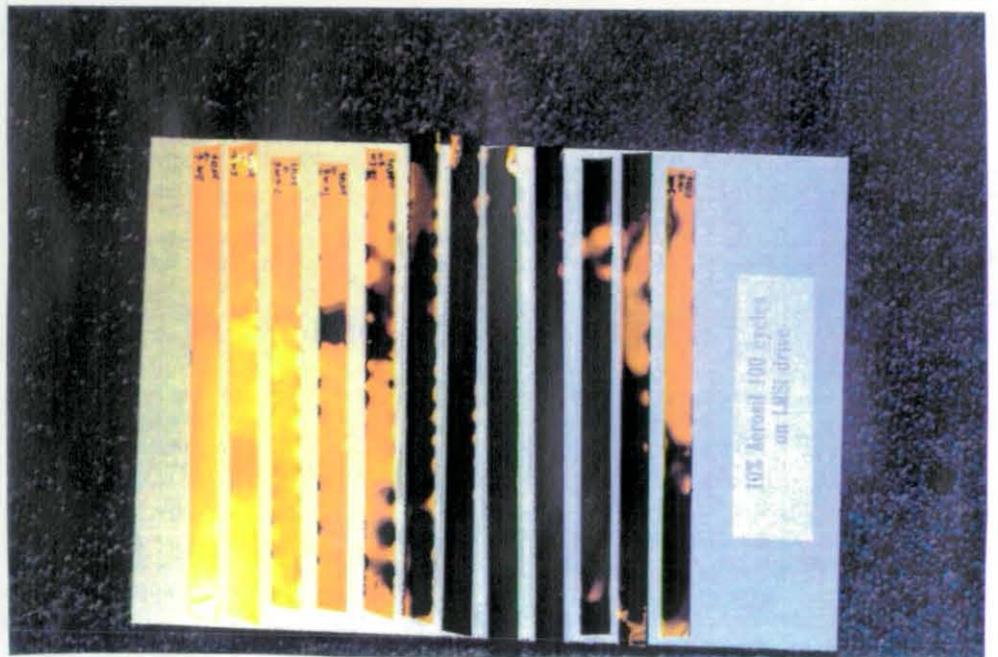
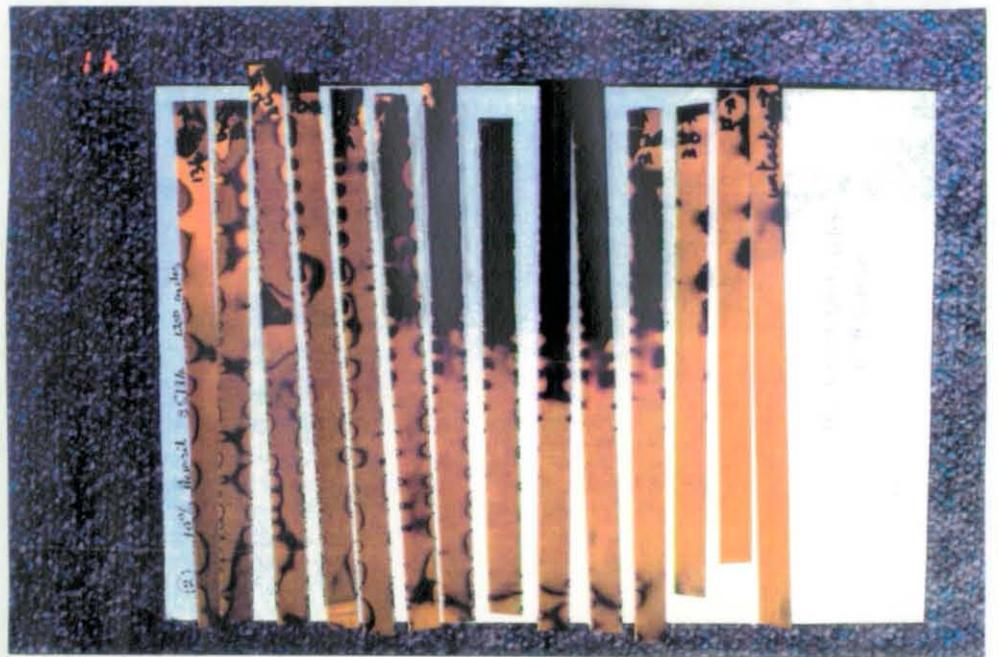
### 3.12 Discussion: 23 $\mu$ m Tapes

#### 3.12.1 Aerosil R972 Tapes

Some edge damage was apparent before microscope examination, the tapes had 'scalped'. (Fig 35) Scalping made examination difficult, as the microscope focus had to be adjusted to allow for the change in height position of the scalped edges. Scalping was caused when the edges of the tape were stretched becoming 'frilled'. Edge damage was thought to be a product of the guidance system which consisted of sprung metal fingers. On the top edge of the tape and a solid plate at the bottom edge, which ensured that the tape remained in vertical alignment to maintain head/tape orientation, during winding, reading and writing. If the tapes were allowed to move in the vertical direction tracks of information, written in a serpentine fashion along the length of the tape, would not remain steady in front of the laser, leading to read/write errors. As tapes touched these guides, force was applied to the web and differential stretching occurred resulting in scalped edges. Scalping tended to be worst on the reference edge (bottom edge) which was solid, allowing no tape movement.

Magnetic media edge guides were used. IBM had found that their sprung steel guides were not sufficiently resistant to abrasion by magnetic media, and had to be replaced frequently. Ceramic tipped edge guides which were much more abrasion resistant were developed to replace the steel guides, it was possible that these guides were too harsh for optical media. (51)

Scalping was seen to be slightly worse on the 5% Aerosil R972 filled tape. Measured friction and surface roughness values were similar, for both coatings it was thought that the difference in degree of scalping may have been caused by machine variations rather than any formulation differences, as the drive was an early unoptimised prototype.



Scalloped Tapes

Figure 35

Although the wind quality was the best thus far the amount of damage after only 4,000 cycles was considered to be significant and too high for market purposes. As there was no optical head to confirm this with bit error rates, the standard method on optical tape. Since there were no standards for comparison of wear after a set number of cycles or a recognised failure point it was felt that the degree of damage over 40,000 cycles continued to increase at the rate found over 4,000 cycles it would be so severe that the tapes would be unacceptable.

### **3.12.2 Damage to Aerosil R 972 tapes**

Photomicrographs showed the type of damage occurring: (Figs 31a, 31b, 31c, 33, 34, 34a & 34b)

- Tumbling debris; source believed to be a mixture of debris from poor quality edge slitting, (Fig 31a), from damage produced along the edges during wearing, (Fig 33a) large agglomerates removed from the back coat surface, debris from other damaged areas, dust and external debris. (Fig 31c)
- Tumbling debris produced patterns described as horse shoes where debris contacted, gouged and tumbled again. Some of these areas were severe enough to break the surface of the active layers and produce further debris. (Fig 31b) In other areas the active layer was merely deformed, it was likely that these deformations would eventually wear producing more debris. Written data would almost certainly be lost from these areas. Photomicrograph (Fig 31c) shows a mixture of tumbling debris, gouged debris and debris sitting on the surface.
- Sharp debris; source edge debris, photomicrograph (Figs 33 & 34) show fractures in the active layers produced during the slitting process that have crack propagated. A proposed mechanism for edge debris production is presented. (Fig 32)

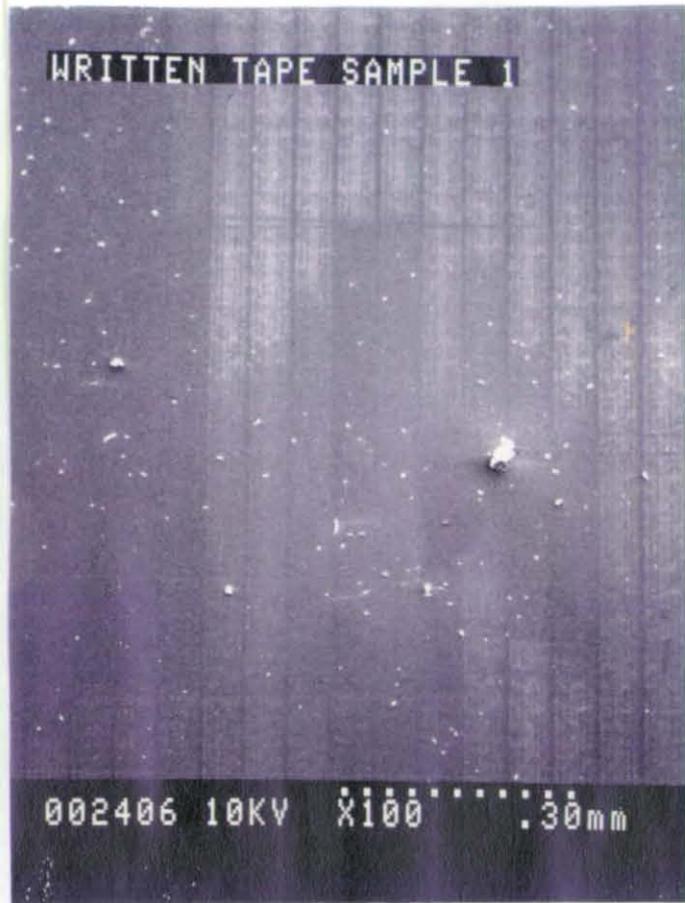
- These sharp flakes of debris are free to either gouge into the surface or tumble across the surface and produce more generally finer debris which again either tumbles, gouges or sits on the surface, all of which increase wear. Photomicrographs (Fig 36a & 36b) show at higher magnification, gouges, debris and rub outs on 75 $\mu$ m media with a filled back coat, the wear is the same as that seen on 23 $\mu$ m media. Dye and overcoat can be seen piled up around the edges of the area, with some surface debris behind the damaged area. The photomicrograph shows that debris has ploughed into the surface, removing the coatings and then piling some up at the side and releasing some which will cause further damage. It can be seen that the damage started from the left and moved right leaving further debris in its wake, much as a plough through a field.
- Edge damage; appears to be more severe on the top edge as opposed to scalloping which is worst on the bottom edge, this edge is guided in the vertical plane by sprung metal edge guiders with a ceramic tip. (52) It is believed to be the bouncing action of these edge guiders that induced the excessive edge damage. It seems reasonable to suggest by optimising the slitting process and improving the adhesion of the various active layers to each other that either onset of edge damage would be delayed or prevented entirely, thus improving the lifetime of the media.

### **3.12.3 Discussion: Monarch 1000 back coats.**

A softer filler, carbon black was introduced, this had not been investigated previously because of possible contamination in the class 10,000 cleanroom where web manufacture took place, as it required a complete set of dedicated carbon black containers and coating aids. However, this required filtration to be carried out distant from the

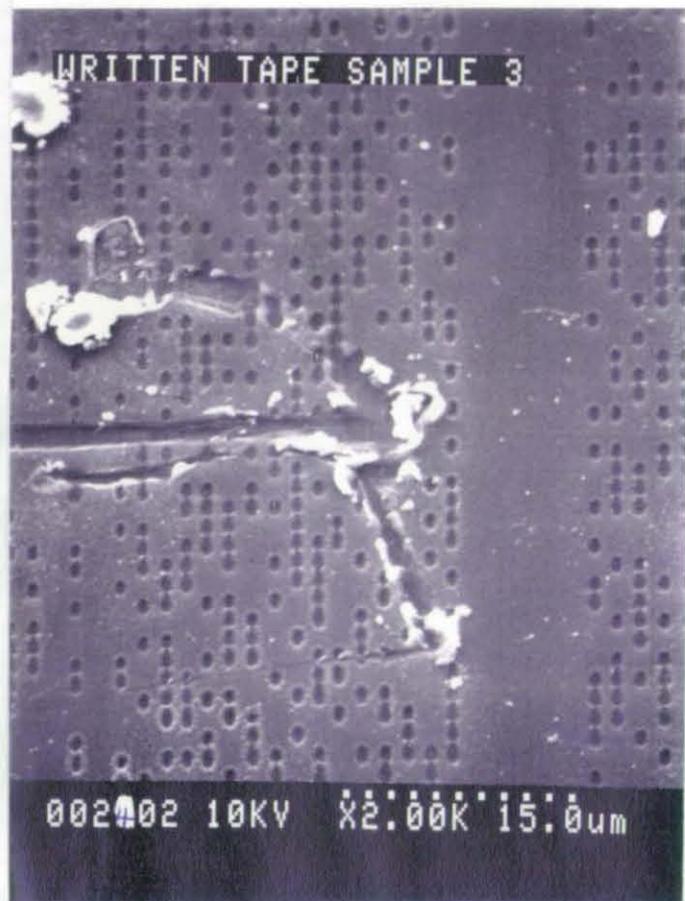
X Rub out  
on written  
a rea of  
75 $\mu$ m tape

Figure 36 A



Damage on  
written area  
of 75 $\mu$ m  
tape

Figure 36 B



coating machines allowing for the possibility of flocculation or agglomeration between the two processes.

#### 3.12.4 Damage to Monarch 1000 back coats

It seems likely that when the tape was worn, the thin layer of back coat resin covering carbon black agglomerates was removed, leaving a well of black pigment which was then transferred to the active layers in streaks. (Fig 37) The amount of resin removed from the larger particles would be minimal, especially when compared with chunks removed from the edges of tapes, hence the low level of physical damage. Carbon black is a soft filler thus the transfer of pigment to the active layer surface resulted in streaks without causing physical damage to the sensitive active layers. Although damage did not occur, these smears would have interfered with the focus of the laser and hence with reading and writing.



**Figure 37** Shows carbon black particle coated and uncoated after the wearing process.

Tapes 85 149 A & B were the best produced with minimal damage and no scalloping after 4,000 test cycles.

The major source of damage was still debris from the edges of the tapes. A geometric error rate (GER) map across the width of a tape shows the GER to be higher at the edges than in the centre of the tapes, this is also the case with unworn tapes. (Fig 38) The drive manufacturers intended that the tapes should have a 0.4 mm (400µm) guard band at the edge of the tape, where no information would be written. It was thus concluded that these coatings would be the route forward when optimised for production.

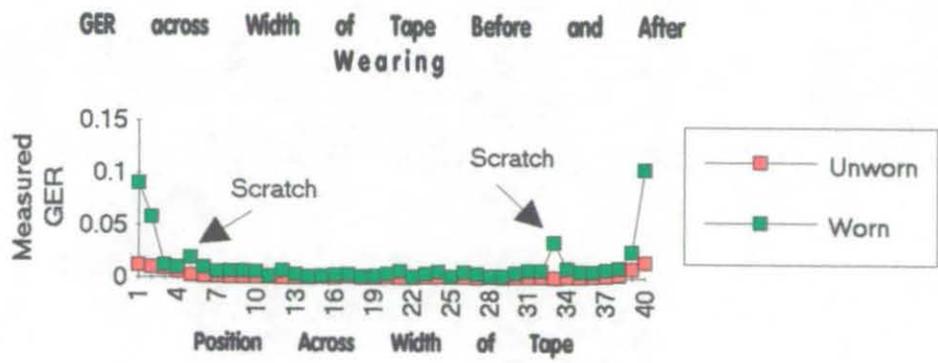


Figure 38

## **4.0 Discussion**

### **4.1. High filler loading back coats on 75µm tape.**

Handling and wear results showed that using formulations similar to those use by the magnetic media industry would not be feasible because of the high levels of wear. Overcoats were very badly worn by the back coats, with active layers being removed entirely in some areas. This was unacceptable with information being easily lost and the useable lifetime of the tapes short. In addition high filler loadings tended to imprint into the soft optically smooth active layers, possibly causing additional read/write errors. Optics for the tape drive were not available at this stage of the investigation and so this could not be tested. It became clear that lower filler loadings would have to be investigated.

### **4.2 Low filler loadings on 75µm tape.**

Low filler loadings proved to have better handling and wear characteristics, with Bacosol 2A SD showing some promise. However, back coat areas around filler particles proved susceptible to microblocking, by a process described as tenting. Aerosil R 972 back coats required further optimisation to improve wear and handling, however they did prove to be less susceptible to micro-blocking. New overcoats proved to be the most susceptible to micro-blocking, possibly due to incomplete cure.

### **4.3 Low filler loadings on 23µm tape**

Bacosol 2A SD had proved to be unacceptable as a filler because it was not small enough in particle size. To produce coatings with sufficient particles would require higher filler loadings that had already been discounted or a smaller particle size material.

Thus the research was continued using Aerosil R 972 a hydrophobic grade of fumed silica. Back coats with Aerosil R 972 gave good wear properties, but still required significant improvement.

Carbon black was investigated and despite early difficulties with dispersion a stable solution was produced which, when coated gave excellent handling properties with minimum damage to the active layers. It was discovered that most of the damage and debris came from the edges of the tape. Fractures formed in the active layers during the slitting process, crack propagated during handling, resulting in the worst cases in loss of active layers that tumbled across the tape surface producing further damage. Analysis showed GER to be higher at the edges of tapes before slitting, this became more pronounced after wearing.

The Monarch 1000, Solsperse, Ebecryl 5 129 formulations were considered to be fit for development grade tapes.

## 5.0 Conclusions

### 5.1 75 $\mu\text{m}$ Backcoats

- Highly filled backcoats are too abrasive to be considered as backcoats for optical media of the type discussed. They remove all the active layers, resulting in a tape with a very short lifetime. Therefore it is possible to say that the techniques and formulations used for backcoats in the magnetic media industry would not be suitable for optical tapes.
- By replacing the alumina hydrate/cellulose acetate/DIOP filler with a filler without additives, the susceptibility of optical tape to static build up is reduced, implying that the cellulose acetate and DIOP are the cause of static build up. *— expt to prove*
- The hexa-functional aliphatic polyester polyurethane recommended as a replacement for a di-functional epoxy acrylate provides the backcoat with a binder which has: excellent adhesion to both PET base and fillers thus preventing shedding of the backcoat: sufficiently high cure to prevent the solvent from subsequent coatings having a deleterious effect on the backcoat: flexible enough to not edge crack when slit and therefore not be a source of debris.
- Calcium carbonate, aluminium hydrate, both in chip and powder form, and china clay do not provide the required properties in solution or when coated for a backcoat for optical tape. Fumed silica does not provide the required physical properties required of a filled backcoat, the solution properties of the material are excellent producing well-dispersed solutions which remain in suspension for many months.

- Although filled backcoats were not optimised on 75 $\mu\text{m}$  media it is believed that a suitable filled backcoat could have been developed for the application.
- The frequency of filler particles has to be sufficient to prevent backcoats from tenting but low enough to have minimal abrasivity. Size of particles or agglomerates in the coated material is required to be under 1.0 $\mu\text{m}$  preferably under 0.8 $\mu\text{m}$ .
- Microblocking is not simply a function of particle size and distribution, but also of the frictional properties and age of the overcoat. Microblocking results when optical tapes are subjected to:
  - a) pressure, i.e. in a wound tape,
  - b) archive conditions e.g. time, temperature and humidity
  - c) close contact due to lack of filler particles holding the surfaces apart.
- It is believed that microblocking could be reduced if adhesion between both overcoat and dye and dye and aluminium could be improved to a point where adhesion is greater than the forces created between a backcoat and overcoat in close contact.

## 5.2 23 $\mu\text{m}$ Backcoats

- Fumed silica, does not provide the appropriate properties required for a backcoat with the required wear and lifetime properties on 23 $\mu\text{m}$  tape, being too abrasive and causing microblocking. However, the solution properties are ideal, the filler remained in suspension for months.

✓

- Carbon black filler with a low surface area is the most appropriate filler for backcoats, providing the following properties:
  - a) Low surface area blacks are easy to mill to the requisite particle size and produce solutions which have the required particle size distribution.
  - b) Sufficiently low particle size to produce a surface roughness (Ra) which whilst furnishing the tape with low coefficient of friction values, with the particles being large enough and closely spaced to prevent intimate contact of the backcoat and overcoat surfaces hence preventing microblocking.
  - c) Having sufficient conductivity (although not measurable by techniques available) to provide a degree of surface resistivity and hence aid in prevention of static build up.
  - d) When used with Solsperse dispersion aids producing a solution which was stable for a suitable time. This was deemed to be three months by production personnel.
  - e) The backcoat produced has the appropriate frictional properties for the tape drive it was designed for use with. The friction is low enough to allow the tape to settle into a natural position in the reel without causing scratching or in the worst case foul up of the drive as do high friction backcoats.
  
- Carbon black backcoats gave class one passes in the layer to layer microblocking test, indicating that the archive lifetime of the tapes would be acceptable.
  
- Although the Malvern Auto Measure is a more sensitive method of measuring particle size, the Fritsch Analysette is more suitable for this type of work because it analyses flowing solutions which require little or no dilution. The Malvern requires significant dilution of solutions, a possible source of error from, agglomeration and settlement.

*Surely* Conclusion & Discussion sections have been reversed

## 6.0 Recommended Further Work

- Ensure that milling conditions, (formulation, milling time, mill chamber temperature and re-circulation speed) for the final carbon black formulation always produce a millbase which, within reasonable tolerances, is repeatable in terms of: particle size: size distribution: viscosity and that when let down in the same way each time the final solution is also repeatable.
- Investigate with a view to improving it, adhesion properties of dye/binder to aluminium and of dye/binder to overcoat. Thus enabling microblocking to be reduced, increasing the lifetime of tapes.
- Investigation of adhesion should extend to the effect of adhesion of active layers on slit edge quality and the wear thus arising.
- Investigate slitting quality and its effect upon edge and tape damage.
- Determine if there is a lower limit as well as upper limit to the required particle size.
- Determine optimum filler and dispersion aid concentrations.
- Determine the effect of print through on read/write quality.
- Carbon black backcoats should be investigated for use on 75  $\mu\text{m}$  tapes.

## **Appendix II**

**Thesis access form**

**Certificate of originality**

## Appendix I



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