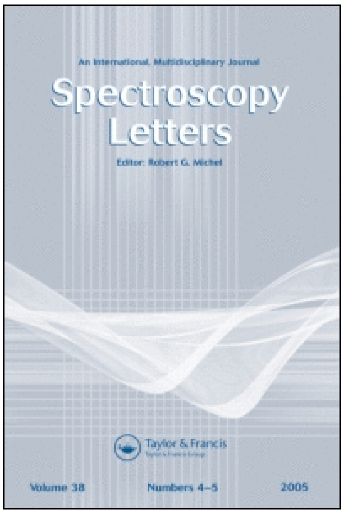

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The Identification of Dropouts in Magnetic Media Using Infrared Reflectance Microspectroscopy

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THE IDENTIFICATION OF DROPOUTS IN MAGNETIC MEDIA USING INFRARED REFLECTANCE MICROSPECTROSCOPY

KEYWORDS: Infrared Microspectroscopy, Magnetic Media, Dropouts, Specular Reflectance, Diffuse Reflectance, Kramers - Kronig Transform

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ABSTRACT

Dropouts are a major source of poor quality video tape in the magnetic media industry. A dropout can arise from any defect, contamination or hole in a magnetic tape which causes a loss of magnetic signal and results in poor video picture quality. This article reports on the application of infrared microspectroscopy to the analysis of dropouts originating from small contaminations that result in magnetic signal losses. The technique uses infrared reflectance (both specular and diffuse) and is accomplished with very little sample preparation prior to the analysis.

INTRODUCTION

Fourier transform infrared microspectroscopy can be a powerful tool in identifying small particle inclusions in polymeric materials (1-5). By operating in the reflectance mode, meaningful information can be obtained with minimal sample preparation related to contamination on the surface of many materials of interest (6).

The magnetic media industry presents a very interesting challenge for the analytical spectroscopist because the materials that are most commonly analyzed contain components not amiable to infrared spectroscopy. Most notable is the presence of significant levels of carbon black (7). To complicate the analysis, the defects to be analyzed can be stuck or even buried in the magnetic coating making them very difficult if not impossible to remove from the magnetic media. One alternative is to do the analysis insitu by infrared reflectance.

A dropout can be described as any defect, contamination or hole in a magnetic recording tape that results in a physical separation of the recording head from the magnetic media. This mechanism is depicted in Figure 1 showing the recording head losing contact with the magnetic tape. The end result is a magnetic signal loss accompanied by poor picture quality on the

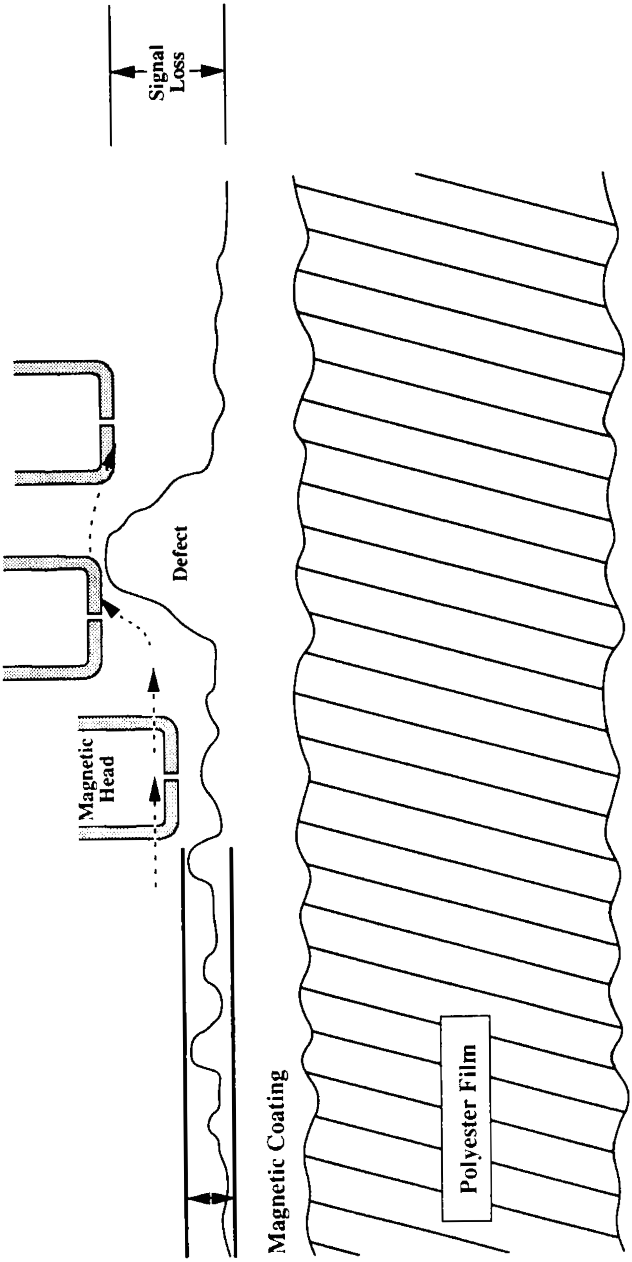


FIG 1: Representation of a magnetic head losing contact with the video tape resulting in a dropout.

television set. The physical size of the defect can range from submicron to hundreds of microns (normally 10 to 50 microns) and it has been shown in previous papers that defect size is directly proportional to magnetic signal loss (8). The dropout types of interest in this paper are organic in nature and result from small particle inclusions that can arise from materials associated with the coating formulation, contaminations from the base film or environmental contamination. These types of inclusions can cause bumps, ridges or areas with contamination on the magnetic surface. When the read/write head of the recorder is physically separated from the recording media by these inclusions, the end result is a dropout which is seen as the presence of white lines on the television screen.

Magnetic media is usually made up of a polyester base film coated with a magnetic recording material designed to adhere to the film. The polyester base film usually has a certain amount of small inorganic particles that are dispersed throughout the film. These particles are added to make the film less tacky so the material can be processed without sticking to itself or to conveying hardware. Magnetic coating formulations vary from manufacturer to manufacturer but the core ingredients for the most part, are the same. The component in highest concentration is the magnetic pigment. This material is usually made up of some type of iron oxide or a cobalt iron oxide material (9). The component in the next highest concentration is the organic binder. The binder is added to provide an adhesive for the pigment particles (10) and to give the coating better wear characteristics. Most binder materials are composed of crosslinkable polyurethanes that possess both a hard and soft sector in the polymer chain or a mixture of polyurethane and polyvinyl chloride/polyvinyl acetate. These are added to provide both structural rigidity and flexibility to the magnetic coating (11, 12). The last component that is important in dropout analysis is the lubricant. The lubricant is usually made up of long chain aliphatic esters or silicon oils. These materials are added to minimize coating abrasion problems (13). In this paper, reflectance infrared microspectroscopy is used to identify the source of defects resulting in losses of signal on magnetic tapes. The infrared spectra, both specular and diffuse, are evaluated using the Kramers - Kronig and Kubelka - Munk algorithms respectively. Data are then presented reporting some common causes of dropouts in the magnetic media industry.

EXPERIMENTAL

The infrared microscope used was an IR-PLAN from Spectra Tech Inc. (Stamford, Conn.). The microscope uses 15X and 32X IR/Vis reflecting cassegrainian objectives with on axis optics to focus the infrared beam onto the sample. The microscope has a matched focus dedicated narrow band (0.25 mm X 0.25 mm) liquid nitrogen cooled MCT detector for maximum sensitivity. The microscope also uses a remote aperture to mask the sample. The FTIR used was a Perkin Elmer 1760 (Danbury, Conn.). The signal to noise ratio for this instrument using 100 scans at 4 cm-1 resolution and a 100 micron pinhole is ~1700:1. 256 co-added scans were used and the spectra were ratioed to an infrared reflecting mirror background. The oxygen etching apparatus used was an SPI Plasma Prep II from Structure Probe (West Chester, PA).

SAMPLE PREPARATION

Samples were mounted on glass microscope slides to prepare them for infrared analysis. The samples were then placed under a visible light microscope to determine if the defect that resulted in a dropout was on the surface of the magnetic tape or buried beneath the magnetic coating. If the defect was on the surface, the dropout could be analyzed without any further preparation. If the defect was buried underneath the magnetic coating, the sample is then placed in an oxygen etching apparatus for two minutes. After etching, a piece of adhesive tape was placed on the dropout. The adhesive tape was then given a fast firm pull to remove it from the magnetic tape. Upon removal, the oxygen etched portion of the magnetic tape was removed leaving the dropout exposed for reflectance analysis.

DETECTOR AND OBJECTIVE CONSIDERATIONS

Commercial MCT detectors are available in narrow, medium and wide band windows. Narrow and medium band windows are most commonly used in infrared microscopes. It was found in this work that a medium band MCT window (1.0 mm X 1.0 mm) did not possess sufficient sensitivity to detect the dropouts commonly encountered in our lab. It was only when a narrow band window (0.25 mm X 0.25 mm) was purchased that sufficient sensitivity was achieved to acquire infrared spectra.

The overall resolution of the infrared microscope is inversely proportional to the numerical aperture of the system. This is related by the equation (14) :

$$R = \frac{0.61\lambda}{N.\ A.} \qquad (1)$$

where R is the resolution, λ is the wavelength of light used and N. A. is the numerical aperture. Holding all other parameters constant, the resolution can be directly related to the numerical aperture of the reflachromat objectives used in the infrared microscope. The infrared microscope in the authors lab uses 15X and 32X reflachromat objectives with numerical apertures of 0.58 and 0.65 respectively. Using equation 1 to calculate the resolution for each objective it is seen that an 11% increase in resolving power can be realized by using the higher numerical aperture objective at a given wavelength. Under normal conditions this increase in resolution is not significant, but as the particles approach the theoretical diffraction limit of the technique, such as in the analysis of dropouts, this enhancement becomes very important.

THEORY

Depending on the surface roughness of the magnetic coating and the size of the particle to be analyzed, there are three types of reflectance spectra that can be obtained. These are diffuse, specular or a mixture of both.

Diffuse reflectance can be described as light scattered in directions unrelated to that of the incident beam radiation (15-17). For the most part in dropout analysis, the surface roughness and particle size are sufficient to make diffuse reflectance dominant over specular reflectance. This can be rationalized by realizing that the size of these dropouts in many instances are approaching the wavelength of the infrared beam and it has been shown previously, that specular reflectance is minimized when this occurs (18). Diffuse reflectance spectra are evaluated by the well known Kubelka-Munk relationship shown below for an infinitely thick scattering material (17).

$$f(R_\infty) = (1-R_\infty)^2/2R_\infty = K/S \qquad (2)$$

Where R_∞ is the absolute reflectance, K is the molar extinction coefficient and S is the scattering coefficient.

Specular reflectance originates from surface reflectance of an optically thick sample or from particles that are large compared to the infrared beam. The acquired spectrum looks much like a first derivative spectrum and at times can be difficult to interpret. In the event specular reflectance is dominant in the acquired spectrum of a dropout, the data may be evaluated using the Kramers - Kronig transformation shown below (19).

$$\eta_{(\nu)} = \eta_{\infty} + \frac{2}{\pi} \int_{\infty}^{+\infty} \frac{K_{(\nu)}\nu}{\nu^2 - \nu_C^{\ 2}} \, \partial\nu \qquad (3)$$

Where η is the refractive index, K is the extinction coefficient, and ν is the frequency. The Kramers - Kronig algorithm evaluates the specular reflectance in terms of K, the extinction coefficient which can then be expressed in terms of absorbance, and N, the refractive index spectrum (20). When evaluating spectra using the Kramers - Kronig transform, extreme caution should be exercised because the data points that are chosen for the algorithm must be completely specular in nature and have little or no contamination from the diffuse component. The normal procedure, used in the author's lab is to isolate and evaluate small areas of the spectrum that are known to contain the highest amount of specular component.

RESULTS AND DISCUSSION

Dropouts that originate from lubricant materials, appear on the magnetic tape as a discoloration on the surface (Figure 2A).

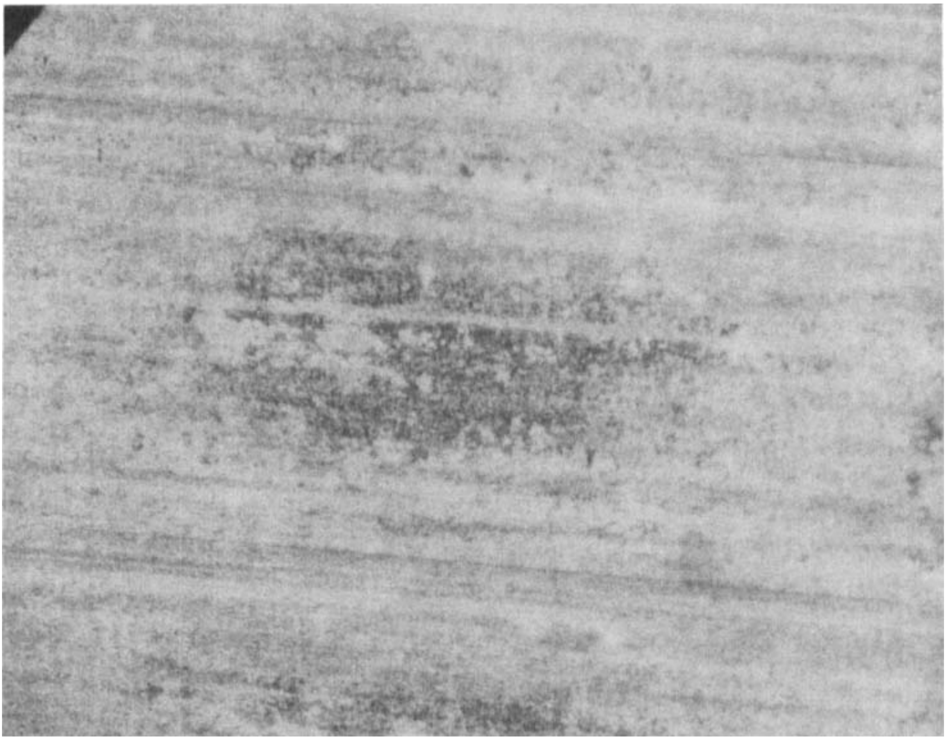
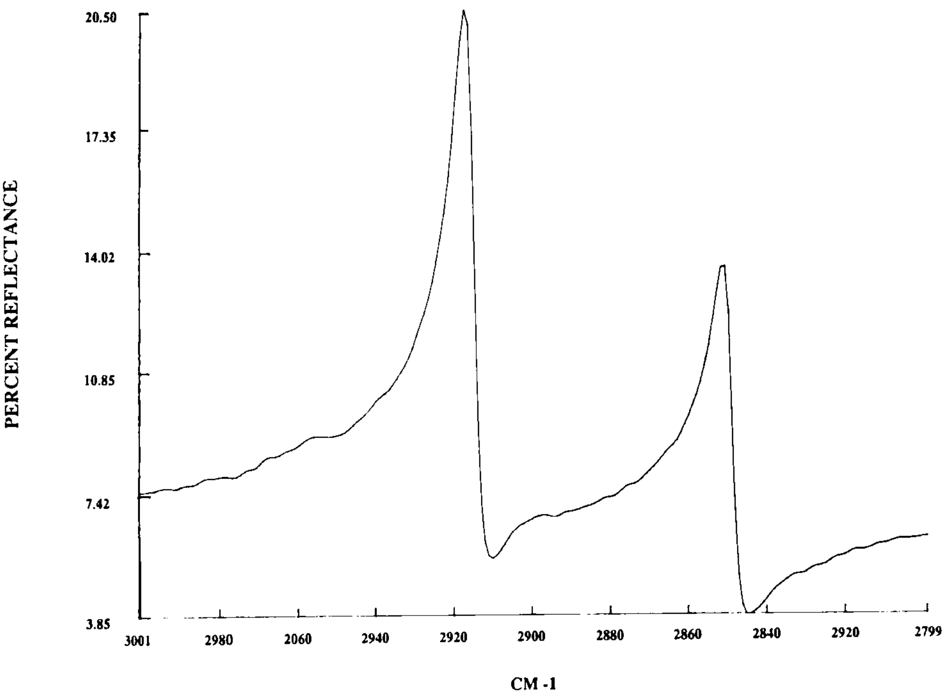


FIG 2A: Interference light micrograph taken at 166X of a contamination on the surface of a video tape.

FIG 2B: Infrared specular reflectance trace of the contamination shown in Figure 2A before the Kramers - Kronig transform.

FIG 2C: Infrared specular reflectance trace of the contamination shown in Figure 2A after the Kramers - Kronig transform. The material was identified to be an aliphatic stearate lubricant.


PERCENT REFLECTANCE
20.50
17.35
14.02
10.85
7.42
3.85
3001
2980
2060
2940
2920
2900
2880
2860
2840
2920
2799
CM -1

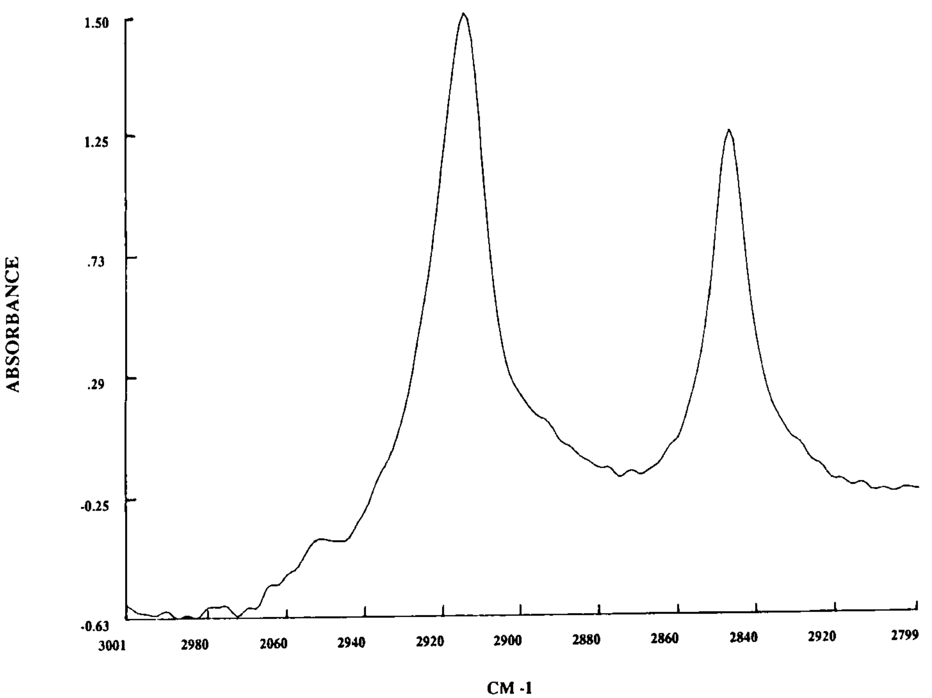

ABSORBANCE
1.50
1.25
.73
.29
-0.25
-0.63
3001
2980
2060
2940
2920
2900
2880
2860
2840
2920
2799
CM -1

FIG 3A: Scanning electron micrograph taken a 5000X of a deposit on the magnetic coating of a video tape.

Since the surface of these areas contain little or no magnetized pigment particles, there will be a weak magnetic signal sent from the recording head. Figure 2B represents the infrared reflectance spectrum of the aliphatic region of the micrograph shown in Figure 2A. This portion of the spectrum is specular in nature and is not representative of the magnetic coating background. By applying the Kramers - Kronig transform only to the region from 3001 to

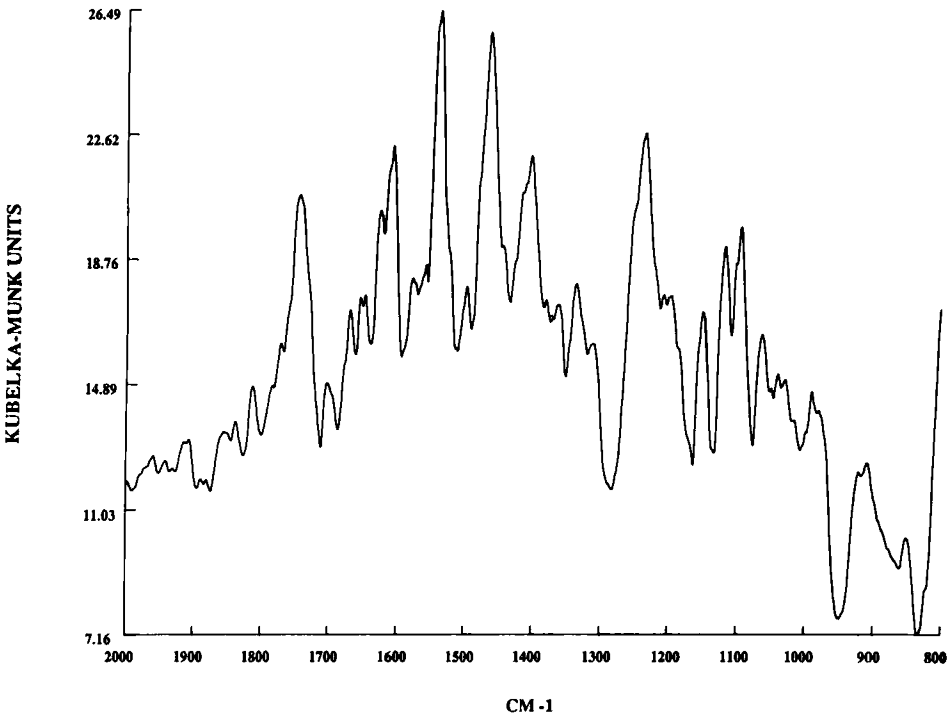


FIG 3B: Infrared diffuse reflectance spectrum of the deposit in Figure 3A identified to be polyurethane.

2799 cm-1 (Figure 2C), this spectrum was identified to be the methylene stretches of butoxy ethyl stearate which is a common lubricant component in the magnetic media industry.

Polyurethane materials, because of their type of functionality, are sensitive to hydrolytic degradation with time. This type of degradation is mainly a function of humidity and temperature which usually results in the formation of gel

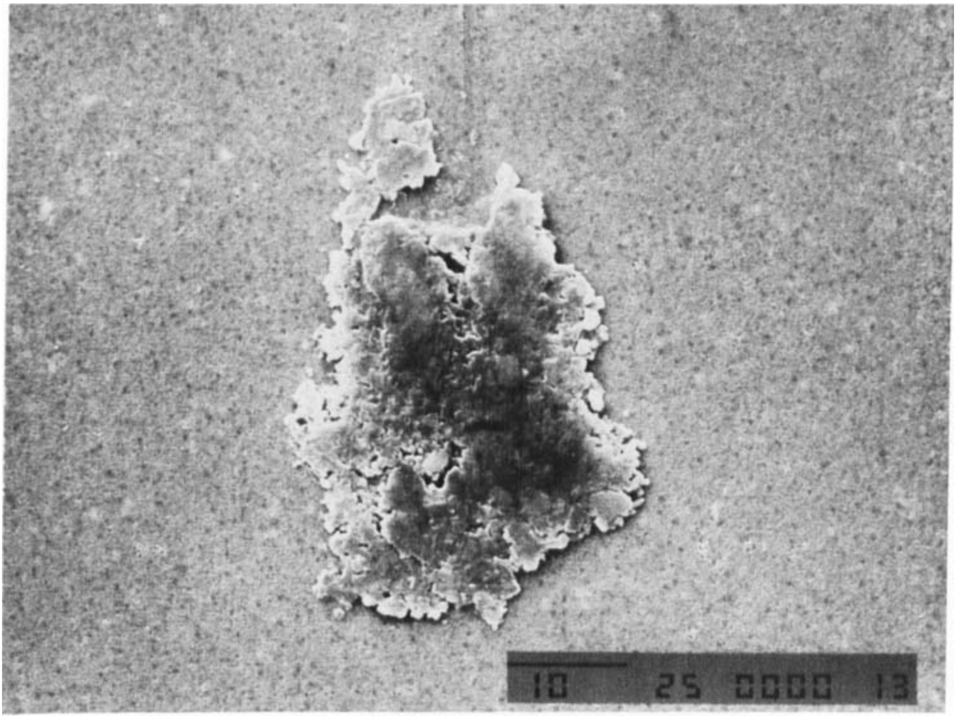


FIG 4A: Scanning electron micrograph taken at 1500X of a deposit on the surface of a magnetic coating.

components in the binder mix. The gel causes improper dispersion in the coating bath and the final result is small insoluble particles that can cause dropouts on the magnetic tape. Figure 3A is a scanning electron micrograph taken at 5000X of a dropout approximately 10 microns in size that was found on the surface of a magnetic tape. The diffuse reflectance spectrum from 2000 to 800 cm-1 of this defect is shown in figure 3B. This spectrum was

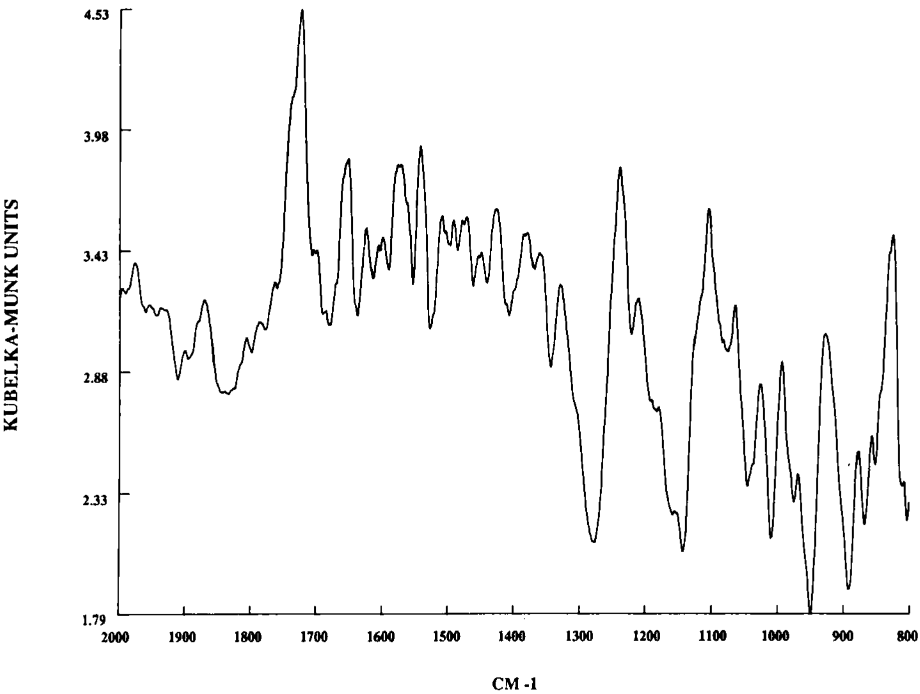


FIG 4B: Infrared diffuse reflectance spectrum of the deposit shown in Figure 4A identified to be polyester.

identified to be the polyurethane binder material from the carbonyl peak at 1730 cm-1 and the deformations from the ester side of the molecule at 1240 and 1110 cm-1.

In some rare instances, dropouts have been attributed to the polyester film. This can occur if debris collects on the base film when it is being slit into rolls for the magnetic coaters. Figure 4A is a scanning electron micrograph taken at 1500X of a dropout approximately 25 microns in size. Figure 4B shows the

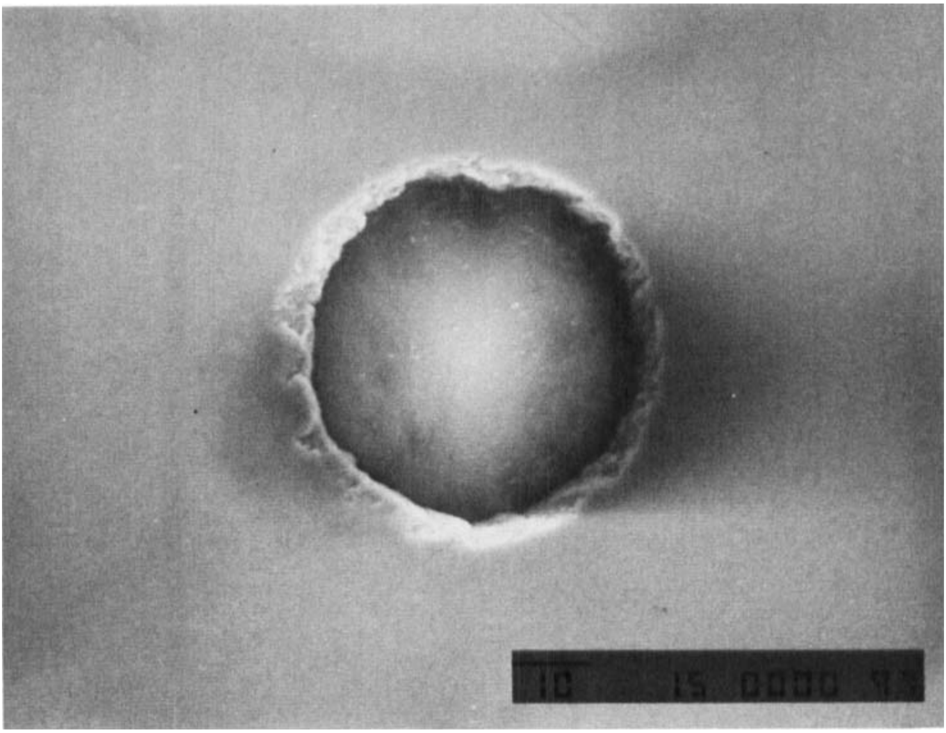
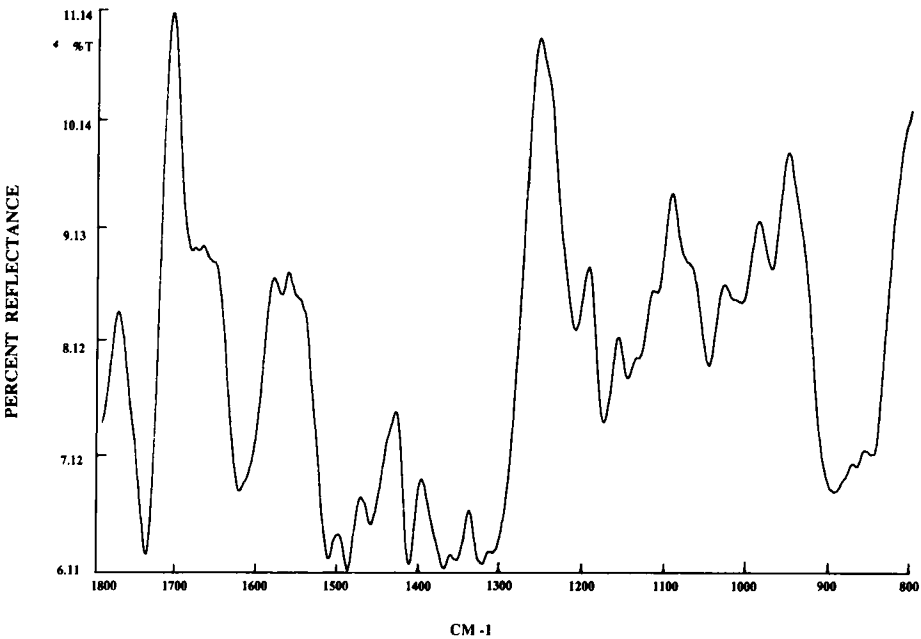
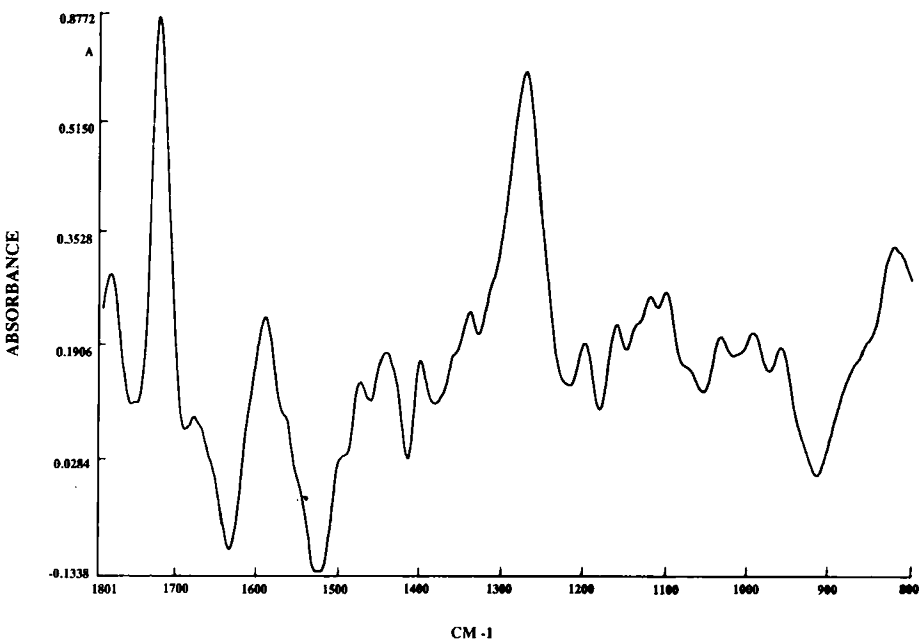


FIG 5A: Scanning electron micrograph taken at 1000X of a hole in the magnetic coating that resulted in a dropout.

FIG 5B: Infrared specular reflectance trace of the dropout shown in Figure 5A before the Kramers - Kronig transform.

FIG 5C: Infrared specular reflectance spectrum of the dropout shown in Figure 5A after the Kramers - Kronig transform. The spectrum is that of the polyester base film.


11.14
%T
10.14
9.13
8.12
7.12
6.11
PERCENT REFLECTANCE
1800
1700
1600
1500
1400
1300
1200
1100
1000
900
800
CM -1


0.8772
A
0.5150
0.3528
0.1906
0.0284
-0.1338
ABSORBANCE
1801
1700
1600
1500
1400
1300
1200
1100
1000
900
800
CM -1

diffuse reflectance spectrum from 2000 to 800 cm-1 of the dropout identified to be a polyester flake from the carbonyl band at 1724 cm-1 and the ester deformations at 1260 cm-1 and 1125 cm-1.

Some dropouts occur because of the complete absence of magnetic coating. In many cases a hole that exposes the base film is the only evidence that is seen (Figure 5A). The infrared reflectance spectrum from 2000 to 800 cm-1 of this defect is completely specular in nature and is shown in Figure 5B. The Kramers -Kronig transform was performed on this spectrum and it was identified to be the polyester base film (Figure 5C) from the major peaks at 1725, 1260 and 1125 cm-1. Defects of this kind usually occur because a particle in the magnetic coating at some point in the process was removed leaving the base film exposed.

CONCLUSIONS

Infrared microspectroscopy has been shown to be a valuable technique for the insitu analysis of dropouts in magnetic tape. The dropouts analyzed in this paper originated from magnetic coating agglomerations, lubricant and the polyester base film. In practice, magnetic coating agglomerations account for over 60% of the dropouts analyzed in the author's lab. Less than 10% can be attributed to lubricant and the polyester base film. The remaining 30% are due to impressions placed on the magnetic media by the coater's process.

Improvements in the instrument's sensitivity can be realized by using a narrow band window MCT detector and fully reflecting cassegrainian objectives with a high numerical aperture. Finally, the technique requires little sample preparation and capitalizes on both specular and diffuse reflectance spectroscopies for analysis.

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