MONOGRAPH

magnetic recording media under the microscope

Memorex Corporation /1180 Shulman Avenue / Santa Clara, California 95050/(408) 248-3344

magnetic recording media under the microscope

by Donald C. Gaubatz

ABSTRACT

The average user is unaware of the effort that has gone into the production of high quality magnetic recording tape, which is deceptively simple in appearance. The painstaking methods by which it was produced are not apparent from a casual scrutiny. One vital aspect of the control exercised is an examination of microscopic characteristics throughout the production of magnetic recording tape from raw material through the final precision product. Similar controls must be exercised during the equally critical production of disc packs. This monograph discusses and illustrates several techniques which reveal the micrographic character of magnetic recording tapes and discs.

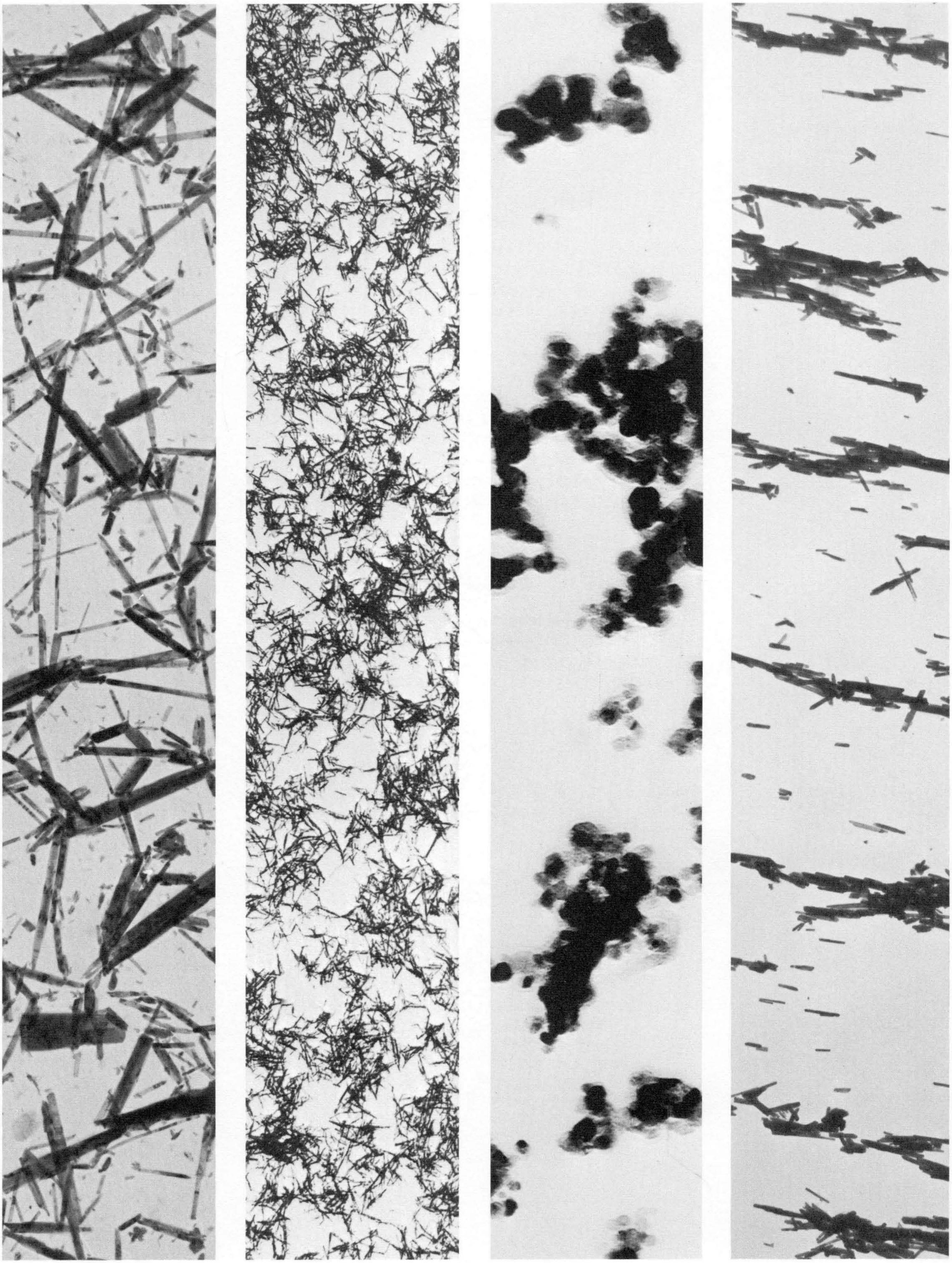
INTRODUCTION

Many techniques have been developed by Memorex to observe the subtle characteristics of high quality precision magnetic recording media. These techniques involve the use of the electron microscope, optical microscope, optical reflectometer, and stylus-type profilometer. It is the intent of this monograph to discuss these instruments and their use in observing the microscopic characteristics of the surface, interior, and ingredients of magnetic tapes and discs.

The surface of a magnetic recording medium is but one facet of a complex structure. As a slice of bread reveals the interior of the loaf, so does a single slice of magnetic recording medium exhibit the interior structure of the product. This cross-section accurately depicts the thickness of the coating and base film and shows the makeup of the composite structure.

Base film and coating defects, agglomeration, particle size and shape, voids, binder-rich areas, inclusions, particle orientation, uniformity of particle dispersion, nodules, loose oxide surface particles, and surface roughness are characteristics readily shown by a micrographic examination of a given product. It is the control of these, in addition to many other properties, that set a precision product above all others.

electron microscope



ELECTRON MICROSCOPE

The primary tool to observe the microscopic characteristics of a magnetic recording medium is the electron microscope. It is an instrument capable of providing 250 times better resolution than an optical microscope. A good optical instrument is able to resolve two points that are 2500 Angstroms (10 microinches) apart. A high quality electron microscope provides a resolution of less than 10 Angstroms (0.04 microinches). With this degree of resolution, the electron microscope is able to reveal microscopic details of magnetic recording media well beyond the capability of its optical counterpart. Typically, the maximum magnification of optical instruments is 1500 times. This represents the lowest magnification generally used with the electron microscope. Direct magnifications of well over 100,000 times are possible with the electron microscope. Further, photographic enlargement makes possible the attainment of much higher magnifications.

CROSS-SECTION

The electron microscope represents the only practical method for studying a cross-section of a magnetic recording medium. A thin cross-sectional slice, 2 to 6 microinches in thickness, can be studied in detail at almost any desired magnification up to the maximum capable with the electron microscope. In these cross-sections, it is possible to make accurate measurements of coating thickness and to study such parameters as surface roughness, particle size, degree of particle orientation, and uniformity of the oxide-binder mixture.

In the production of a thin cross-section, a sample of the material is held in a potting resin. When the resin has cured, the potted section is mounted in the specimen holder on an ultramicrotome. A diamond knife is then used to slice a section. This section of the sample is floated onto the surface of a liquid as it leaves the knife edge. A small copper grid is used to retrieve and hold the section for direct observation when placed in the electron microscope.

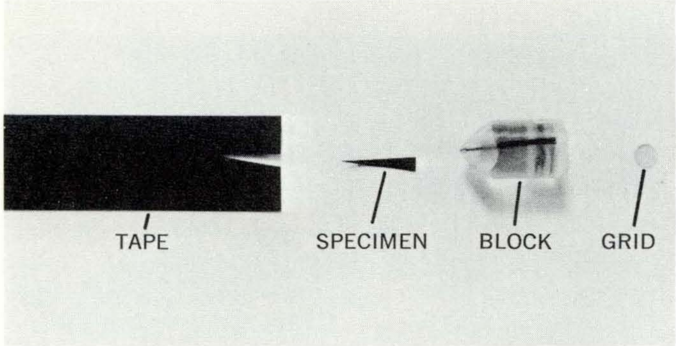


Figure 1. Steps in preparing a transverse section of magnetic recording tape for microscopic examination. Proceeding from left to right, a small triangular section is removed and suspended in a potting resin for microtoming. The mounted sample is shown on the small copper grid at the right side of the photograph.

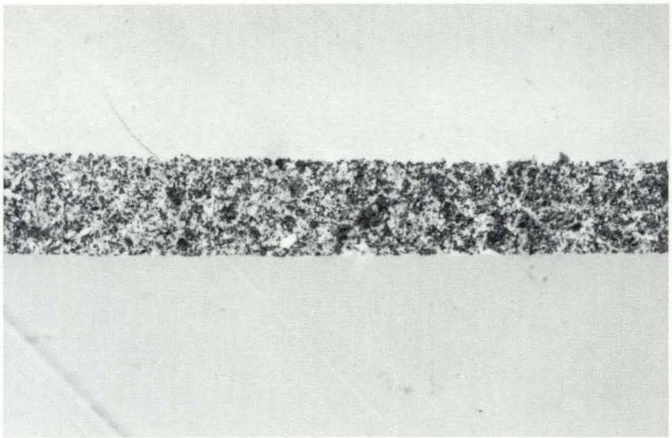


Figure 2A. Coating cross-section of a currently marketed magnetic recording tape (2000X).

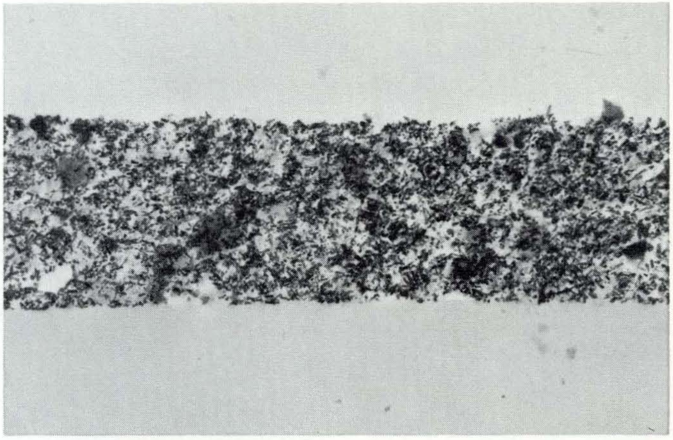


Figure 2B. Coating cross-section illustrated in Figure 2A. (6000X).

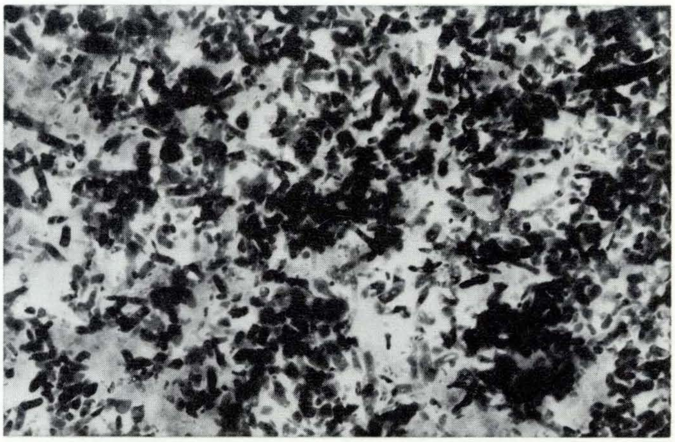


Figure 2C. Coating cross-section illustrated in Figure 2A. (20,000X).

Figures 2A, 2B and 2C show a cross-section of a currently marketed magnetic recording tape at three different magnifications. The lowest magnification is useful to see the complete thickness of the base film and the coating. This magnification permits a longer section of the coating to be judged for such qualities as uniformity of dispersion throughout the coating thickness. The highest magnification permits a detailed examination of the individual particles.

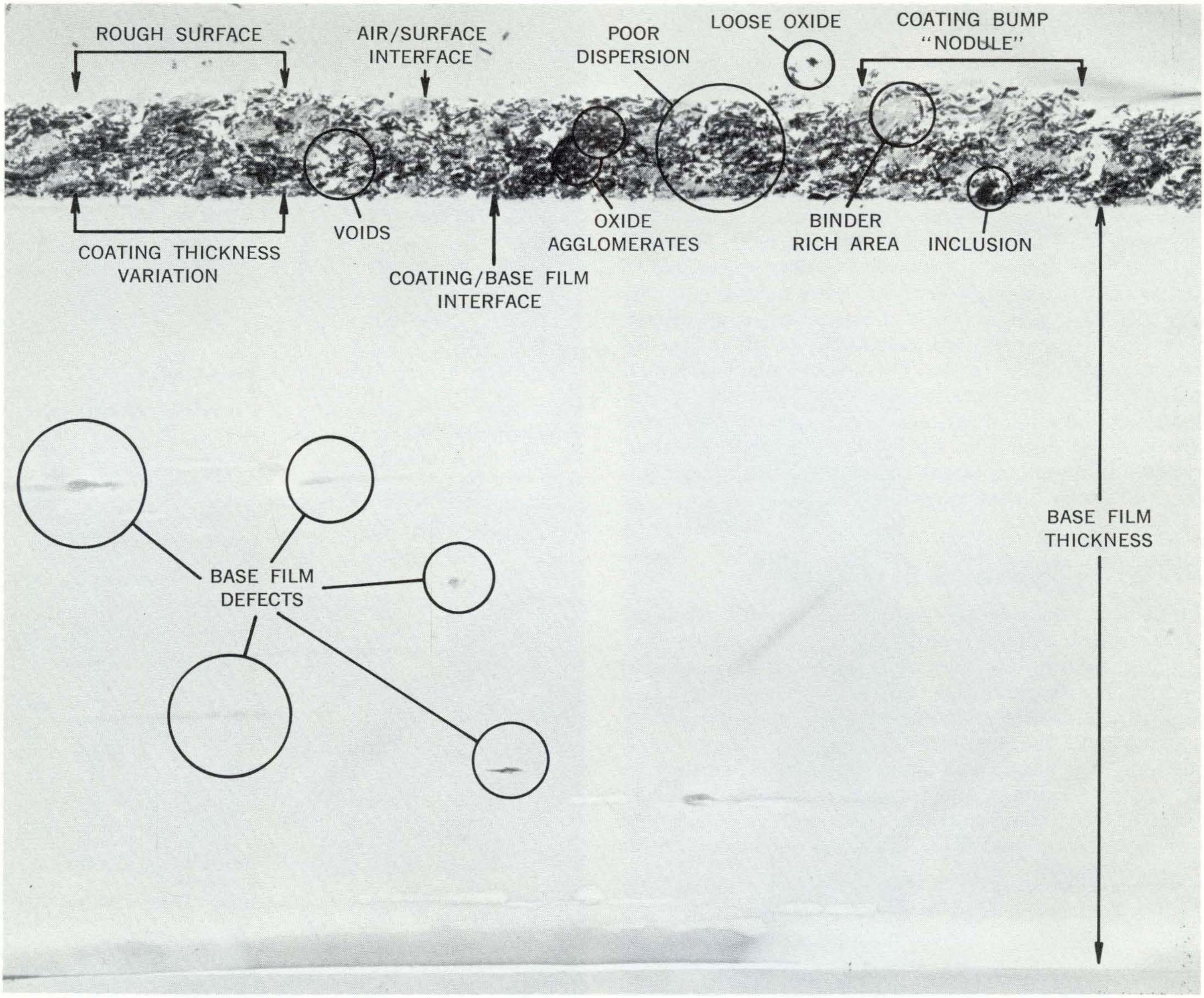


Figure 3. Many of the characteristics to be seen in a cross-section of magnetic recording tape. (6000X).

The ability to isolate and identify defects eases their elimination. A cross-sectional view assists in understanding a magnetic medium, promotes process improvement, and aids the development of new products. A thorough understanding of the microstructure of its products has enabled Memorex to progress to its present position of leadership.

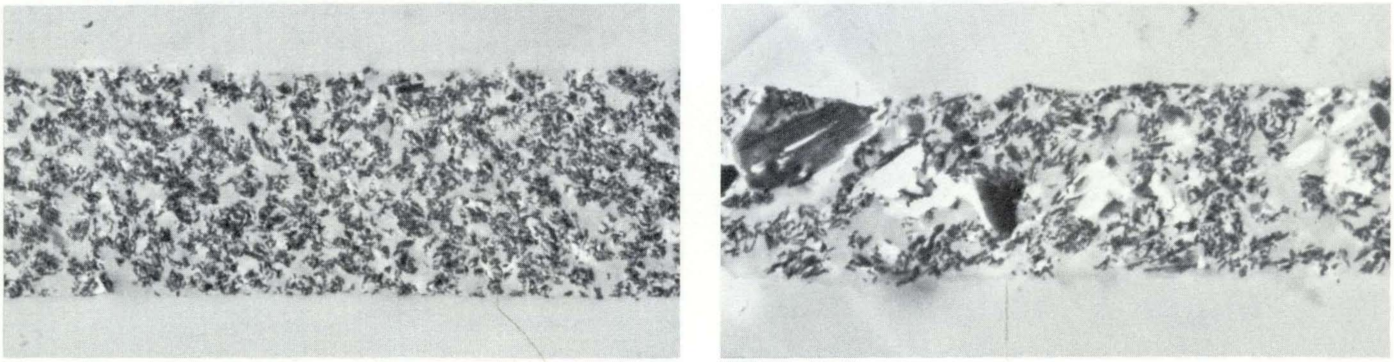


Figure 4. Disc Coating Cross-sections (6000X).

A comparison of particle and coating uniformity showing the superiority of a Memorex disc versus that of a leading competitor.
Left — Memorex Mark 1 Disc Pack. Right — Leading competitive Disc Pack.

POWDER OBSERVATION

For the observation of the particles of magnetic recording material, considerable skill is required by the microscopist to produce well-dispersed, agglomerate-free samples. Regular gamma-ferric oxide is generally an acicular particle with a length of 10 to 20 microinches and a width of 2 to 4 microinches. Some experimental materials have a maximum dimension of less than a microinch. In an examination of these powders, the electron microscope provides information concerning particle shape, size, uniformity, and porosity.

In the preparation of a sample of powder for the electron microscope, a small amount of powder is placed on a microscope slide which has been moistened with a solution such as collodion. A second microscope slide is used to spread the material and produce a mixing and break-up of the agglomerates through a shearing action. The final motion between the two microscope slides is to slide them apart and leave a thin film of magnetic material and collodion. Once the collodion has dried, the sample can be floated off the microscope slide onto the surface of a liquid. A small copper grid may be used to pick up a sample of this film for direct observation in the electron microscope. Depending upon the type of magnetic material to be observed, other techniques such as mechanical or ultrasonic vibration can be used to create a good dispersion and a minimum of agglomeration within the collodion-magnetic particle mixture.

SURFACE OBSERVATION

The electron microscope, with typical magnification of 4000 to 50,000 times, permits the observation of details of a magnetic recording surface that are an order of magnitude smaller than a common magnetic particle. A large amount of detail is shown in a surface photomicrograph, but the high magnification necessary for great detail limits the area which can be viewed in a single picture. Therefore, it is usually desirable to study the surface of a magnetic recording medium with both the optical and electron microscopes to provide the best overall and detailed conception of surface features.

When the structure of magnetic recording media or other objects is too thick to be penetrated by an electron beam or when conventional reflection methods are inadequate or impractical, the objects may be studied through the replication of their surface on a thin film. In one replication process, a solution of plastic is applied to the surface in question. The film is allowed to dry, then is separated from the surface. This process provides a negative replica, so called because protrusions and depressions are reversed with respect to the original surface.

Figures 6A and 6B illustrate the orientation visible in the study of two different cross-sections of an oriented magnetic oxide tape. In Figure 6A, the length of the particles is readily seen, whereas the particles in Figure 6B have been sectioned and are seen as an end view.

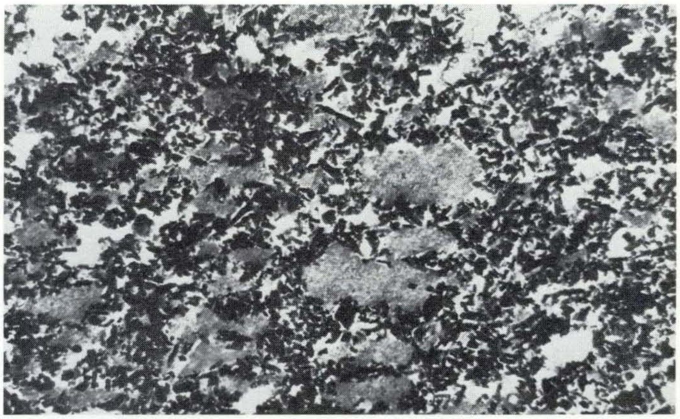


Figure 5. Coating cross-section with a poor distribution of magnetic particles in the oxide-binder mixture (6000X). The large grey areas are binder rich and show an almost complete absence of magnetic particles.

Figure 6A. Longitudinal cross-section of the magnetic oxide layer of an oriented tape (40,000X).

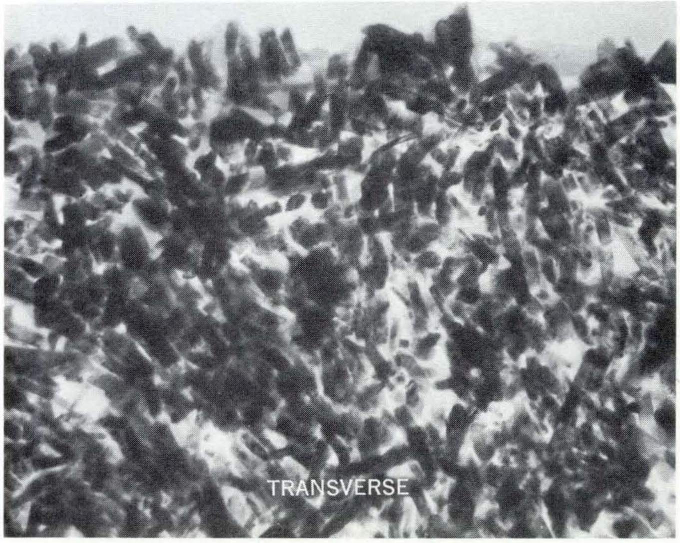


Figure 6B. Transverse cross-section of the magnetic oxide layer of an oriented tape (40,000X).

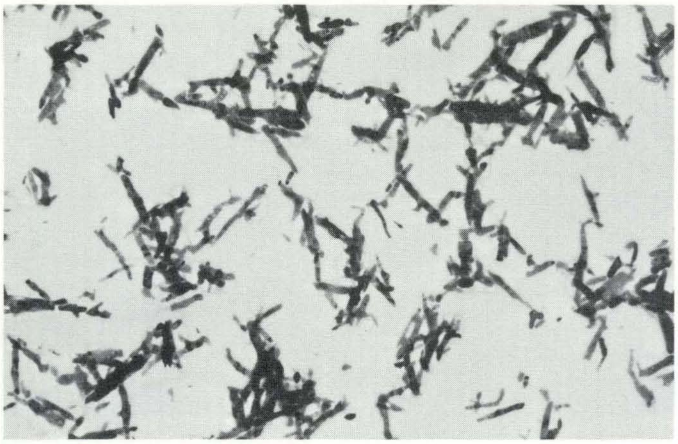


Figure 7A. A common acicular gamma-ferric oxide used in magnetic recording tape (16,000X).

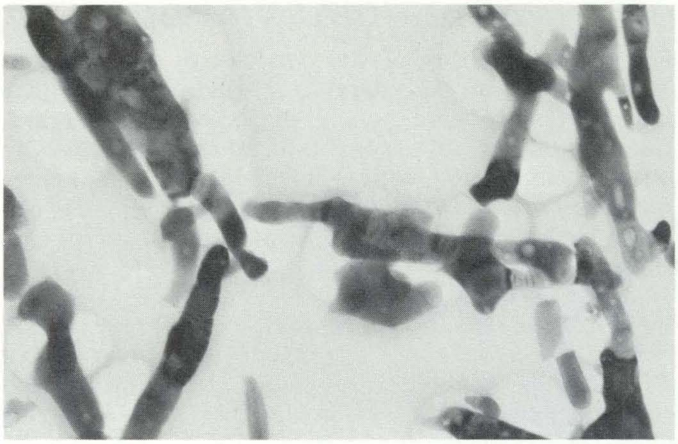
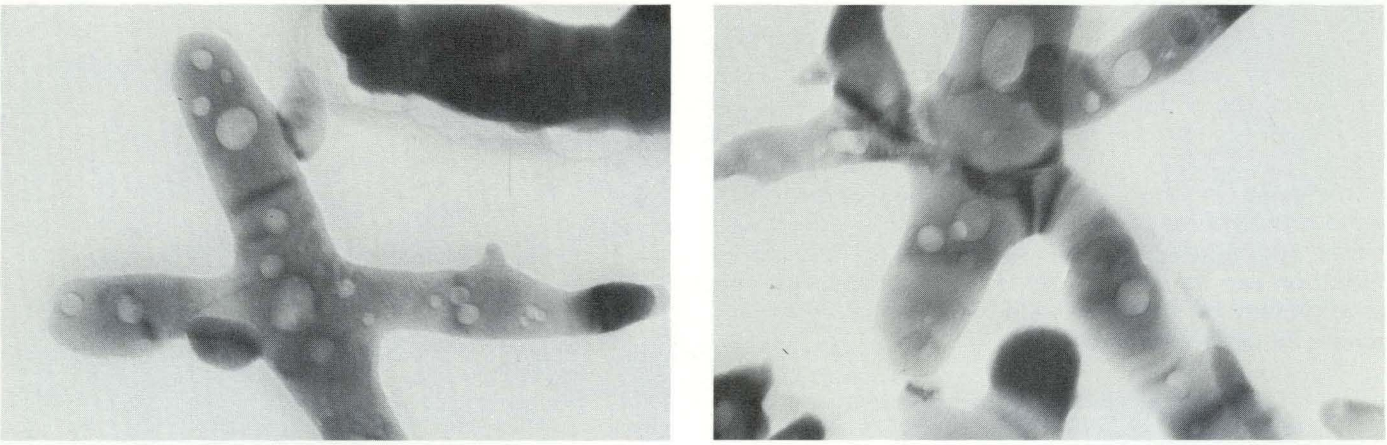


Figure 7B. The acicular gamma-ferric oxide illustrated in Figure 7A (80,000X). Note the porosity of individual particles.



Figures 7C-7D. Selected particles of gamma-ferric oxide illustrated in Figure 7A (200,000X).

Figures 7C and 7D illustrate some of the many problems that can be observed in a detailed micrographic examination of a magnetic oxide. Both photomicrographs illustrate particles with a high degree of porosity and non-uniform crystal growth.

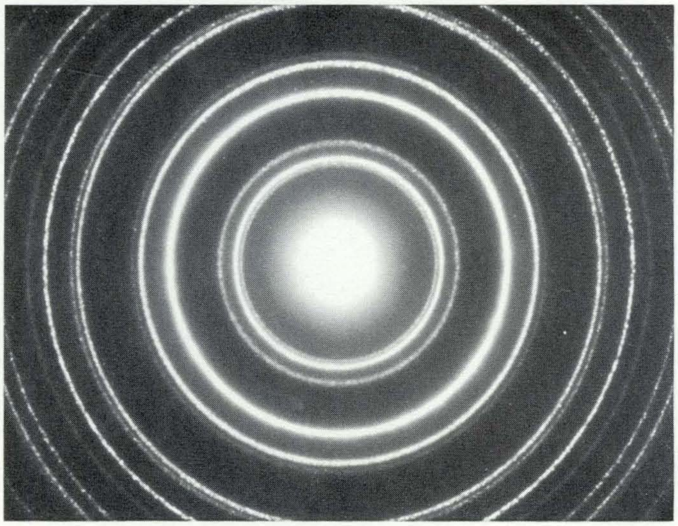


Figure 8A. Electron diffraction pattern from a pure gold film.

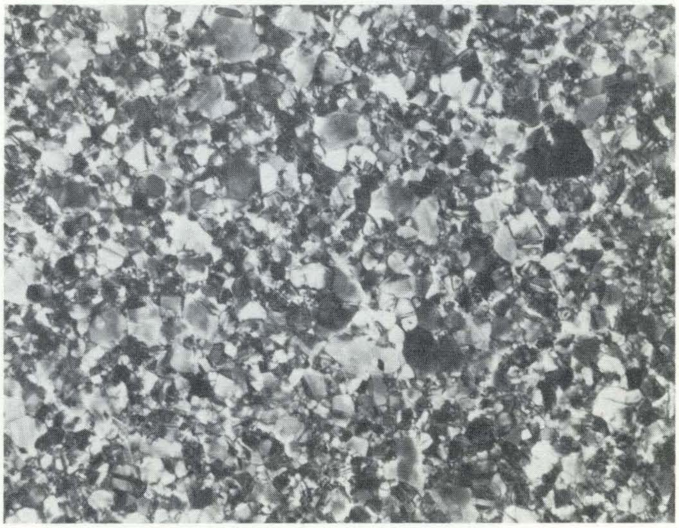


Figure 8B. Transmission photomicrograph of the pure gold film to produce the electron diffraction pattern shown in Figure 8A. (40,000X).

For the purpose of investigating the structure of magnetic recording materials, electron diffraction can be used. Electron diffraction is an interference phenomena that results from the interaction of electrons with the crystal lattice of a given specimen. The diameter of the rings illustrated in Figure 8A and their relative intensities can be classified and compared with the diffraction patterns of known materials to obtain an identification of small individual particles.

The replica films may be examined directly without shadowing, but when the surface variations are of the order of a few microinches, the negative replica may appear to lack detail. Shadow casting is a technique which brings out the finer structure, renders the image relatively independent of film thickness, and aids in the interpretation of height and depressions. In the shadow casting process, metal evaporated from an electrically heated filament in a vacuum impinges on the specimen placed at an appropriate distance so that the metal atoms arrive along almost parallel straight lines. An object projecting a distance h above the surface casts a shadow of length $L = \tan \Theta$, where Θ is the impingement angle. The angle is given in terms of the ratio of shadow length to object height, typically 2:1. Shadow casting is effective for revealing surface features not otherwise visible and gives an accurate method for the determination of the height of a roughness element by the measurement of the length of the cast shadow.

Replication is useful for examination of other objects, particularly those that are too large to be placed under the microscope or to be brought to the microscope. This technique provides a non-destructive method for observing surfaces in general. Even the narrow gaps of the finest reproduce heads may be studied without fear of damage to the head. The dimensional stability of the replicating material and the magnification of the electron microscope permit direct quantitative measurements to be made on such parameters as gap length, lamination dimensions, gap misalignments, gap smearing, wear, etc.

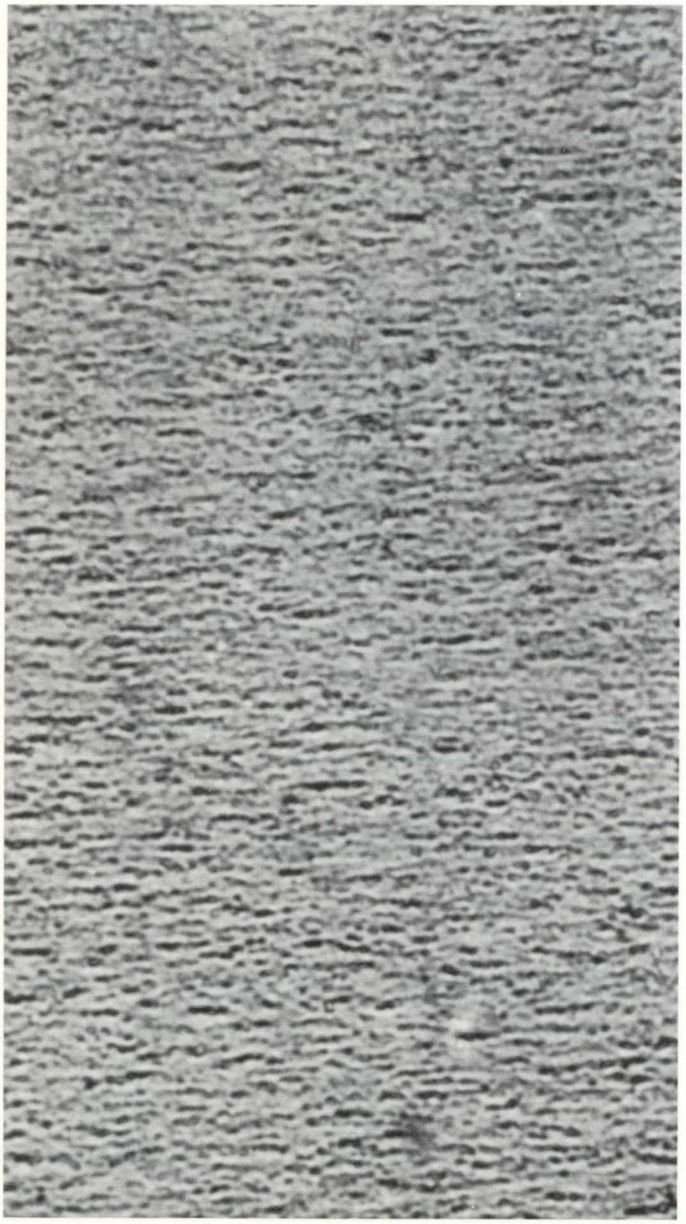


Figure 9A. Shadow cast replica of the surface of a typical computer tape (200X).

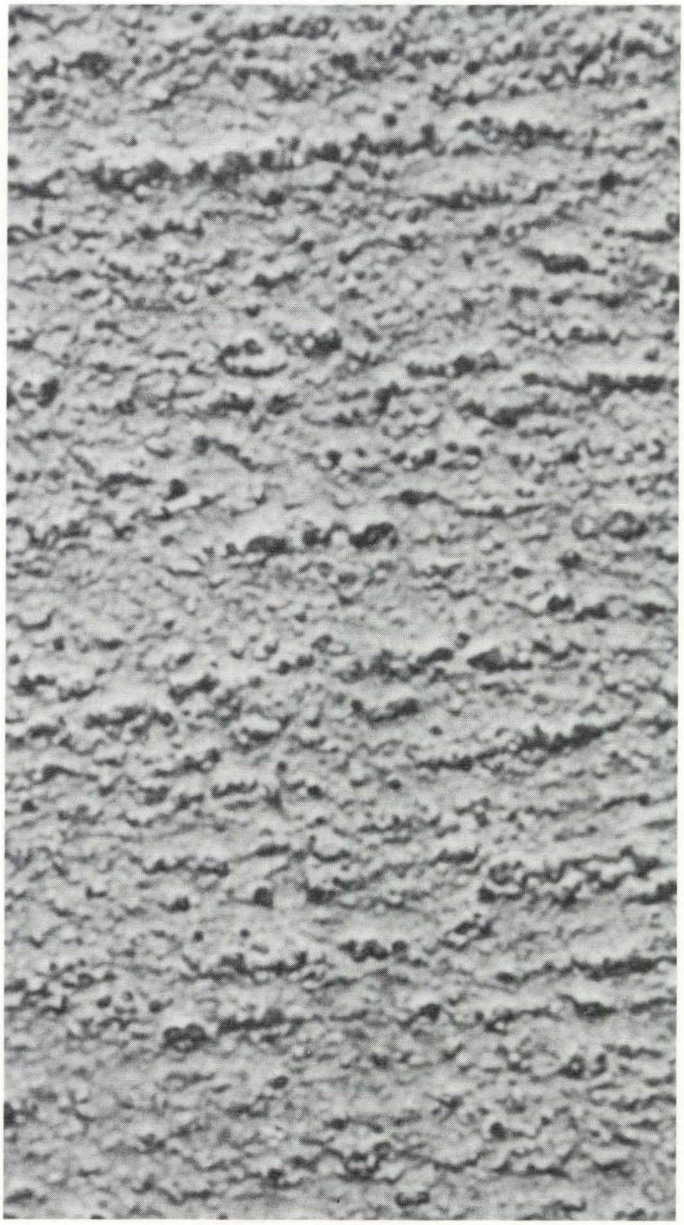


Figure 9B. Shadow cast replica of the surface of the typical computer tape shown in Figure 9A (1000X).

CROSS SECTION OF SURFACE TO BE REPLICATED

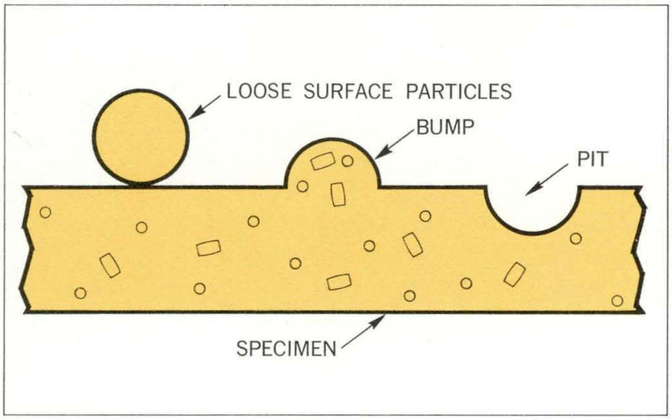


Figure 10A. Surface to be replicated showing a loose particle on the surface, a bump, and a pit.

PLASTIC REPLICA MATERIAL

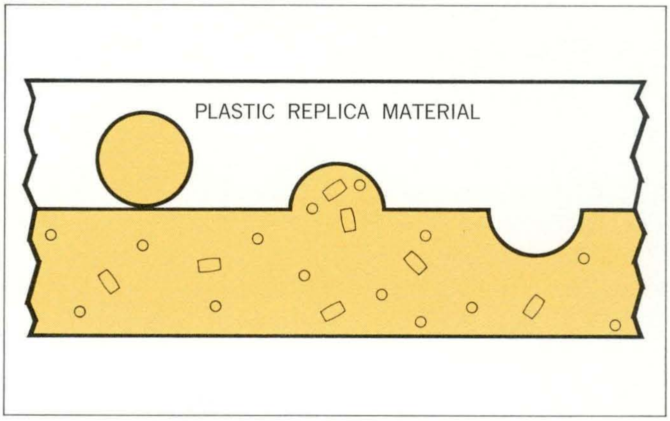


Figure 10B. Surface with replicating material applied.

NEGATIVE REPLICA AFTER STRIPPING FROM SPECIMEN

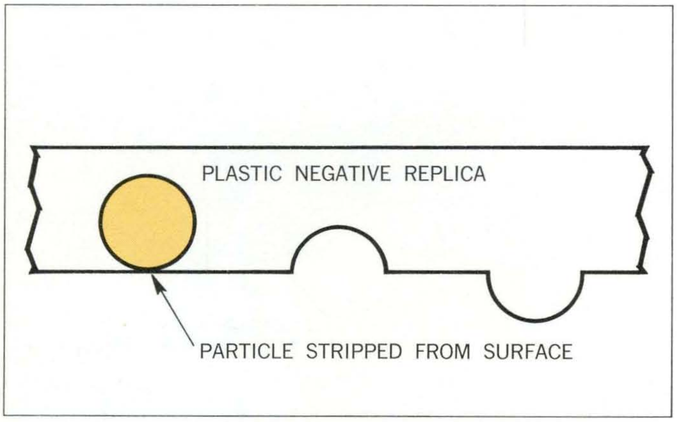


Figure 10C. Negative replica of a surface.

SHADOW CASTING BY EVAPORATION OF METAL

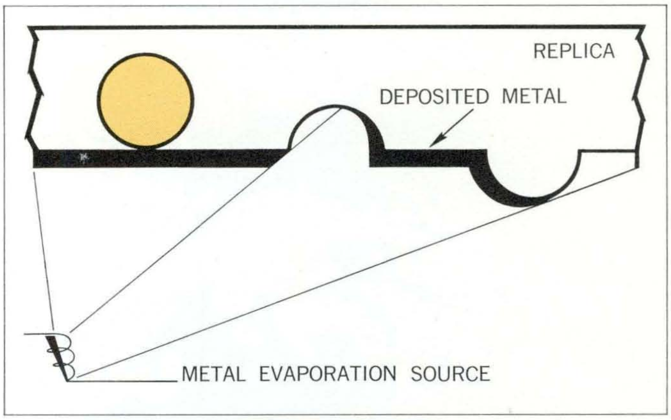


Figure 10D. Shadow casting of a negative replica by the evaporation of metal in a vacuum atmosphere.

EXPOSURE & RESULTANT SURFACE PHOTOMICROGRAPH

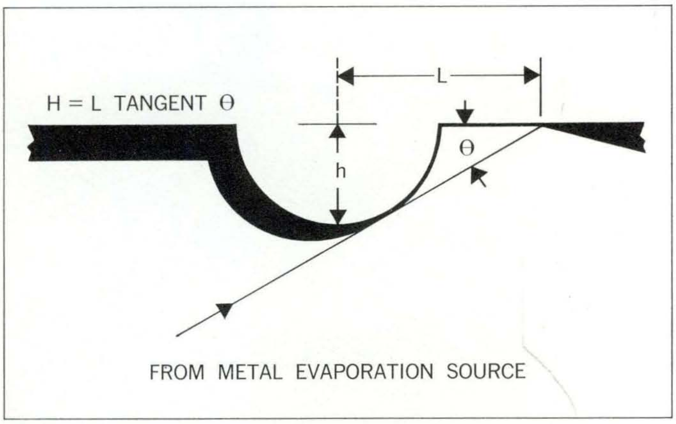


Figure 10E. Observation of a shadow cast negative replica.

HEIGHT (h) OF OBJECT FROM MEASUREMENT OF LENGTH (L) OF SHADOW

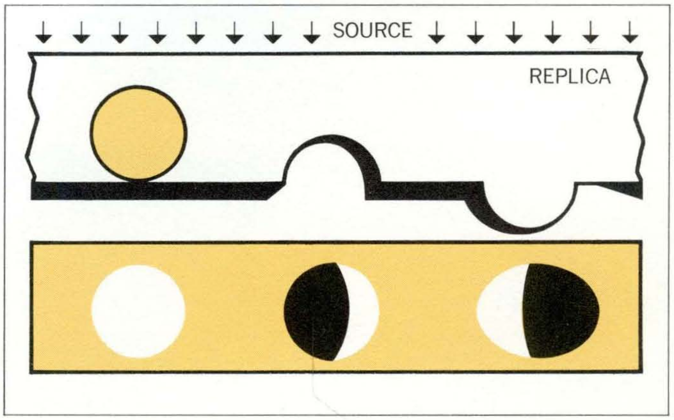


Figure 10F. Determination of the height of an object as seen on a shadow cast negative replica.

optical microscope

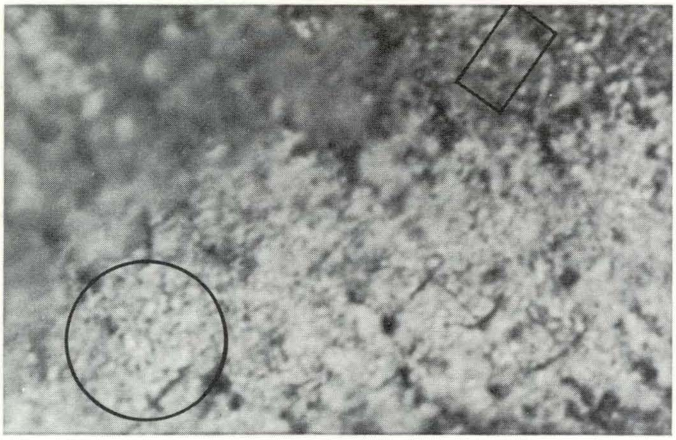


Figure 11A. Portion of a scratch on a tape surface viewed directly through the optical microscope (2000X).

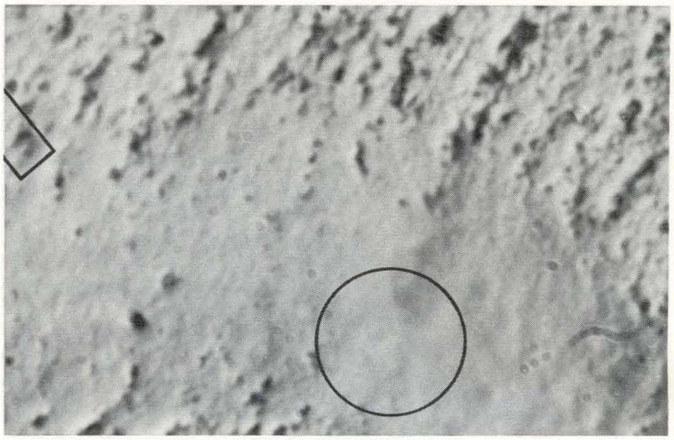


Figure 11B. Negative shadow cast replica of the scratched tape surface shown in Figure 11A as observed with the optical microscope (2000X).

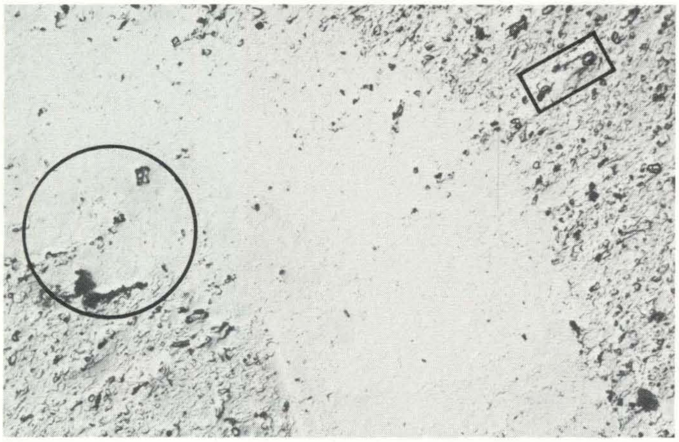


Figure 11C. Electron photomicrograph of the shadow cast replica showing the scratch on a tape surface as illustrated in Figure 11A (2000X).

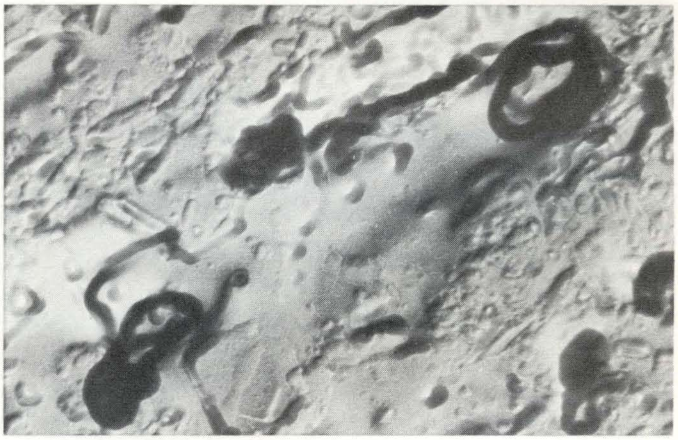


Figure 11D. An electron photomicrograph of the same scratch shown in Figure 11A but at a magnification of 20,000X.

The photomicrographs, Figures 11A through 11D, illustrate the potential of the shadow cast replication technique to observe the micrographic surface structure of a magnetic recording medium. Figures 11A and 11B represent the highest magnification commonly used with the optical microscope and depict the limit of resolution of that instrument, whereas Figures 11C and 11D are representative of a comparatively low magnification with the electron microscope. Many surface features can be seen in the four photomicrographs. Loose surface particles, bumps, holes, inclusions that protrude through the surface and a slight "roping" due to magnetic orientation are readily detected.

OPTICAL MICROSCOPE

The electron microscope is used to provide a detailed examination of magnetic recording tape. However, the high magnification necessary to bring out the fine detail has a disadvantage in that it is only possible to view a limited area in any one photomicrograph. Therefore, it is usually desirable to study the magnetic medium at lower magnification with the optical microscope to provide a complementary concept of the microscopic features.

The optical microscope is frequently used for viewing scratches and dropout-causing mechanisms, and for inspecting heads and surfaces of tape guides. A problem is encountered when the optical microscope is used to observe the smooth surface of a magnetic recording medium. If the sample is illuminated from any angle other than a low grazing angle, one finds a bright specular surface lacking in detail. Further, the angle of lighting cannot be well enough controlled to provide a quantitative measurement of the height of various surface features.

Replication techniques familiar to the electron microscopist have been employed to overcome many of these limitations. As in electron microscopy, the surface structure of objects which are too thick for transmission observation may be studied through the replication of their surface on a thin film. The shadow cast replica is effective for revealing surface structures not otherwise visible and allows a quantitative determination of the height of a roughness element by the measurement of the length of the cast shadow.

Figure 12A. Optical photomicrograph of the surface of shadow cast base film material (1000X).

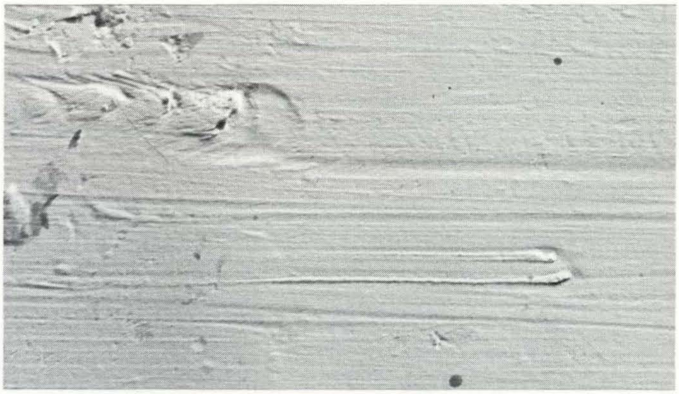


Figure 12B. Electron photomicrograph of a shadow cast negative replica of the surface of base film material (12,000X).

The optical photomicrograph, Figure 12A, illustrates many minute scratches existing in the surface of a base film material. The same features are visible in the electron photomicrograph, Figure 12B. Further, the electron photomicrograph illustrates some of the striking surface features in an apparently smooth material.

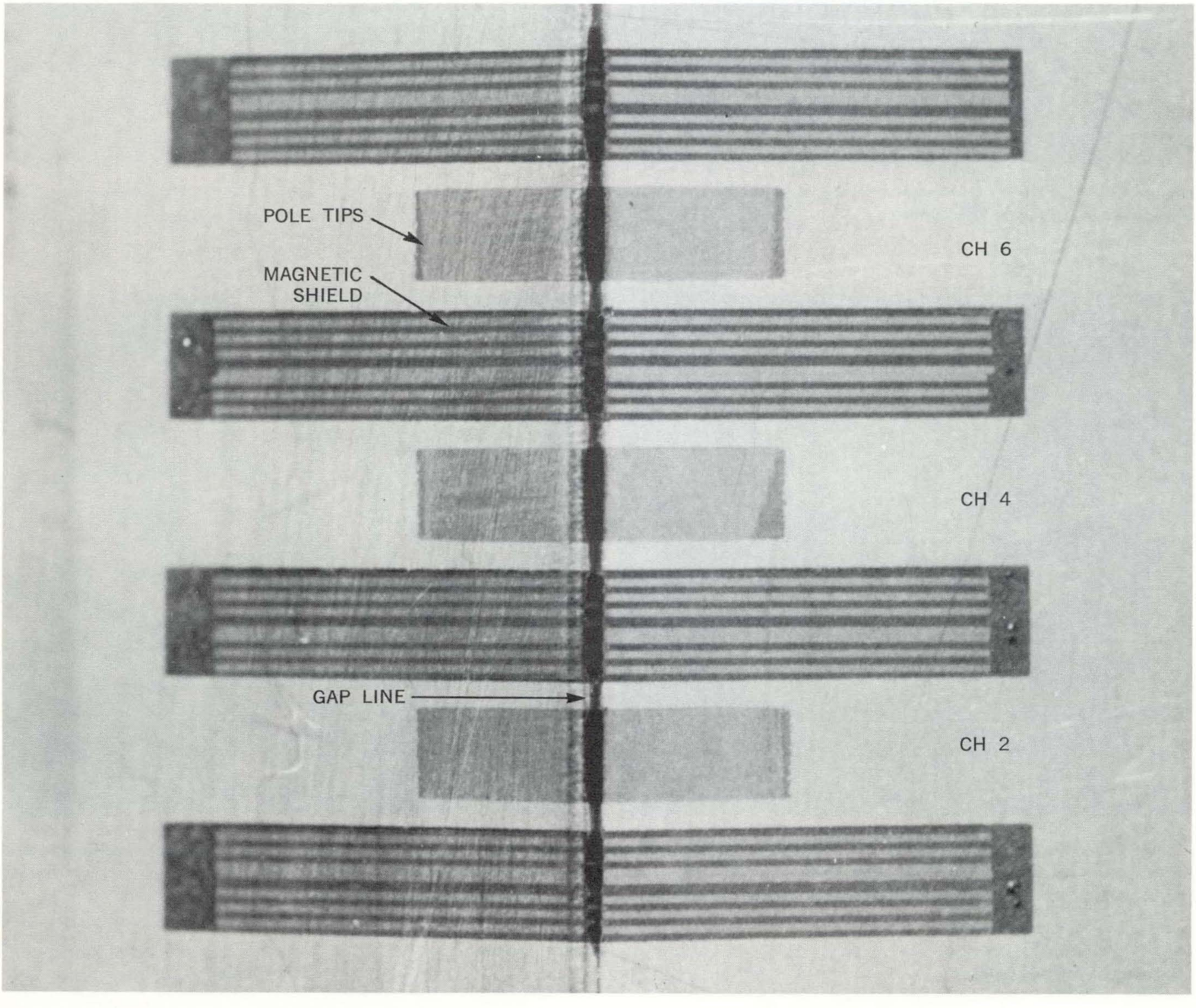


Figure 13A. Three-channel broadband reproduce head stack.

In examining the surface of a magnetic recording medium, two standard magnifications are generally required to give the most comprehensive evaluation. At Memorex, these two standard magnifications have become 155X and 790X. The lower magnification is useful for gaining an overall impression of the surface of the specimen, whereas the higher is useful for the presentation of a surface for quantitative analysis. It is left to the electron microscope to present a more detailed view of a given surface.

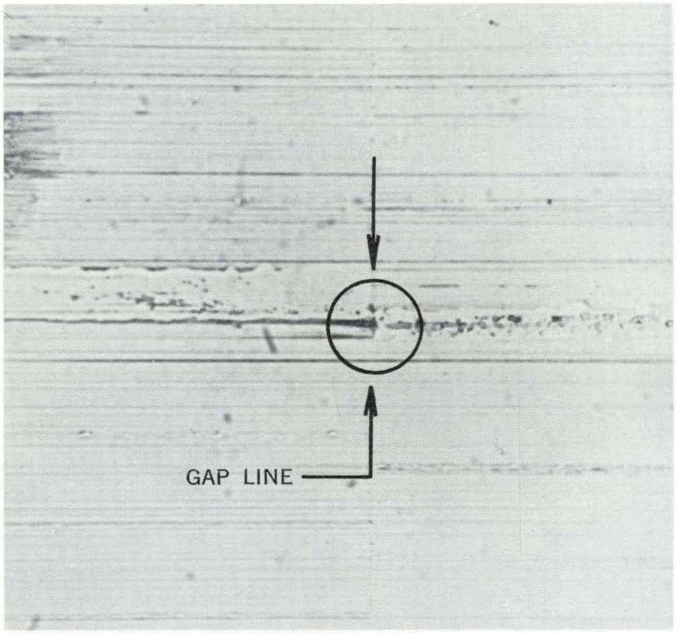


Figure 13B. Shadow cast replica of the gap of the broadband reproduce head of Figure 13A (200X).

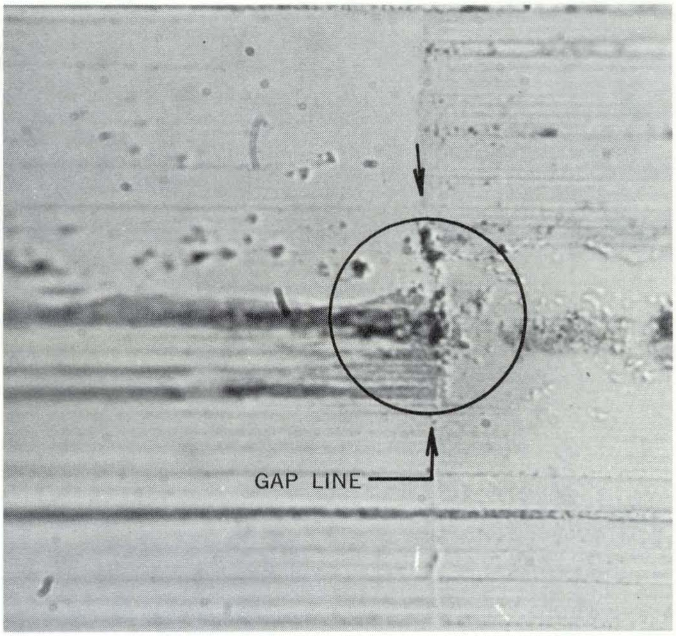


Figure 13C. A shadow cast replica of the broadband reproduce head of Figure 13A. Gap misalignment is readily apparent in this optical photomicrograph (1000X).

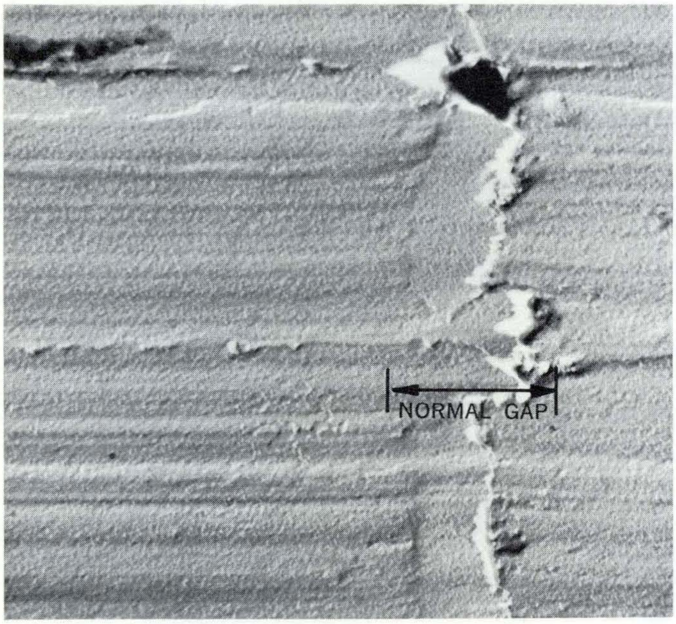


Figure 13D. Shadow cast replica of the broadband reproduce head illustrated in Figure 13A. Note the gap smearing. In this instance, the gap length has been reduced to about one-half of normal (20,000X).

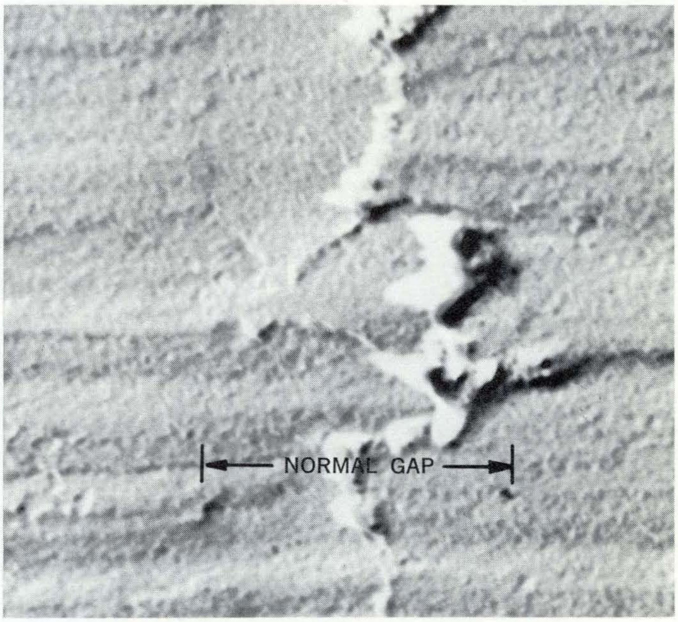


Figure 13E. Shadow cast replica of the broadband reproduce head illustrated in Figure 13A. Greater detail of the gap smearing is present at a higher magnification (40,000X).

The above illustrations demonstrate the usefulness of the shadow cast negative replication technique. In this instance, it is possible to study the microscopic detail of a precision reproduce head. The gap length of this head is of the order of one micron. This length has been reduced by the smearing of poletip material across the gap with consequent performance degradation. Further, Figure 13C illustrates the gap misalignment observed in one channel of this head.

surface instrumentation

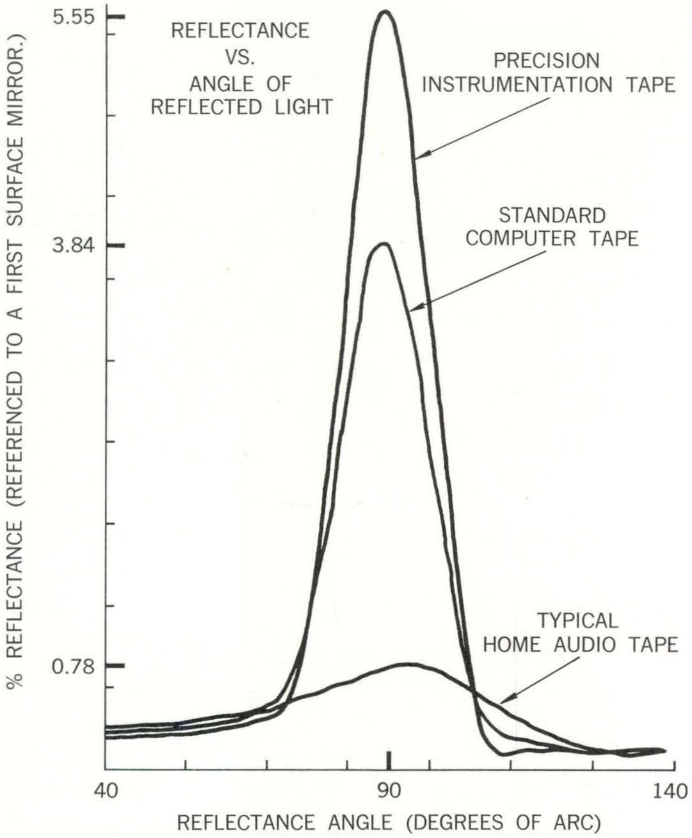


Figure 14. Reflectance versus angle of reflected light. The X-Y trace from an optical reflectometer. Full-scale (Y axis) represents the amount of light reflected from a first-surface mirror.

SURFACE INSTRUMENTATION

REFLECTOMETER

One characteristic of magnetic recording media to which the human eye is quite sensitive is the gloss or reflectance of a surface. The eye provides a judgment of one surface next to another, or an estimate of the uniformity of a given surface. However, judgment from one day to the next or comparison between nearly similar products is difficult.

Memorex has designed a reflectometer that projects the image of a slit of light (measuring 0.002 inch by 0.050 inch) onto the surface to be studied. The light reflected from the sample is then picked up by means of a flexible section of fiber optics. The fiber optic pick-up is swung through a 100° arc above the surface to be observed. The light transmitted by the fiber optic tube is sensed by a photo-multiplier. The output signal is amplified and used as the Y coordinate on an X-Y plotter. The angle of arc of the fiber optic pick-up is plotted as the X coordinate. Although surface roughness is a contributing factor to reflectance, constituents and manufacturing methods are the major elements determining the optical characteristics of magnetic recording material. Thus, the reflectometer is useful in the evaluation of tape surfaces in that it provides a quantitative determination that is consistent from day to day, sensitive enough to provide valid comparisons between similar products and suitably stable for the observation of surface uniformity.

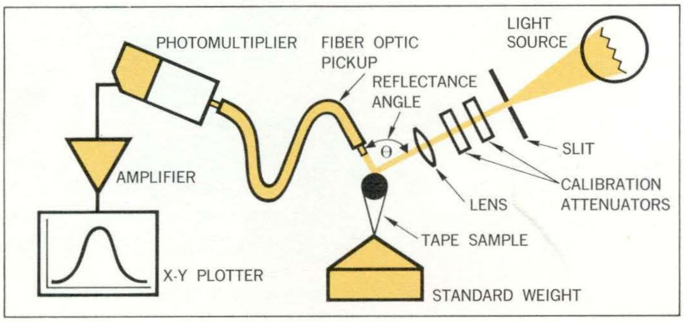


Figure 15. Memorex reflectometer.

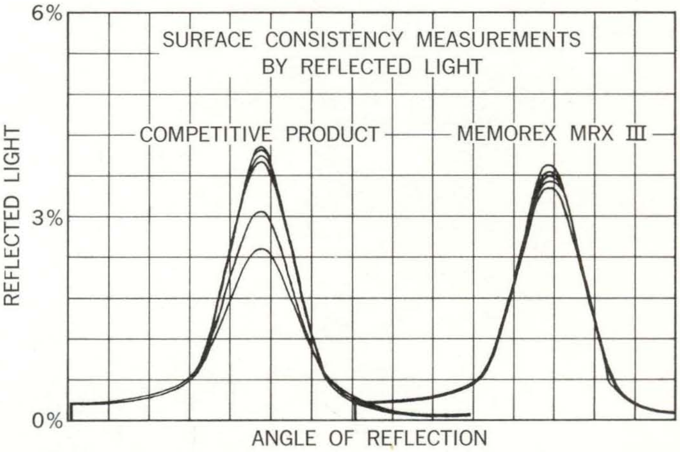


Figure 16. A reflectance comparison, between two of the more popular high precision magnetic recording tapes demonstrating surface uniformity for various points along the samples.

STYLUS-TYPE PROFILOMETER

The stylus-type profilometer is a device used to measure surface roughness. A needle-sharp diamond (0.0001 inch radius, 0.1 gram pressure) is caused to traverse a given surface. The vertical motion (2.5 microinches per 0.25 inch) of the stylus caused by irregularities of the surface is transduced, amplified, and used to reproduce a magnified profile of the surface. This profile measurement can be the basis for a specification concerning the surface of magnetic recording media.

In the description of a magnetic recording surface profile, center line average (CLA) and peak-to-valley roughness measurements give a good characterization of a surface. The peak-to-valley measure provides an indication of the minimum separation between a magnetic record-reproduce head and the bulk of the magnetic material in the medium. The center line average involves a determination of the average area enclosed by the total profile. A high center line average reading is indicative of a high frequency "sharp" profile.

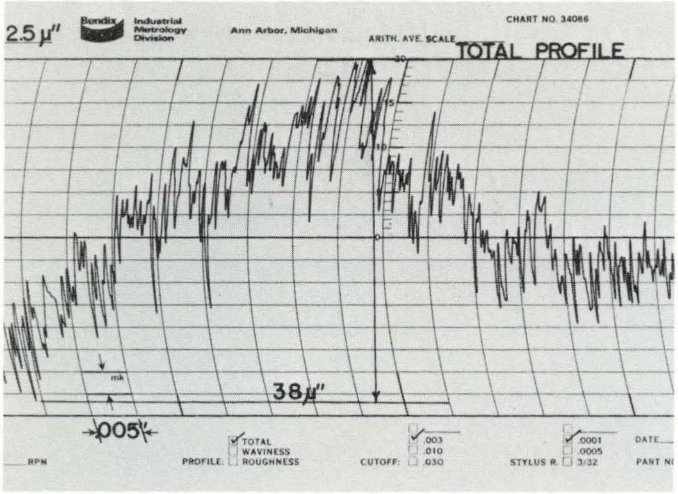


Figure 17A. Stylus-type profilometer trace showing the total profile of a tape surface with a drop-out causing defect at the surface.

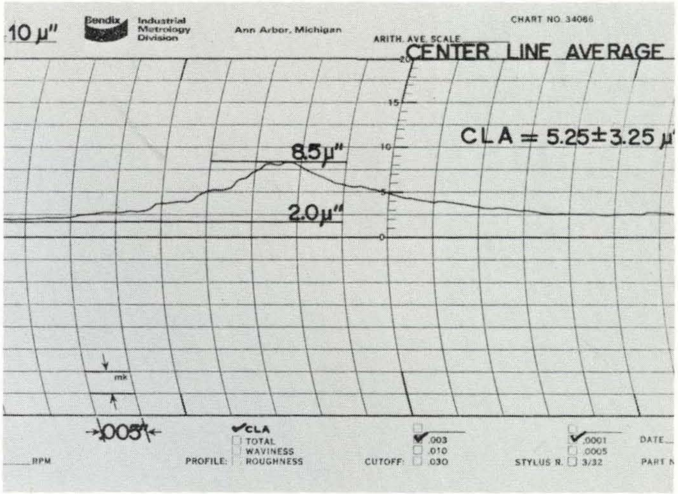


Figure 17B. Stylus-type profilometer trace showing the Center Line Average profile of the tape surface of Figure 17A.

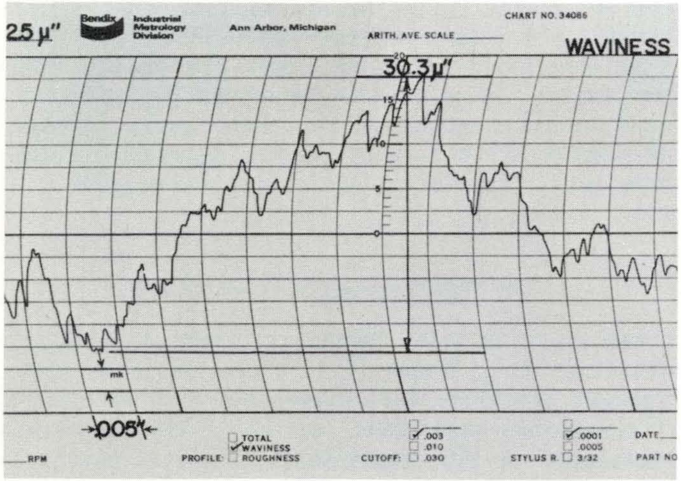


Figure 17C. Stylus-type profilometer trace. Waviness profile of the tape surface of Figure 17A.

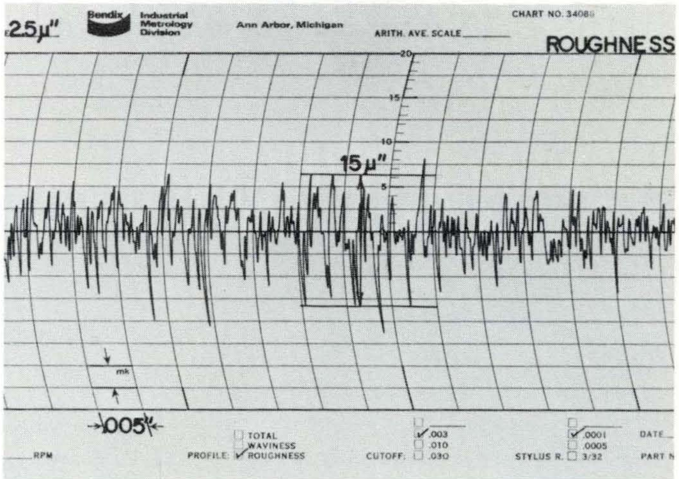


Figure 17D. Stylus-type profilometer trace. A roughness profile of the tape surface of Figure 17A.

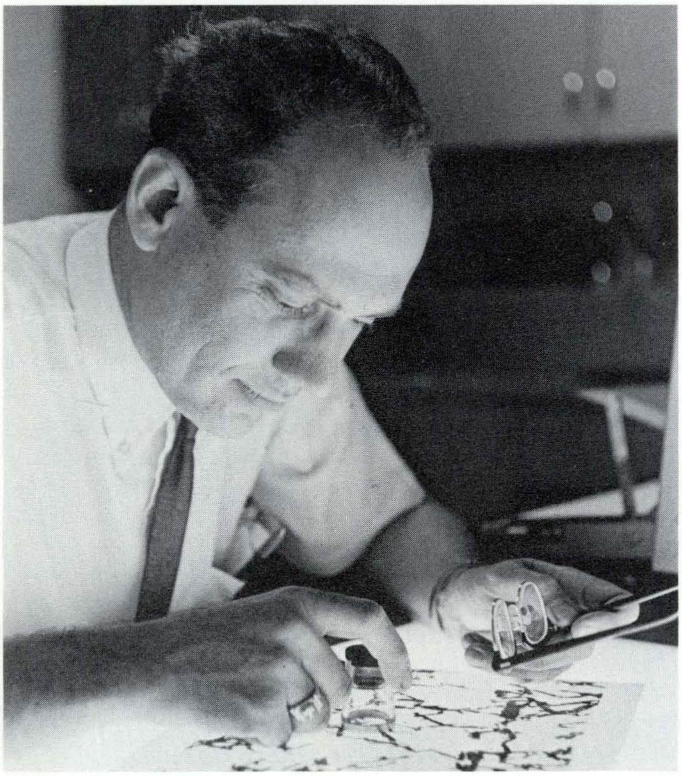
A low center line average reading is indicative of a low frequency "undulating" surface profile. The variation of the center line average from one region to the next provides a measure of the uniformity of the surface roughness. A surface examination of Memorex and leading competitive products (at the time of writing) results in the following stylus-type profilometer ranges:

Computer Tape	11-15μ″ peak-to-valley 2-3μ″ center line average ±0.25 to ±0.75 μ″ CLA uniformity
Instrumentation and Video Tape	4-10μ″ peak-to-valley 1-2.5μ″ center line average ±0.25 to ±0.50μ″ CLA uniformity
Magnetic Recording Discs	6-15μ″ peak-to-valley 1.5-3.5μ″ center line average ±0.25 to ±1.5μ″ CLA uniformity

The stylus profilometer depends upon the ability of a diamond point to follow the profile of the surface. A stylus must have some pressure to force it to follow the profile, or it will bounce over a bump and not return to the surface. The load on a 0.0001 inch radius stylus is typically one-tenth gram. This gives a local pressure of several thousand pounds per square inch. Like an indention hardness test, the stylus-type profilometer senses hardness as well as roughness elements. In examining plastic materials, hardness is especially prominent for profiles of less than 30 microinches peak-to-valley. Comparison with optical surface measurements shows as much as a two to one difference between indicated and actual profiles. Although the stylus-type profilometer does not give an exact representation of a profile, it is still a valuable instrument to be considered when comparing a set of recording medium specifications. The measurements are reproducible within a given formulation and are suitable for quality control and comparison purposes. It is left to the optical and electron microscopes to observe the true surface of a magnetic recording medium.

CONCLUSION

This monograph has presented some of the techniques developed to observe the subtle properties that characterize high-quality, precision magnetic recording materials. These micrographic techniques are employed in the control of the high quality of Memorex products from magnetic materials and plastic resin systems to the final test of tapes, discs, and other components. One of the striking features of a precision magnetic recording medium is that each step in its production must continue flawlessly or the product will be unusable. There is no possibility to go back and rework as in equipment manufacture. It is this single fact which makes the production of premium quality magnetic recording materials one of the most exacting tasks in the world. The micrographic techniques discussed here have helped Memorex to meet this state-of-the-art challenge and to continue its position of leadership in the production of magnetic tape, cards, and discs of premium quality.



ACKNOWLEDGEMENTS

I wish to thank D. K. Stellman, Supervisor of Micrographics; G. L. Toms, R. L. Warncke, J. H. Vega, Optical Laboratory Technicians; and R. G. Mendonca, Photographic Technician, for their cooperation and assistance in the preparation of the photomicrographs and other materials for this monograph.

ABOUT THE AUTHOR

Donald C. Gaubatz is Manager of Physical Research, Memorex Corporation. His responsibilities include operation of the facility for analysis of recording materials, development of magnetic standards, and design of special test equipment and devices.

He received his degree in Physics from the University of California. He is professionally associated as a member of IEEE, Magnetic's Group; American Vacuum Society; and the Thin Film Society of Northern California.

M606 20M 10/68 CA Printed in U.S.A.