Model 590

Application Laboratory Report 11

Preparation of Hard Disk Drive Magnetic Recording Media for XTEM Analysis

Sample Preparation

1.0: Purpose

To develop and help refine a TEM specimen preparation protocol for the successful production of XTEM specimens of magnetic recording hard disk drive materials. The specimen preparation process should be relatively fast, produce high quality specimens with large thin areas, and be repeatable. Several processes and methods will be attempted with all of them evaluated based on the given criteria. Upon successful completion of specimens TEM micrographs will be presented to show the quality of the specimen produced.

2.0: Materials and Methods

Several different methods and materials equipment were used in this experiment to help characterize the ideal preparation process for these materials. The disk media which was to be sectioned was a 3.7" (94mm) diameter magnetic hard disk drive. The specimen was approximately 800 µ thick prior to cutting and sectioning. Figure 1 shows a schematic cross section of the disk drive media which is to be prepared in cross section.

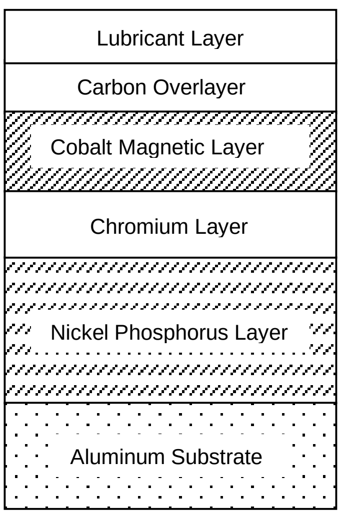

Figure 1: Schematic cross section of the magnetic media to be prepared in cross section. The layers of primary interest are the top four layers which include: Carbon protection layer, Cobalt magnetic layer, and the Chromium under layer. The NiP and Aluminum layers are not of major interest, although ideally they should be uniformly thinned as well.

Cutting and Sectioning

Several approaches to cutting the disk media could be taken, all depending upon the amount of tolerable damage, efficiency of process, cutting time, and repeatability. Based on these criteria several different cutting methods were employed to evaluate the best method. These methods included Diamond Band Sawing, Wire Sawing, Ultrasonic Cutting, and Core Drilling. All four methods have certain advantages and disadvantages based upon the criteria listed above. Below is a chart outlining the results obtained from the various cutting processes used.

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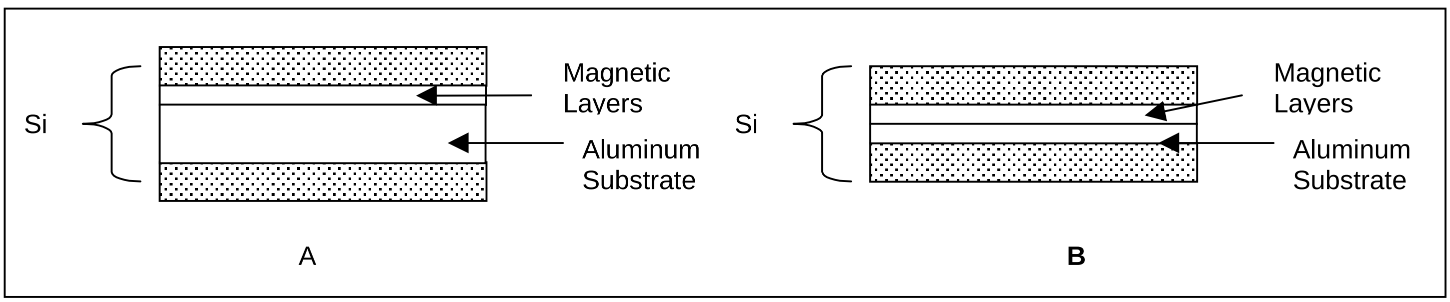
Instrument	Abrasive Size / Type	Cutting Time (per cut)	Damage	Number of Cuts for 1 Sample	Rating	Comments
Model 860 Diamond Band Saw	~ 75 µ / diamond	1.5 minutes	High	4 total	Poor	Fast cutting; high damage; good for general bulk reduction
Model 650 Low Speed Diamond Wheel Saw	~ 45-55 µ / diamond	15-20 minutes	Low	4 total	Good	Slow cutting; low damage; poor throughput
Model 850 Wire Saw	14 µ / boron carbide	2.5 minutes	Low	4 total	Excellent	Fast cutting; low damage; high specimen throughput
Model 380 Ultrasonic Disc Cutter	23 µ / boron carbide	35-40 minutes	Medium	1	Poor	Slow cutting; medium damage; somewhat unreliable with Al
Model 360 Abrasive Slurry Disc Cutter	23 µ / boron carbide	5-10 minutes	Medium/Low	1	Good	Slow cutting; low damage; only produces discs

Based upon the evaluation of cutting processes, it was determined that using the Model 850 Wire Saw was the best method. Delamination of the top films of the specimen during processing is a problem which was encountered using the other, more aggressive techniques such as ultrasonic cutting and diamond band sawing. The Model 850 was very gentle on the films and produced no delamination which was evident during final thinning processes.

Configuring the Specimen

During the cutting process the specimens were cut into 4mm x 6mm rectangles (approximately). Following this process, there were two approaches taken. In the first approach, the specimens were sandwiched between two pieces of blank Si which were used to both monitor the specimen thickness during thinning and to provide support to the specimen at the final stages of preparation. The second approach was the same except the Al portion of the specimen was mechanically ground to within 10 µ of the NiP layer. This was done to reduce any problems which may have been associated with the Al substrate such as specimen curling, uneven thinning rates, and delamination from the Si. Following the pre-preparation of these parts they were epoxied together using Epotek 353 ND. Below is an illustration of the specimen configurations used in this procedure.



Final Thinning

Once the specimen had been configured in the manner desired, the specimens were then final thinned using the Model 590 Tripod Polisher™. The Tripod™ technique was chosen for many reasons. It is very site specific which will be needed for future preparations of these disk drives; it allows mechanical thinning of a wide range of materials at or near electron transparency thicknesses; is relatively inexpensive and is relatively fast compared with other techniques.

Using the Tripod Polisher™ to final thin the specimen can be done several ways. The basic process involves thinning one side of the specimen using finer and finer diamond abrasive films to produce a very smooth, flat surface without any scratches or topography. This is important as the quality of the first side polish dictates the overall quality of the final TEM thin section. Once the first side has been polished smooth, the specimen is then flipped over and thinned down again at a slight angle. This produces a thin portion at the top of the specimen which becomes progressively thicker towards the back of the specimen.

Adjustment of the amount of tilt angle which is placed onto the specimen can be done at the user's discretion. Traditionally steep wedge angles have been used for difficult to thin materials such as sapphire, InP, and others. Shallow wedge angles are typically used for sturdy, easy to thin materials such as Si and SiC. In this case the wedge angle used was relatively small (about 10-25 small divisions). A small wedge angle was used to allow the maximum amount of material to be removed and to minimize any ion milling time which may be needed at the final stages of preparation.

The process flow for preparing a TEM section using the Tripod Polisher™ is shown below:

First Side Polish	Second Side Polish
30 micron diamond film	30 micron diamond film to within 500 microns of surface
15 micron diamond film	15 micron diamond film to within 250 microns of surface
9 micron diamond film	9 micron diamond film to within 100 microns of surface
3 micron diamond film	3 micron diamond film to within 25-40 microns of surface
1 micron diamond film	1 micron diamond film to slightly reddish color in Si
0.5 micron diamond film	0.5 micron diamond film to 1/2 removal of Si top layer
0.05 Colloidal Silica on Multitex Cloth	0.05 Colloidal Silica on Multitex Cloth to fringes

Once the specimen had been polished to the desired thickness, the specimen was then removed and mounted onto a TEM grid using MBond 610. The final thickness still needed to be reduced using ion milling techniques, and will be discussed in the next section.

Ion Milling

Following the final thinning and mounting of the specimen from the Tripod Polisher, ion milling techniques were employed to thin the specimen for TEM viewing. The advantages obtained with low angle ion milling such as minimizing atomic number and topographic thinning effects, lower heat production, and smoother surfaces are well established. Using the Model IV3 Research Grade Ion Milling Instrument these advantages are easily taken into practice. The IV3 allows very low angle milling coupled with high powered, low kV capability, making it ideally suited to a variety of material systems. The following parameters were used with the specimens prepared:

Gun Voltage:	6 kV
Gun Current:	4 mA
Incident Angle:	5 degrees
Beam Current:	30 microamps
Rocking Angle:	50 degrees
Process Time:	2 hours

3.0: Conclusions

Using these processing steps high quality TEM specimens of hard disk media were obtained and are easily reproduced. Production of these types of specimens can be completed in 1 day using the techniques described here.

- If you found this information useful, please contact:

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