Effect of Substrate Cleanliness on Permalloy Thin Films*

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The use of mechanical and chemical cleaning techniques in the production of a clean, random, and smooth microslide glass substrate surface for the vacuum deposition of uniaxial thin Permalloy film elements has been studied. The principal cleaning techniques intensively investigated were: (1) a mild detergent wash followed by a vapor degreasing cycle in isopropyl alcohol; (2) a chalk paste scrub followed by an ultrasonically agitated distilled water rinsing cycle and a forced hot air drying process; and (3) hydrofluoric acid etching followed by a distilled water rinsing cycle and a forced air-drying process. The surface condition of the cleaned microslide glass was assessed with electron micrographs of preshadowed carbon replicas (magnification 88,000×). The micrographs show that the chalk-cleaned glass substrate has the smoothest surface. The variously cleaned substrates were then used for the vacuum deposition of 1700-A 4-mm-diam Permalloy film elements (melt composition 83% nickel, 17% iron). This work was carried out in a 10⁻⁴ mm Hg vacuum system. Twenty-five evaporation processes were made with a total of 54 elements per evaporation. The Permalloy film elements deposited on the differently cleaned glass substrates were then examined by means of a 1000-cycle hysteresis loop apparatus and the following measurements were made: (1) coercive force \( H_C \); (2) saturation flux \( \Phi_s \); (3) orientation of the anisotropy axis \( \theta \); (4) magnetoelastic strain coefficient \( \nu \); and (5) anisotropy field \( H_K \). Dispersion measurements of the easy axis were made on a 1000-cycle crossed field hysteresis loop apparatus. Measured in this manner, the chalk-cleaned glass substrates yielded consistently the lower average value of \( H_C \); i.e., 1.4 oe. The average value of \( H_K \) for the three different cleaning techniques was 2.5 oe. Within the total range of values of coercive force and the anisotropy field, the chalk-cleaned and the acid-etched glass substrates yielded the same values; i.e., \( H_K \leq 0.2 \) oe, \( H_K \leq 0.3 \) oe. Measurements of angular dispersion varied from 8° to less than 2° with an average value of less than 3°. The chalk-cleaned substrates yielded the lowest values of angular dispersion.

INTRODUCTION

A VARIETY of mechanical and chemical cleaning techniques has been used for the preparation of an absolutely clean, random, and smooth glass substrate surface for the vacuum deposition of thin magnetic films. T. Putter has reported that a detergent washing combined with vapor degreasing produces glass surface cleanliness nearly equal to that of discharge cleaning. K. Behrndt and F. S. Maddocks report that chemical cleaning alone may not be sufficient to produce the homogeneity needed for the reproducibility of magnetic parameters of thin magnetic films. They found an SiO coating after chemical cleaning decreased the scattering of coercive force \( H_C \). S. Nielson has reported that vacuum melted glass yields a smooth surface and better reproducibility of magnetic parameters for thin magnetic films. J. C. Lloyd and R. S. Smith have reported a correlation between substrate roughness and coercive force \( H_C \) and anisotropy field \( H_K \) for electrodeposited films.

The cleaning media studied in this investigation are the following: a solution of hydrofluoric acid and water or nitric acid; a sulfuric acid and chromic acid solution; a solution of sodium hydroxide, water, and alcohol; sodium carbonate; a detergent wash and vapor degreasing and a chalk scrub. Each of the foregoing cleaning techniques was investigated to determine the relative cleanliness and smoothness it is capable of producing on a glass substrate and the resultant effect on the magnetic parameters of the uniaxial Permalloy thin films.

EXPERIMENTAL

The test substrates were 1X3-in. soda lime microslide glass. The surface condition of the cleaned microslide glass substrates was assessed by their wetting properties and electron microscopy. The preshadowed carbon replicas (magnification 88,000×) indicate that the chalk-cleaned and the sulfuric-chromic acid etched glass substrates yield the smoothest surfaces. The chalk cleaning consisted of rubbing a chalk paste onto the glass substrate surface, then rinsing the substrate with ultrasonically agitated distilled water, and finally drying with forced hot air. From the resulting micrographs, wetting properties, and preliminary vacuum studies, three different cleaning procedures were chosen for intensive investigation of the magnetic parameters of vacuum-deposited Permalloy thin films. The procedures used were: (1) an acid etching (2% hydrofluoric acid at room temperature); (2) a simple washing (mild detergent and vapor degreasing); (3) a mechanical cleaning (chalk scrub). Micrographs of these three glass surfaces are shown in Fig. 1. After a series of slides was cleaned by each of the three procedures outlined above, an array of eighteen 4-mm and one 8-mm-diam Permalloy film element was then vacuum deposited on each slide. The evaporation was made in a glass chamber in which oil diffusion pumps yield a 10⁻⁴ mm Hg ultimate pressure as measured by an ionization gauge tube. The evaporation process

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source was 83% nickel, 17% iron wire wrapped around a radial tungsten wire. The test substrates were baked in vacuum for two hours by three radiative infrared bulbs at a recorded temperature of 375°C. An iron­Constan tan thermocouple junction cemented with Saureisen cement to a microslide glass was used as a temperature sensor. Deposition was initiated by passing a current through the tungsten filament and was continued for approximately 30 sec in a 100-oe magnetic field. Film thickness was approximately 1700 Å as measured by a crystal monitor.  

Next, 24 consecutive evaporations were made, and the resulting films were measured in a 1000-cycle hysteresis loop tracer. A crossed field 1000-cycle hysteresis loop tracer was used to measure dispersion of the "easy" axis for the one 8-mm-diam Permalloy film.

RESULTS

Table I shows the average value, the maximum value, the minimum value, the standard deviation, and the percentage of standard deviation for the coercive force $H_C$ and the anisotropy field $H_K$ for a single evaporation and for a series of 25 evaporations. All of the eighteen 4-mm-diam Permalloy films were tested on each test microslide glass substrate for the first evaporation, whereas for the next 24 evaporations, only six of the eighteen 4-mm-diam Permalloy films were tested in the

<table>
<thead>
<tr>
<th>Method of cleaning</th>
<th>Acid</th>
<th>Detergent</th>
<th>Chalk</th>
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<tbody>
<tr>
<td>Coercive force</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Average value (oe)</td>
<td>1.7</td>
<td>1.7</td>
<td>1.5</td>
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<tr>
<td>Maximum value (oe)</td>
<td>1.9</td>
<td>1.7</td>
<td>1.5</td>
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<tr>
<td>Minimum value (oe)</td>
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<td>1.2</td>
<td>1.1</td>
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<tr>
<td>Standard deviation</td>
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<td>0.30</td>
<td>0.09</td>
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</table>

<table>
<thead>
<tr>
<th>Angular dispersion</th>
<th>Average value (deg)</th>
<th>Maximum value (deg)</th>
<th>Minimum value (deg)</th>
<th>Standard deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>3.9°</td>
<td>7.3°</td>
<td>2.2°</td>
<td>1.1°</td>
</tr>
</tbody>
</table>

1000-cycle hysteresis loop tracer. These six were chosen in a sequence so that after every third evaporation all of the Permalloy films had been tested for a given substrate area. With this procedure any variation in film thickness or temperature is accounted for in the mathematical analysis. The cleaning techniques did not appear to alter $H_K$ values significantly. The chalk-cleaned microslide substrate produced the lowest measured average value, the least deviation, and the lowest percent of standard deviation for the coercive force $H_C$. The acid-etched substrate has a measured deviation of $H_C$ equal to the chalk-cleaned substrate. (Hysteresis loop tracer reliability ±5% for $H_C$.) These values are typical for more than one hundred evaporations in which a variety of vacuum systems and vacuum procedures were used.

Table I also shows the measured average value, standard deviation, and percentage of standard deviation of $\theta_{90}$, the angular dispersion of the "easy" axis. The chalk-cleaned microslide glass substrates yielded the lowest average (3.2°) value and lowest percent of standard deviation (21%) of the three cleaning techniques, i.e., acid 3.9°, 27% detergent 4.4° 30%. However, the values of $\theta_{90}$, the angular dispersion of the "easy" axis, are not of a magnitude as great as the values of coercive force.

CONCLUSIONS

Thin Permalloy magnetic films have been vacuum deposited onto glass substrates which have been cleaned in a variety of ways. Chalk-cleaned glass substrates have a very smooth and random surface as viewed by an electron microscope. In measured values of coercive force, the chalk-cleaned glass substrates yield the lowest average value, the least amount of scattering, and even more important better reproducibility from evaporation.