Evaporatively Deposited Co-MgF2 Granular Materials for Thin-Film Inductors

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Evaporatively Deposited Co-MgF_2 Granular Materials for Thin-Film Inductors

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***Abstract*--Co-MgF_2 granular thin films prepared by thermal evaporation are reported. The fabricated materials show promise as high-frequency, low-loss cores for microfabricated magnetic devices in microprocessor power delivery applications. Transmission electron microscopy (TEM) reveals Co grains of about 3.2 nm embedded in a MgF_2 matrix 1 to 2 nm wide. Films were prepared with 20% to 89% volume cobalt at substrate temperatures between -21°C and 311°C. Good soft magnetic properties in a range of 40% to 55% volume cobalt with high dc resistivity of 500 μΩ-cm to 100 Ω-cm are measured for films deposited at room temperature. Low intrinsic coercivity, H_{ci} of 0.76 Oe and susceptibility, χ of 26 in the easy axis direction with quality factor, Q over 100 and saturation flux density, B_s of 0.7 T are obtained.**

***Index Terms*—Co-MgF_2, evaporation, evaporative deposition, granular magnetic film, inductors, thin films.**

I. INTRODUCTION

RECENT work on metal-nonmetal granular soft magnetic films has attracted much interest [1]-[9]. The combination of moderate susceptibility (χ) up to high frequencies, high electrical resistivity (ρ), large saturation magnetization (B_s), and low coercivity (H_c) make them attractive for a wide range of applications. Previous work on the magnetic properties of such films have primarily used sputtering [1]-[9]. Good magnetic properties including susceptibilities of as high as 1000 up to hundreds of MHz, electrical resistivities over 500 μΩ-cm, saturation flux densities of about 1 T, and coercivities as low as tenths of an oersted have been achieved [2],[4]-[9].

These materials are particularly promising for inductors used in high-frequency power converters [11]-[13]. Our design analysis [12],[13] indicates that they will enable dramatic improvements in performance compared to inductors built with conventional thin-film materials [11], particularly for addressing the emerging problem of supplying high current (near 100 A) at low voltage (near 1 V) for future microprocessors and other digital electronics.

For high volume production of relatively thick films (e.g. 10 μm), vacuum evaporation may be preferable to sputtering. To investigate this possibility, we studied thermal evaporation of Co-MgF_2 films. Magnesium fluoride was chosen instead of alumina or other ceramics typically used in similar sputtered films because it is easily evaporated, because of its propensity to separate from a co-evaporated metal, and because its use in films for optical applications is well established. Cobalt was used as the magnetic metal due to its high saturation flux density, low vapor pressure as compared to other ferromagnetic materials, and moderate susceptibility, which is desirable for high-frequency operation and for minimizing conductor AC-resistance effects in inductors [10],[11].

In this paper, the structural, electrical, and magnetic properties of evaporatively deposited Co-MgF_2 films are presented for a range of compositions and substrate temperatures during deposition.

II. DEPOSITION

Granular soft magnetic Co-MgF_2 films were prepared by evaporative co-deposition of cobalt and magnesium fluoride onto glass substrates. Cobalt was deposited from an alumina-coated tungsten wire crucible while magnesium fluoride was deposited using a tungsten boat. The two sources, positioned 9 cm apart, were located 22 cm below the substrate. To avoid mixing of the two materials before contact with the substrate, a 13 cm high divider was placed between the sources. Composition was controlled by varying the relative deposition rates as monitored by two quartz crystal monitors. Cobalt deposition rates were typically 5 Å/s; magnesium fluoride rates were between 0 and 8 Å/s. Higher deposition rates would be used in production. Film compositions ranged from 20% to 89% volume cobalt. A custom designed Halbach array measuring 75 mm in diameter was used to induce a highly uniform, 400 Oe magnetic field throughout the substrate region. Base pressures of 2 x 10^{-6} Torr were achieved with a diffusion pump. Substrate temperature was controlled by a resistive heater block to which the substrate was clipped. A thermocouple was used to monitor the heater block temperature, which ranged from -21ºC to 311ºC.

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III. ANALYSIS TECHNIQUES

Magnetic properties of the films were measured using a vibrating-sample magnetometer (VSM). Resistivity of the films was determined by the four-point probe method. A JOEL 100CX transmission electron microscope (TEM) was used to examine films with 200,000x magnification at 200 keV accelerating voltages with resolutions as good as 0.1 nm. About 100 Å of the overall deposition was deposited onto Formvar coated copper TEM grids for this analysis. Energy dispersive x-ray spectroscopy (EDS) was also carried out with this TEM to determine elemental composition. Analysis of TEM images was performed using NIH image software where a fast Fourier transform (FFT) of 256 x 256 pixels was used to determine cluster size. Stylus profilometry was performed on the films after deposition. A knife-edge technique was used at the edges of the samples to re-confirm relative percentage compositions in the films [13]. Film adhesion was also tested. A Crale high-frequency permeameter was used with an HP 8753C network analyzer and an RF preamplifier for permeance measurements up to 300 MHz. Our equipment limited the high-frequency measurements to small-signal excitation only.

IV. RESULTS AND DISCUSSION

A. Structure

In TEM images (Fig. 1), a network-like structure of nanoscale grains of Co (dark areas) and thin intergrain boundaries of MgF_2 (light areas) can be seen. The grain size is between 2.7 and 3.7 nm in diameter with intergrain regions between 1 and 2 nm wide.

Energy dispersive x-ray analysis (EDS) has been performed using the TEM elemental analysis tool. A typical energy spectrum for these materials is shown in Fig. 2. The K-α lines for Co, Mg, and F are most pronounced. The copper peak is a result of the TEM grid material. The small peak between Co and Cu in this plot is confirmed to be a Co L-α line. Small, although not insignificant percentages of C and O are also present in the final film.

B. Electrical Resistivity

The percolation threshold occurs at about 60% volume fraction cobalt. For favorable magnetic properties, films with 35% to 50% volume Co are preferred. In this range, dc resistivities of 1,000 to 100,000 μΩ-cm are obtained,

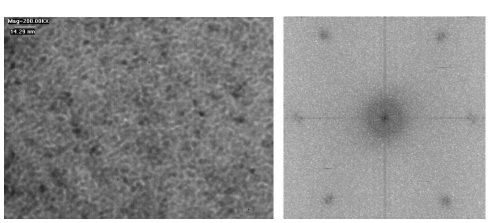

Fig. 1. TEM image (a) with associated FFT (b) for particle size determination. Inside edge of first circular region in FFT multiplied by 82% yields particle size. Shown above is a 41% cobalt sample deposited at 150°C.

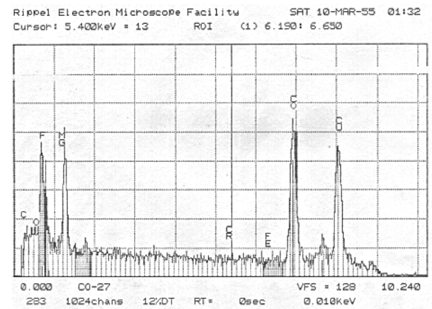

Fig. 2. EDS for a Co-MgF_2 TEM sample showing elemental spectral peaks.

sufficient to control eddy current loss in thicknesses up to at least 10 μm for applications in power converters operating in the 5-10 MHz range, as calculated in [12,13]. One sample's resistivity was tested with ac signals up to 500 kHz, but it did not change significantly in the range measured.

C. Magnetic Properties

A symmetrically driven field of +/- 0.1 T was used in a VSM to generate a minor hysteresis loop and determine intrinsic coercivity (H_{ci}), susceptibility (χ), and large-signal, low-frequency quality factor (Q)—which is the ratio of energy stored to energy dissipated in the magnetic material [14]. Higher fields, up to 1 T, were used on some samples to measure their saturation characteristics.

The best Co-MgF_2 granular thin film for our application had H_{ci} of 0.76 Oe with χ of 26 both measured in the easy axis direction. The sample contained 42% volume cobalt and had a resistivity of 2 x 10^7 μΩ-cm, B_s of 0.7 T, and Q of over 100. A hysteresis plot for this sample is shown in Fig. 3.

The product of Q times χ (Fig. 4) is a useful single-number measure of the overall performance of a magnetic material in an inductor. Ideally, both of these numbers should be large, resulting in a very large product. The energy stored is inversely proportional to χ. Since Q is directly proportional to energy stored, the product of Q times χ is constant if χ varies while other parameters stay fixed. Conventionally, air gaps have been used to decrease the effective value of χ but cannot increase it; thus a minimum χ is typically needed for a

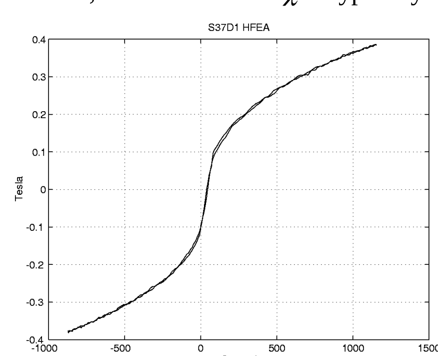

Fig. 3. Hysteresis loop for a 42% volume cobalt sample in the easy axis direction. $H_{ci||}$ = 0.76 Oe, $\chi_{||}$ = 26, $Q_{||}$ = 100, B_s = 0.7 T, and ρ = 2 x 10^7 μΩ-cm.

given application. A high $Q\chi$ product, then, is a quick way to identify good candidate magnetic materials for further study.

Samples were deposited with substrate temperatures between -21°C and 311°C. The variation in magnetic properties we observed showed no significant correlation with temperature, indicating that uncontrolled deposition system variables, such as contamination, may have been more important in determining the variations we observed in magnetic properties.

Volume percentage cobalt was seen to significantly affect magnetic properties. A clear trend toward higher coercivities with higher percentages of cobalt is found in the range tested. Susceptibility also increases with percentage cobalt between 20% and 60% volume cobalt and decreases above the percolation threshold of 60%. Highest values of the quality factor occur at lowest cobalt percentages. For the applications we are studying, the best range is between 40% to 55% volume cobalt in order to achieve simultaneously high susceptibility and low coercivity. This can be most clearly seen by evaluating materials on the basis of the $Q\chi$ figure of merit; selecting a material for a particular application would require evaluating both Q and χ individually.

Some further investigation of these materials was carried out. Higher deposition rates of 10 Å/s for cobalt and 16 Å/s for magnesium fluoride do not show noticeable improvements over the lower deposition rates we used for most films. Annealing at 250°C increases the measured coercivity of samples while other magnetic properties remain unchanged. The addition of a chromium underlayer does not seem to improve magnetic properties nor does it initially seem to result in better adhesion of Co-MgF_2 to glass slides. Preliminary measurements of high-frequency permeability and quality factor show approximately flat responses, near their measured DC values in the range up to 300 MHz that we measured.

V. CONCLUSION

New granular thin films of Co-MgF_2 look promising for application as low-loss, high-frequency core materials in power converters. Films have been evaporatively co-deposited using separate cobalt and magnesium fluoride resistively heated sources. Properties were characterized in terms of structure, electrical, and magnetic behavior. Good soft magnetic characteristics are observed for cobalt particle sizes around 3.2 nm, with resistivities greater than 1000 μΩ-cm. Films deposited onto room-temperature substrates with 40% to 55% volume cobalt achieved the best compromise between low coercivity, high susceptibility, and high quality factor with a saturation flux density on the order of 1T.

The best Co-MgF_2 granular thin film produced in terms of its magnetic properties in this study showed an intrinsic coercivity of 0.76 Oe, susceptibility equal to 26, and quality factor over 100. Furthermore, the saturation flux density was 0.7 T with a resistivity of 2 x 10^7 μΩ-cm at 42% volume cobalt deposited onto a room temperature substrate.

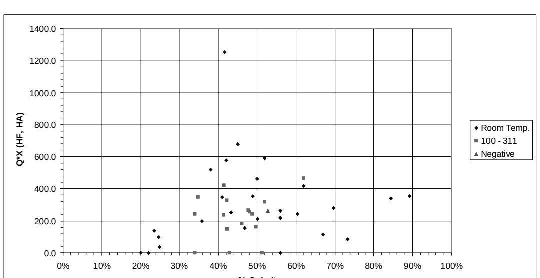

Fig. 4. $Q\chi$ product versus volume percentage cobalt. Measured in the hard-axis direction with an applied field of +/- 1000 Oe.

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