Chemical Vapor Deposition of Metallic Thin Films Using Homonuclear and Heteronuclear Metal Carbonyls

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The purpose of this work has been to demonstrate a simple chemical vapor deposition (CVD) technique that employs easily handled solid metal carbonyls as precursors for the formation of metallic thin films. Co, Fe, Ru, and Os metallic thin films were deposited in a chemical vapor deposition (CVD) apparatus under a vacuum of 10^{-5} Torr using $Co_2(CO)_8$, $Fe_3(CO)_{12}$, $Ru_3(CO)_{12}$, and $Os_3(CO)_{12}$ at 150, 200, 175, and 225 °C, respectively. $FeCo_3$ and $FeRu_3$ metal alloy films were deposited from $HFeCo_3(CO)_{12}$ and $H_2FeRu_3(CO)_{13}$ at 150 and 200 °C, respectively. A Co/Ru/Co multilayer film was prepared by consecutively depositing each layer from the respective carbonyls at the aforementioned temperatures. The method employed differs from previously reported procedures in that a carrier gas is not used, but a dynamic vacuum of 10^{-5} Torr is found sufficient to transport the precursor metal carbonyls to the heated substrate. The precursor carbonyls $Co_2(CO)_8$, $Fe_3(CO)_{12}$, and $H_2FeRu_3(CO)_{13}$, were sufficiently volatile that they could be maintained at room temperature. $HFeCo_3(CO)_{12}$ and $Os_3(CO)_{12}$ were maintained at 50 °C while $Ru_3(CO)_{12}$ was held at 75 °C. The films were characterized by Auger electron spectroscopy (AES), X-ray photoelectron spectroscopy (XPS), X-ray diffraction (XRD), scanning electron microscopy (SEM), and stylus profilometry.

Introduction

The use of metal carbonyls as precursors for the chemical vapor deposition (CVD) of metallic thin films has been of continuing interest since Mond first reported the preparation of Ni films from $Ni(CO)_4$ in 1890.[1a] A number of metal carbonyls are reasonably air stable at ambient temperatures and have sufficient volatility to be transported in the vapor phase at relatively low temperatures. Since the metals within these compounds are formally in the zerovalent state[2] and the CO ligand is by itself a stable noncondensable gas, thermolysis even in the absence of reducing environments can in principle produce high-quality metallic films. The deposition of single metal films, multilayer films, and alloy films at low temperatures is of great interest to the electronics industry.[1] Metal carbonyls can provide an avenue to achieve these goals.

Previously, CVD of Fe, Ru, and Os has been accomplished using metal pentacarbonyls as precursors. Fe was deposited from $Fe(CO)_5$ using thermolysis,[3−5] laser CVD,[6] UV photolysis,[4] and electron beam deposition,[4] while Ru and Os films have been thermally deposited from $Ru(CO)_5$ and $Os(CO)_5$.[7] However, these pentacarbonyls are not convenient to handle because they are air-, light-, and temperature-sensitive liquids. Furthermore, $Ru(CO)_5$ and $Os(CO)_5$ are prepared from $Ru_3(CO)_{12}$ and $Os_3(CO)_{12}$, employing demanding conditions.[7] While the substituted metal pentacarbonyls, $M(CO)_4[C_2(CF_3)_2]$ (M = Ru, Os),[8] are stable, solid precursors, they require relatively higher temperatures (500 °C for Ru, 600 °C for Os) for the deposition of the metal film than the unsubstituted metal pentacarbonyl precursors.

In the present investigation it has been our intention to demonstrate a simplified, practical CVD technique that employs easily handled, solid metal carbonyls as precursors. Instead of a carrier gas, the procedure employs a dynamic vacuum at sufficiently low pressure such that the precursor is easily transported in the vapor phase to the heated substrate. A dynamic vacuum promotes the release and removal of CO from the metal carbonyl and reduces the possibility of CO dissociation to form metal carbides and oxides. Applications of this procedure are described below in the preparation of metal films of Fe, Ru, Os, and Co from the trinuclear precursors $M_3(CO)_{12}$ (M = Fe, Ru, Os) and the dinuclear precursor $Co_2(CO)_8$ molecule. Thin-film alloys of $FeCo_3$ and $FeRu_3$ obtained from $HFeCo_3(CO)_{12}$ and $H_2FeRu_3(CO)_{13}$ and Co/Ru/Co multilayer films were also produced by this CVD procedure. Studies on the magnetic properties of these films have been published previously.[9]

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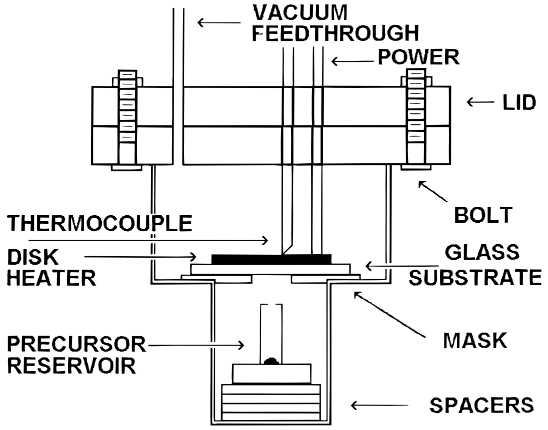

Figure 1. Diagram of chemical vapor deposition apparatus.

Experimental Section

General Comments. All manipulations were carried out using standard high-vacuum-line techniques or in a drybox under N_2. $Co_2(CO)_8$ (Alfa Chemical Co.) was recrystallized from hexane dried over sodium metal. $Fe_3(CO)_{12}$ (Strem Chemical Co.) was recrystallized from CH_2Cl_2 dried over P_2O_5. $Os_3(CO)_{12}$ (Strem Chemical Co.) was used as received. $Ru_3(CO)_{12}$,[10] $HFeCo_3(CO)_{12}$,[11] and $H_2FeRu_3(CO)_{13}$[12] were prepared by the methods that are described in the literature. Recrystallized $Co_2(CO)_8$, $Fe_3(CO)_{12}$, $HFeCo_3(CO)_{12}$, and $H_2FeRu_3(CO)_{13}$ were stored at −30 °C in the refrigerator of the glovebox.

Instrumentation. Auger electron spectroscopy (AES) data for Co, Fe, Ru, and $FeRu_3$ films and X-ray photoelectron spectroscopy (XPS) data for Os and $FeRu_3$ films were collected on a Perkin-Elmer Model 550 ESCA/Auger spectrometer containing a double-pass cylindrical mirror analyzer (DPCMA) with a primary electron beam energy of 3 keV and a beam current of 1 μA. Sputter cleaning and depth profiling were achieved with an Ar ion sputtering gun operating at 2 keV. This instrument gives an AES and XPS survey accuracy of ±2 eV along the x axis, an AES profile accuracy of ±2−3% atomic concentration, and an XPS high-sensitivity scan accuracy of ±4% atomic concentration. AES spectra for Os and $FeCo_3$ films were collected on a V. G. Scientific ESCALAB MkII spectrometer. The instrument utilizes a 150° spherical sector analyzer and a LEG200 electron source that operates up to 10 keV using a LaB_6 filament. It has a spot size capability of better than 0.2 μm. Sputter cleaning and depth profiling were accomplished with an AG21 argon ion source. X-ray diffraction (XRD) patterns were collected on a Rigaku Geigerflex diffractometer utilizing a Cu target. Scanning electron microscopy (SEM) photomicrographs were obtained on a JEOL JSM 820 SEM instrument with an attached Link Oxford EDS unit. Profilometry measurements were performed on a Tencor Instruments Alpha Step 200 Stylus Profiler.

Substrate Preparation. Precleaned glass microscope slides were prepared by submersion in boiling water (containing detergent) for several hours followed by several boiling water rinses. The slides were then suspended vertically in beakers and dried in an oven at 120 °C overnight.

Chemical Vapor Deposition Apparatus. The CVD apparatus used in this study is illustrated in Figure 1. A stainless steel canister holds the precursor and supports the substrate and mask. The stainless steel lid houses the disk heater, thermocouple, and vacuum feedthrough. When the lid is bolted to the canister an O-ring provides a vacuum-tight seal. The disk heater rests against the substrate; its temperature is monitored by the thermocouple. The heater is maintained at the desired temperature by means of an external time proportioning digital temperature controller. The precursor rests in the bottom of the canister which may be externally heated to facilitate precursor transport. Sublimation of the precursor under vacuum is followed by decomposition of the vapor at the heated substrate surface.

General Deposition Procedure for Metallic Thin Films. In a glovebox approximately 5−10 mg of the desired metal carbonyl was placed in the reservoir and loaded into the canister. The mask and substrate were then positioned and the stainless steel lid was bolted to the canister. The complete CVD apparatus was then removed from the glovebox and attached to the vacuum line. Electrical and thermocouple leads from the temperature controller were attached. The CVD system and reservoir were then evacuated and pumped on dynamically at 10^{-5} Torr. The temperature controller was turned on and the deposition temperature for the desired metal carbonyl was set {150 °C for $Co_2(CO)_8$ and $H_2FeRu_3(CO)_{13}$, 175 °C for $Ru_3(CO)_{12}$, 200 °C for $Fe_3(CO)_{12}$ and $HFeCo_3(CO)_{12}$, and 225 °C for $Os_3(CO)_{12}$}. The temperature of the metal carbonyl reservoir was maintained at room temperature for $Co_2(CO)_8$, $Fe_3(CO)_{12}$, and $H_2FeRu_3(CO)_{13}$, raised to 50 °C for $HFeCo_3(CO)_{12}$ and $Os_3(CO)_{12}$, and increased to 75 °C for $Ru_3(CO)_{12}$. After 5 h (10 h for Os films) the temperature controller was turned off and the system was returned to room temperature. Single-layer films were then removed in the air after the CVD system was vented to atmospheric pressure. For multilayer films, the CVD system was transported under vacuum to the glovebox along with a clean metal carbonyl reservoir which was subsequently loaded with another metal carbonyl. The CVD system was vented under nitrogen and disassembled to install the newly filled metal carbonyl reservoir. The system was then reassembled using the previously grown film as the substrate. The next film was grown over the previous metallic layer. By repeating these steps, multilayer films were produced.

Results and Discussion

The films prepared were mirrorlike in appearance and showed no signs of peeling upon exposure to air. Deposition parameters, thicknesses, particle sizes (from SEM photomicrographs), and levels of impurities for each film are given in Table 1. Precursor temperatures below the values in Table 1 resulted in either thin films or no film deposition while temperatures higher than those in Table 1 resulted in either colored films or premature precursor decomposition evident as a gray or black ash in the bottom of the chamber. Recorded impurity levels of less than 3% for AES profile data and less than 4% for XPS high-sensitivity scans are within instrumental error (see Instrumentation section).

Co Films. Typical film thicknesses ranging from 630 to 750 Å were produced from $Co_2(CO)_8$ at 150 °C. Particles sizes ranged from 300 to 1700 Å. Besides expected Co peaks (775, 716, 656 eV), the AES survey scan of a Co film prior to sputter cleaning showed weak peaks for carbon (272 eV) and oxygen (503 eV) due to surface contamination from air. An AES depth profile of the Co film (Figure 2a) showed 4−5% carbon and 2−3% oxygen impurity levels throughout the film. This indicates some carbon impurity is actually present, the oxygen impurity is negligible. XRD measurements indicated the weak diffraction of the (100) and (002) planes of α-Co. Gross[2] and co-workers produced epitaxially grown Co films on Si wafers with carbon and oxygen impurities below the 1.0% and 1.5% detection levels, respectively. Ogrin and Haycock[13] deposited Co

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Table 1. Metallic Thin-Film Data

	Co	Fe	Ru	Os	$FeCo_3$	$FeRu_3$
precursor	$Co_2(CO)_8$	$Fe_3(CO)_{12}$	$Ru_3(CO)_{12}$	$Os_3(CO)_{12}$	$HFeCo_3(CO)_{12}$	$H_2FeRu_3(CO)_{13}$
precursor temp (°C)	room temp	room temp	75	50	50	room temp
deposition temp (°C)	150	200	175	225	200	150
deposition time (h)	5	5	5	10	5	5
carbon impurity (%)	4−5	2−3	very low	4−5	2−3	very low
oxygen impurity (%)	2−3	3	<1	1−2	5	2−3
thickness (Å)	630−750	365−830	900−1200	440−475	50	910−1070
particle sizes (Å)	300−1700	450−4500	300−3500	1600−3200	900−4500	300−500

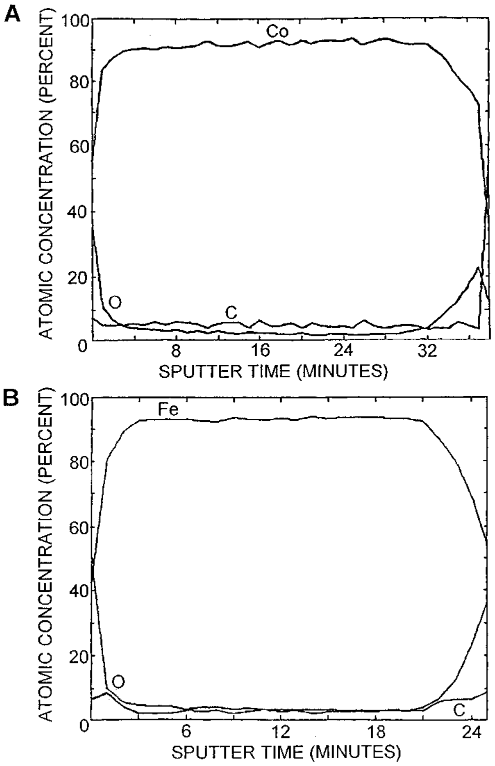

Figure 2. (a) AES profile of a cobalt film. (b) AES profile of an iron film.

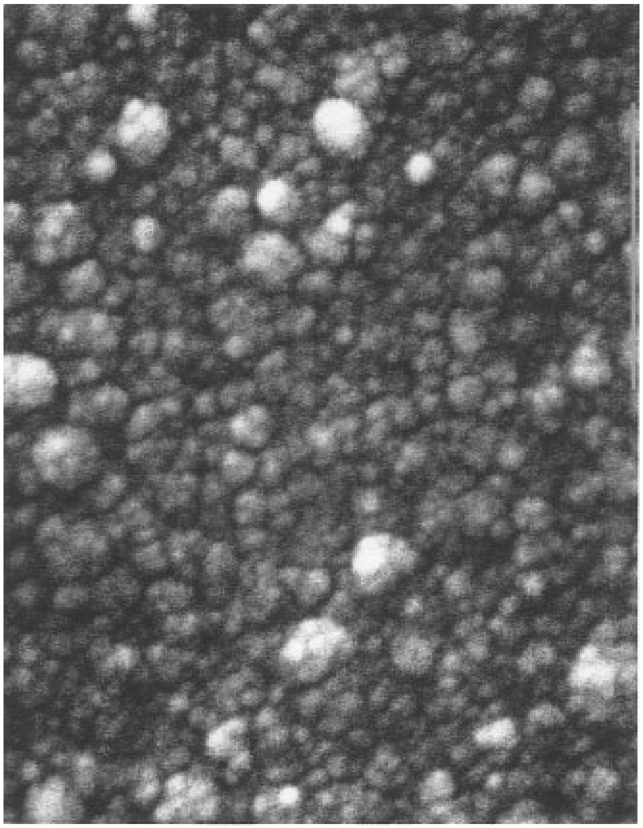

Figure 3. Scanning electron micrograph of an iron film (white bar equals 1 μm).

on microscope slides by MOCVD using $CpCo(CO)_2$ and $Co_2(CO)_8$. Although they produced thinner films (35−220 Å), they did not discuss impurity levels or deposition parameters.

Fe Films. Typical film thicknesses ranging from 365 to 830 Å were produced from $Fe_3(CO)_{12}$ at 200 °C. A scanning electron micrograph (Figure 3) indicated particle sizes of 450−4500 Å. It shows that the larger particles consist of smaller particles that are 450 Å in diameter. Light sputter cleaning was performed, after which an AES depth profile (Figure 2b) showed a carbon impurity level of 2−3% and an oxygen impurity of 3% throughout the film. These values indicate that virtually little, if any, carbon and oxygen impurities are present. XRD measurements indicated a preferred orientation of the (110) plane of α-Fe in the film.

Films obtained here show that CO dissociation from $Fe_3(CO)_{12}$ occurs cleanly. Additionally, solid $Fe_3(CO)_{12}$ is preferable to $Fe(CO)_5$ (a liquid) as a thin-film precursor since it is easier to handle being only a slightly air and light sensitive solid.

Ru Films. Typical film thicknesses ranging from 900 to 1200 Å were produced from $Ru_3(CO)_{12}$ at 175 °C. Particle sizes ranged from 300 to 3500 Å. Light sputter cleaning was performed before an AES depth profile survey was obtained. Unfortunately, the KLL line of carbon at 272 eV overlaps with an MNN line of Ru at 273 eV in the AES survey scan[8] and the C_{1s} peak overlaps with the Ru $3d_{3/2}$ peak in the XPS survey scan. This prevented the possibility of directly scanning for carbon content by either method. However, Green et al.[14] noted that another MNN Ru line at 231 eV does not overlap with any carbon lines. By comparing the relative intensities of the MNN Ru line at 273 eV and the other MNN Ru line at 231 eV with those of an Auger survey scan of a pure Ru standard, they were able to claim a minimal extent of carbon incorporation in their films. The peak intensity ratio (I_{273}/I_{231}) of pure Ru[14] was found to be 2.64; a similar ratio, 2.63, occurs in a published AES survey scan.[15] From our AES survey scan, a ratio of 2.61 was obtained which indicates that our Ru film is close to the Ru standard[15] with respect to carbon impurities. The film prepared by Green and co-workers [14] gave a value of 2.7. The AES depth profile data from our Ru films (Figure 4a) showed an essentially pure Ru sample with 1% or less oxygen incorporation comparable to Ru films prepared by Green.[14] XRD measurements of our film showed a

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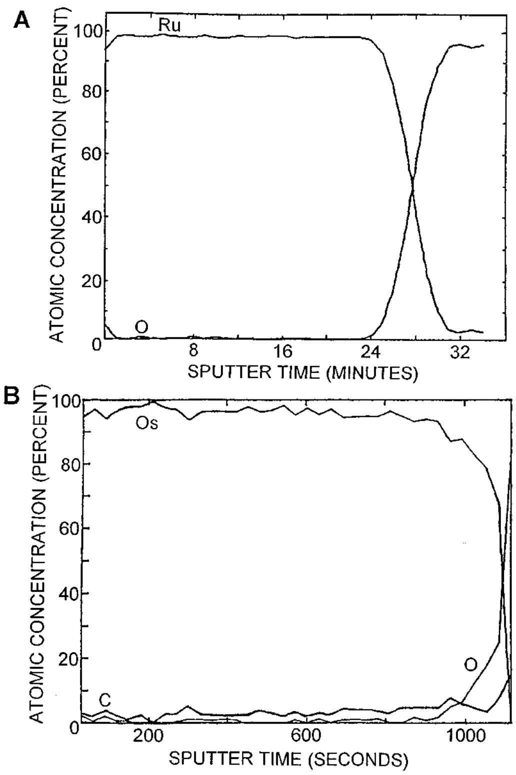

Figure 4. (a) AES profile of a ruthenium film. (b) AES profile of an osmium film.

preferred orientation of the (110) plane of hexagonal Ru with weaker diffraction from the (100) and (101) planes.

The Ru films reported here, although comparable to those obtained by Green et al.,[14] were produced at a substantially lower temperature and higher vacuum (175 °C and 10^{-5} Torr compared to 300 °C and 0.09 Torr). A higher vacuum allows the growth of Ru films from $Ru_3(CO)_{12}$ to be conducted at lower temperatures without increasing impurity levels.

Os Films. Film thicknesses ranging from 440 to 475 Å were produced from $Os_3(CO)_{12}$ at 225 °C. Particle sizes ranged from 1600 to 3200 Å. The XPS survey showed the characteristic peaks for Os[16] after 5 min of light sputtering. No peaks for oxygen (745−780 eV) were observed, and carbon peaks (980−1020 eV) appear weak in intensity. The AES survey scan prior to sputter cleaning showed peaks for Os (176 eV), carbon (272 eV), and oxygen (503 eV). The AES depth profile (Figure 4b) showed impurity levels of 4−5% for carbon and 1−2% for oxygen throughout the film. These values indicate an actual carbon impurity of 2% or less with little or no oxygen. XRD measurements indicated a preferred orientation of the (002) plane of hexagonal Os in the film.

Besides methods mentioned in the Introduction, polycrystalline Os films 5−10 μm thick have also been grown pyrolytically by Lehwald and Wagner[17] from $OsCl_4$ at 1250 °C on Mo and W substrates in a vacuum of 5×10^{-3} Torr over 20−30 min. Although this method avoids possible carbon and oxygen incorporation in films, the method reported here employs a significantly lower deposition temperature.

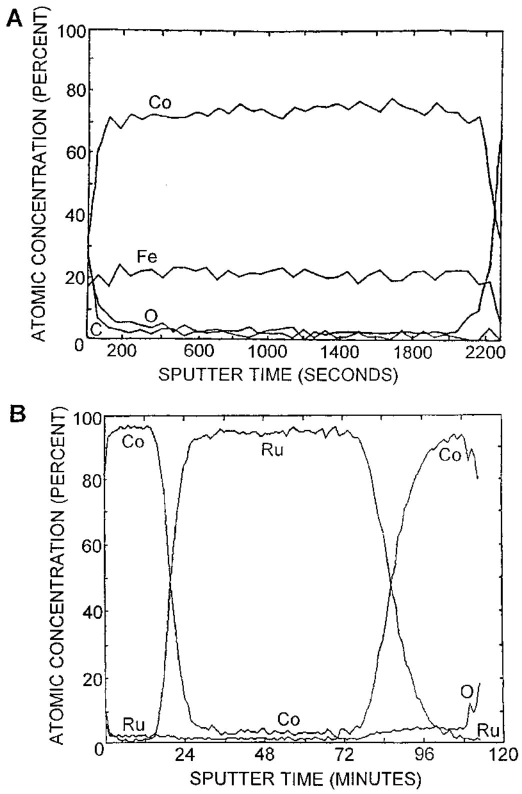

Figure 5. (a) AES profile of a $FeCo_3$ alloy film. (b) AES profile of a Co/Ru/Co layered film.

$FeCo_3$ Alloy Films. Film thickness ranging from 50 to 70 Å were produced from $HFeCo_3(CO)_{12}$ at 150 °C. An SEM indicated particle sizes ranged from 900 to 4500 Å and also showed extensive growth parallel to the substrate surface. After light sputter cleaning, the AES depth profile (Figure 5a) showed a carbon impurity level of 2−3% and an oxygen impurity level of 5%. The Fe/Co ratio from AES depth profile concentration data is 1Fe/3.15Co which is within instrumental error of the ideal 1Fe/3Co ratio. The film was amorphous by XRD.

These results compare well with those from films obtained photochemically by Liu et al.[18] in terms of overall metal stoichiometry and are closer to the ideal Fe:Co stoichiometry of 1Fe/3Co. The $FeCo_3$ alloy films produced by Geoffroy and Czekaj[19] at 300−350 °C showed an excess of Fe, perhaps indicating Co loss by formation of $Co_2(CO)_8$.

$FeRu_3$ Alloy Films. Film thicknesses ranging from 910 to 1070 Å were produced from $H_2FeRu_3(CO)_{13}$ at 200 °C. Particle sizes ranged from 300 to 500 Å. An AES survey scan after 5 min of light sputter cleaning showed Ru peaks (273, 231, 200, 150 eV), less intense Fe peaks (703, 651, 598 eV), and a weak oxygen peak (503 eV). Measurement of the Ru I_{273}/I_{231} ratio (see Ru Films) gave a value of 2.60 which is close to 2.63, the value for pure Ru.[15] As stated earlier, the carbon content cannot be directly determined by either AES or XPS methods. However, the XPS high-sensitivity scan data after 5 min of sputtering gave an Fe/Ru ratio of 1Fe/3.33Ru, compared to the theoretical value of 1Fe/3Ru, assuming there is negligible contribution from carbon in the spectrum. The oxygen content was 2−3%. It would be of interest to compare our AES data to that

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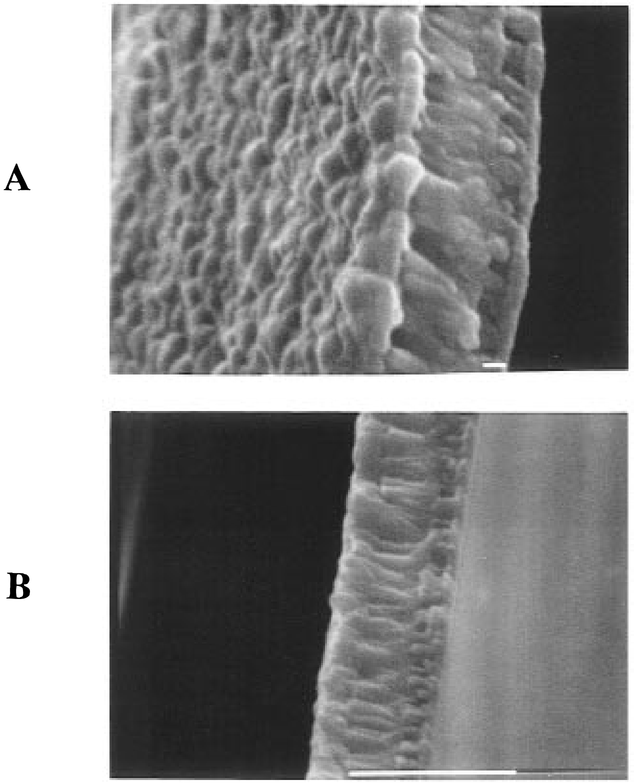

Figure 6. Scanning electron micrograph of Co/Ru/Co layered film: (a) showing edge and surface features (white bar equals 100 nm); (b) showing thicknesses of each layer (white bar equals 1 μm).

of an actual $FeRu_3$ alloy standard. However, to our knowledge such a stoichiometric alloy has not been reported. The film was amorphous by XRD.

Co/Ru/Co Layered Film. To demonstrate multilayer capabilities, a Co/Ru/Co layered film was grown. Scanning electron micrographs of the film edge were obtained and clearly show individual film layers. Figure 6a depicts a sample of the multilayer film peeled from the substrate surface. Particle sizes for the top Co layer are comparable to those of the single layer Co film. Figure 6b shows a direct end-on orientation with the substrate attached. Figure 6b clearly shows the thickness of the top Co layer to be 200 Å, the middle Ru layer to be 300 Å, and the bottom Co layer to be 200 Å. Particles in the bottom Co layer appear more spherical while those in the top Co layer and middle Ru layer are elongated in the direction perpendicular to the substrate. A scanning electron micrograph of the edge of a single Ru film showed no indication of columnar growth. This would indicate that new particle growth in multilayer films occurs in the vertical direction on top of particles in the previous metallic layer rather than randomly as in the initial layer. This type of growth may be facilitated by our chemical vapor deposition system in that our substrate is inverted with film growth occurring accordingly.

An AES depth profile (Figure 5b) shows the existence of a layered film and the lack of impurities in each layer. Atomic concentrations of Co, Ru, and oxygen were determined throughout the depth of the film. While carbon could not be directly determined due to overlap with Ru peaks, Figure 5b clearly shows an oxygen impurity level averaging 2−5% throughout all three layers. The interface between the top (outermost) Co layer and Ru layer is quite sharp. The bottom Co layer is thinner than the top Co layer, and as a result, passage into the substrate is abrupt.

To further determine impurity levels in each film, the AES depth profile (Figure 5b) was periodically interrupted to run an AES survey scan on each metallic layer. The AES survey scan of the top Co layer showed a small carbon peak (272 eV) and no apparent oxygen peak (503 eV). The AES survey spectrum of the Ru layer showed no oxygen impurity and no apparent Co impurity. Thus the apparent Co impurity level in the Ru layer of the AES profile (Figure 5b) is not due to Co. A I_{273}/I_{231} ratio gives a value of 2.52 compared to 2.63 for pure Ru.[15] An AES survey spectrum of the bottom Co layer showed a trace amount of carbon at 272 eV and oxygen at 503 eV. The peak at 272 eV is complicated by noise and could also indicate, besides a carbon impurity, a slight Ru impurity (273 eV) which could have resulted from the return of Ru to the Co surface after sputtering.

XRD measurements indicated that both the top Co layer and the Ru layer could be observed simultaneously. Both layers exhibited diffraction from multiple crystal planes indicative of hexagonal Ru and α-Co. Both layers have a preferred orientation for their respective (110) plane.

Summary

A simple CVD apparatus operating at 10^{-5} Torr in the absence of a carrier gas was used to deposit metal films with characteristics comparable to their respective bulk metals[9] while stoichiometric alloys have been produced with low carbon and oxygen impurity levels. Since the CVD system was designed to be small enough to fit inside a conventional glovebox, multilayer films were produced without exposing layer interfaces to atmospheric contaminants. SEM micrographs of single metal and alloy films show uniform particle sizes comparable to those of similar films from other groups. Particle growth on previous metallic layers seems to occur on existing particles and elongate in the direction perpendicular to the substrate. The preparation of $FeRu_3$ and $FeCo_3$ alloys from $H_2FeRu_3(CO)_{13}$ and $HFeCo_3(CO)_{12}$ by our method suggests that other alloy films may be equally accessible from mixed-metal clusters and deposited in a stoichiometric fashion with little contamination from impurities.

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