

Single-Source MOCVD of Fe/Sn Alloy Thin Films

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Two organometallic compounds containing an Fe–Sn bond, $\text{CpFe}(\text{CO})_2(\text{SnMe}_3)$ (Cp : η^5 -cyclopentadienyl) and *cis*- $\text{Fe}(\text{CO})_4(\text{SnMe}_3)_2$, have been used as single-source low-pressure CVD precursors to grow Fe/Sn alloy thin films at 300–420 °C. Deposited films were characterized by various surface analytical techniques. Electron probe microanalyses show that Fe and Sn elements are evenly distributed in the films. X-ray powder diffraction patterns indicate that these are polycrystalline films. Polycrystalline FeSn films have been successfully prepared from $\text{CpFe}(\text{CO})_2(\text{SnMe}_3)$ with the deposition rates of 78–175 Å/min. The films composed of FeSn_2 phase with a minor constituent of FeSn were obtained by using *cis*- $\text{Fe}(\text{CO})_4(\text{SnMe}_3)_2$ as precursor and the deposition rates were 130–330 Å/min.

Introduction

The chemical vapor deposition of metal alloy or mixed-metal oxide thin films from organometallic complexes containing two metals in the ratio required for a particular stoichiometry is of great current interest. It is due to the fact that this approach has the potential to control the film stoichiometry, to simplify the precursor delivery, and to gain better film homogeneity.¹ To be eligible as a “single-source MOCVD precursor”, an organometallic complex has to meet some important requirements including (i) appropriate metal stoichiometry, (ii) reasonable volatility, (iii) low heteroatom content in the ligand, and (iv) controllable deposition chemistry. This strategy has been successfully employed to produce the III–V semiconductor thin films.² However, relatively few examples of metal alloy thin films deposited by this method are known and listed in Table 1. Czekaj and Geoffroy³ reported the preparation of Fe/Co alloy and oxide films from Fe–Co carbonyl clusters. Liu, Lai, and Chin⁴ have prepared Fe–Co and Fe–Ni thin films from the corresponding heterobinuclear metal carbonyl compounds by low-temperature photochemical vapor deposition. Shore and co-workers⁵ reported the CVD of FeCo_3 and FeRu_3 alloy films from $\text{HFeCo}_3(\text{CO})_{12}$ and $\text{HFeRu}_3(\text{CO})_{13}$, respectively. Kaesz and co-workers^{6–9} and Fis-

cher and co-workers^{10–18} have used a variety of organometallic compounds containing transition metal–group 13 metal bonds to deposit the corresponding metal alloy thin films. In addition, Aylett’s group has used silyl transition metal carbonyl compounds to deposit metal silicide films such as CoSi , $\beta\text{-FeSi}_2$, and Mn_5Si_3 .^{19–22} Transition metal/tin alloys which are generally manufactured by electrochemical deposition are known to be useful for anticorrosion protection, solar energy devices, and magnetic tape.^{23–25} Deposition of thin films of polycrystalline Ni_3Sn_4 incorporated with SnO_2 from $[\text{Ni}_2\text{Sn}_2(\text{O}^{\prime}\text{Bu})_8]$ was reported by Veith et al.²⁶ Recently, we used two organometallic compounds containing an Co–Sn bond, $\text{R}_3\text{SnCo}(\text{CO})_4$ ($\text{R} = \text{Me}$ and Ph), as precursors to deposit thin films of binary CoSn alloy.²⁷ To continue our effort in this research area, we have explored other

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Table 1. Reported Single-Source CVD of Metal Alloy Thin Films^a

precursor	technique	T _{Dep} (°C)	result	ref
HFeCo ₃ (CO) ₁₂	CVD with carrier gas of CO	300–350	amorphous Fe/Co alloy (Fe:Co = 0.5)	3
CpFeCo(CO) ₆	CVD with carrier gas of CO	300–350	amorphous Fe/Co alloy (Fe:Co = 1.2)	3
CpFe ₂ Co(CO) ₉	PCVD ^b with carrier gas of CO	~50	amorphous Fe/Co alloy (Fe:Co = 0.85)	4
CpFeCo(CO) ₆	PCVD ^b with carrier gas of CO	~50	amorphous Fe/Co alloy (Fe:Co = 1.19)	4
HFeCo ₃ (CO) ₁₂	PCVD ^b with carrier gas of CO	~50	amorphous Fe/Co alloy (Fe:Co = 0.31)	4
Cp ₂ FeNi(CO) ₃	PCVD ^b with carrier gas of CO	~50	amorphous Fe/Ni alloy (Fe:Ni = 0.98)	4
HFeCo ₃ (CO) ₁₂	LPCVD ^b	200	amorphous Fe/Co alloy (Fe:Co = 0.32)	5
H ₂ FeRu ₃ (CO) ₁₃	LPCVD ^b	150	amorphous Fe/Ru alloy (Fe:Ru = 0.30)	5
(CO) ₄ CoGaCl ₂ (THF) ^a	CVD with carrier gas of H ₂	500	polycrystalline CoGa	6–8
Pt(dmg·GaMe ₂) ₂ ^a	CVD with carrier gas of H ₂	500	polycrystalline PtGa ₂ (with little Pt ₂ Ga ₃)	6, 7
(py)(Et)Co(dmg·GaEt ₂) ₂ ^a	CVD with carrier gas of H ₂	560–630	polycrystalline β -CoGa	9
Ni(dmg·GaMe ₂) ₂ ^a	CVD with carrier gas of H ₂	560	polycrystalline Ni/Ga alloy with mixed phases	9
(CO) ₅ MnGaEt ₂ (NR ₃)	LPCVD ^b	250–420	polycrystalline MnGa	10, 11
[(CO) ₅ Mn] ₂ Ga[(CH ₂) ₃ NMe ₂]	LPCVD ^b	250–420	polycrystalline Mn ₂ Ga	11
Cp(CO) ₂ FeAl[(CH ₂) ₃ NMe ₂](BH ₄)	LPCVD ^b	300–350	amorphous Fe/Al alloy (Fe:Al = 1.31)	12
Cp(CO) ₂ FeGa[(CH ₂) ₃ NMe ₂](BH ₄)	LPCVD ^b	250–350	amorphous Fe/Ga alloy (Fe:Ga = 1.26)	12
{(CO) ₄ FeGa[(CH ₂) ₃ NMe ₂]} ₂	LPCVD ^b	250	amorphous Fe/Ga alloy (Fe:Ga = 1.00)	13
(CO) ₄ Fe ₂ [Ga[(CH ₂) ₃ NMe ₂]·(Bu)] ₂	LPCVD ^b	200	amorphous Fe:Ga alloy (Fe:Ga = 0.53)	13
(CO) ₄ CoGaH ₂ (NMe ₃)	LPCVD ^b	200	polycrystalline β -CoGa	14
(CO) ₄ CoGa[(CH ₂) ₃ NMe ₂]·(Bu)	LPCVD ^b	350	polycrystalline β -CoGa	15
(CO) ₄ CoGaEt ₂ (NMe ₃)	LPCVD ^b	300–400	polycrystalline β -CoGa and α -CoGa	15, 16
(CO) ₄ CoIn[(CH ₂) ₃ NMe ₂] ₂	LPCVD ^b	250–350	polycrystalline Co/In alloy with mixed phases	17
[(CO) ₄ Co] ₂ In[(CH ₂) ₃ NMe ₂]	LPCVD ^b	250	polycrystalline Co/In alloy with mixed phases	17
[(CO) ₄ Co] ₃ In	LPCVD ^b	280	polycrystalline Co/In alloy with mixed phases	17
Cp(CO)NiIn[(CH ₂) ₃ NMe ₂] ₂	LPCVD ^b	300–350	polycrystalline ϵ -NiIn	15, 18
[Ni ₂ Sn ₂ (O'Bu) ₈]	LPCVD ^b	450–550	polycrystalline Ni ₃ Sn ₄	26
(CO) ₄ CoSnMe ₃	LPCVD ^b	250–300	polycrystalline CoSn	27
(CO) ₄ CoSnPh ₃	LPCVD ^b	300–400	polycrystalline CoSn (with little Co ₂ Sn)	27

^a THF, tetrahydrofuran; dmg, dimethylglyoximato; py, pyridine. ^b PCVD, photochemical vapor deposition; LPCVD, low-pressure chemical vapor deposition.

Table 2. Precursor, Growth Condition, and Results of CVD of Fe/Sn Alloy Thin Films

precursor	deposition temperature (°C)	film color	deposition rate (Å/min)	grain size (μm)	crystallinity XRD	Fe/Sn ratio ^a	
						EDS	AAS
CpFe(CO) ₂ (SnMe ₃)	300	silver-gray	78	0.2–0.5	amorphous	1.25	0.95
	330	silver-gray	100	0.8–1.3	FeSn	1.11	1.06
	360	silver-gray	127	1.0–1.5	FeSn	1.25	0.93
	390	silver-gray	150	0.8–1.5	FeSn	1.00	1.08
	420	silver-gray	175	1.2–1.8	FeSn	1.25	1.07
cis-Fe(CO) ₄ (SnMe ₃) ₂	300	silver-gray	130	0.2–0.5	FeSn ₂	0.48	0.55
	330	silver-gray	208	0.5–0.8	FeSn ₂ /FeSn	0.41	0.61
	360	silver-gray	260	1.0–1.5	FeSn ₂ /FeSn	0.45	0.54
	390	silver-gray	305	1.2–1.6	FeSn ₂ /FeSn	0.42	0.58
	420	silver-gray	330	1.4–2.0	FeSn ₂ /FeSn	0.50	0.57

^aThe relative errors of the given values of the ratio Fe/Sn are $\pm 20\%$ for EDS and $\pm 5\%$ for AAS.

potential single-source precursors to deposit transition metal/tin alloy thin films. Herein, we present CVD of FeSn and FeSn₂ thin films at 300–420 °C from two organometallic precursors containing an Fe–Sn bond, CpFe(CO)₂(SnMe₃) (Cp: η^5 -cyclopentadienyl) and *cis*-Fe(CO)₄(SnMe₃)₂, respectively.

Experimental Section

Precursor Preparation. All operations were carried out by using standard vacuum-line techniques or in a glovebox under N₂. Two yellow, liquid organometallic precursors, CpFe(CO)₂(SnMe₃) and *cis*-Fe(CO)₄(SnMe₃)₂, were prepared by the methods described in the literature^{28,29} and were purified by distillation under reduced pressure (ca. 0.1 Torr). These compounds were characterized by IR (Perkin-Elmer Model 16 PC FTIR) ¹H, ¹³C NMR (Bruker Avance DPX-400) spectroscopic methods.³⁰

Substrate Preparation. Substrates of three types, *n*-type Si(100) wafer, Si wafer coated with 2000 Å of thermally grown

SiO₂, and glass, were used. The substrates, typically 1.5 × 1.5 cm², were cleaned with deionized water, acetone, and 1,1,1-trichloroethane and then heated to 100 °C for 20 min before use.

Chemical Vapor Deposition Apparatus and Procedure. The low-pressure CVD experiments were carried out in a cold-wall, lamp-heated, turbomolecular-pumped reactor described elsewhere.²⁷ The precursor (\approx 1.0 g) was loaded into a precursor reservoir in a nitrogen glovebox. After the reactor was loaded with a substrate and the precursor reservoir connected, the system was evacuated to a base pressure of 8×10^{-6} Torr. The precursor was then heated to an appropriate temperature (50 °C for CpFe(CO)₂(SnMe₃) and 60 °C for *cis*-Fe(CO)₄(SnMe₃)₂) and was evaporated into a reaction chamber without carrier gas. The system pressure was kept constant (0.06 Torr for CpFe(CO)₂(SnMe₃) and 0.05 Torr for *cis*-Fe(CO)₄(SnMe₃)₂).

(30) Characterization data for two precursors are listed as follows. For CpFe(CO)₂(SnMe₃): IR (hexane) ν (CO): 1992 (s), 1942 (s) cm⁻¹. NMR (C₆D₆, 25 °C) ¹H δ 4.71 (s, C₅H₅), 0.33 (s, with Sn satellites), ²J_{H-Sn} = 46.4 Hz, Sn(CH₃)₃ ppm. ¹³C{¹H} δ 216.3 (s, CO), 82.1 (s, C₅H₅), -4.0 (s, with Sn satellites), ¹J_{C-Sn} = 183 Hz, Sn(CH₃)₃ ppm. For *cis*-Fe(CO)₄(SnMe₃)₂: IR (hexane) ν (CO): 2056 (s), 1998 (m), 1987 (m), 1968 (s) cm⁻¹. NMR (CDCl₃, 25 °C) ¹H δ 0.48 (s, with Sn satellites), ²J_{H-Sn} = 47.3 Hz, Sn(CH₃)₃ ppm. ¹³C{¹H} δ 207.3 (s, CO), -3.2 (s, with Sn satellites), ¹J_{C-Sn} = 250 Hz, Sn(CH₃)₃ ppm.

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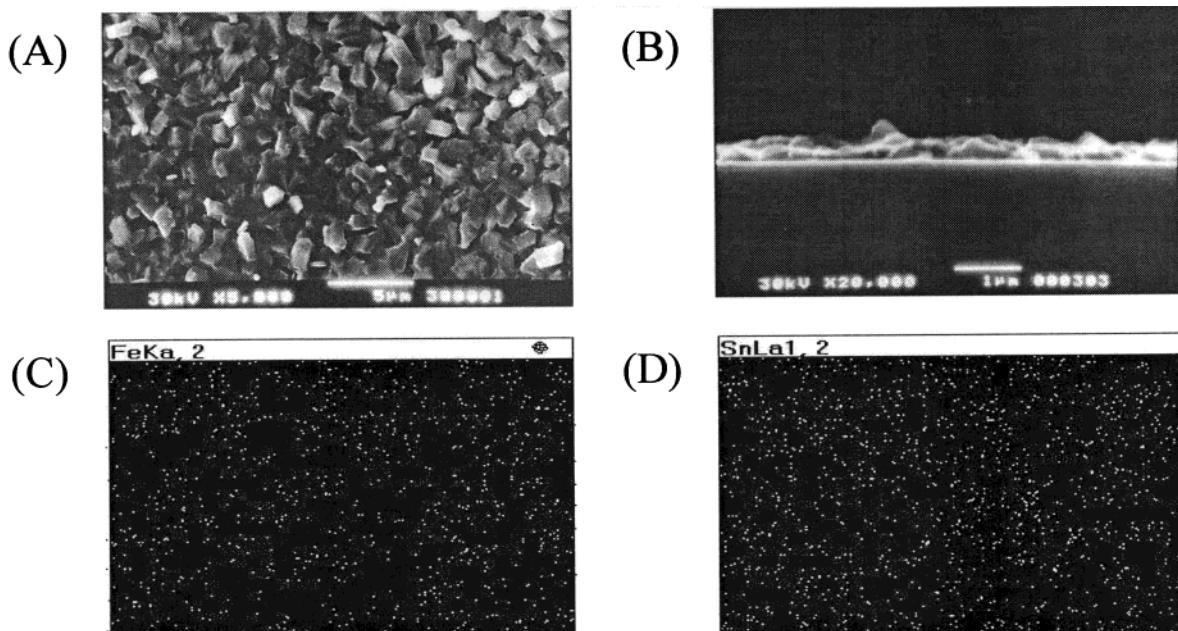


Figure 1. Scanning electron micrograms [(A) top view, (B) cross section] and EPMA pictures [(C) Fe and (D) Sn] of the films deposited on Si from $\text{CpFe}(\text{CO})_2(\text{SnMe}_3)$ at 360 $^{\circ}\text{C}$.

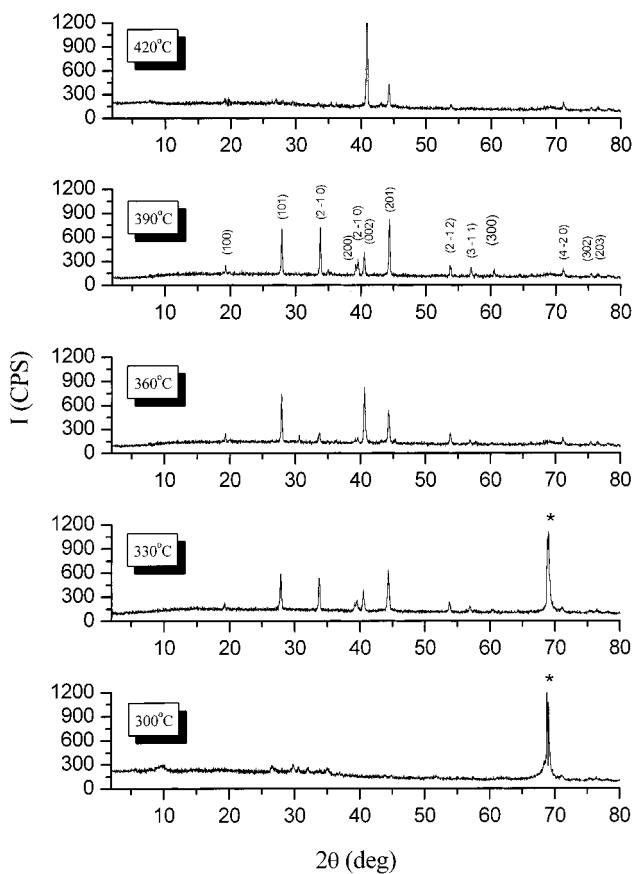


Figure 2. XRD patterns of the films deposited from $\text{CpFe}(\text{CO})_2(\text{SnMe}_3)$ at 300–420 $^{\circ}\text{C}$. (*Si substrate)

($\text{SnMe}_3)_2$) by adjusting the pumping speed. The substrate was heated from the backside by a mercury lamp and the temperature was measured by a thermocouple touching the front side of the substrate.

Film Characterization. Deposited films were characterized by scanning electron microscopy (SEM, JEOL JSM-5400), X-ray diffraction (XRD, Shimadzu XRD-6000), Auger electron spectroscopy (AES, VG Microlab 310D), electron spectroscopy

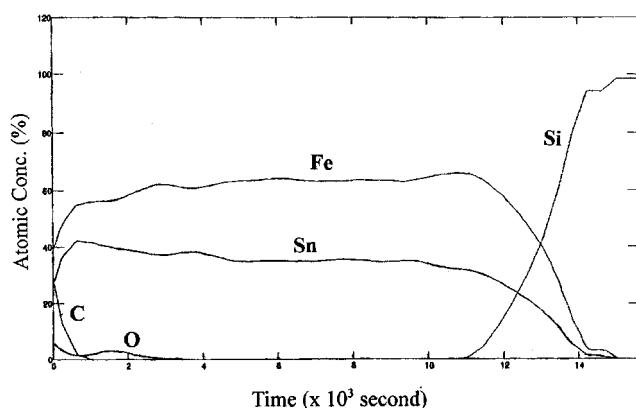


Figure 3. Auger depth profile of the film deposited from $\text{CpFe}(\text{CO})_2(\text{SnMe}_3)$ at 360 $^{\circ}\text{C}$. (Sputtering rate: 50 $\text{\AA}/\text{min}$.)

for chemical analysis (ESCA, VG ESCA 210), energy-dispersive spectroscopy (EDS, Oxford Link ISIS), and atomic absorption spectroscopy (AAS, Perkin-Elmer 3110). The film thickness was roughly measured from the side view of SEM images.

Results and Discussion

The CVD experiments were undertaken over the temperature range 240–420 $^{\circ}\text{C}$ for both $\text{CpFe}(\text{CO})_2(\text{SnMe}_3)$ and *cis*- $\text{Fe}(\text{CO})_4(\text{SnMe}_3)_2$. No significant deposition was observed at the temperatures below 300 $^{\circ}\text{C}$. Dense, uniform thin films with silver-gray color were grown on a Si wafer, SiO_2 -coated wafer, and glass at a temperature of 300–420 $^{\circ}\text{C}$. All films had good adhesion according to the Scotch tape test. No apparent difference in SEM and XRD data was observed for the films deposited on three different substrates. Typical experimental results of the films deposited on Si substrate are summarized in Table 2. Deposition rates increased with increasing deposition temperature and ranged over 78–175 and 130–330 $\text{\AA}/\text{min}$ for $\text{CpFe}(\text{CO})_2(\text{SnMe}_3)$ and *cis*- $\text{Fe}(\text{CO})_4(\text{SnMe}_3)_2$, respectively.

Deposition from $\text{CpFe}(\text{CO})_2(\text{SnMe}_3)$. The surface morphologies of the films grown from $\text{CpFe}(\text{CO})_2(\text{SnMe}_3)$ at

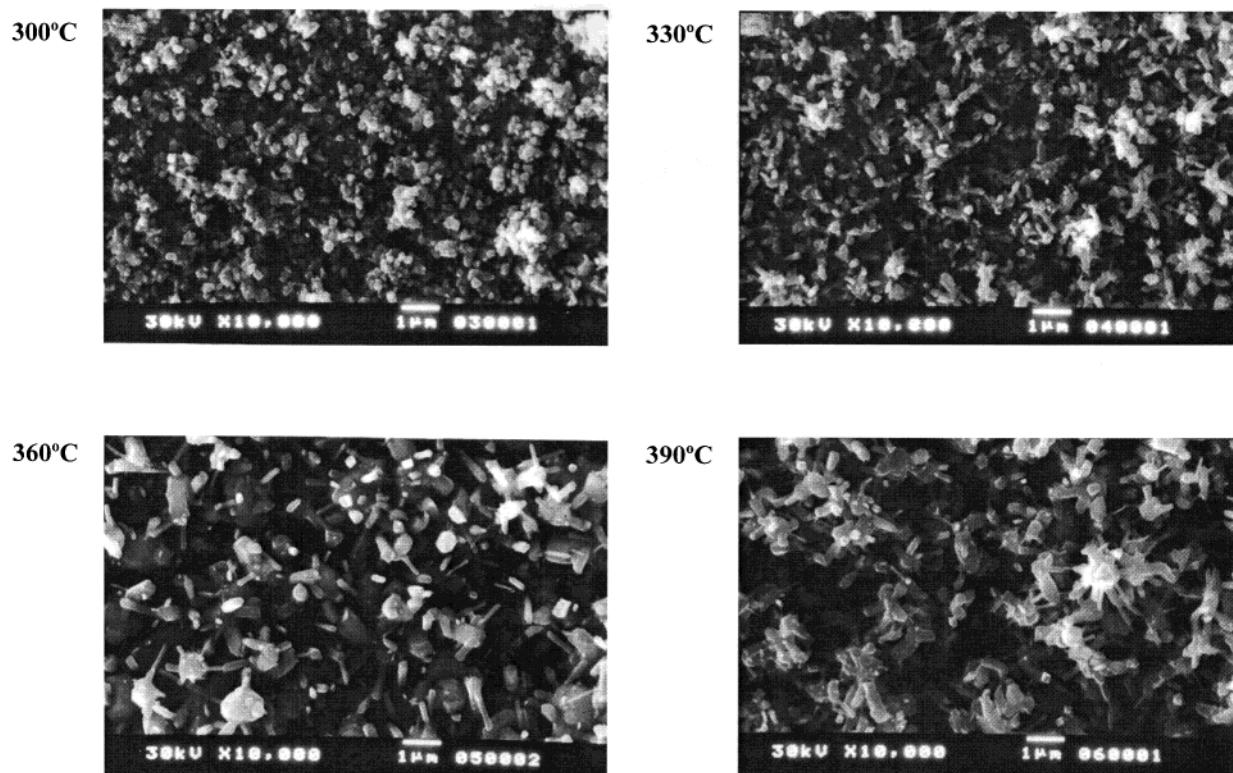


Figure 4. Scanning electron micrograms of the films deposited on Si from *cis*-Fe(CO)₄(SnMe₃)₂ at different temperatures.

(SnMe₃) were examined by scanning electron microscopy; a typical example deposited on a Si substrate at 360 °C is shown in Figure 1 A,B. The film was composed of irregular-shaped grains. The grain size increased with increasing deposition temperature and ranged from 0.2 to 1.8 μ m. Electron probe microanalysis (EPMA) indicated that Fe and Sn were evenly distributed in the films. The Fe and Sn EPMA micrograms of the film deposited at 360 °C are shown in Figure 1C,D, respectively.

X-ray powder diffraction spectra of the films deposited on Si wafers at a temperature of 300–420 °C are shown in Figure 2. The spectrum of the sample grown at 300 °C exhibits no diffraction peaks and reveals the formation of an amorphous film. Diffraction patterns of the films deposited at 330–420 °C indicate the existence of polycrystalline material and correspond to the single-phased FeSn alloy.³¹ These results are in agreement with other surface analysis data. The compositions of the films were mainly determined by atomic absorption spectroscopy. The AAS analysis data (Table 2) demonstrate that the Fe/Sn ratios are close to 1. An Auger electron depth profile of a sample prepared at 360 °C (Figure 3) exhibits Fe/Sn in nearly constant ratio from the top to the bottom of the film. Carbon and oxygen impurities existing on the top of the film are likely resulted from the surface contamination.

Deposition from *cis*-Fe(CO)₄(SnMe₃)₂. The scanning electron micrograms of the films deposited from *cis*-Fe(CO)₄(SnMe₃)₂ on a Si substrate at 300–390 °C are presented in Figure 4. The grain size increased with increasing deposition temperature and ranged from 0.2 to 2 μ m. Electron probe microanalyses also show even distribution of Fe and Sn elements in the films.

X-ray diffraction analyses of these films show the existence of polycrystalline materials. Diffraction pat-

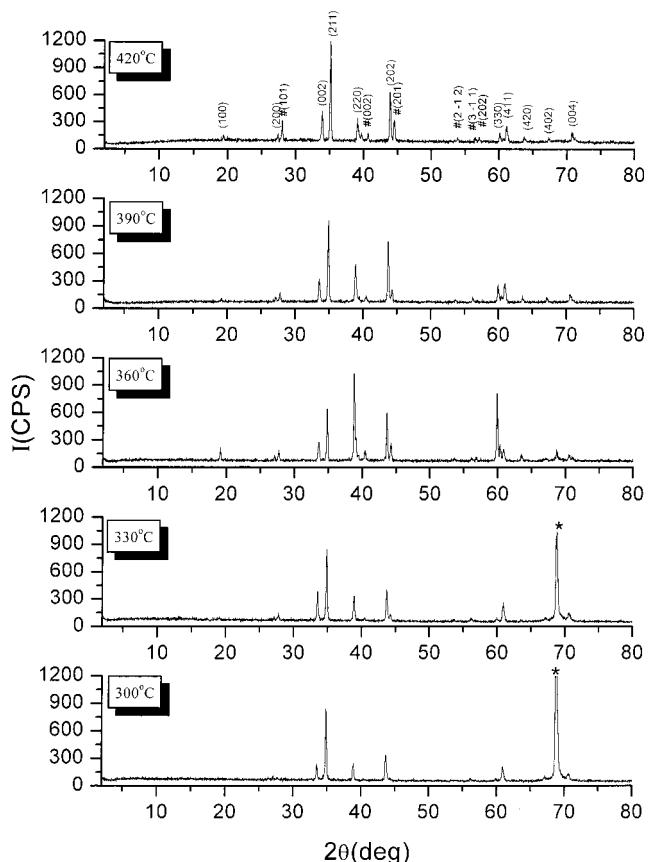


Figure 5. XRD patterns of the films deposited from *cis*-Fe(CO)₄(SnMe₃)₂ at 300–420 °C. (*Si substrate; # FeSn phase).

terns of the films deposited on Si from *cis*-Fe(CO)₄(SnMe₃)₂ at 300–420 °C are shown in Figure 5. The pattern of the film grown at 300 °C indicated the

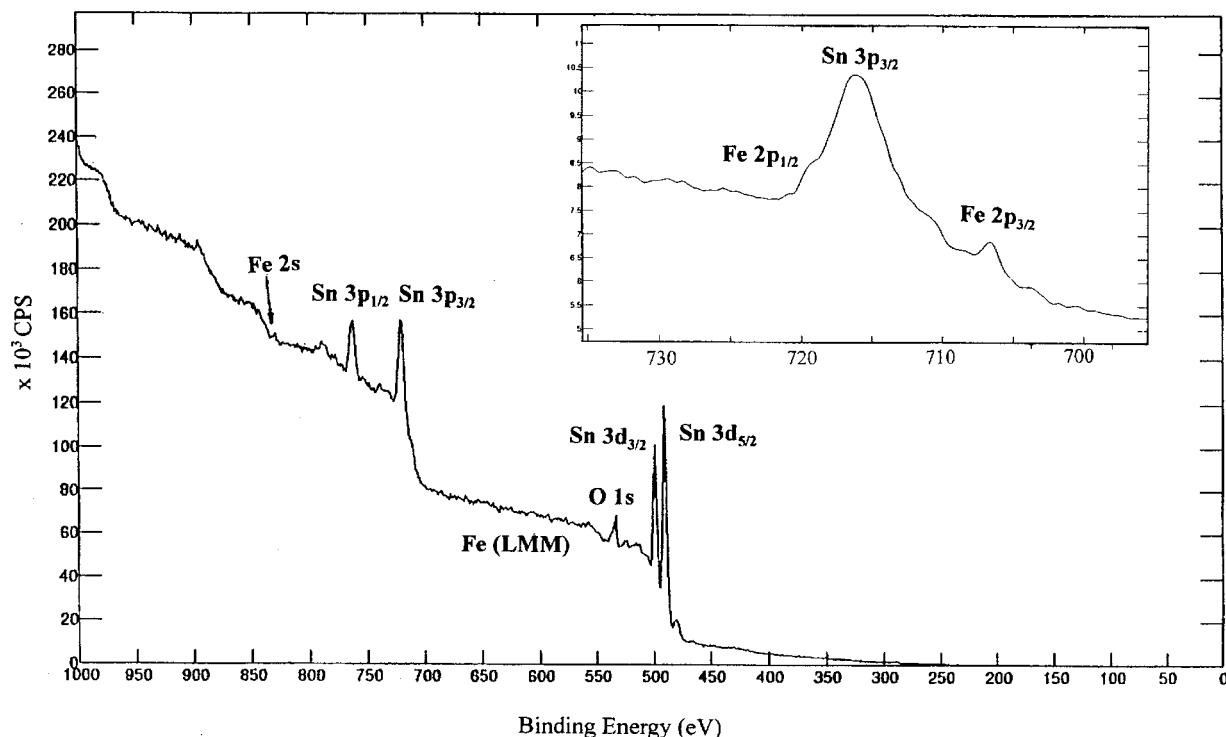
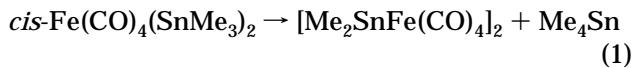


Figure 6. Auger electron spectrum for the film deposited from *cis*-Fe(CO)₄(SnMe₃)₂ at 390 °C. Inset shows Fe 2p_{3/2} and Fe 2p_{1/2} peaks.

existence of FeSn₂ phase. As the deposition temperature increased, the pattern of a minor constituent of FeSn phase appeared in the spectra.³¹ Energy-dispersive spectroscopic and atomic absorption spectroscopic analyses (see Table 2) demonstrate that Fe/Sn ratios in the films are slightly greater than 0.5. An Auger electron spectrum of a sample prepared at 390 °C (Figure 6) reveals that Fe and Sn exist in the film. Oxygen impurities likely result from the surface contamination since the spectrum was taken without sputtering the surface. These data suggest that the films consist of the FeSn₂ phase with a small amount of the FeSn phase. It is known that the compound undergoes slow decomposition according to eq 1 to give [Me₂SnFe(CO)₄]₂ and Me₄Sn.³²



This decomposition may occur when the precursor is travelling near the hot substrate. The minor component of FeSn phase is likely a result of the pyrolysis of a small amount of [Me₂SnFe(CO)₄]₂.

Both FeSn and FeSn₂ are stable in the atomic range of iron between 50 and 67% at reaction temperatures.³³

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Thus, the formation of the products seems to be thermodynamically controlled. Hot-wall CVD experiments have also been conducted, however, providing amorphous films with random Fe/Sn composition. These are probably due to the decomposition of precursors prior to their reaching the substrates.

Conclusion

We have successfully used two organometallic complexes containing a Fe–Sn bond in different Fe/Sn ratios, CpFe(CO)₂(SnMe₃) and *cis*-Fe(CO)₄(SnMe₃)₂, to manufacture the polycrystalline FeSn and FeSn₂ thin films, respectively. It demonstrated the stoichiometric control of the deposition by using the appropriate precursors. These results are encouraging for further development of heterobimetallic, single-source precursors to deposit intermetallic thin films.

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