found to be reduced (Table V). Hence, the mesomorphic behavior displayed by the Az (alkoxy, alkyl) series may be mainly attributed to the nature of the para,para' groups.

Concluding, a further point concerning the role played by the ligand's aromatic molecular core can be elucidated by comparing the nature of the mesophases exhibited by the different palladium complexes with reference to the uncomplexed ligands. In particular, it may be pointed out that the nematic (enantiotropic or monotropic) azobenzene species, and the smectic imine give nematic or smectic complexes, respectively. The nematic imine Im (CH_3O, C_4H_9) gives a smectic and nematic complex while the nematic 2-phenylpyrimidine species gives smectic complexes only (Table V). With respect to the previously discussed trend, which accounts for the presence of more ordered mesophases in the palladium derivatives rather than in the ligands, we can then conclude that such an effect is enhanced for the 2-phenylpyrimidine complexes. Nevertheless, this behavior can be explained by looking at the structural features of the Az, Im, and Pp cyclopalladated dimers (Chart IV).

Thus, evident is the similarity between the Az and Im structures, wherein the nonmetalated ring is free to rotate, and their difference with the comparatively more rigid Pp geometry imposed by the heteroaromatic nitrogen atom involved in the cyclopalladated ring. Recently, Espinet and Serrano, reporting on palladated azine dimers,[10d] ob-

served that a greater mobility to the non-ortho-palladated moiety may cause either the lowering of the transition temperatures (Table V) or the occurrence of the mesophases. Hence, in the present case, the mesomorphic behavior displayed by the 2-phenylpyrimidine complexes might be ascribed to their extremely rigid molecular structure.

Growth and Characterization of Thin Films of Thallium(III) Oxide by Organometallic Chemical Vapor Deposition

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Films containing thallium(III) oxide were grown by organometallic chemical vapor deposition in an oxygen-rich atmosphere on MgO, Al_2O_3, and Si substrates. Three organothallium precursors were used: thallium acetylacetonate, dimethylthallium acetylacetonate, and cyclopentadienylthallium. Films were characterized by using X-ray diffraction techniques and X-ray photoelectron, Auger electron, and infrared spectroscopies. The results indicate the presence of Tl_2O_3 as the major component with possible smaller amounts of Tl_2CO_3. Deposition on Si resulted in the formation of a silicate from interaction between the film and the substrate.

Introduction

Thallium(III) oxide has electrical and optical properties that make it potentially useful in solar cells and transparent electrical contacts.[1] Interest in this material has grown further with the discovery of the high-temperature superconducting material Tl–Ba–Ca–Cu oxide.[2] Although thin films of metal oxides have been deposited by a variety of techniques including evaporation, sputtering, hydrolysis, and organometallic chemical vapor deposition (OMCVD),[3] studies of Tl_2O_3 have been limited by the unique set of problems involving the toxicity, volatility, and stability of thallium compounds. To our knowledge, films of Tl_2O_3 have not been grown by OMCVD, although Tl–Ba–Ca–Cu

oxide has been deposited by this technique.[4,5] In this study, we report the growth and characterization of Tl_2O_3 films from thallium acetylacetonate, dimethylthallium acetylacetonate, and cyclopentadienylthallium on MgO, Al_2O_3, and Si substrates.

Experimental Section

Organometallic Precursors. Thallium acetylacetonate (Tl(acac)) was purchased from Strem Chemicals, Inc., and sublimed at 90–120 °C and 10^{-5} Torr before use. Dimethylthallium acetylacetonate ($Me_2Tl(acac)$) was synthesized according to published procedures from the reaction of trimethylthallium and acetylacetone[6] and characterized by IR, ^1H NMR, and elemental

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Table I. Comparison of X-ray Diffraction Data for Tl$_2$O$_3$ and Films Deposited on MgO

hkl	Tl$_2$O$_3$[11]		Tl(acac)		Me$_2$Tl(acac)		CpTl (350 °C)		CpTl (450 °C)	
	d	I/I$_0$	d	I/I$_0$	d	I/I$_0$	d	I/I$_0$	d	I/I$_0$
222	3.042	100	3.041	100	3.041	96	3.041	100	3.039	26
400	2.635	40	2.634	79	2.634	100	2.634	47	2.633	100
			2.627	44	2.628	55	2.627	26	2.627	55
440	1.863	35			1.864	2				
600	1.758	1	1.756	3	1.756	3	1.765	2	1.756	5
			1.753	1	1.752	2	1.753	1	1.753	3
622	1.589	25								
444	1.522	6	1.522	18	1.521	18	1.522	28	1.522	10
			1.518	9	1.517	10	1.518	15	1.518	6

analyses. The IR and ^{1}H NMR spectra were in agreement with published data.[7] Me$_2$Tl(acac): Anal. Calcd: C, 25.21; H, 3.93. Found: C, 25.61; H, 4.05 (E + R Microanalytical Laboratory, Inc., Corona, NY). A melting point of 245–248 °C with decomposition was recorded in contrast to 214–215 °C reported previously.[8] Cyclopentadienylthallium (CpTl) was synthesized according to published procedures and identified by its infrared spectrum.[9]

Film Deposition. Film depositions were carried out at ambient pressure in a vertical Pyrex reactor. Substrates were cleaned sequentially with detergent, distilled water, isopropyl alcohol, boiling TCE, and isopropyl alcohol, dried under a stream of nitrogen, and placed on an inductively heated, rotating graphite susceptor. Results of depositions with substrate temperatures of 350 and 450 °C are reported here. Precursor transport lines were wrapped with heater tape, and source temperatures varied in the ranges 125–130 °C for CpTl, 135–140 °C for Me$_2$Tl(acac), and 140–145 °C for Tl(acac). Nitrogen was passed over the heated precursors at 150 cm^3/min and mixed with oxygen flowing at 1000 cm^3/min prior to entering the reactor.

X-ray Photoelectron Spectroscopy (XPS). A Surface Science Instruments Model 100-03 spectrometer with a base pressure of 1 × 10^{-8} Torr or less was employed to obtain XPS spectra. Initial wide-scan spectra were taken to identify the elements in the surface region with a pass energy of 100 V and a spot size of 0.6 mm. Narrow-scan spectra of the detected elements were subsequently recorded at a pass energy of 50 V and a spot size of 0.3 mm. Under the conditions of the narrow scans, the full width at a half-maximum (fwhm) for the Au 4f$_{7/2}$ peak was approximately 0.9 eV.

Samples of thallium(I) carbonate and thallium(III) oxide were mounted on indium foil to be used as standards. Films from depositions using Tl(acac) on MgO and Si substrates were examined. Charging effects observed with some samples were overcome by using a flood gun with a nominal 2.5 V and a charge-neutralization screen.

Peaks were fitted with a nonlinear least-squares program supplied with the spectrometer. All peaks were shifted to compensate for charging so that the adventitious carbon peak was at 284.6 eV.

Auger Electron Spectroscopy (AES). The AES spectra were recorded with a Physical Electronics Ind. Model 10-155 instrument using a cylindrical mirror analyzer. The samples used for XPS noted in the previous section were also examined with AES.

Infrared Spectroscopy. The IR spectra were acquired on a Digilab FTS-90 Fourier transform infrared spectrometer (Digilab Division, Bio-Rad Inc., Cambridge, MA) with either a narrow-band or wide-band mercury–cadmium–telluride detector with respective cutoffs of 700 and 450 cm^{-1}. Transmission spectra of the films on Si and MgO were taken with a 4× beam condenser in transmission mode (Harrick Scientific, Ossining, NY), giving a resultant nominal spot size of 0.75 mm. Spectra were recorded at either 4- or 2-cm^{-1} resolution, and 256–1024 scans were co-added to obtain adequate signal-to-noise. Triangular apodization was used, and all computations were done in double precision (32 bits). Reference spectra of chromic acid cleaned Si wafers and freshly

cleaned MgO as previously described were used as required to obtain absorbance spectra.

Microscopy, X-ray Diffraction, and Resistivity Measurements. Nomarski photographs were taken at 50× magnification by using a Nikkon Optiphot microscope. X-ray diffraction spectra were obtained on a Philips APD 3520 powder diffractometer equipped with a Huber horizontal goniometer and a Cu Kα radiation tube. Resistivity measurements were made using a four-point probe technique consisting of an Alessi four-point probe head and a Keithly 225 current source and 197 digital multimeter.

Results and Discussion

As noted above, the source temperatures of all three precursors were quite similar. Cyclopentadienylthallium appeared to be the most volatile compound, followed by Me$_2$Tl(acac) and Tl(acac) in that order. Vapor pressures at the source temperatures used during deposition were low and were believed to be in the range of 2 Torr or less based on mass-loss calculations. No evidence of decomposition at these temperatures was observed for either CpTl or Me$_2$Tl(acac), but a dark brown residue was left by Tl(acac).

Deposition on MgO. Cyclopentadienylthallium was the only precursor to give a deposit on MgO at the lower substrate temperature of 350 °C. The use of oxygen saturated with water vapor did not improve the efficiency of deposition and actually inhibited film growth at 350 °C with CpTl. Substrate temperatures above 400 °C were required to deposit films with the acetylacetonates as source materials.

Films deposited on MgO from all precursors were similar in appearance, consisting of crystallites ranging in size from 10 to 100 μm as shown in the dark areas of the Nomarski photographs at 50× in Figure 1. Coverage was not continuous except for large quantities of deposited material, and considerable growth perpendicular to the substrate surface was observed. This suggested an island or layer plus island growth mode in which the deposited species were bound more strongly to each other than to the surface.[10]

Data from the X-ray diffraction patterns of all films on MgO are listed in Table I along with the relevant peaks for Tl$_2$O$_3$.[11] There is excellent agreement between the d values of the films and those of Tl$_2$O$_3$. The difference in intensities of the (222) and (400) reflections from film to film represents an unexplained variation in orientation. Other thallium-containing materials such as Tl metal,[12] Tl(OH),[13] Tl$_2$O,[14] Tl$_4$O$_3$,[15] and Tl$_2$CO$_3$[16] also contain re-

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(12) JCPDS No. 30-1341.

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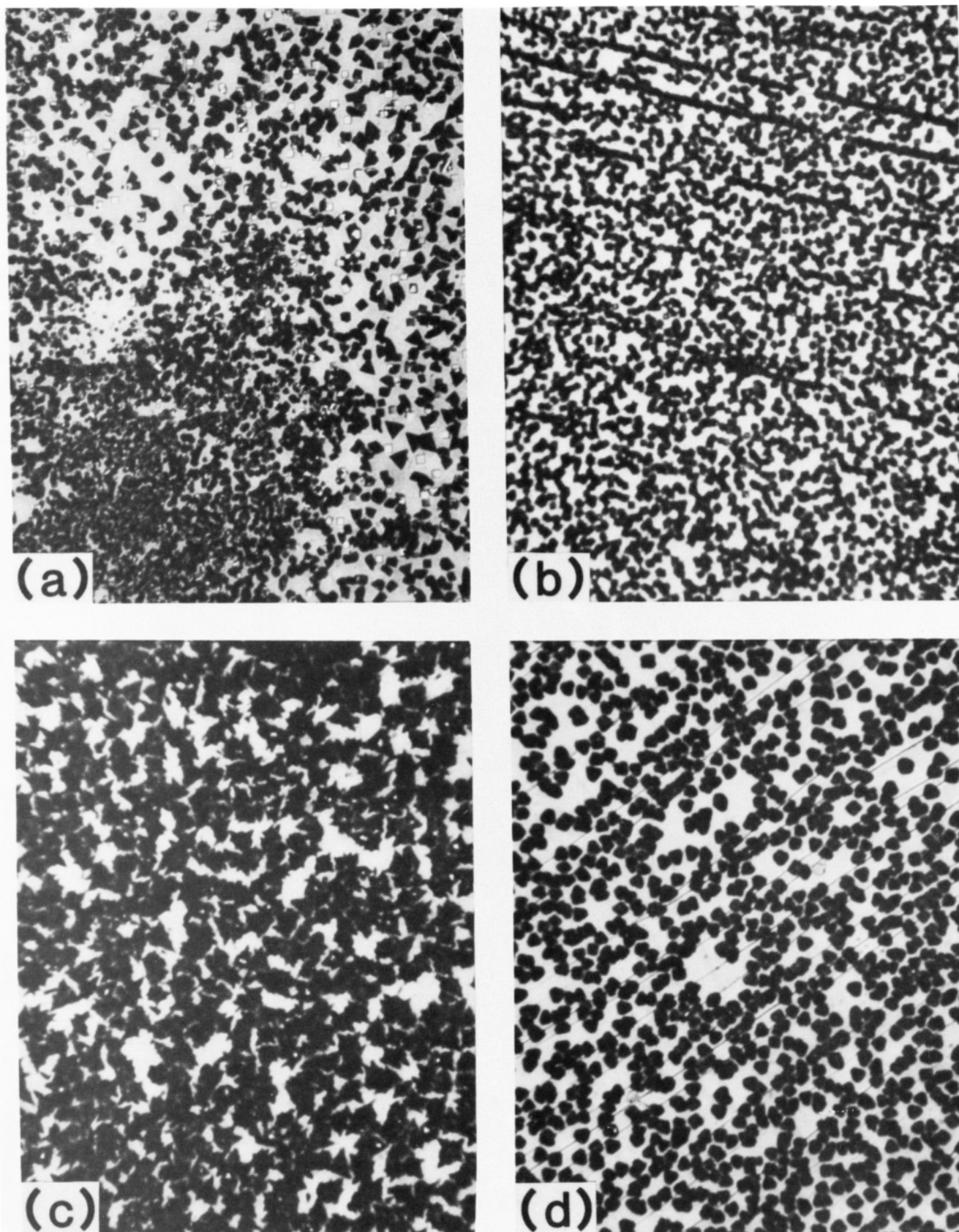(14) JCPDS No. 23-584.

(15) JCPDS No. 23-585.

(16) JCPDS No. 20-1259.

Figure 1. Nomarksi photographs at 50× for films on MgO deposited from (a) Tl(acac) + O_2, 450 °C, (b) Me_2Tl(acac) + O_2, 450 °C, (c) CpTl + O_2, 450 °C, and (d) CpTl + O_2, 350 °C.

flections close to those of (222) and (400) and consequently must be considered as possible film components on the basis of X-ray data alone. An evaluation of the properties of these materials and the results of several analytical techniques ultimately provided the means for film characterizations.

A qualitative consideration of physical and chemical properties revealed that it would be unlikely for several of these materials to be present as the principal film components under the deposition conditions used. For example, Tl metal oxidizes readily under ambient conditions and would not be expected to exist in the high-tem-

**Table II. XPS Binding Energies^a (electronvolts) for Various Tl Samples**

XPS line	Tl$_2$O$_3$	Tl$_2$CO$_3$	Tl(acac) + O$_2$ (on MgO as deposited)	Tl(acac) + O$_2$ (on Si as deposited)	Tl(acac) + O$_2$ (on Si after swab)
Tl(4f$_{7/2}$)	117.0		116.8	116.8	117.0
	118.2	118.3	118.2	118.1	118.8
C(1s)	284.6	284.6	284.6	284.6	284.6
	287.5	286.1	286.7		
		288.3	288.7	288.0	
O(1s)	528.3		528.3	528.2	
	530.5	530.1	530.9	530.4	530.5
			532.2	532.2	532.2
Mg(2s)			88.6		
Si(2p)					102.6

^{a} All peaks have been shifted such that the main C(1s) line is at 284.6 eV.

perature, oxidative deposition environment. Thallium hydroxide, which would form from the hydration of Tl$_2$O via eq 1, is volatile (vapor pressure = 13 Torr at 46 °C)

$$Tl_2O + H_2O \rightarrow 2Tl(OH) \qquad (1)$$
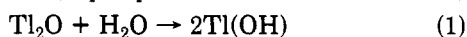
and absorbs CO$_2$ to form Tl$_2$CO$_3$ when exposed to air as shown by eq 2.[17] Furthermore, the hydroxide would be

$$2Tl(OH) + CO_2 \rightarrow Tl_2CO_3 + H_2O \qquad (2)$$
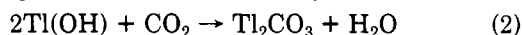
expected to give rise to O–H stretching vibrations in infrared spectra of the films, and this was not seen as discussed below. Thallium(I) oxide is hygroscopic as described in eq 1 and is volatile,[18] which makes it unlikely that a large amount of this would be present. Thallium carbonate is a white crystalline solid that melts at 272 °C and shows loss of CO$_2$ above 175 °C to form Tl$_2$O;[19] consequently, it should not be present in appreciable quantities at our deposition temperatures.

The qualitative effect of temperature on the amount of material in these films and their X-ray diffraction patterns was examined by a post-deposition heat treatment of a film deposited from CpTl on MgO at 450 °C. After 2 h at 450 °C in a nitrogen atmosphere, there was little change in appearance or in the X-ray pattern. Additional heating at 550 °C for 2 h resulted in a decrease in the intensity of the sample X-ray peaks compared to that of the MgO substrate and minor changes in relative intensities; moreover, the visible amount of deposit on the substrate had decreased significantly.

From the above information, we believe that the oxides Tl$_2$O$_3$ and Tl$_4$O$_3$ are the most likely possibilities for the principal component(s) of our films. Thallium(III) oxide is a brown, black solid that melts at 716 °C under 1 atm of oxygen[19] and vaporizes in the range 530–675 °C according to eq 3.[20] As described earlier, the X-ray pattern

$$Tl_2O_3(s) \rightarrow Tl_2O(g) + O_2 \qquad (3)$$
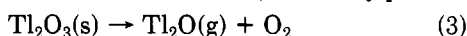
of Tl$_2$O$_3$ contains two intense peaks that are in agreement with those observed in our films. Tetrathallium trioxide, Tl$_4$O$_3$, is a black, mixed oxide (3Tl$_2$O·Tl$_2$O$_3$) prepared by heating Tl$_2$CO$_3$ and Tl$_2$O$_3$ at 450 °C in an inert atmosphere.[19] However, since the X-ray diffraction pattern of Tl$_4$O$_3$ contains no peak with a d value of 2.63 Å,[15] as observed in our films, it cannot be the sole component in our films.

Results from a mass spectral analysis of material deposited on MgO using CpTl suggested the presence of an oxide and a carbonate. Heating the sample in a direct-insert probe produced an increase in peaks at 44 and 32 amu, corresponding to CO$_2$ and O$_2$, beginning at 300 °C and continuing up to 500 °C. No evidence for Tl-containing peaks in the vapor was found. The observation of CO$_2$ and O$_2$ under these conditions was consistent with the thermal decomposition products of Tl$_2$CO$_3$[19] and Tl$_2$O$_3$,[20] but it did not provide any information about their relative amounts.

The presence of both oxide and carbonate in films deposited from Tl(acac) on MgO was also supported by XPS results as shown in Table II. Note that only the position of the 4f$_{7/2}$ peak in the Tl 4f$_{7/2}$–4f$_{5/2}$ doublet is reported. Data from the Tl$_2$O$_3$ and Tl$_2$CO$_3$ standards provided some interesting qualitative information about these compounds. For example, the observation of two Tl 4f$_{7/2}$ and two O 1s peaks in Tl$_2$O$_3$ suggested two forms of Tl and O were present. It has been proposed that two different crystallographic sites exist for Tl in Tl$_2$O$_3$,[21] which may account for this behavior. Furthermore, earlier XPS spectra reported for Tl$_2$O$_3$ were taken at a resolution that would not have resolved Tl peaks about 1 eV apart.[22] The position of the lower binding energy peak for O in this sample was somewhat unusual but is consistent with that reported for some high-temperature superconductors[23] and heavy metal oxides.[24] In contrast, the Tl$_2$CO$_3$ standard had only one Tl 4f$_{7/2}$ and one O 1s peak. The C peak at 284.6 eV in all samples corresponded to adventitious C, as noted in the Experimental Section. The peaks at 287.5 and 286.1 eV for Tl$_2$O$_3$ and Tl$_2$CO$_3$, respectively, appeared as small shoulders on this main peak and were also attributed to surface contamination. The highest energy C 1s peak at 288.3 eV in Tl$_2$CO$_3$ was consistent with a carbonate species.

Spectra of as-deposited films on MgO contained two Tl 4f$_{7/2}$ and three O 1s peaks. The Tl peaks and two of the three O peaks had binding energies close to those found in Tl$_2$O$_3$. Moreover, the Tl and O peaks (118 and 530 eV, respectively) and the highest energy C 1s peak were also consistent with the spectrum of Tl$_2$CO$_3$. The O 1s peak at 532 eV suggested the presence of a hydroxy or single-bonded carbon–oxygen species. The Mg 2s peak in the film on MgO may have resulted from a nonuniform deposit or Mg diffusion. Attempts to differentiate between these possibilities using scanning Auger microscopy were not successful.

Auger spectroscopy of an as-mounted sample on MgO from a deposition using Tl(acac) proved to be less defi-

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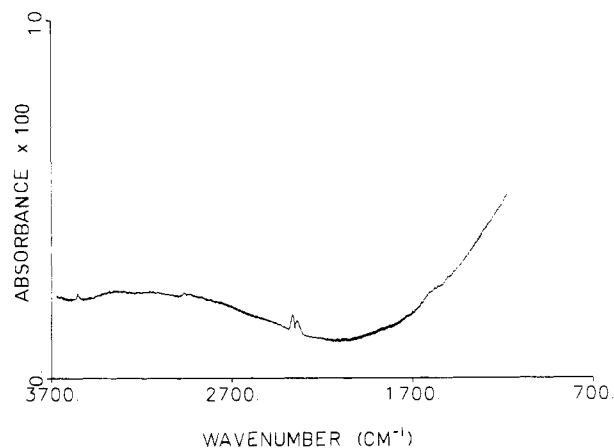
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76 *Chem. Mater., Vol. 3, No. 1, 1991*

Berry et al.

Figure 2. Infrared spectrum of film deposited from Tl(acac) + O₂ on MgO at 450 °C.

**Table III. X-ray Diffraction Data for Tl(acac) Films Deposited on Si**

350 °C		450 °C	
d	*I/I₀*	*d*	*I/I₀*
3.790	49	3.795	76
		3.156	43
3.035	10		
2.537	100	2.537	5
		2.105	100
1.903	6	1.898	13

nitive than XPS. Peaks corresponding to Tl, O, and adventitious C were observed, and the latter was removed after sputtering for 3 min with Ar^+. The spectra of Tl_2CO_3 and Tl_2O_3 on indium foil also contained similar Tl and O peaks and showed no distinction between the Tl(I) and Tl(III) oxidation states. No C peak was seen in the Tl_2CO_3 spectrum, which was analogous to the behavior of the Ca, Sr, and Ba carbonates.[25,26]

Infrared spectroscopic analyses of films grown from Tl(acac) and CpTl on MgO were consistent with Tl_2O_3, not Tl_2CO_3, being the primary film component. The spectra were essentially featureless throughout the mid-infrared region from 4000 to 458 cm^{-1} (Figure 2), as were those of Tl_2O_3 in KBr pellets. This was consistent with the behavior of conductive materials that are optically dense with few or no spectral features. The absence of bands in the O–H stretching region near 3600 cm^{-1} or in the carbonate stretching region between 1300 and 1500 cm^{-1} ruled out the presence of substantial quantities of Tl(OH) or Tl_2CO_3.

The conclusions from infrared data are further supported by four-point probe resistivity measurements on a film grown from CpTl on MgO. As with all of our films, the surface proved to be too rough for thickness measurements by profilometry, but this sample appeared to be continuous under a light microscope. Assuming a thickness of 1 μm, resistivities of 50–110 μΩ cm were calculated by using the formula $\rho = V\pi t/(I \ln 2)$, where *t* is the film thickness.[27] The resistivity of single-crystal Tl_2O_3 is approximately 80 μΩ cm at room temperature.[21]

Deposition on Si. Films deposited on Si from Tl(acac) were considerably different from those on MgO. Reflective surfaces with interference fringes were observed in the early stages of growth. Longer deposition times produced
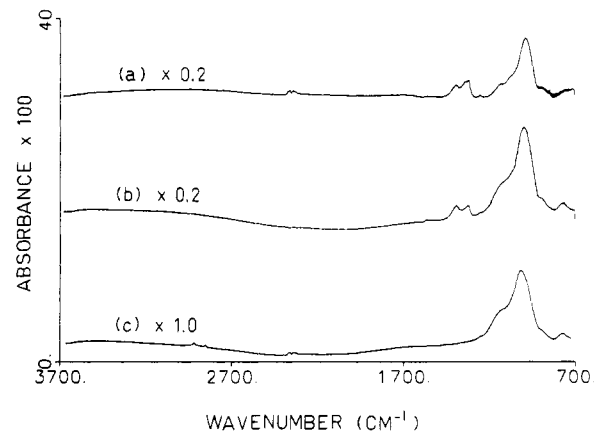
Figure 3. Infrared spectra of films deposited from Tl(acac) + O₂ on Si: (a) at 350 °C, (b) at 450 °C, and (c) at 450 °C after swabbing with petroleum ether. Absorbance values in spectra a and b have been multiplied by 0.2.

a hazy appearance with scattered, shiny, metallic-like areas. X-ray data from films deposited at 350 and 450 °C are listed in Table III and contain reflections at positions found in serveral compounds including Tl_2O_3,[11] Tl_2O,[14] Tl_4O_3,[15] Tl_2CO_3,[16] and $Tl_8Si_5O_{14}$.[28] The infrared spectra shown in Figure 3a,b contain bands near 1400 (m), 1330 (m), 1250 (w), 1130 (sh), 1000 (s), 900 (sh), and 780 (s) cm^{-1}. The film deposited at 450 °C was investigated with a longer wavelength detector, and an additional band was observed at 455 cm^{-1}. The terminology "near" with respect to the band maxima is used since the frequencies of the bands shifted slightly for the two deposition temperatures. The relative intensities of the bands at 1390 and 1325 cm^{-1} changed with deposition temperature, and a splitting of the 1325-cm^{-1} band occurred in the film deposited at 350 °C. This meant that either the peak at 1390 cm^{-1} and the one at 1325 cm^{-1} were due to different species, or the spectral changes were due to a change in the crystalline environment of the same species. We prefer the former interpretation for the following reasons:

(a) The 1390-cm^{-1} band showed substantially less shift (±4 cm^{-1}) at the two temperatures than did the 1325-cm^{-1} band (±10 cm^{-1}).

(b) The band at 1390 cm^{-1} could be assigned to the degenerate asymmetric stretch for the carbonate ion (observed at 1390 cm^{-1} for our sample of Tl_2CO_3 in a KBr pellet).

(c) The band at 1325 cm^{-1} could not easily be associated with Tl_2CO_3 and remains unassigned. Although David and Ault[29] observed a band at 1311 cm^{-1} in matrix-isolated Tl_2CO_3, this was the weaker component of the split degenerate pair of the asymmetric CO_3^{2-} stretch. The second stronger component occurred near 1500 cm^{-1}, and no feature at that frequency was observed in any of our spectra.

The species responsible for the higher energy bands were not strongly bound to the surface, as they could easily be removed by rubbing the film with a swab containing petroleum ether; this left a brown residue on the swab and a comparatively brighter deposit on the substrate. Since the surfaces of both the as-deposited and the swabbed films were too rough to obtain reliable profilometry measurements, it was not possible to estimate the relative amount of material removed in this manner. Both bands in the 1300–1400-cm^{-1} range disappeared, and the lower

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frequency bands (below 1300 cm^{-1}) all remained (Figure 3c), implying a stronger interaction between this material and the Si. The formation of silicate type species in the high-temperature oxidative environment during deposition would account for the adherent species. Indeed, the infrared spectrum of Zn_2SiO_4 published in the Sadtler handbook[30] is very similar to the low-energy infrared profile observed from our films deposited on Si substrates. Considering that a myriad of $Si_xO_y{}^{n-}$ species exhibit a series of infrared bands[30] below 1200 cm^{-1}, it seems reasonable to attribute the low-frequency bands of the films on Si to silicate species. This might also account for the shifts in frequency observed for these bands under different deposition conditions. Different experimental conditions could give rise to slightly different $Si_xO_y{}^{n-}$ species. Moreover, this would be consistent with a mechanism whereby an original silicate-type species is formed during the initial stages of deposition. Subsequently, purely physisorbed thallium species, including Tl_2O_3 (which has no bands in the mid-infrared) and Tl_2CO_3, are deposited over this silicate layer. X-ray diffraction analysis of the swabbed substrates revealed no distinguishable peaks, indicating that the remaining films were amorphous.

Results from an XPS analysis of a film grown on Si using Tl(acac) (Table II) supported the hypothesis that physisorbed Tl_2O_3 and Tl_2CO_3 were deposited over an initial thallium silicate layer. A spectrum of the as-deposited film was similar to that grown on MgO, which suggested the presence of both oxide and carbonate on the surface as discussed above. The spectrum of the swabbed sample

contained significant differences from that of the as-deposited material. First, the high-energy C peak attributed to the carbonate species was not present; this was consistent with the infrared data showing the removal of bands in the 1300-1400-cm^{-1} range by swabbing. Second, the spectrum of the swabbed sample contained a Si peak at a binding energy consistent with oxidized Si.[31] This was also in agreement with infrared data indicating the formation of a silicate from the interaction of the film and substrate. The absence of Si in the spectrum of the as-deposited sample could have been the result of its being covered by a subsequent layer as suggested above or the relatively low sensitivity of Si in the X-ray photoelectron spectrum. For the swabbed sample, the absence of the low-energy O(1s) peak indicated that the surface region was not similar to that of Tl_2O_3.

Depositions on Other Substrates. Depositions were carried out on other substrates in attempts to produce continuous films at lower quantities of deposited material. On Al_2O_3 at 450 °C, CpTl gave a film whose appearance and X-ray pattern were similar to those of material deposited on MgO. On a buffer layer of $SrCO_3$ deposited on MgO, CpTl also gave a discontinuous black film whose strongest peaks in the X-ray pattern were found at 2θ = 34.00° (d = 2.634 Å) and 29.36° (3.039 Å).

(30) *The Sadtler Infrared Spectra Handbook of Minerals and Clay*; Ferraro, J. R., Ed.; Sadtler Research Laboratories: Philadelphia, PA, 1982.

(31) Grunthaner, F. J.; Grunthaner, P. J. *Mater. Sci. Rep.* **1986**, *1*, 65.

Surface Characterization Study of InP(100) Substrates Using ISS, AES, and ESCA. 3. Experimental Artifacts

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A surface characterization study has been performed on InP(100) substrates. Both solvent-cleaned, native oxides and sputter-cleaned InP have been examined by using multiple surface characterization techniques including ion scattering spectroscopy (ISS), Auger electron spectroscopy (AES), and electron spectroscopy for chemical analysis (ESCA). The data obtained indicate that the results may be difficult to interpret due to experimental artifacts including compositional alteration by ion sputtering and electron stimulated desorption (ESD) and chemical state alteration. This combination of techniques is powerful because ISS, AES, and ESCA probe varying depths beneath the surface. When used carefully, these techniques yield a nondestructive depth profile of the near-surface region of the sample.

Introduction

Ultrahigh-vacuum (UHV) surface analytical techniques have grown in popularity and use in the past 20 years. Today, it is fairly common for an analytical laboratory to have a stand-alone, computer-interfaced Auger electron spectroscopy (AES) or electron spectroscopy for chemical analysis (ESCA or XPS) system. Other UHV surface analytical techniques including secondary ion mass spectrometry (SIMS), ultraviolet photoelectron spectroscopy (UPS), scanning Auger microscopy (SAM), scanning

electron microscopy (SEM), and electron dispersive X-ray analysis (EDX), ion scattering spectroscopy (ISS), etc., are also being used more extensively. Although the development of the field of surface science is still in its early stages, the power of these techniques has been demonstrated and is generally accepted. Unfortunately, a belief has developed that the sample is inserted, an automated analysis is performed, and the concentration or other desired information is read off a computer screen or an X–Y plot. The goal of this paper is to describe some of the experi-