Textures and Microstructures, 1991, Vols 14–18, pp. 543–554
Reprints available from the publisher
Photocopying permitted by license only

TEXTURE DEVELOPMENT IN THIN METALLIC FILMS

D.B. KNORR, D.P. TRACY, AND T.-M. LU
Center for Integrated Electronics
Rensselaer Polytechnic Institute
Troy, New York 12180-3590 U.S.A.

ABSTRACT

Aluminum films 1μm in thickness are deposited on SiO_2/Si(100) by the self-ion assisted PIB technique and by sputtering. Texture analysis of the four deposition conditions reveals an {111} texture component superimposed on a random grain distribution. The relative fraction of the two components depends strongly on deposition conditions. Annealing at 400°C for 0.5 hr sharpens the {111} texture component at the expense of the random while inducing substantial grain growth where mean grain size increases by a factor of 3.5 to 5. One condition is truly a fiber texture, and the other three have textures with some deviation from axial symmetry.

INTRODUCTION

Thin films deposited by physical vapor deposition (PVD) processes often show a preferred orientation. The degree of orientation can vary from weakly textured to epitaxial where only one single orientation is present. The character of the texture (strength, principle components, etc.) depends on substrate, PVD technique, and deposition conditions. The substrate can be either single crystal, polycrystalline, or amorphous. PVD techniques include evaporation, sputtering, and ion-assisted deposition with either inert gas ions or self-ions. A variety of deposition parameters are important such as vacuum level, background pressure in sputtering, atom or ion energy (usually measured in eV per atom), substrate temperature, substrate cleanliness, source-target geometry, and deposition rate. Thus, it is evident that the formation and microstructural development of thin films is a complex process dependent on a large number of variables.

If as-deposited grain size is very fine, substantial grain growth occurs upon annealing. Normal grain growth kinetics are extensively studied in thin metallic films. An important metric is the grain size as compared to the film thickness. The phenomenon of stagnation results when the grain size exceeds the thickness of the film followed by abnormal grain growth at higher temperature.

Very few systematic studies of texture development in thin metallic films have been reported. Often[1,2,3], a texture index such as $I_{\{111\}}/I_{\{200\}}$ derived from a Bragg diffraction scan is used to assess the strength of the texture. The information derived is limited due to the small sampling of points in orientation space. Pole figures reveal substantially more information on the intensity and distribution of texture components. Thin metallic films of Al[4], Cu[5], and Au[6] deposited by PVD techniques are known to form fiber textures.

The effects of deposition conditions and annealing treatments on microstructural development are reported for pure aluminum thin films deposited on oxidized (amorphous) Si(100) substrates. Texture, grain size, and grain size distribution are reported as a function of depositon condition and of subsequent annealing treatment to 400°C.

EXPERIMENTAL PROCEDURES

Thin Film Processing

Two deposition techniques are used: 1) partially ionized beam[7] (PIB), and 2) sputtering. In the PIB technique a small fraction of evaporated aluminum atoms is ionized while a potential is applied to the substrate during deposition. Both neutral aluminum atoms and self-ions are deposited on the substrate simultaneously. The energetic ions provide enhanced surface mobility during deposition and effective in-situ cleaning. A schematic diagram of the experimental apparatus is shown in Figure 1.

The substrate bias potential (ion energy) is fixed at 2 kV. Aluminum films are deposited at approximately 10Å/sec on Si(100) wafers TEOS oxidized to give approximately 1000Å of amorphous SiO_2. Ion contents of the beam are 1% and 2%. The substrate is held at room temperature, and background pressure is 10^{-4} Pa during deposition. Sputtered films were provided by

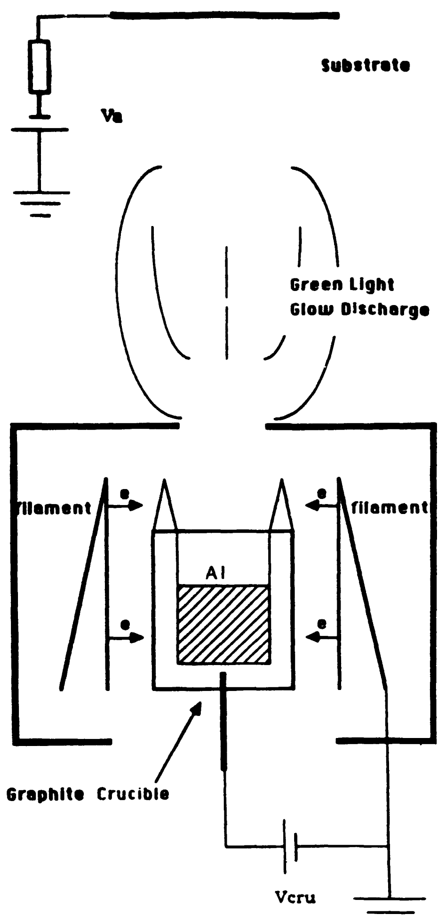

Figure 1. Schematic of PIB deposition system.

an outside source where the power settings on the sputtering unit were set at either 2 kV or 4 kV. All Al films are about 1µm in thickness.

Samples from all four deposition conditions are annealed in an Ar - 3% H_2 atmosphere at annealing temperatures of 250, 300, 350, and 400°C for 0.5 hours.

Microstructural Analysis

Texture is measured by {111} pole figures. Data are obtained in reflection to a maximum tilt angle ϕ of 85°. Two correction factors must be applied to yield accurate relative intensity values. Geometric defocusing is corrected with a random powder sample.

Absorption is important because the X-ray penetration depth exceeds the film thickness. The correction factor due to Schultz[8] is used. Corrected and normalized (random = 1) data are plotted either as {111} intensity versus tilt angle ϕ for fiber textures or as pole figures when sample symmetry is less than axial.

Grain size and grain size distributions are determined by transmission electron microscopy. The silicon wafer is thinned from the backside, then ion milled until the specimen becomes electron transparent. Grain size is measured from photomicrographs taken in the TEM. The photographs are point counted on a digitizing pad by the linear intercept method to determine the grain size distribution. Between 427 and 1090 grains are counted for the various conditions with an average of 616 grains. The grain size distributions are plotted as cumulative probability versus log grain size. Mean grain size is the value at the 50 point on the distribution.

RESULTS

Four deposition histories are reported: 1) PIB with 2 kV and 1% ions (PIB-2/1), 2) PIB with 2 kV and 2% ions (PIB-2/2), 3) sputtered at 2 kV power (Sp-2), and 4) sputtered at 4 kV power (Sp-4). The most extensive analysis is done on the as-deposited films and after a 400°C/0.5 hr annealing treatment.

A typical cumulative grain size probability plot is shown in Figure 2 for condition PIB-2/2. A straight line implies that the distribution is log normal. The cumulative probability plots give mean grain size which is plotted versus temperature in Figure 3. Typical microstructures in both as-deposited and annealed 400°C/0.5 hr are shown in Figure 4.

Texture results for both as-deposited and 400°C/0.5 hr conditions are shown in Figures 5-7. {111} intensity versus tilt angle ϕ are given for condition PIB-2/1 in Figure 5. Pole figures from condition PIB-2/2 are shown in Figure 6. Pole figures for both sputtered conditions are presented in Figure 7. Two important quantities derived from the pole figure data are peak {111} intensity which is at or near the center of the pole figure and random {111} intensity which

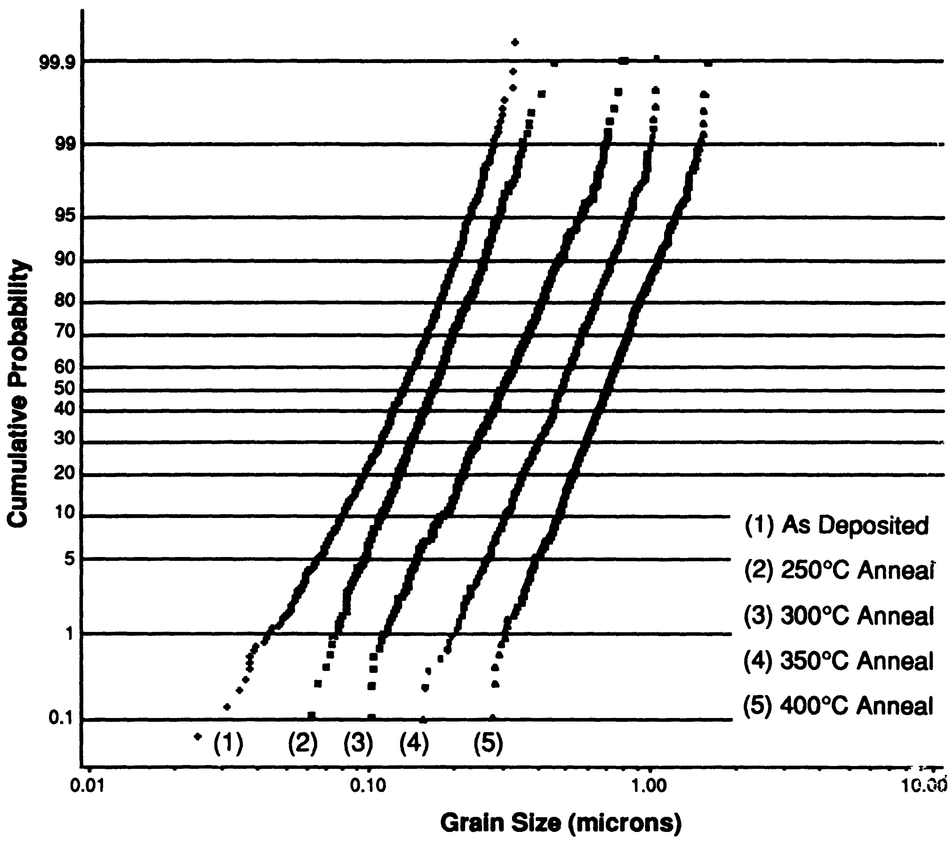

Figure 2. Cumulative probability plot of grain size for condition PIB-2 kV/2% ions.

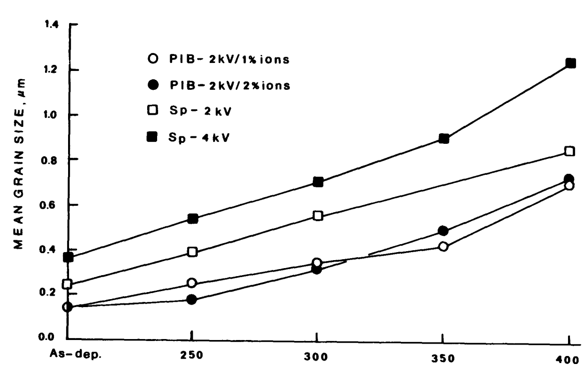

Figure 3. Mean grain size as a function of processing for four deposition conditions.

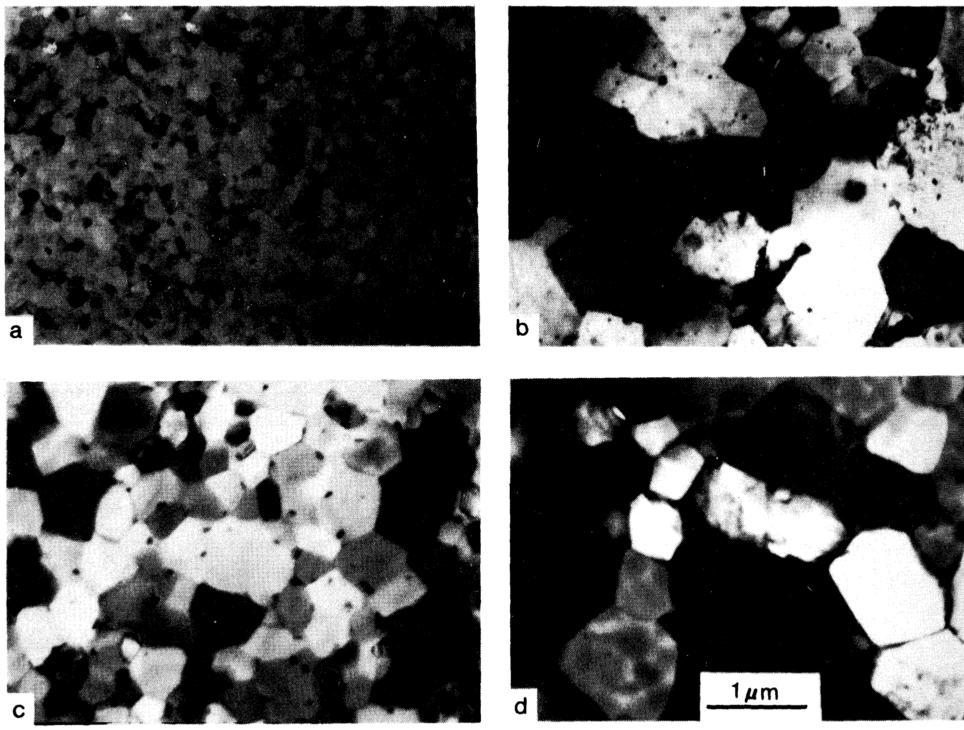

Figure 4. TEM micrographs showing grain structure: a) PIB-2/1, as-deposited: b) PIB-2/1, 400°C/0.5 hr; c) Sp-4, as-deposited; d) Sp-4, 400°C/0.5 hr. The marker in micrograph d) is common for all micrographs.

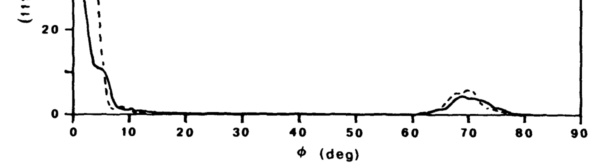

Figure 5. {111} pole intensity as a function of tilt angle ϕ or condition PIB-2 kV/1% ions. Intensity units are multiples of random.

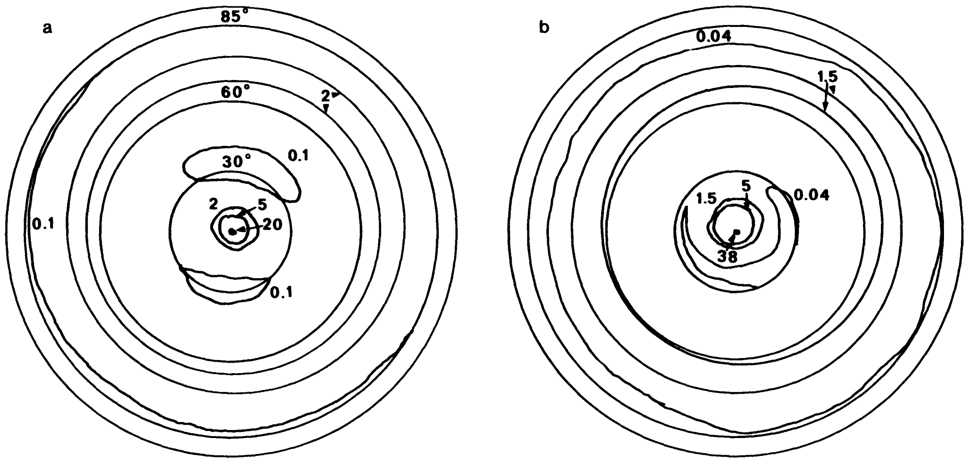

Figure 6. {111} pole figures for condition PIB - 2 kV/2% ions: a) as-deposited; b) 400°C/0.5 hr.

is taken at 35-40° in tilt angle to avoid both the major {111} peak at or near the center and the smaller complementary peak about 70.5° in tilt angle. The grain size and normalized intensity values are listed in Table 1.

Table 1.

Summary of Microstructure Data

Deposition Condition	Processing History	Mean Grain Size (μm)	Peak Intensity	Random Intensity
PIB-2/1	As-deposited	0.14	48	0.25
	400°C/0.5 hr	0.71	74	0.10
PIB-2/2	As-deposited	0.14	23	0.11
	400°C/0.5 hr	0.74	38	0.02
Sp-2	As-deposited	0.24	2.2	0.82
	400°C/0.5 hr	0.85	5.9	0.68
Sp-4	As-deposited	0.36	5.8	0.31
	400°C/0.5 hr	1.25	9.8	0.16

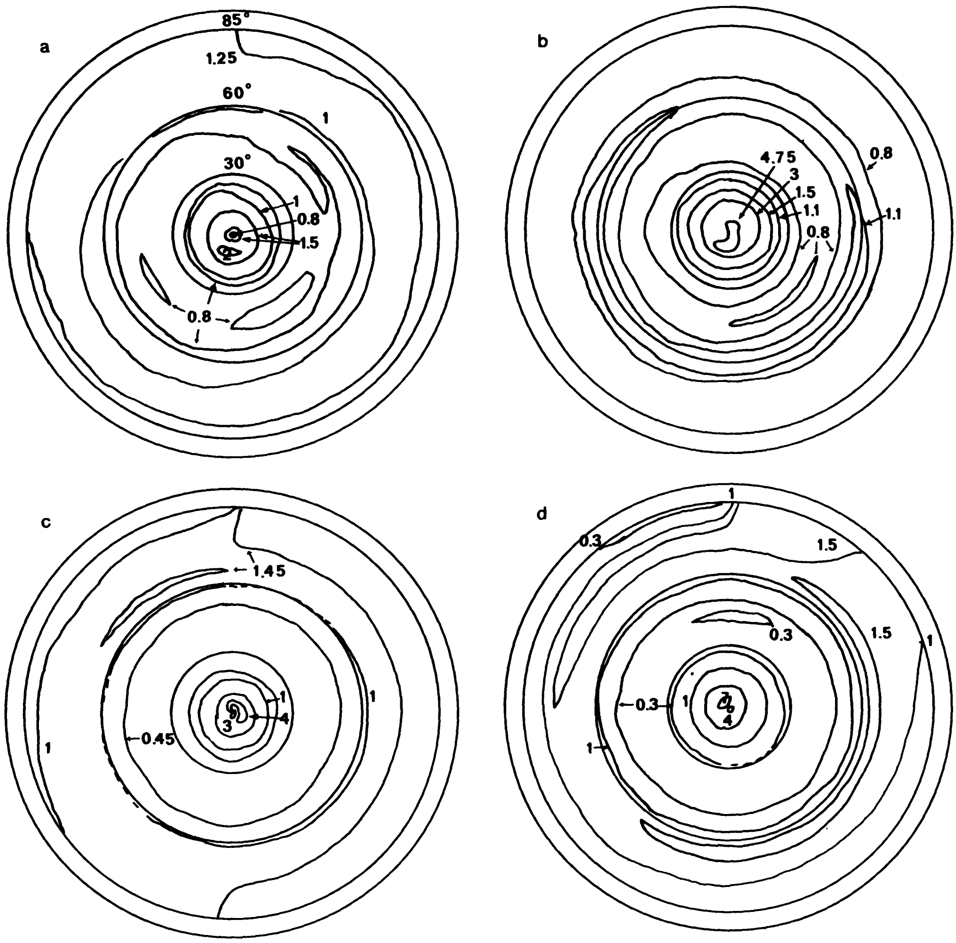

Figure 7. {111} pole figures for sputter deposited materials:
a) SP - 2 kV, as-deposited;
b) Sp - 2 kV, 400°C/0.5 hr;
c) Sp - 4 kV, as-deposited;
d) Sp - 4 kV, 400°C/0.5 hr.

DISCUSSION

Two texture components are evident in all conditions where an {111} texture component is observed to superimpose on a random background. The relative quantities of random and {111} vary depending on both deposition condition and annealing history. Two attributes of the {111} texture component are the peak intensity value and the spread as measured at the base of the component by the angle ω. The arrangement is shown schematically in Figure 8. The fraction of each texture component and ω are summarized in Table 2 while peak intensity values are given in Table 1.

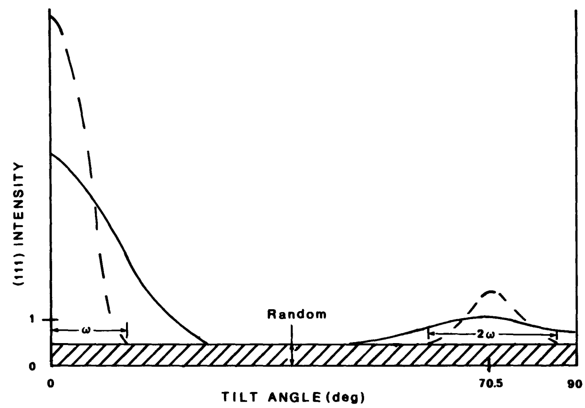

Figure 8. Schematic representation of random and {111} texture components. The sharper {111} component has a higher intensity and smaller ω.

Table 2.

Summary of Major Texture Components

Deposition Condition	Processing History	Fraction of Texture Component: Random	Fraction of Texture Component: {111}	ω(deg in ϕ)
Sp-2	As-deposited	0.82	0.18	21
	400°C/0.5 hr	0.68	0.32	18
Sp-4	As-deposited	0.31	0.69	18
	400°C/0.5 hr	0.16	0.84	16
PIB-2/1	As-deposited	0.25	0.75	13
	400°C/0.5 hr	0.10	0.90	10
PIB-2/2	As-deposited	0.11	0.89	12
	400°C/0.5 hr	0.02	0.98	11

As a group, sputtered films are more weakly textured than PIB films. The Sp-2 condition has a very weak

{111} texture component while Sp-4 is somewhat stronger, similar to an evaporated condition previously reported[4]. The PIB films have much stronger textures[4] which depend on deposition condition. Higher deposition energy appears to strengthen the {111} texture component. The higher sputtering power at 4 kV deposits more energy in the film as evidenced by the larger as-deposited grain size. Likewise, the relatively high average atom energy during PIB deposition produces sharp textures. The process of texture evolution and microstructural coarsening during deposition is complex as seen in the deposition and microstructure study of Vaidya and Sinha[1] so care should be taken in generalizing these observations.

Annealing has similar effects on all four conditions. A substantial increase in grain size is observed while the grain size distribution is well represented by log normal behavior[8]. The maximum mean grain sizes after the 400°C/0.5 hr anneal are on the order of the film thickness so grain growth stagnation should just be becoming important. The most striking changes are in the textures as evident by the characteristics reported in Tables 1 and 2. The {111} component is sharper and occupies a greater fraction of the grains at the expense of the random component when compared to the respective as-deposited condition. Grain growth appears to favor grains with {111} orientation due to both a decrease in the random component and sharpening of the {111} component.

A surprising aspect of the texture analysis is the breakdown of the expected axial symmetry in three out of four conditions. PIB-2/1 is the only true fiber texture. PIB-2/2 has its peak intensity at the center of the pole figure, but the {111} distribution is skewed toward the top of the pole figure as drawn in Figure 6. The same trend is observed after annealing. The two sputtered conditions show the most deviation from a fiber texture. The peak intensity is located off the center of the pole figure. Annealing moves the peak intensity slightly toward the normal direction where the tilt angle changes from 6° to 4.5° for Sp-4 and from 8° to 7° for Sp-2. The assymmetry could be due to source-target geometry. Deposition at an oblique angle tends to induce texture at an angle corresponding to normal incidence of the metal vapor[9,10]. This hypothesis cannot be tested without specific details on deposition geometry.

CONCLUSIONS

Deposition of thin aluminum films using four different deposition conditions and subsequent annealing of the films produces several interesting trends in texture and its evolution.

1. An {111} texture component superimposes on a random texture component. The relative fraction of the two components depends on deposition condition.

2. Annealing at 400°C for 0.5 hr substantially increases mean grain size by a factor of 3.5 to 5. The {111} texture component sharpens and increases in volume fraction at the expense of the random component.

3. Deviations from a fiber texture are found for three out of four conditions. The sputtered films have peak {111} intensity 6° to 8° off specimen normal. The location of the peak intensity is close but not equivalent to a fiber axis.

REFERENCES

1. S. Vaidya and A.K. Sinha, *Thin Solid Films,* 75, 253 (1981).

2. A.S. Yapsir, L. You, T.-M. Lu, and M. Madden, *J. Mater. Res.,* 4, 343 (1989).

3. P. Bai, G.-R. Yang, T.-M. Lu, and L.W.M. Lau, *J. Vac. Sci. Technol.,* A8, 1465 (1990).

4. D.B. Knorr and T.-M. Lu, *Appl. Phys. Lett.,* 54, 2210 (1989).

5. D.B. Knorr and T.-M. Lu, *Textures and Microstructures,* in press.

6. F. Witt, R.W. Vook, and M. Schwartz, *J. Appl. Phys.,* 36, 3686 (1965).

7. S.-N. Mei and T.-M. Lu, J. *Vac. Sci. Technol.,* A6, 9 (1988).

8. B.M. Tracy, P.W. Davies, and D. Fanger, *Microstructural Science for Thin Film Metallizations in Electronic Applications* (ed. by J. Sanchez, D.A. Smith, and N. Delanevolle, TMS, Warrendale, PA, 1988), p.157.

9. T. Kamimori, H. Fujiwara, T. Hashimoto, and K. Hara, *Thin Solid Films,* 81, 377 (1981).

10. T. Hashimoto, K. Okamoto, K. Hara, M. Kamiya, and H. Fujiwara, *Thin Solid Films,* 91, 145 (1982).