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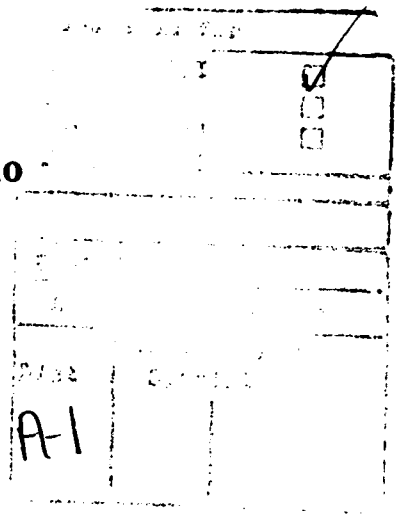

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NON-DESTRUCTIVE EVALUATION OF RESIDUAL STRESSES IN THIN FILMS VIA X-RAY DIFFRACTION TOPOGRAHY METHODS

by

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ABSTRACT

Quantitative x-ray diffraction topography techniques have been used to measure the residual strain magnitude and uniformity of deposition for Mo and W sputtered films on Si(100) substrates. High sensitivity rocking curve measurements were able to determine differential strains for films as thin as 2.5nm; while Bragg angle contour mapping had similar sensitivity and was also able to assess coating uniformity and stress distribution over areas covering a whole wafer. Measurements of strain versus film thickness over a range of 2.5nm to 80nm showed that a critical thickness exists for maximum residual strain. Growth beyond this range produces stress relaxation. This non-destructive type of analysis could be employed on a wide range of film-substrate combinations.

I. INTRODUCTION

Thin film refractory metal coatings on Si substrates are of interest in the semiconductor industry for a variety of purposes, including diffusion barriers, high temperature contacts and potential x-ray lithography masks. Dimensional stability of the film-substrate couple is an important consideration in processing and ultimate utilization. Studies of the strain and concomitant residual stress properties of these films have been reported for a number of Si-Metal systems. For an overview of the past developments in this field the reader is referred to a number of comprehensive reviews.[1,2,3,4,5,6,7] However, almost all studies of Si-metal couples have been performed either on thick films (>100 nm) under non-UHV conditions (i.e. not atomically clean) or on very thin films under UHV conditions (i.e. atomically clean, but not reflecting the actual growth situation for many engineering applications). It is apparent that the early stages of film growth play a critical role in determining the ultimate microstructure even for thick films (arbitrarily defined here as films > 100 nm). To better understand the mechanisms controlling film microstructure and to enable eventual design of film properties for a wide variety of applications, characterization studies beginning at a very early stage of film growth under non-UHV conditions is needed.

Sputtering, one of the most widely used film deposition processes in industry, has been chosen as our film growth method. Non-destructive x-ray analysis using asymmetric double crystal diffraction topography (DCDT) to quantitatively measure strains and map them out in two dimensions over areas comparable to wafer dimensions was the major tool

for assessing the residual strains as a function of growth thickness. This method has a high strain sensitivity, of the order of $\Delta d/d \approx 10^{-7}$, is capable of exploring large areas, is not affected by surface roughness: since the curvature of the crystal lattice planes is determined and not the curvature of the physical surface, and finally it is completely non-destructive. Major improvements in DCDT techniques, over those previously reported,[8,9] have been achieved by using a modified double crystal camera system of higher sensitivity and stability and also by employing image processing tools which have allowed strain sensitivities to extend down to examinations of layers as thin as 2.5 nm. The focus of this report will consider the development of residual strain (stress) in W and Mo metallizations on Si(100) as a function of film thickness, during the early stages of growth, using this non-destructive method.

II. EXPERIMENTAL PROCEDURE

Double crystal x-ray diffraction (DCD) has been commonly employed in studies of epitaxial films to measure rocking curves for particular hkl reflections; usually observations of the (400)-(400) reflections in the parallel (+,+) mode.[10,11,12,13,14,15] Rocking curves of single crystals can also be used to assess the residual strain in the substrate from measurements of the line broadening, usually expressed in terms of full width at half maxima, FWHM. This is a sensitive method but does not allow one to visualize the distributions of the strain (stress) over the full dimensions of a wafer sample.

In earlier work by Renninger it had been shown that for a bent sample only that portion of the crystal lattice which is in the Bragg

condition will create a topographic image on a x-ray photographic film detector.[16,17,18] By rotating the sample about an axis perpendicular to the plane of diffraction (i.e. the plane defined by the incident and diffracted beam vectors) a series of contours can be observed which map out equi-inclination of the crystal lattice planes. From this information the crystal lattice curvature of the sample can be determined.[9] Improvements in collimation, energy resolution, goniometer design and computer control have made it possible to measure the differential curvatures for even a few monolayers. Refractory metal films are especially amenable to this technique. Consider the experimental observation as shown in Fig. 1(a) for an uncoated Si(100) wafer. In this case the x-rays are almost evenly reflected from the (400) planes of the sample and the microdensitometry trace shown in Fig. 1(b) exhibits a large flat plateau. This latter observation indicates a virtually strain-free initial condition. However, when non-uniform residual strains are present in the initial wafer, which is the typical situation, then the x-ray image and microdensitometry is quite different as pictured in Fig. 1(c) and (d). Most of our as received wafers were in this latter condition, which would be acceptable for measuring large strains in thick films, but is unacceptable where high sensitivity observations are required. Therefore in the present experiments only those wafers having uniform initial flatness were used to assess the strains associated with subsequent deposition.

The set-up of the DCDT method is shown schematically in Fig. 2. A nearly perfect, asymmetric-cut, Si(100) single crystal is used as a monochromator crystal. Highly parallel x-rays corresponding to 8 KeV are obtained from the (400) diffraction peak of this first crystal. Using grazing angle incidence Cu K(α) x-rays, a large area coverage is achieved which is

essential for examining film deposition uniformity and stress distribution over the whole Si sample wafer under study (i.e. the 2nd crystal).[19] The sample is adjusted, by rotating it about an axis perpendicular to the plane of diffraction, such that the monochromator-sample crystal pair is tuned for the (400)-(400) reflections to minimize the dynamical effects on the rocking curve. Polaroid high speed x-ray film camera is put in the path of diffracted beam as shown on the right hand side of Fig. 2 and a line (no. 1) is recorded which maps out the trace of the region of the wafer that is in the Bragg condition. Subsequent rotation, in increments ω, produces an additional line (no. 2, Fig. 2) which maps out a new contour which is now equi-inclined to the incident beam. When uniform curvature exists, a set of bands on the image of equal spacing are found. For more complex bending, or for small strains where high sensitivity is needed, the spacing between Bragg angle contour lines can be readily found by frame-grabbing the image with a TV camera, then using microdensitometry to find the optical density and hence the exact spatial position. Note that high resolution x-ray photographic film is not necessary. By knowing the rotation angle, and the contour line spacing, the curvature, K, of the bent sample can be readily found as has been previously reported.[20]

$$K = 1/R = \omega \sin \theta / D \qquad (1)$$

Where R is the radius of curvature, ω is the rotation angle (as shown on Fig. 2), θ is the Bragg angle (Si(400) = 34.58°) and D is the distance between successive Bragg angle contours on the x-ray photographic image. An example of Bragg angle contour results from a 80nm (800Å) Mo metallization, before and after sputtering, using this methodology is shown

in Fig. 3 (a). Fig. 3 (b) depicts the corresponding microdensitometry where it can be seen that the contour line spacing changes by approximately 50% from that determined for the same wafer prior to metallization.

Once the curvature is known the corresponding film stress is determined by using the modified Stoney equation.[21,22] This equation was originally derived on the following assumptions: the substrate is a plate that is thin, elastically isotropic, the film coating has uniform thickness, much less than the substrate thickness, and a uniform isotropic plane stress exists. For Si single crystal substrates, these assumptions may not be totally satisfied. However, recent work has shown that the anisotropy associated with certain crystalline substrates is completely consistent with the original equation.[23] It is not the focus of the present work to evaluate the details of the theory of determining film stress from curvature observations, but rather to introduce a new, and highly accurate method of sample curvature analysis; therefore, the Stoney approach will be assumed valid. Thus:

$$\sigma = \frac{E t_o^2 (K - K_o)}{6 (1 - \nu) t} \qquad (2)$$

Where E is Young's modulus for the Si substrate, ν is Poisson's ratio for Si, t_o is the thickness of the Si wafer, K is the curvature after coating, K_o is the initial curvature of the uncoated wafer and t is the sputtering metallization thickness.

Both DCDT and rocking curves measurements are made concurrently with the same Double crystal camera system. The x-ray photographic film detector is interchangeable with a scintillation counter which can be readily used to determine the rocking curve of the (400)-(400) Si

diffraction peak. The FWHM of the rocking curve will reflect the distortion in the direction normal to the wafer surface and can also be used to check whether the film is epitaxially grown; while the DCDT is used to map out the strain field in the plane parallel to the substrate surface due to film sputtering over the whole sample area. Rocking curves and DCDT were taken on all samples before and after sputtering.

P-type Si(100) prime wafers with resistivities of 1- 3 Ω·cm from UniSil Corporation were used for all experiments. The Mo films were deposited on these substrates by DC sputtering in a planar-type sputter system, TORUS, at ambient temperature. The basic parameters for the sputtering process were: base pressure, 10^{-6} torr; Ar pressure, 3×10^{-3} torr; sputtering current 0.3A; and a sputtering rate ≈ 0.08 nm/sec. Metallization thicknesses of 2.5, 5.0, 10.0, 20.0, and 80.0nm, were prepared for Mo and comparable range for W (excluding a 2.5nm sample). The thickness is controlled by sputtering time according to the calibrated sputtering rates. For the thinnest films Auger sputter depth profiling was performed to verify the thickness calibration which were found to be in good agreement.

III. RESULTS AND DISCUSSIONS

Figs. 3(a) and 4(a) show topographic images of 20nm (200Å) and 80.0nm (800Å) Mo films, before and after sputtering, and the corresponding image analysis for determination of the D values (i.e. spacing between DCDT lines) is shown in Figs. 3(b) and 4(b). This type of

data is typical of observations for all film thickness values. The curvature here is about an axis normal to the plane of diffraction and nominally in the (010) direction of the Si substrate. By rotating the sample 90° about an axis perpendicular to the surface (i.e. the (100) direction), one can assess the curvature in the (001) direction. When simple bending is involved the DCDT image would shrink to a point upon this rotation, but when compound bending occurs a new set of DCDT lines (of possible different spacing for the general anisotropic case) would appear. In these experiments, all films showed compound bending with nearly isotropic strains in two orthogonal directions (010) and (001). Thus samples were shaped like a dish rather than a simple bent beam indicating a uniform biaxial stress.

Figs. 5 and 6 display plots of the differential curvature ($K - K_o$) before and after sputtering as determined from equation (1). A rather complex relationship exists between the strains induced by deposition and film thickness, especially below 20nm. Originally all uncoated wafers are slightly concave. Focussing on Mo metallizations in Fig. 6, for the thinnest 2.5 nm film, the sample becomes less concave; then as film thickness increases the sample reaches a minimum differential curvature at 5.0nm (i.e. sample relaxes back towards its original curvature). Further sputtering makes the sample more and more concave again. Recall that differential curvature is being plotted, and since the sample is initially concave, film strains never reach sufficient magnitude to change the sign of the curvature to convex. The situation is qualitatively similar for W films as can be seen in Fig. 5.

The physical consequences of the film deposition stresses were also monitored with rocking curve measurements. This data is shown in Table I for Mo.

The intrinsic FWHM of perfect Si(400) is approximately 2.2 arcsec. Thus Table I demonstrates the sensitivity of the rocking curve measurements. The present double crystal camera system is capable of making observations in FWHM to within 0.1 arcsec. Note that the strain determined here from rocking curve analysis is the average .alue over the whole penetration depth of the x-rays into the wafer. In this case, for Si which has very low x-ray absorption, one averages over the whole 380 μm wafer thickness. Also, DCDT gives the physical curvature of the lattice planes parallel to the crystal surface while the rocking curves give the average $\Delta d/d$ in the direction perpendicular to the film surface. The rocking curves show that once strains are sufficiently high it is relatively easy to make quantitative assessments. For example, consider Mo metallization at 80nm in Table I, significant line broadening occurs and the calculated stress from rocking curve analysis is approximately 500MPa. For very thin films < 20nm, there is qualitative agreement using rocking curve analysis in the sign of the differential curvature and the strains are of the order expected. Thus both methods, rocking curve analysis and DCDT, indicate complex stress-thickness relationships for refractory metal films below 20nm.

Table I

Si(400)-(400) rocking curves results: before and after metallization.

Sample thickness (nm)	FWHM (arcsec) Before Coating	FWHM (arcsec) After Coating	Strain* (10^{-6})
2.5	3.5	2.7	-8
2.5	3.4	2.7	-7
2.5	3.1	2.6	-5
2.5	3.0	2.8	-2
5.0	3.2	3.0	-2
5.0	3.1	2.9	-2
5.0	2.8	3.0	+2
5.0	3.4	2.9	-5
10.0	2.8	3.1	+3
10.0	2.7	3.2	+5
10.0	3.0	3.1	+1
10.0	3.3	3.1	-2
20.0	3.2	3.4	+2
20.0	2.9	3.0	+1
20.0	2.8	2.6	-2
80.0	2.5	10.0	+75
80.0	2.6	9.2	+66
80.0	2.9	9.2	+63
80.0	2.9	7.2	+43

*Note: Strain, $\Delta d/d$, is determined from the difference in FWHM between values after film deposition and that observed before coating.

Figs. 7 & 8 illustrate the film stress determined from DCDT using the Stoney formulation. Positive stress values refer to tensile and negative to compressive states in the film. Earlier work on films > 20nm thickness level had indicated that the stress-thickness dependence for refractory metal films on Si was not monotonic.[24] In fact, in the initial stages of growth it would be surprising if the stress-thickness relationship was simple. Most observations fail to report data below 20nm, as far as can be determined from a search of the literature. Thus, it is difficult to compare the present results on very thin Mo and W films with the work of others. Nevertheless, it is possible in sputtered films that a number of factors can affect the early stages of growth stress-thickness dependence: first the apparent thickness for full area coverage must be reached, the state of crystallinity of the film must be determined (i.e. does the film start out crystalline or amorphous and is there a transition thickness from one type of structure to the other), the nature and concentration of interfacial impurities would have a much more profound effect on the early stages film growth compared to thicker microstructures, and the nature of the evolving microstructure must be considered, eg. nanocrystalline vs grain growth and possible grain texture development. These are some of the issues that must be understood to gain a full appreciation of the mechanisms for film stress as function of thickness in the early stages of film growth. Such information is essential to gain control over the resultant microstructure for particular applications as the film thickens. Now that detailed means are available for measuring the stress-thickness dependence of film growth, as described in the present work, experiments are planned to evaluate the microstructural and microchemical causes. The present techniques should be capable of evaluating stress dependency

in a variety of other cases such as strained-layer superlattice materials combinations and so on, as long as the displacements are in the ranges of sensitivity of the method ($\approx 10^{-7}$). The apparent critical thickness where stresses peak as a function of film thickness for Mo, for example, is probably due to the competition of the various microstructural aspects of growth and is not the same as that usually considered in heteroepitaxy.[25]

CONCLUDING REMARKS

The DCDT method described in the present report is capable of measuring the strain (stress) state of thin films in a simple, fast, highly sensitive and non-destructive fashion. Another feature of this method is that it actually measures the curvature of the crystal lattice planes of the substrate while most other methods, like laser scanning and cantilever beams, measure the curvature of the physical surface. The strain measured is due to the bending of the crystal lattice, and is not obscured by any possible roughness of the surface. The method is so simple and quick that it may potentially be used in-situ if a high intensity source such as a synchrotron were available with a growth chamber at an end station.

The stress state of thin Mo and W films on Si(100) substrates have been shown to have complicated mechanisms which will be the subject of future investigations.

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FIGURE CAPTIONS

Fig. 1 (a) and (b), Double Crystal Diffraction Topographic (DCDT) image of a good quality (nearly zero residual stress) original wafer and a corresponding microdensitometry plot, respectively. Optical density is determined along the trace approximately in the middle of the wafer in part (a). Each division on the distance scale in (b) corresponds to 5mm.

(c) and (d), DCDT image of a original wafer with non-uniform residual stress and corresponding microdensitometry plot, respectively. Optical density is determined along the trace approximately in the middle of the wafer in part (c). Each division on the distance scale in (d) corresponds to 5mm.

Fig. 2 Schematic of DCDT experimental set-up. Monochromator is the first Crystal and Sample is the second Crystal. As sample is rotated in direction shown contour line moves to left, from position 1 to 2.

Fig. 3 (a) DCDT image, of a 80.0nm Mo film. The image on the left is the substrate before sputtering. The same sample is shown on the right after sputtering. The image processing system stores them side-by-side and then the microdensitometry scan is taken across the white trace.

(b) Corresponding microdensitometry analysis of the DCDT line spacings. The scan of the substrate before sputtering goes from 0.0 to 30mm and the scan from 30 to 56mm corresponds to the same

sample after. Before sputtering DCDT bands are 7.8mm apart and after average 3.8mm apart.

Fig. 4 (a) DCDT image, before (left) and after (right) sputtering of a 20.0nm Mo film. Taken with image processor same as Fig. 3.
(b) Corresponding microdensitometry analysis of the DCDT line spacing. Before scan from 0.0 to 30.0mm and after, 30.0 to 57.2mm.

Fig. 5 Differential Curvature $K-K_o$, i.e. the difference in the curvature between the value after and before sputtering, for Tungsten films as a function of thickness.

Fig. 6 Differential Curvature $K-K_o$, i.e. the difference in the curvature between the value after and before sputtering, for Molybdenum films as a function of thickness.

Fig. 7 Tungsten film stress calculated from equation 2, as a function of thickness.

Fig. 8 Molybdenum film stress calculated from equation 2, as a function of thickness.

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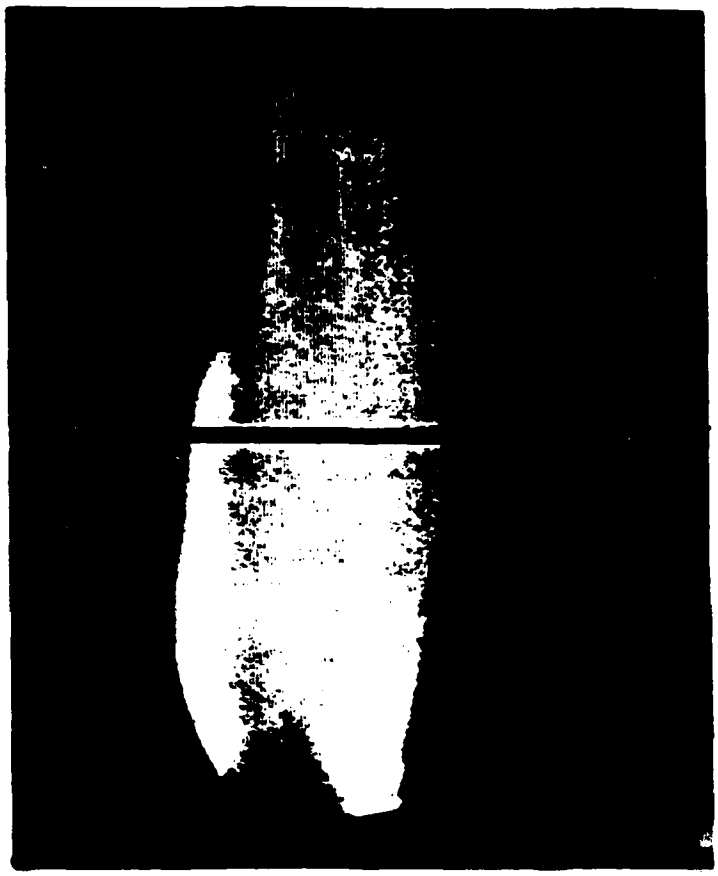

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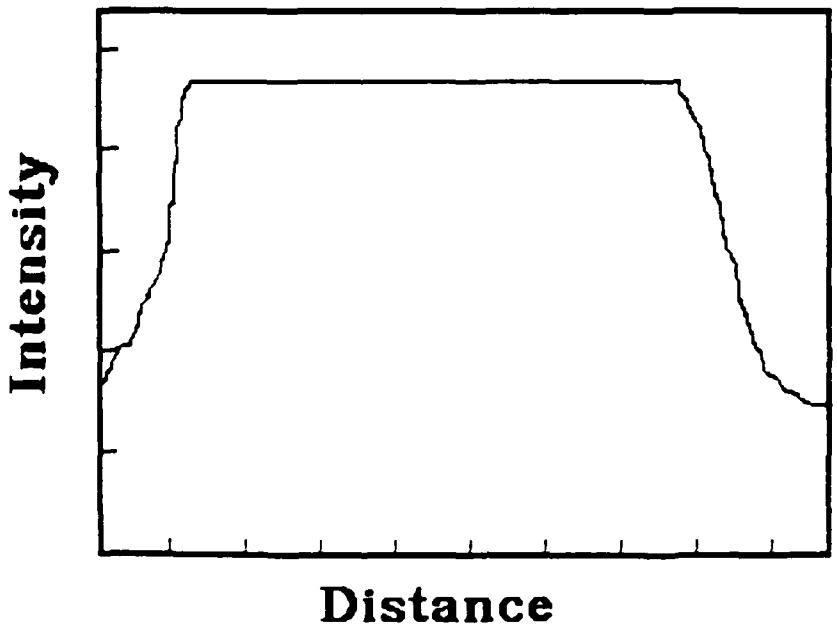

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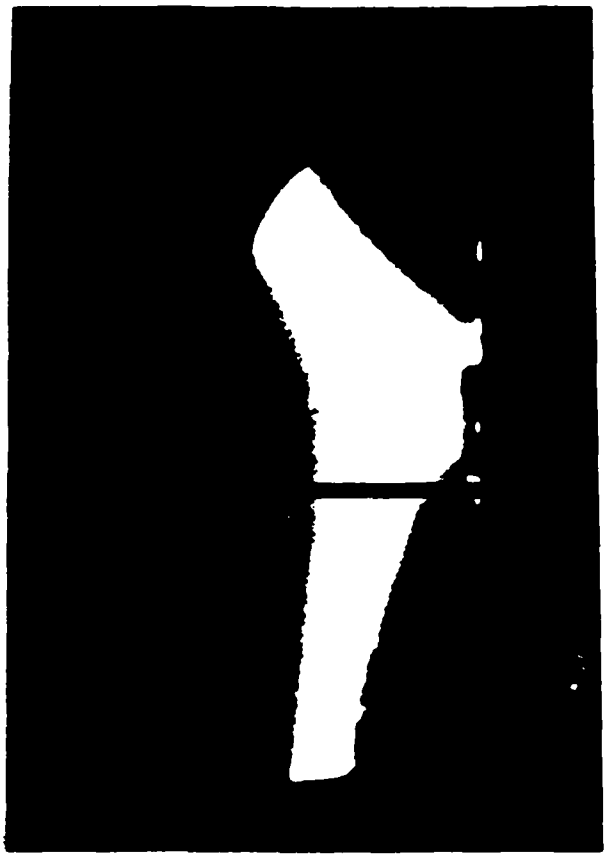

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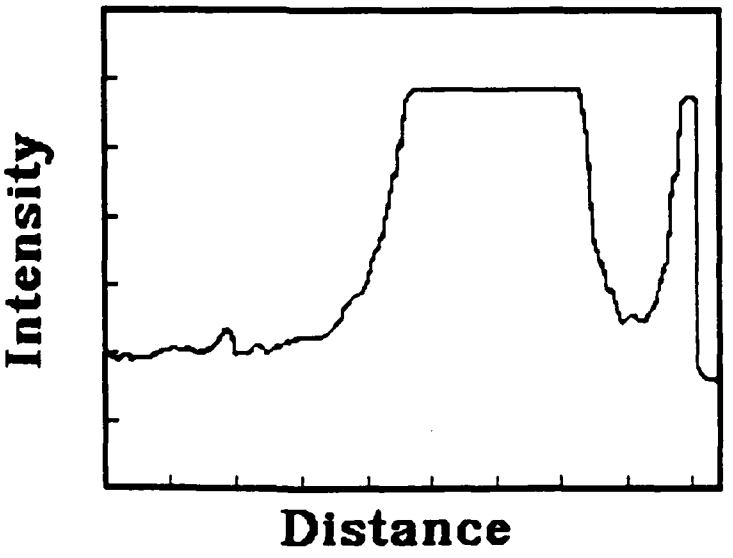

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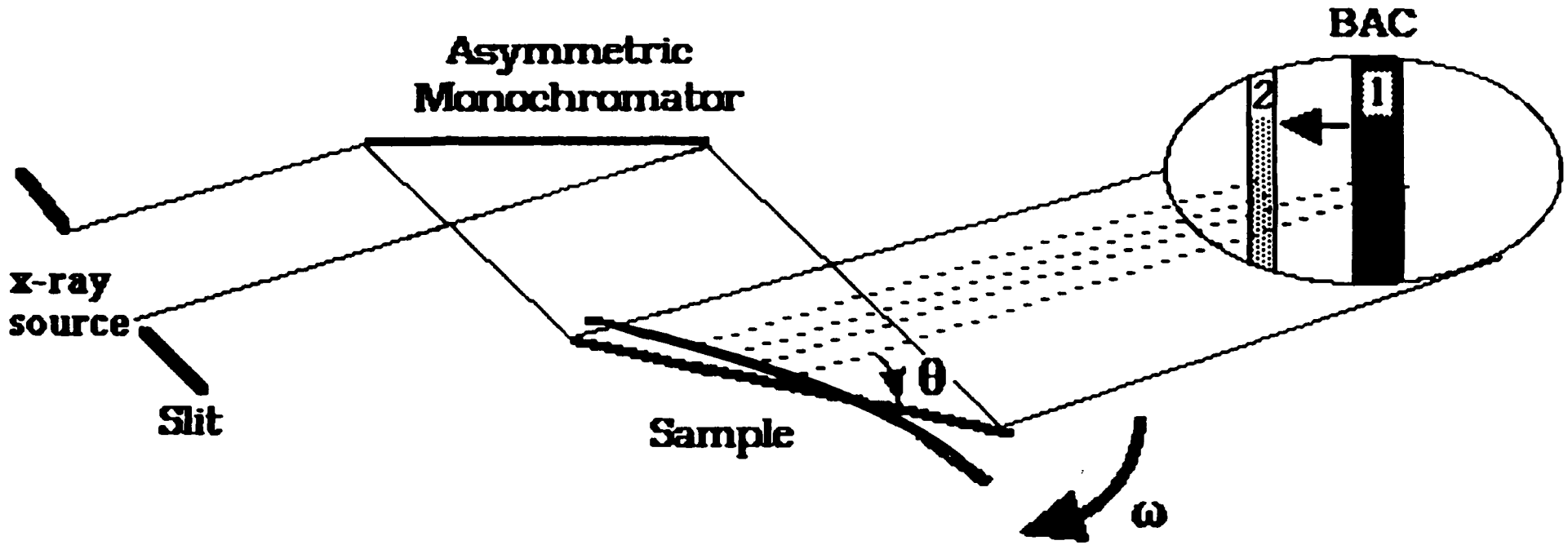
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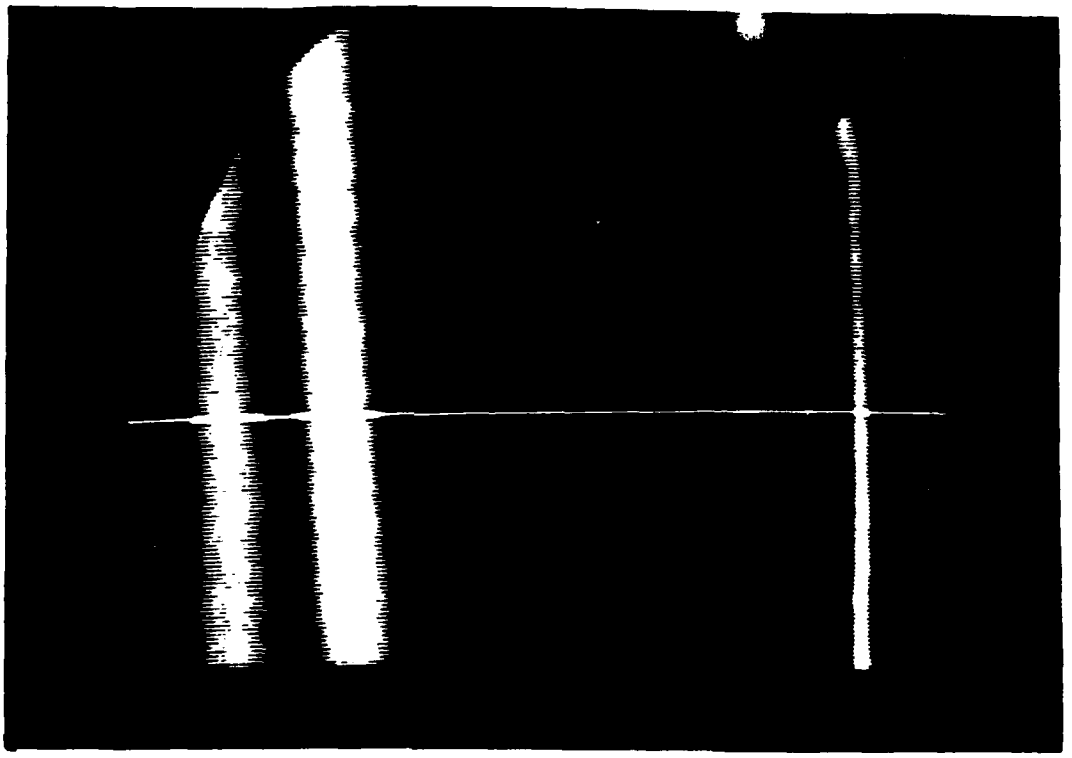

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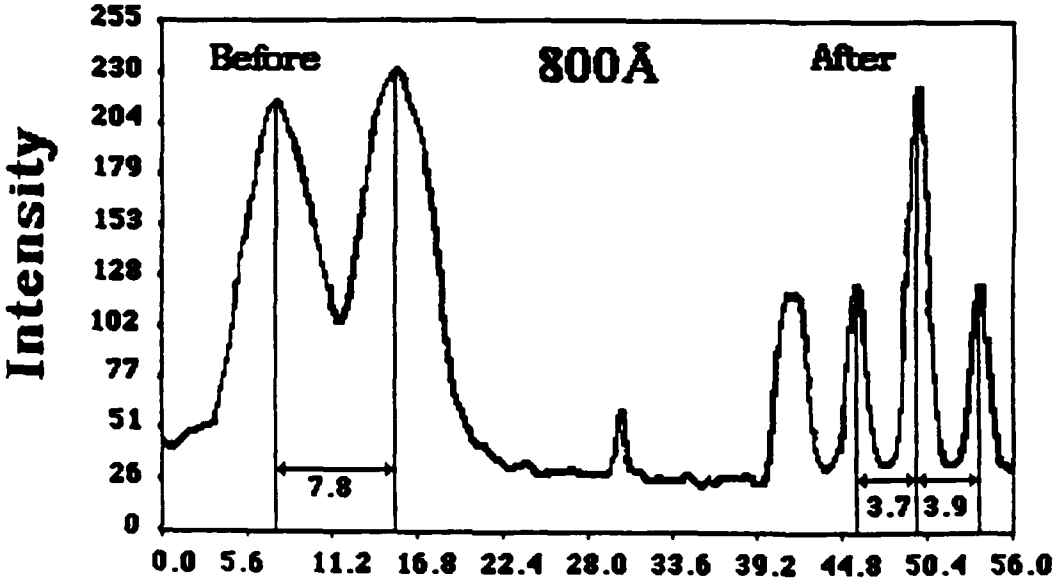
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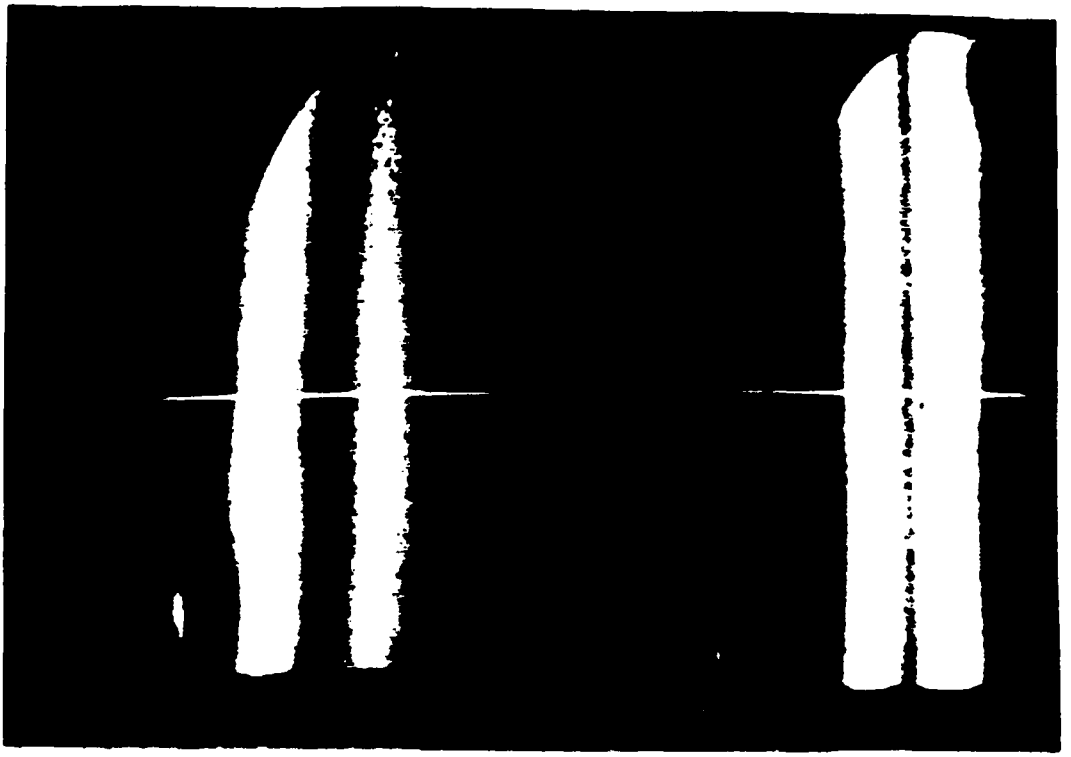

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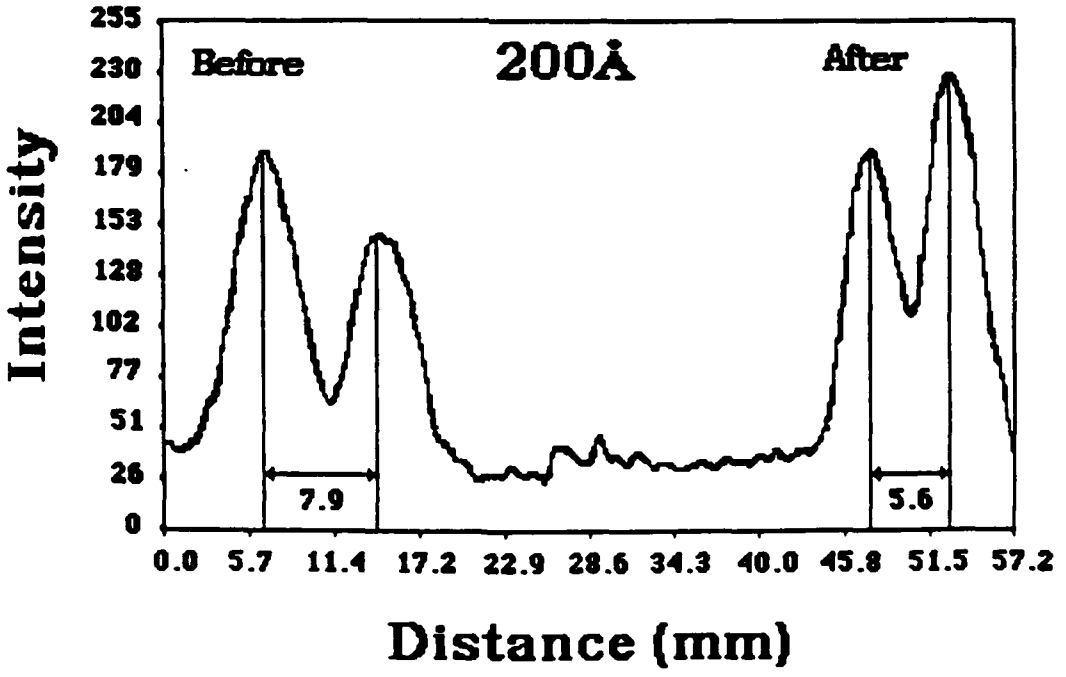
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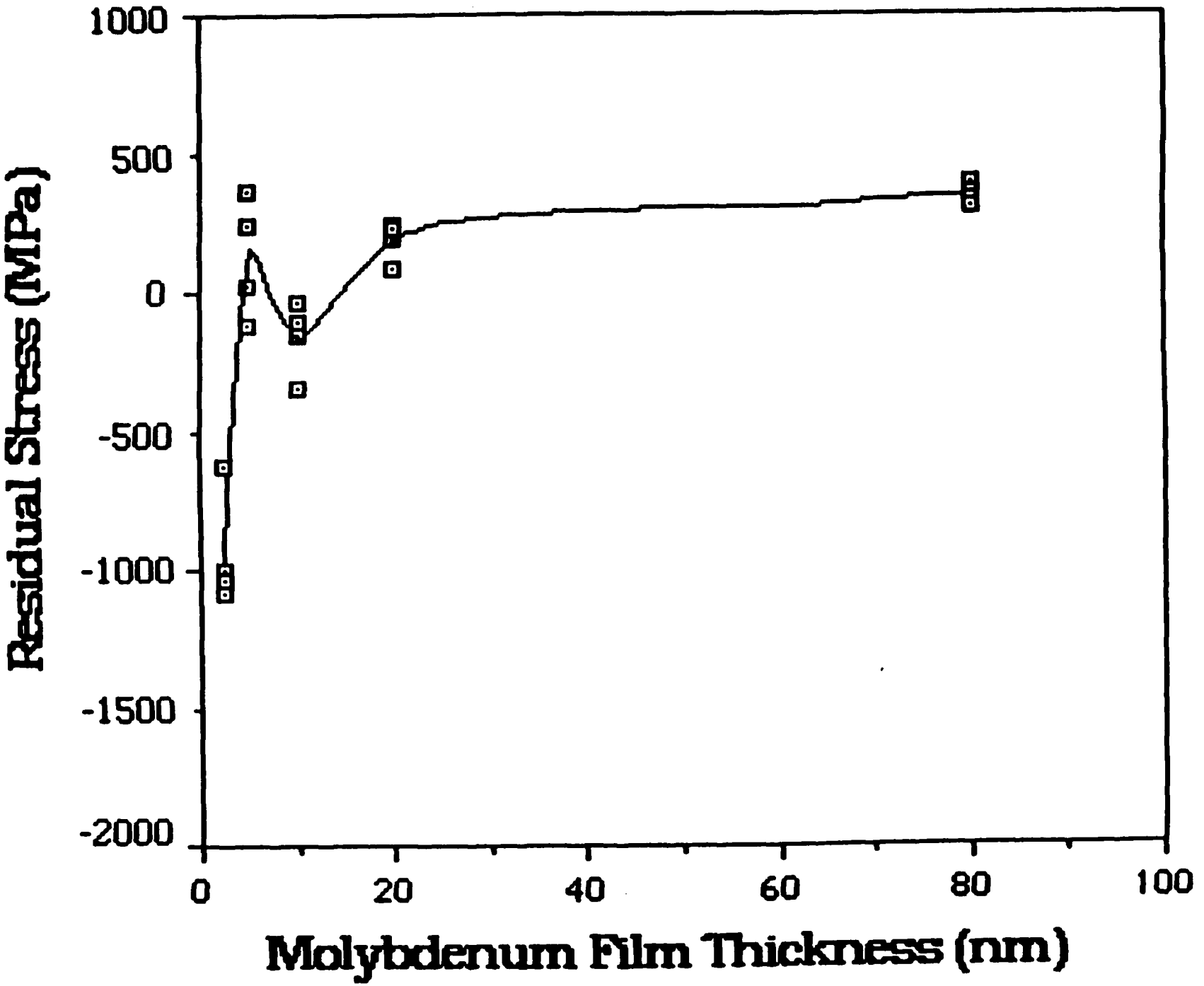
200Å
After
Intensity
255
230
204
179
153
128
102
77
51
26
0
7.9
5.6
0.0
5.7
11.4
17.2
22.9
28.6
34.3
40.0
45.8
51.5
57.2
Distance (mm)

(b)

Differential Curvature ($m^{-1} \times 10^{-8}$)
0
-2
-4
-6
-8
0
20
40
60
80
100
Tungsten Film Thickness (nm)

Differential Curvature ($m^{-1} \times 10^{-8}$)
8
6
4
2
0
-2
0
20
40
60
80
100
Molybdenum Film Thickness (nm)

Residual Stress (MPa)
0
-200
-400
-600
-800
-1000
Tungsten Film Thickness (nm)
0
20
40
60
80
100


1000
500
0
-500
-1000
-1500
-2000
Residual Stress (MPa)
0
20
40
60
80
100
Molybdenum Film Thickness (nm)

APPENDIX I

List of publications and presentations acknowledging Army Research Office Contract no. DAAL 03-87-K-0049

PUBLICATIONS and PRESENTATIONS:

"Identification of the Failure Mechanism of a Compressively Loaded Thin Film on a Thick Substrate by Means of Synchrotron X-ray Topography Combined with Transmission Electron Microscopy", A. H. King, W. Ng, and D. Goyal, amd J. C. Bilello, Materials Research Society, Boston, MA, December (1987).

"Microstructure Development in Thin Films During Deposition From the Vapor Phase", A. H. King, D. Goyal and J. C. Bilello, Materials Research Society, Boston, MA, December (1987).

"Synchrotron X-Ray Topography of Dislocation Arrays", Proceedings Materials Research Society, Symposium on New Materials Characterization Techniques, Published in: Characterization of Defects in Materials, eds, R.W. Siegel, Julia Weertman and R. Sinclair, MRS, Pittsburgh, PA, Vol. 82, 197, (1987).

"Depth Profiling of Defects in Epi-layer Semiconductor Materials Using Synchrotron X-Radiation Topography", C. L. Kuo and J. C. Bilello, Jour. of Applied Physics, 62, 137, (1987).

"Identification of the failure mode of a compressively loaded thin film on a thick substrate by means of synchrotron x-ray topography combined with transmission electron microscopy, "W. Ng, D. Goyal, J.C. Bilello and A.H. King, American Physical Society, Spring Meeting (1987).

"Microstructure development in thin films during deposition from the vapor phase", D. Goyal, A.H. King and J.C. Bilello, Materials Research Society, Symposium Series , Published in: Electronic Packaging Materials Science III, Eds. Jaccodine, Jackson and Sundahl, vol. 108, (1988).

"Identification of the failure mechanism of a thin film on a thick substrate by means of synchrotron x-ray topography combined with transmission electron microscopy" , D. Goyal, W. Ng, A.H. King and J.C. Bilello, Materials Research Society Series , Electronic Packaging Materials Science III, Eds. Jaccodine, Jackson and Sundahl, vol. 108, (1988).

"Electron microscope observations of mechanisms of thin film delamination from substrates", D. Goyal and A.H. King, MRS Symp. Series , Adhesion in Solids, Eds. Mattox, Baglin, Gottschall and Batich, vol. 119, (1988).

"Microstructure development and its effect on the development of stress in thin films during deposition from the vapor phase", D. Goyal and A.H. King, AIME Topical Symposium on Microstructural Science for Thin Film Metallization in Electronics , Eds. deLanerolle, Sanchez and Smith, (1988).

"Studies of Lattice Distortion and Crystal Curvature in Arsenic-Implanted Silicon", (with C. L. Kuo, Y. H. Kao, Emil Arnold), and J. C. Bilello, Jour. Appl. Physics, 64 (4), 1791, (1988).

"Synchrotron Topography Techniques", Invited talk at the Engineering Foundation, Engineering Week Conference in Palm Coast, Florida, J. C. Bilello, February, (1988).

"TEM observations of the mechanism of thin film delamination from substrates", D. Goyal and A.H. King, TMS-AIME Fall Meeting (1989).

"White Beam and Section Topographic Survey of Mo Thin Films on Silicon Substrates", L.H. Lee, H. Y. Wang, H. J. Wu, Z. U. Rek, and J. C. Bilello, Stanford Synchrotron Radiation Laboratory Annual Activity Report, (1990).

"TEM *observations of the failure mechanism of thin-film-on-substrate* systems", D. Goyal and A.H. King. J. Mater. Res. (submitted), (1990).

"Non-destructive Evaluation of Residual Stresses in Thin Films via X-ray Diffraction Topography Methods", J. Tao, L. H. Lee and J. C. Bilello, Symposium on Stress Relaxation and Misfit Accommodation in Epitaxial Thin Films II, TMS-AIME, Annual meeting, New Orleans, LA, Feb., (1991).

"Non-destructive Evaluation of Residual Stresses in Thin Films via X-ray Diffraction Topography Methods", J. Tao, L. H. Lee and J. C. Bilello,. Submitted, Jour. Elec. Mat. (submitted), (1991).

APPENDIX II

List of personnel supported on this contract and degrees earned where appropriate.

Personnel:

Dr. J. C. Bilello, Professor of Materials Science and Engineering and Professor of Applied Physics, University of Michigan, Ann Arbor, MI. (Formerly: Professor of Mechanical Engineering and Dean of Engineering and Computer Sciences, California State University, Fullerton, Fullerton, CA).

Dr. A, H. King, Professor of Engineering and Vice-Provost for Graduate Studies, SUNY Stony Brook, Stony Brook, NY.

Dr. C. L. Kuo, Post-Doctoral Research Associate, Visiting Scholor PRC, full Professor Shanghai Institute of Ceramics.

Dr. L. H. Lee, Post-Doctoral Research Associat, Ph. D. Rutgers University before working on this program.

D. Goyal, Graduate Student, Ph. D., SUNY Stony Brook, 1990.

H. Y. Wang, Graduate Student, California State University, Fullerton.

H. J. Wu, Graduate Student, California State University, Fullerton.

M. Vill, Graduate Student, M.S. (1991), California State University, Fullerton, continuing as a graduate student at University of Michigan.

J. Tao, Graduate Student, University of Michigan, continuing.