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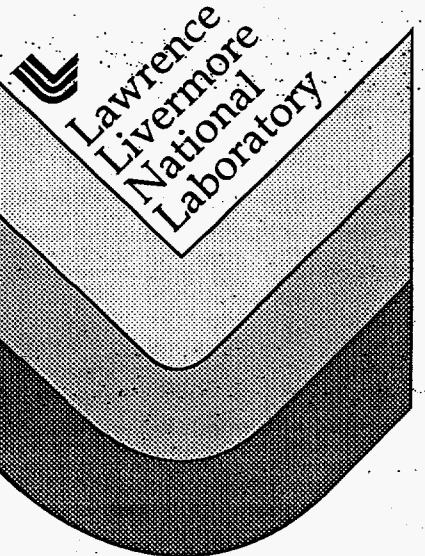
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## Reactive Sputter Deposition of Boron Nitride

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### ABSTRACT

The preparation of fully dense, boron targets for use in planar magnetron sources has led to the synthesis of Boron Nitride (BN) films by reactive rf sputtering. The deposition parameters of gas pressure, flow and composition are varied along with substrate temperature and applied bias. The films are characterized for composition using Auger electron spectroscopy, for chemical bonding using Raman spectroscopy and for crystalline structure using transmission electron microscopy. The deposition conditions are established which lead to the growth of crystalline BN phases. In particular, the growth of an adherent cubic BN coating requires 400-500 °C substrate heating and an applied -300 V dc bias.

### I. INTRODUCTION

Boron-based coatings are of interest for electronic applications, optical filters, wear resistance and high hardness applications. Thin film processing techniques as laser ablation and ion beam sputtering have been developed to deposit boron nitride films. A long-standing objective has been to stabilize the growth of the cubic phase of boron nitride for the above mentioned applications. Typically, the source material for these physical vapor deposition methods has been boron nitride in its hexagonal phase. To date, the stabilization of the cubic phase in thin films has typically been achieved through the use of very energetic deposition processes, e.g. bias sputtering or ion beam bombardment of the substrate.<sup>[1-4]</sup> Consequently, the cubic boron nitride (cBN) films have large intrinsic stresses. Therefore, in addition to

preferential sputtering effects<sup>[5]</sup>, a mechanism for stabilization of cBN in films has been attributed to the presence of large stresses.<sup>[6]</sup> However, such highly stressed films are mechanically unstable and often have poor adherence. A less energetic physical vapor deposition process could have advantages in forming cBN films.

The recent development of fully dense, pure boron targets has made it possible to initiate an investigation of boron nitride deposition without the hexagonal phase precursors.<sup>[7,8]</sup> The ablation or sputtering of a target material often results in the transport of molecular clusters to the substrate.<sup>[9]</sup> This is clearly the case for a target consisting of an equilibrium phase such as hexagonal boron nitride (hBN). The deposition of even small amounts of the target material could promote the epitaxial formation of the hexagonal phase. An energetic bombardment is therefore required to transform the hexagonal phase into the less stable or metastable cubic phase. In this study, we assess the deposition conditions needed to rf sputter pure boron targets in order to stabilize crystalline growth in boron nitride films. A comparison of the free energies of formation indicates that the thermodynamic equilibrium for the transformation of hBN to cBN is less favorable than the formation of cBN from pure boron.<sup>[10]</sup>

We initiate the study by examining the influence of working gas composition and substrate temperature variation on the formation of crystalline boron nitride phases. Next, we explore the energetic conditions needed to stabilize cBN growth in boron nitride films as synthesized with the application of substrate bias. Characterization of the boron nitride films includes chemical evaluation through Auger electron spectroscopy (AES), structural analysis through the use of both light and electron microscopies as well as an assessment of chemical bonding through Raman and Fourier transform infrared (FTIR) spectroscopies.

## II. EXPERIMENTAL METHOD

The synthesis of the boron nitride films proceeds from the rf sputter deposition of fully dense, pure boron targets using unbalanced planar magnetrons. The patented process used to

create these unique boron targets has been described in detail elsewhere.<sup>[11]</sup> In brief, boron powders are compacted in an evacuated tantalum container which is then hot isostatically pressed to yield a right circular cylinder of fully dense (i.e. less than 0.15% porosity) boron. The manufacture of boron sputtering targets then proceeds by a patented process involving the brazing of a compatible backing plate to prevent catastrophic target failure at initiation of the sputtering process.<sup>[11]</sup> X-ray diffraction analysis reveals the targets to be the rhombohedral crystalline phase of boron.

Two deposition chambers are used to evaluate the synthesis of boron nitride using a reactive gas mixture of Ar-N<sub>2</sub>. The first deposition chamber is used to establish the baseline effects of gas pressure, flow and substrate temperature on the composition and phase formation of boron nitride films. This deposition system is cryogenically pumped from atmospheric pressure to a base pressure of  $5.3 \times 10^{-6}$  Pa in 12 hrs including a 4 hr, 100 °C bake out. Some reports have indicated that epitaxial growth of cBN can be enhanced through the use of nickel epilayers.<sup>[12,13]</sup> As such, 20-50nm of nickel are initially sputter deposited onto silicon wafers for use as substrates. The substrate table is positioned 9 cm horizontally away from the center of the 6.4cm diameter boron target. The substrate temperature is controlled using a Boralectric™ heater. The wafers are heated up to 600°C, as above this temperature CuK $\alpha$  x-ray diffraction reveals the formation of an orthorhombic phase of nickel silicide. The Ar sputter gas pressure is nominally selected as 1 Pa (7 mTorr) with a constant flow rate of 28 cc min<sup>-1</sup>. A partial flow of N<sub>2</sub> is incorporated to yield a 0 to 55% composition range. The deposition rate is monitored with a calibrated 6 MHz Au coated quartz crystal. An increase in applied forward power from 100 to 300 W produces a linear increase in deposition rate from to 0.007 to 0.021 nm s<sup>-1</sup>. The boron nitride films are grown to a total thickness of 100-300nm as verified using contact profilometry.

The second deposition chamber is used to examine the effects of applied substrate bias on the formation of BN. Differences from the first deposition chamber are as follows. The second chamber is fixtured so as to accomplish the purpose of applying a BN hard coating to the surface of cutting tools. Provision is made for the initial sputter deposition of 20-50nm of either titanium

or chromium to promote adhesion of the coating. The substrate table is positioned 5 cm vertically below an array of three magnetron sources, each of 3.3cm diameter. To assess the effects of film stress on adhesion, the sputter pressure is varied from 0.2 to 3 Pa. Boron nitride deposition rates of 0.02 to 0.04 nm s<sup>-1</sup> are produced by forward target powers of 50 to 80 Watts. The bias applied to the forming BN coating is produced by drawing off approximately 5% of the applied rf power to the target. The reported 0 to -400 V dc bias is the (negative) component of the rf power applied to the substrate. A small percentage of the ionized sputter gas will then strike the substrate and modify the structure of the coating.

The boron nitride thin films are characterized for composition, morphology, and crystalline structure as well as chemical bonding. The film composition is measured using Auger electron spectroscopy (AES) coupled with depth profiling by Ar sputtering. The intensities of the B, C, N, and O *KLL* Auger electrons (as data accumulated in the derivative mode) are used to compute the atomic concentrations. The detection limit for carbon and oxygen impurities is approximately 2 to 3 atomic % in these BN films. Transmission electron microscopy (TEM) is used for the assessment of film morphology and crystallinity. Bright field imaging of BN films prepared in plan view reveals the grain structure. Dark field imaging coupled with electron diffraction patterns reveal the crystalline state of the films. Raman spectroscopy is used to examine the vibrational states of chemical bonds within the films. Although there are many crystalline phases of BN including rhombohedral and wurtzite, the distinctive structures and chemical bonding are of hexagonal and cubic. The Raman phonon of hBN appears at 1367 cm<sup>-1</sup> as representative of *sp*<sup>2</sup> hybridized, planar bonding. The transverse optical (TO) and longitudinal optical (LO) modes of cBN appear at 1057 and 1306 cm<sup>-1</sup>, respectively, as representative of *sp*<sup>3</sup> tetrahedral bonding. The relative intensity of the TO and LO modes for cBN can vary as BN is noted to have strong Raman anisotropy.<sup>[14]</sup> In addition, the presence of a weak and broad feature at 1245 cm<sup>-1</sup> represents strong plasmon-phonon coupling in cBN. Fourier transform infrared spectroscopy (FTIR) is used as a means to verify the bonding trends observed in these BN films using Raman spectroscopy. Whereas the Raman spectra are generated in reflection, the FTIR

spectra are generated in transmission. Simulations of the IR spectra have also confirmed secondary hBN features as a peak at  $783\text{ cm}^{-1}$  and the increased asymmetry of the  $1367\text{ cm}^{-1}$  peak with increasing film thickness towards a satellite peak at  $1560\text{ cm}^{-1}$ .<sup>[2]</sup> The primary  $1367\text{ cm}^{-1}$  hBN feature is associated with in-plane B-N bond stretch while the secondary feature at  $783\text{ cm}^{-1}$  is associated with the out-of-plane B-N-B bond bend.<sup>[4,15]</sup> Finally, conventional light microscopy is used to compare the effects of intrinsic film stress on macroscopic film morphology.

### III. EXPERIMENTAL RESULTS & ANALYSIS

The sputter gas composition and substrate temperature are initially varied to determine effects on BN film formation. AES analysis indicates that the coatings have a constant composition through the depth of the film. As an example, the depth profile is shown in Fig. 1 of a film deposited at a substrate temperature of  $600^\circ\text{C}$  with a Ar-55%N<sub>2</sub> gas mixture. The BN film composition is found to be independent over the gas composition range of 25 to 55% N<sub>2</sub> for the limited range of deposition rates used in this study. Heating the substrate above  $200^\circ\text{C}$  during deposition is seen in Fig. 2 as advantageous to eliminate contamination of the BN films by oxygen. The average BN film composition is found to be 45 atomic % Boron from the AES analysis. The Raman spectra of 150-200nm thick BN films deposited over the 50 to  $600^\circ\text{C}$  temperature range are shown in Fig. 3. Note that the individual curves are offset on the intensity axis to accentuate the Raman spectral features. As the substrate temperature increases from 50 to  $350^\circ\text{C}$  the intensity of the primary hBN peak increases. Above  $350^\circ\text{C}$ , the increase in temperature produces an asymmetry in the hBN peak towards the wavenumber corresponding to the LO mode of cBN. The inference from these Raman spectra is threefold. (i) At "room" temperature, an  $sp^2$  bonded structure as hBN is formed with some disorder. (ii) As temperature increases, disordering of the BN phase is reduced resulting in a refined hBN growth. (iii) A further increase in substrate temperature, i.e. a further increase of energy during film formation,



disorders the well ordered  $sp^2$  bonded structure to *possibly* include some small fraction of  $sp^3$  bonding. Confirmation of these substrate temperature effects on BN phase formation is given in the FTIR spectra of Fig. 4 for three of the samples displayed in Fig. 3. The individual curves of Fig. 4 are offset on the intensity axis to accentuate the Raman spectral features. Peak positions for hBN are indicated on the lower wavenumber axis and cBN peak positions on the top axis. For a 50°C substrate temperature, diffuse peaks appear above 1350 and 1550  $\text{cm}^{-1}$  corresponding to a disordered hBN structure, as the turbostratic phase (tBN) or an amorphous structure.<sup>[1,2]</sup> For a 200°C deposition, a well ordered structure is formed with primary and secondary peaks matching the simulated IR spectrum<sup>[2]</sup> of hBN. For a 600°C deposition, the IR spectrum shows diminished intensity for the hBN peaks but includes additional broad peaks about 1100  $\text{cm}^{-1}$ , *possibly* indicative of  $sp^3$  bonding. These deposition related trends in chemical bonding are clearly qualitative. A quantitative assessment of chemical bonding is beyond the scope of this initial study. Further (TEM) characterization for crystalline structure is not yet warranted.

The application of substrate bias is to enhance stabilization of cBN growth. Given the perspective of applying substrate temperature and varying the sputter gas parameters, the deposition conditions established for applying substrate bias are as follows. (i) Vary sputter pressure using an Ar-25%N<sub>2</sub> gas mixture at a nominal flow of 25 to 30  $\text{cc min}^{-1}$ . (ii) Heat the substrate to 400-500°C to promote crystalline formation, yet destabilize the growth of hBN. The effects of substrate bias on BN structure are shown in the Raman spectra of Fig. 5 for 150-200nm thick films deposited using a 1.1 Pa sputter gas pressure, noting that individual curves are offset on the intensity scale to accentuate bonding features. For a -70V bias, a structure with some characteristic  $sp^2$  bonding is formed in the BN deposit. As the bias is increased to -150V, a diffuse peak appears about 1100  $\text{cm}^{-1}$  characteristic of  $sp^3$  bonding as seen in the TO mode for cBN. As the bias is increased further to -300V, the characteristic  $sp^2$  bonding at 1367  $\text{cm}^{-1}$  nearly vanishes while the broad peak at 1100  $\text{cm}^{-1}$  increases in intensity. Further increase in the bias to -400V begins to reduce the intensity of the broad peak at 1100  $\text{cm}^{-1}$ . The application of substrate bias is to destabilize  $sp^2$  bonding as found in hBN and promote  $sp^3$  bonding as found

in cBN. Further microstructural characterization of these bias applied BN structures is warranted with TEM to confirm the change in crystalline structure.

To facilitate TEM plan view examination, samples deposited without a Cr adhesion layer are used since these films are removable from the substrates. The bright field (BF) and dark field (DF) images are shown in Fig. 6 for a BN film deposited on a "room" temperature Si substrate with a -70V bias at 1.1 Pa Ar-40%N<sub>2</sub> gas pressure. The BF image reveals a fine-grained, continuous film with no distinguishing features. An electron diffraction *ring* pattern (Fig. 6 insert) indicates a polycrystalline structure. The interplanar d-spacings are computed from the camera constant as calibrated to a Au standard. The d-spacings are  $0.350 \pm 0.005$ ,  $0.2101 \pm 0.0008$ , and  $0.1205 \pm 0.0002$  nm. The DF image generated from the broad, intense inner ring of the diffraction pattern shows a continuous, fine grained diffracting volume. The structure is probably single phase and nanocrystalline as the dark field illumination of the diffracting 2-5nm sized grains can be easily translated with small sample tilting. The d-spacings for this sample best fit the turbostratic boron nitride (tBN) phase with previously reported d-spacings of 0.356 and 0.212 nm.<sup>[16]</sup> It should be noted that the  $d_{\text{cBN}}(111)$  of 0.2088 nm or the  $d_{\text{hBN}}(10.0/10.1)$  of 0.217/0.206 nm and the  $d_{\text{hBN}}(11.0/11.2)$  of 0.125/0.117 nm are not probable reflections.<sup>[17,18]</sup> The tBN characterization is quite consistent with the results for bonding trends found in the Raman spectra of Figs. 3 and 5. The BN film deposited at low temperature and low bias appears to be a disordered  $sp^2$  bonded structure.

Raman spectra have indicated the presence of  $sp^3$  bonding, possibly for cBN in the -300V deposit of Fig. 5. As the tBN structure is found to be a consistent identification for the Raman and TEM analysis, verification of a cBN phase is now pursued using TEM. The BF and DF images are shown in Fig. 7 for a BN film deposited on a 500°C substrate with a -300V bias at 1.1 Pa Ar-25%N<sub>2</sub> gas pressure. The BF image reveals a continuous film structure containing a mosaic appearance, attributable to some extent of film curvature. The electron diffraction *ring* pattern (Fig. 7 insert) indicates a polycrystalline deposit. The DF image generated from the narrow, most intense inner ring of the diffraction pattern shows a uniformly dispersed, fine

grained diffracting volume. Dark field illumination of the diffracting 2-5nm sized nanocrystals can also be easily translated with small sample tilting. From the innermost ring outward, the computed d-spacings are  $0.2096 \pm 0.0005$ ,  $0.1283 \pm 0.0002$ ,  $0.1093 \pm 0.0001$ ,  $0.0907 \pm 0.0001$ ,  $0.0830 \pm 0.0001$ , and  $0.0741 \pm 0.0001$  nm. These d-spacings can be indexed to (111), (220), (311), (400), (331) and (422) which fit a cubic structure with a lattice parameter of  $0.3625 \pm 0.0003$  nm. The cubic structure and lattice parameter value match the cBN powder diffraction value of 0.3616 nm. The increased lattice parameter may be a result of the nitrogen-rich (53 atomic %) composition in this sample.

The use of optical microscopy further reveals the effect of intrinsic stress on the adhesion of the BN films to the substrate. The formation of cBN has been shown for films deposited on a substrate heated to 400-500°C with a -300V applied bias. The effect of sputter gas pressure further effects the intrinsic film stress. BN films deposited using Ar-25%N<sub>2</sub> sputter gas pressures of 1.1 and 2.1 Pa are shown in the optical photos of Fig. 8. The film deposited at 1.1 Pa is releasing as seen in the crazed pattern whereas the 2.1Pa deposit is adherent showing no such pattern. The presence of cBN in each of these films is verified using Raman spectroscopy producing spectra similar to the -300V bias curve of Fig. 5.

#### IV. DISCUSSION & SUMMARY

It is established that the deposition of a cBN film from a hBN target requires an energetic process.<sup>[1-4]</sup> For example, only tBN and hBN growth is found for pulsed laser ablation of B or hBN targets onto substrates at elevated (600°C) temperature without the energetic component of a coincident ion irradiation of the substrate.<sup>[19]</sup> The apparent stabilization of the cBN phase in the growing film by increasing substrate bias (when sputtering a boron target with Ar-N<sub>2</sub>) is a clear indication that an energetic process is needed in comparison to growth of hBN. Although substrate potentials approaching the target discharge voltage are reached, the current density of incoming Ar<sup>+</sup> ions is clearly much less than that required to noticeably resputter the growing film

due to the applied power distribution and resulting plasma confinement at the planar magnetron target surface. Although unable to measure flux, we provide a quantitative correlation to energy.

The Raman and FTIR spectra are representative of the BN films and not the substrate or epilayers. Raman and FTIR characterization of the epilayer/substrate combinations did not produce any of the spectral features observed in Figs. 3-5. An assessment of enhanced cBN growth using a metal (as Ni) epilayer will require higher substrate temperatures and the use of Ni single crystals. Results reported for cBN growth using hot filament assisted rf plasma chemical vapor deposition required substrate temperatures of 900-1100°C.<sup>[12,13]</sup> The Si substrates of this study are not suitable as silicide formation and unknown artifacts result above 600 °C.

In summary, thin-film fabrication of more than 80 BN samples have been accomplished by reactive sputter deposition from a pure, fully dense boron target. The near stoichiometric BN film composition is found to be relatively insensitive to gas composition (25-55% N<sub>2</sub>) and substrate temperature (200-600 °C) over the range of deposition rates (<0.04 nm s<sup>-1</sup>) studied. This is consequence of a near equilibrium kinetic process between nitrogen and sputtered boron for the range of (low rate) deposition parameters examined. The control of substrate temperature and bias allow for the stabilization of BN films with *sp*<sup>2</sup> and *sp*<sup>3</sup> bonding. Although not all combination have been explored, the following trends are confirmed for nearly stoichiometric films (with a N:B ratio of 1.2) as characterized for chemical bonding using Raman spectroscopy and crystallinity using TEM. Low temperature (<200 °C) and low negative bias (<150 V) produce films with structures characteristic of amorphous or turbostratic BN. Intermediate substrate temperatures (200-350 °C) stabilize the growth of films with structures characteristic of hexagonal BN. The application of intermediate negative bias (150-400 V) for substrates heated to 400-500°C stabilizes the formation of films with structures characteristic of cubic BN.

Ion-assisted processes have been successful in producing cBN films but typically these coatings are highly stressed and poorly adherent. An advantage for using pure Boron targets in the reactive sputter deposition of cBN is the tractable nature of producing adherent films. The

use of sputter gas pressure as a deposition parameter that stabilizes the growth of adherent cBN coatings on flat Si substrates and is therefore promising for cutting tool applications.

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**FIGURE CAPTIONS**

- 1 An atomic concentration depth profile of a boron nitride film deposited at 600 °C.
- 2 The atomic concentration of boron nitride films variation with substrate temperature.
- 3 Raman spectra of boron nitride films variation with substrate temperature (°C) indicates changes in chemical bonding.
- 4 Fourier transform infrared spectra of boron nitride films variation with substrate temperature (°C) confirms the Raman results of Fig. 3.
- 5 Raman spectra of boron nitride films deposited at 400-500 °C variation with applied substrate bias (V) indicates changes in the chemical bonding.
- 6 Transmission electron microscopy reveals of the boron nitride film (deposited at room temperature with a -70V substrate bias) reveals the (a) microstructure in bright field and (b) the diffracting grains in dark field which contribute to the most intense inner ring of the (insert) electron diffraction pattern.
- 7 Transmission electron microscopy reveals of the boron nitride film (deposited at 500 °C with a -300V substrate bias) reveals the (a) microstructure in bright field and (b) the diffracting grains in dark field which contribute to the most intense inner ring of the (insert) electron diffraction pattern.
- 8 Optical micrographs of boron nitride films deposited at (a) 1.1 Pa and (b) 2.1 Pa Ar-25%N<sub>2</sub> gas pressure.

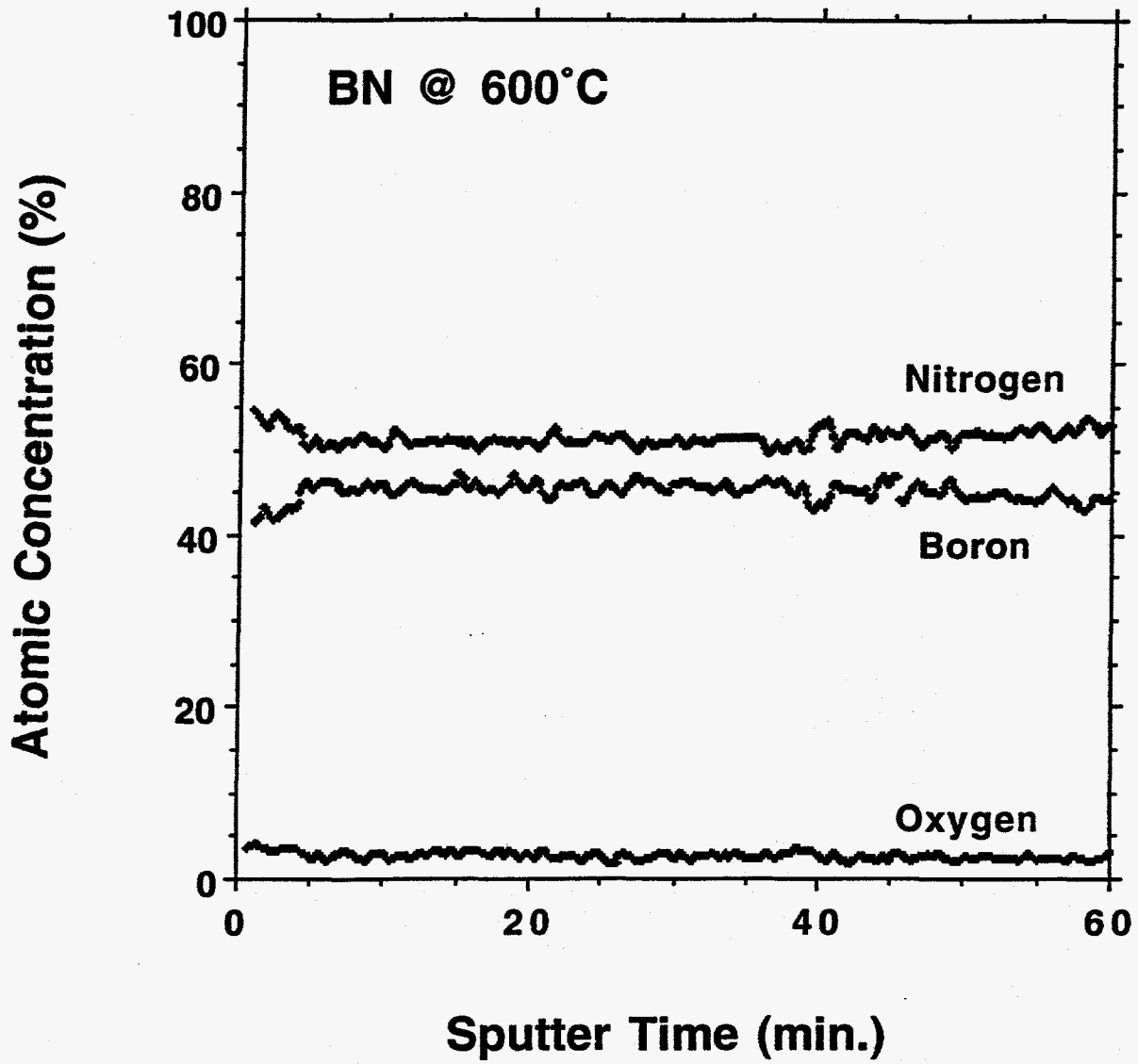


Figure 1



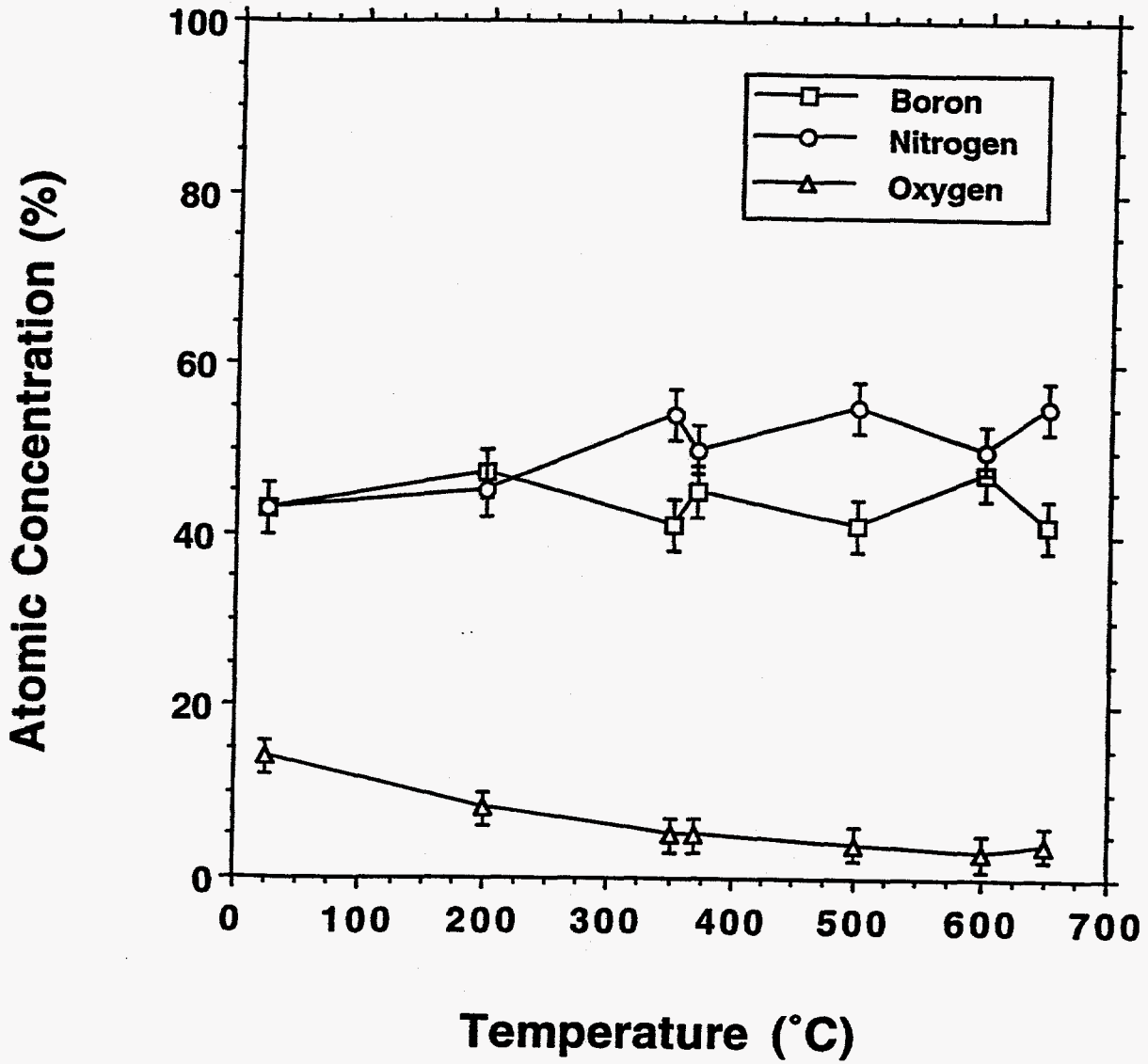


Figure 2

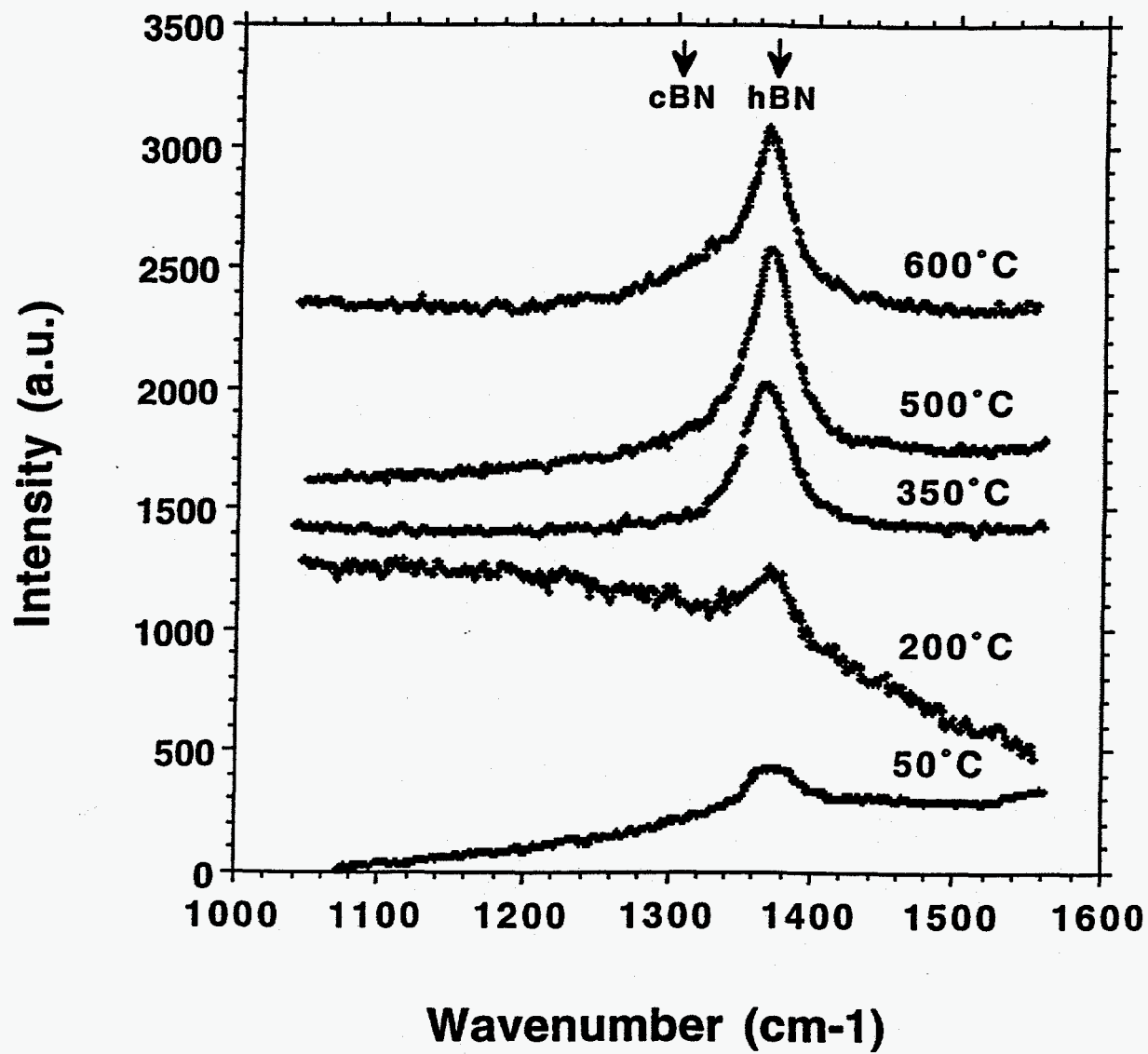


Figure 3

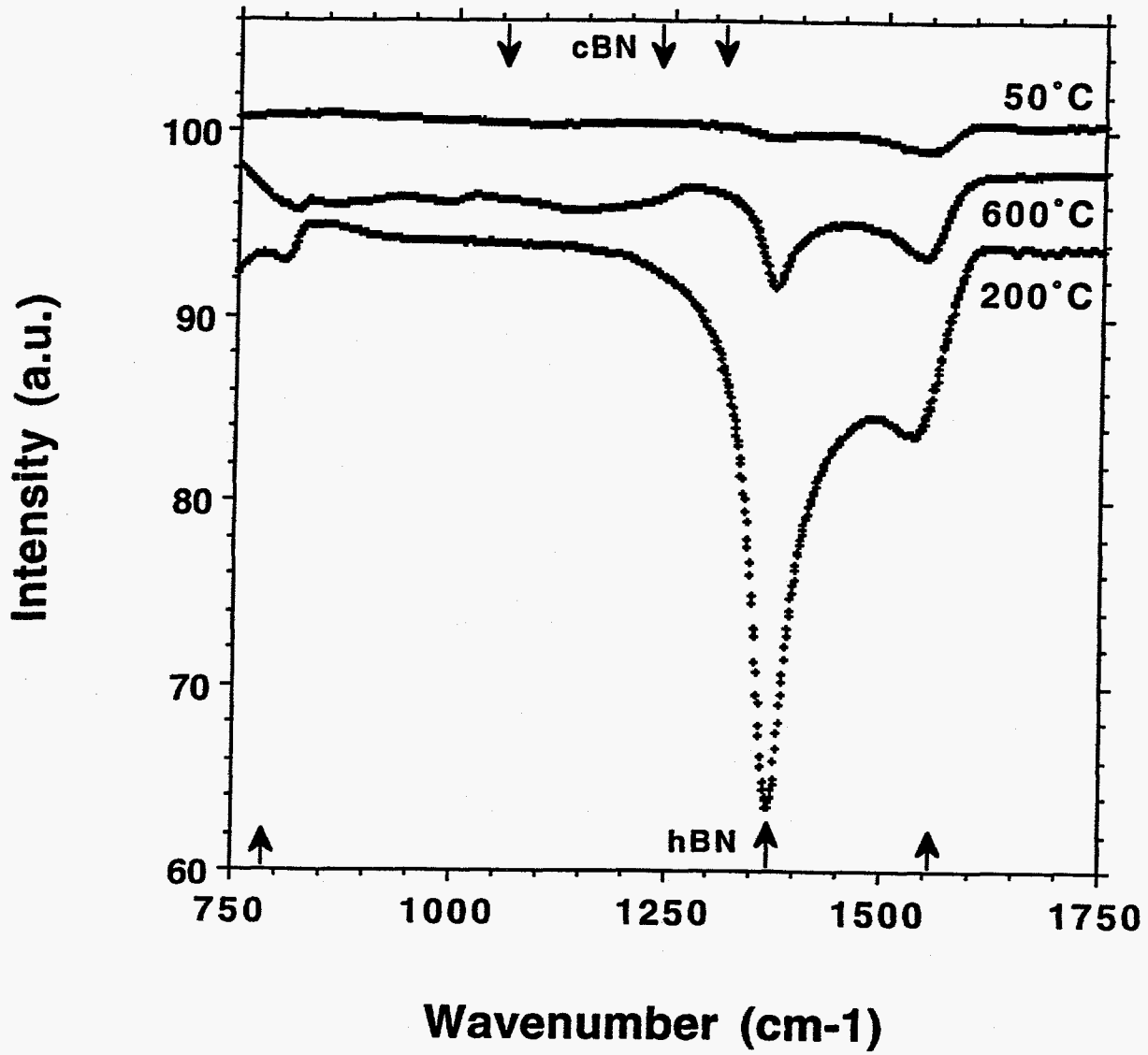


Figure 4

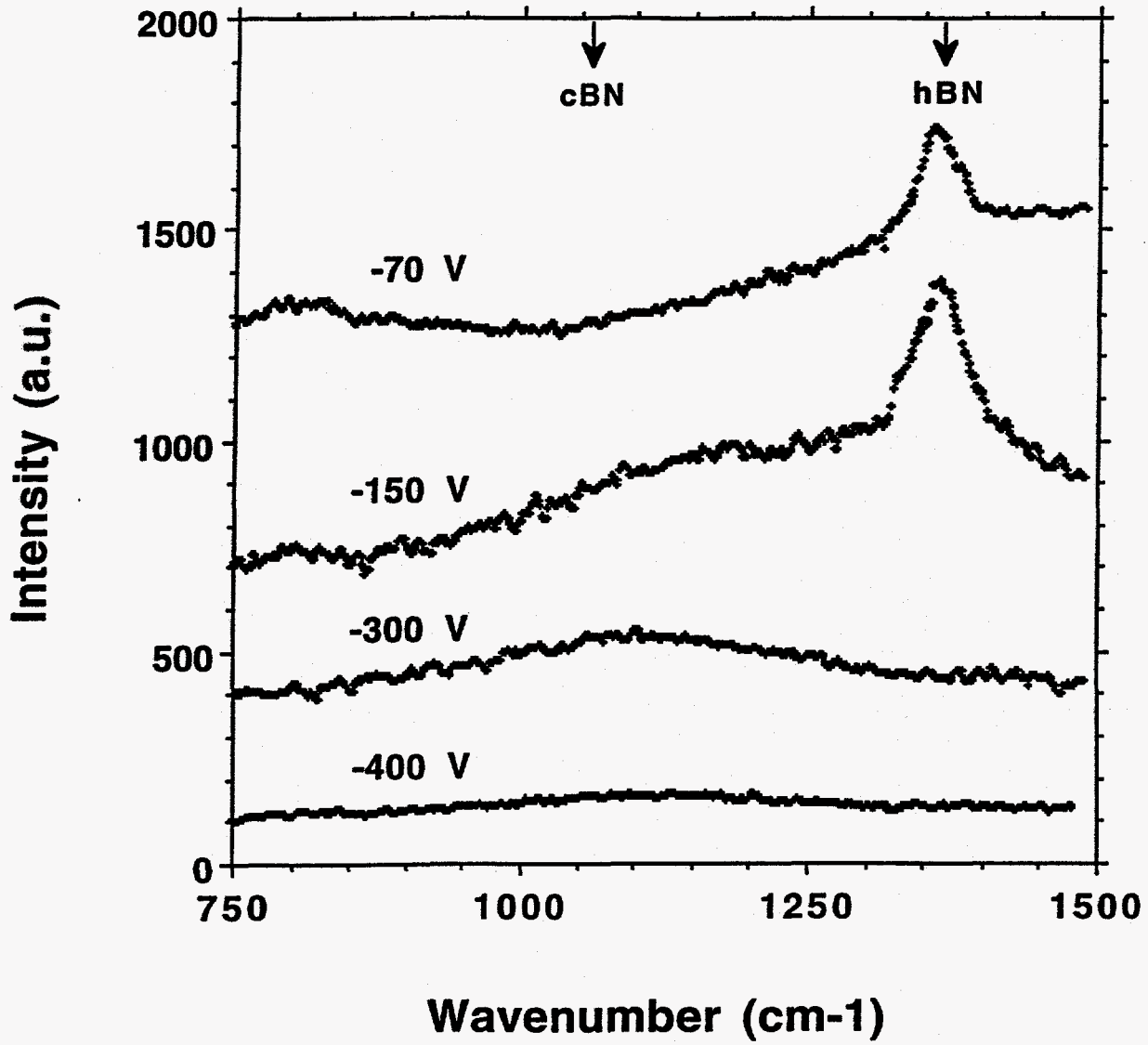


Figure 5

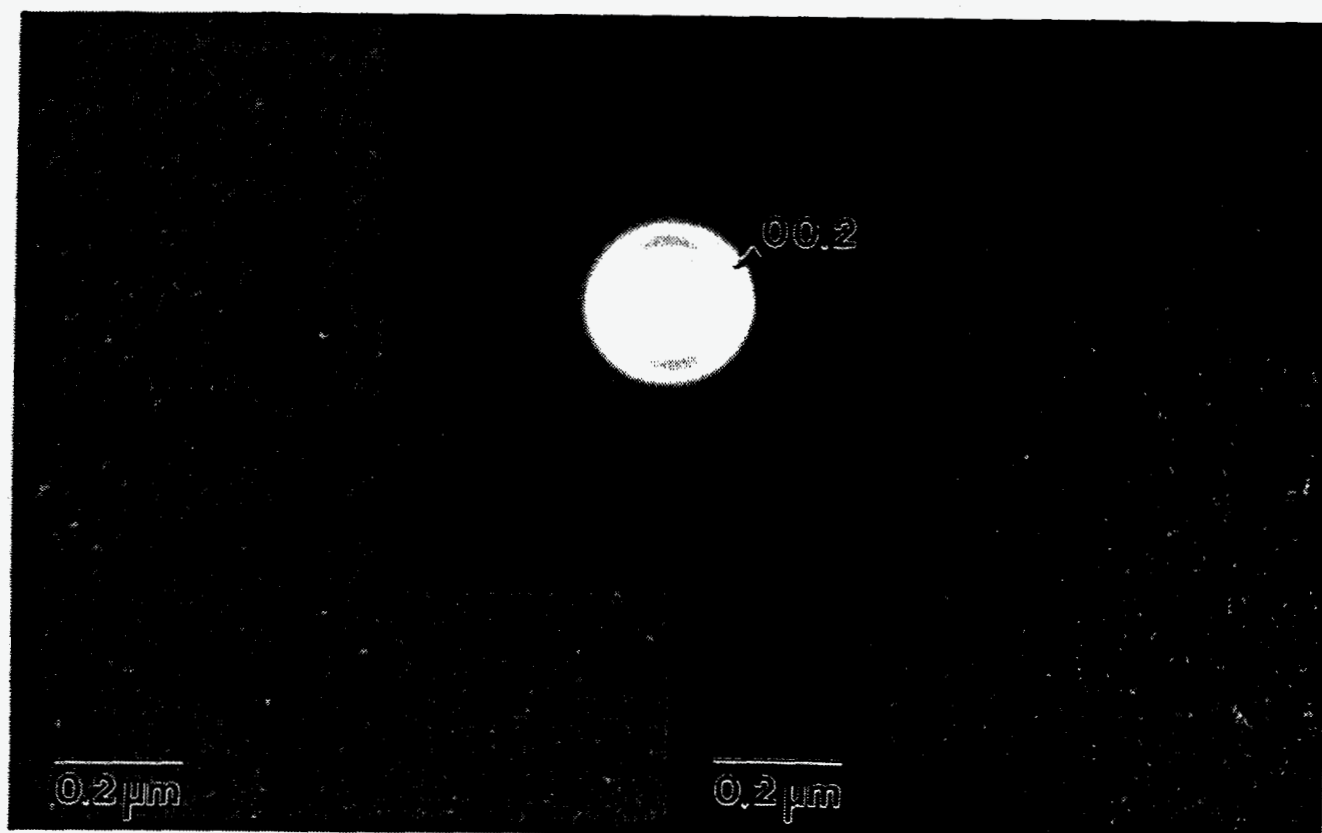


Figure 6

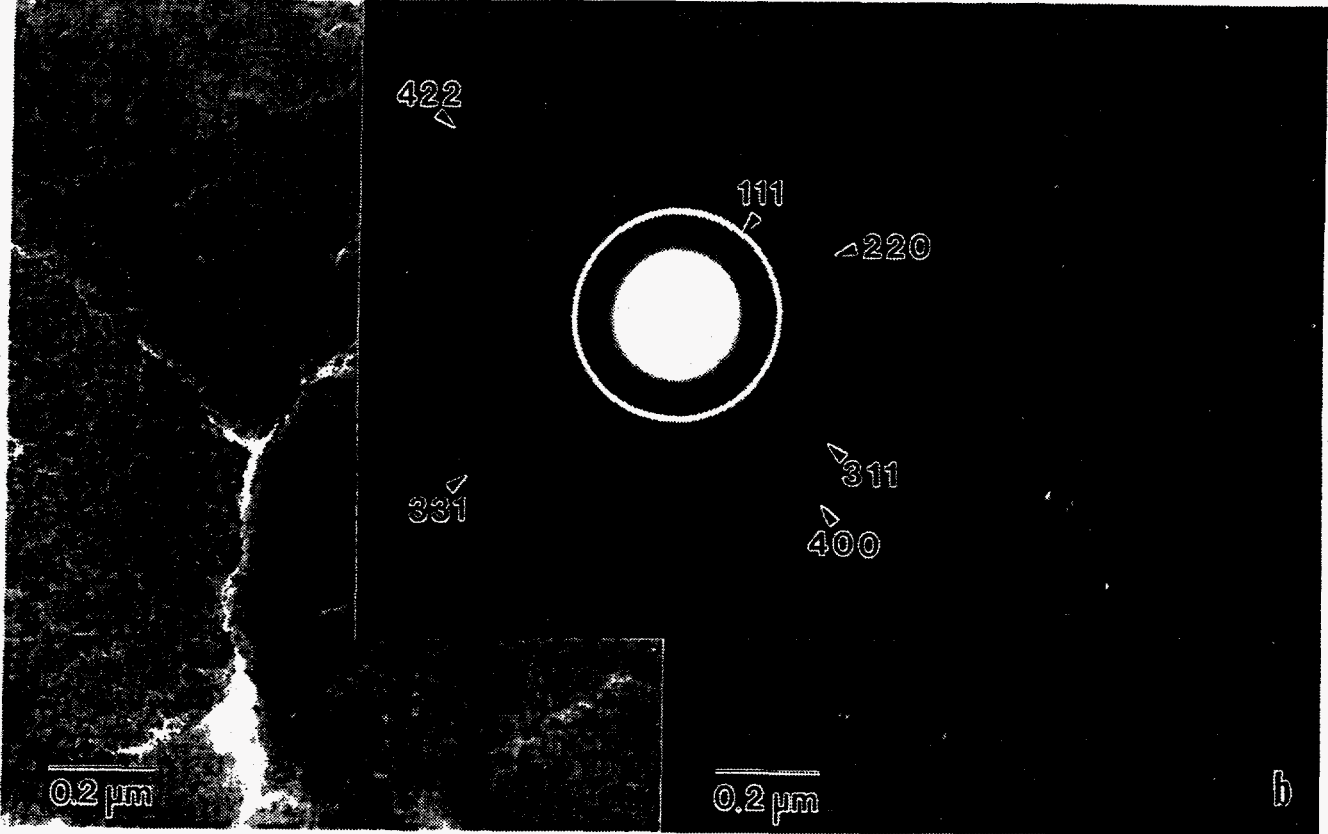


Figure 7



Figure 8