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Eric E. Fullerton, C.H. Sowers, S.D. Bader
Materials Science Division
Argonne National Laboratory
Argonne, Illinois 60439

S. Mini
Experimental Facilities Division
Advanced Photon Source
Argonne National Laboratory
Argonne, Illinois 60439

A.S. Bommannavar
Brooklyn College oc CUNY
Brooklyn, NY 11210

S.N. Ehrlich
School of Materials Engineering
Purdue University
West Lafayette, IN 47904

March, 1993

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*This work supported by the U.S. Department of Energy, BES-Materials Sciences, under contract no. W-31-109-ENG-38

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INTERFACIAL STRUCTURE OF LATTICE MISMATCHED bcc(110)/bcc(110) TRANSITION METAL SUPERLATTICES

ERIC E. FULLERTON,* S. M. MINI,** A. S. BOMMANAVAR,† C. H. SOWERS,*
S.N. EHRLICH,†† AND S. D. BADER*

*Materials Science Division, Argonne National Laboratory, Argonne IL 60439

**Advanced Photon Source, Argonne National Laboratory, Argonne IL 60439

†Brooklyn College of CUNY, Brooklyn, NY 11210

††School of Materials Engineering, Purdue University, West Lafayette, IN 47907

ABSTRACT

We present structural characterizations of a series of sputtered Fe/Nb and V/Nb superlattices by high-angle x-ray diffraction. Diffraction scans were performed with the scattering vector at various angles (χ) with respect to the layers. $\chi=0^\circ$ diffraction spectra (normal to the layers) were fitted to a general structural model to determine the (110) lattice strains, interfacial disorder and interdiffusion. $\chi>0^\circ$ spectra probe the lattice strain of the individual layers and the in-plane interfacial coherence. Both systems form incoherent interfaces above a critical modulation wavelength (Λ_C). At Λ_C , the Fe/Nb system undergoes a crystalline-to-amorphous transition while the V/Nb forms in-plane coherent interfaces.

INTRODUCTION

The study of the interfacial structure of lattice mismatched superlattices has been of increasing interest due to the wide range of anomalous physical phenomena observed in these superlattices [1]. The presence of the interfaces has a profound effect on the magnetic, transport and elastic properties as the layer thicknesses are reduced. The interfacial structure needs to be determined in order to quantitatively understand the anomalous physical properties.

In this paper we study the interfacial structure of sputtered Fe/Nb and V/Nb superlattices with equal layer thickness. Both systems are bcc(110)/bcc(110) oriented with a lattice mismatch of 15 and 8% for Fe/Nb and V/Nb, respectively. The growth procedure and physical property measurements are discussed elsewhere [2,3]. The x-ray diffraction (XRD) spectra were obtained on a Rigaku powder diffractometer using $\text{CuK}\alpha$ radiation and at the National Synchrotron Light Source at Brookhaven National Laboratory on beamline X-18A using 7.5- and 10-keV radiation. Diffraction scans were performed along a number of different crystallographic directions of the superlattice, including (110) in the normal direction, and (211), (200), and (101) which are 30, 45, and 60° from the surface normal, respectively.

DIFFRACTION RESULTS

Shown in Fig. 1 are the diffraction spectra with the scattering vector ($q=4\pi\sin\theta/\lambda$) normal to the superlattice along the (110) direction. If the crystal coherence length is longer than the modulation wavelength (Λ) then the resulting diffraction spectra will be characteristic of the superlattice structure. The diffraction peaks can be indexed by the following relation [4]:

$$2 \frac{\sin\theta}{\lambda_x} = \frac{1}{d} \pm \frac{n}{\Lambda} \quad (1)$$

Figure 1: High angle (110) x-ray diffraction spectra for series of Fe/Nb (left panel) and V/Nb (right panel) superlattices with the scattering vector normal to the layers. Dashed lines indicated expected bulk positions for constituent layers.

where $\bar{d}$ is the average lattice spacing in the growth direction and n is an integer that labels the order of the satellite about the average lattice peak. The only quantities that can be determined from the superlattice peak positions are $\bar{d}$ and Λ . For both systems, the large- Λ spectra are dominated by the scattering from the individual layers in which the Nb layers are slightly expanded in the growth direction. As Λ decreases, well resolved superlattice peaks develop for both systems and the Fe/Nb and V/Nb spectra are similar down to $\Lambda \approx 29 \text{ \AA}$. Below this modulation, the difference between the spectra is quite striking. The Fe/Nb spectrum is a single broad peak characteristic of an amorphous or highly disordered material. However, low-angle-diffraction scans indicate that the sample is still chemically modulated. Below $\Lambda = 28 \text{ \AA}$, the V/Nb diffraction lines sharpen, indicating that the structural coherence length increases and a well defined superlattice structure is maintained for layer thicknesses of less than 3 monolayers.

To quantify the structural parameters of the individual layers requires fitting the diffraction spectra to a general structural model. The solid line in Fig. 2 is the result of the $\Lambda = 18 \text{ \AA}$ V/Nb superlattice using the fitting procedure outline in Refs. 4 and 5. The resulting parameters indicate that the interdiffusion is limited to one monolayer about the interface and

Figure 2: Measured high angle (110) x-ray diffraction spectra for $\Lambda=18\text{\AA}$ V/Nb superlattice (circles) and model calculation (line). Parameters of the calculation are discussed in the text.

both layers are strained in the growth direction. The Nb layer is expanded $\approx 2.5\%$ and the V layer is contracted $\approx 2\%$. For Fe/Nb, the Fe layer expands with decreasing Λ [3]. For the $\Lambda=29\text{\AA}$ sample, the average Fe lattice spacing is expanded $\approx 3.5\%$ and the Nb layer also is expanded $\approx 0.5\%$. This result is consistent with the measured value of $\bar{d}$ which is $\approx 2\%$ expanded over that expected from bulk lattice spacings.

To explore this qualitative difference in the superlattice structure at small modulations, we performed diffraction scans along different crystal directions. Since the scattering vector is no longer normal to the layers and has an in-plane component, the resulting diffraction spectrum will be sensitive to the in-plane coherence of the interface. If the interface is incoherent, the crystal coherence will be limited to individual layers and the layers will scatter independently. The resulting diffraction spectra will be a diffraction peak for each layer, where the position measures the average lattice spacing along that direction and the width will be inversely proportional to the layer thickness. If the interface is coherent, then the lattice planes of the two layers will be correlated and scatter coherently. The resulting diffraction spectra should be characteristic of the superlattices structure with a peak located at the average lattice spacing of the constituent layers and superlattice peaks whose separation correspond to a repeat distance of $\Lambda/\cos\chi$, where χ is the angle of scattering vector with respect to the surface normal.

Shown in Fig. 3 are the diffraction scans along the (101) direction ($\chi=60^\circ$) for the same sample shown in Fig. 1. Since the scattering vector is close to the in-plane direction, the (101) lattice spacing will be sensitive to the in-plane strains. Because of the large lattice mismatch for both systems, coherency strains should result in the Nb being contracted in-plane and the Fe or V layers expanded. For the Fe/Nb samples with $\Lambda > 22\text{\AA}$, a single diffraction peak for the Fe and Nb layers is observed characteristic of an incoherent interface. As the layer thickness is reduced, the peaks broaden, resulting from the finite layer thickness, and shift, indicating an

Figure 3: High-angle (101) x-ray diffraction spectra for series of Fe/Nb (left panel) and V/Nb (right panel) superlattices with the scattering vector angled 60° to the layers. Dashed lines indicate expected bulk positions for constituent layers.

expansion of the Fe and contraction of the Nb(101) spacings. The $\Lambda=22\text{\AA}$ sample shows the same broad diffraction peak as observed in Fig. 1. For $\Lambda>28\text{\AA}$, the V/Nb samples are similar to the Fe/Nb, with a single V and Nb peak characteristic of an incoherent interface showing an expansion of the V and contraction of the Nb. At $\Lambda=28\text{\AA}$, there is a qualitative change in the diffraction spectra. A sharp peak intermediate to the Nb and V position is formed and as Λ is decreased, this average peak sharpens and increases in intensity. This is clear indication of long-range crystalline coherence and that the interface has become commensurate forming a coherent bcc structure.

The transition from an incoherent to coherent interface is more clearly observed in the diffraction scans along the (211) direction ($\chi=30^\circ$) shown in Fig. 4. The $\Lambda=44\text{\AA}$ sample exhibits two broad overlapping diffraction peaks near the expected Nb and V positions. The $\Lambda=18\text{\AA}$ sample has sharp average lattice peaks and clear satellite peaks. The separation of the superlattice peaks gives a repeat distance of $21\text{\AA}$ which corresponds to $\Lambda/(\cos 30^\circ)$.

Figure 4: High-angle (211) x-ray diffraction spectra with the scattering vector angled 30° to the layers for V/Nb superlattices. Dashed lines indicate expected bulk positions for constituent layers.

DISCUSSION

In both systems there is a clear structural transition at a critical modulation wavelength (Λ_C). For the Fe/Nb system, $\Lambda_C \approx 25 \text{ \AA}$ and corresponds to a sudden loss of long-range crystalline order. This type of disorder transition has been observed in a number of bcc/fcc superlattices [6-8]. For the case of W/Ni superlattices [8], the structure after the transition was identified as an amorphous random close pack (rcp) structure. The $\Lambda = 22 \text{ \AA}$ Fe/Nb superlattice spectra are consistent with this type of structure. The first peak in an rcp structure will occur at the same position as the bcc(110) reflection, with no peak at the (200) position [9]. If the structure consisted of small bcc crystallites, then the (200) reflection should be observed. However, there is no evidence of a (200) reflection in any of the $\Lambda = 22 \text{ \AA}$ spectra. For the V/Nb system, $\Lambda_C \approx 35 \text{ \AA}$ and corresponds to the formation of a laterally coherent interface. A similar result was observed for Mo/V superlattices which form a coherent interface at $\Lambda_C \approx 70 \text{ \AA}$ [10].

To understand the different behavior, it is helpful to estimate the critical thickness for the formation of misfit dislocations to relieve interfacial strain. If the layer thickness is below this value, then it is expected that dislocations should disappear and the structure should be coherent. It is important to remember that the critical thickness for dislocations in a superlattice is roughly four times greater than for a single film on a semi-infinite substrate. This is because adjacent layers will take up half the strain at the interface and there are strain contributions from two interfaces per layer. For Mo/V(110), which has a 4% mismatch, the calculated value was

estimated as $\Lambda_C \approx 80 \text{ \AA}$ [10]. By simply scaling this result for the different lattice mismatches, the expected values for V/Nb and Fe/Nb are approximately 40 and 20 $\text{\AA}$, respectively.

The estimated V/Nb value (40 $\text{\AA}$) is consistent with measured value of $\Lambda_C \approx 35 \text{ \AA}$. The estimated Fe/Nb value (20 $\text{\AA}$) is less than the modulation at which the system disorders. Clearly, some additional factor needs to be considered. In a recent article, Fecht proposed a general structural phase diagram in which there is a critical defect concentration at which a crystal will undergo a crystalline-to-amorphous transition at low temperature. When misfit dislocations are introduced to relieve interfacial strain, defects are added to the crystal structure. As the layer thickness is reduced, there will be an increased defect concentration. We propose that for Fe/Nb, the critical defect concentration for amorphization is achieved (at the $\Lambda_C \approx 25 \text{ \AA}$ observed) before the critical thickness to form a coherent interface. This proposal is also consistent with the observation that the Fe expands both in-plane and out-of-plane, since defects tend to result in a volume expansion of the crystal [12].

ACKNOWLEDGMENTS

This work was supported by the U.S. Department of Energy, BES-Materials Sciences, under contract W-31-109-ENG-38

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