EFFECT OF STRUCTURE ON THE ANOMALOUS MECHANICAL PROPERTIES OF METALLIC SUPERLATTICES

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ABSTRACT

The mechanical properties of metallic superlattices have been shown to exhibit anomalous properties. Several of the elastic constants are found to exhibit anomalies which are correlated with structural anomalies in lattice mismatched systems which do not form solid solutions. Lattice matched systems which form solid solutions in their thermodynamics phase diagram, show much smaller elastic anomalies and no structural anomalies. Anomalous plastic behavior, on the other hand, seems to be present in both types of superlattices, indicating that the plastic behavior is possibly defect induced. Detailed quantitative structural measurements combined with comprehensive mechanical properties hold the promise of determining the physical origins of the anomalous properties of metallic superlattices.

INTRODUCTION

It is by now well established that metallic superlattices exhibit anomalous mechanical properties. These include the presence of anomalies in the elastic moduli of lattice mismatched superlattices, and anomalous behavior in the plastic behavior in selected superlattice systems. Simultaneous, quantitative, structural studies indicate that the elastic anomalies are correlated with microscopic structural anomalies and mostly occur in systems which are lattice mismatched and do not form solid solutions in their thermodynamic phase diagram. The anomalous plastic behavior, on the other hand, does not show any clear cut correlations with crystallographic changes or the thermodynamic phase diagram. This indicates that the plastic behavior is possibly modified by defects present at the interfaces.

We present here a review of structural characterization techniques including a new method developed for the quantitative structural refinement studies using X-ray diffraction. These structural studies are correlated with the mechanical properties. The studies presented here show that different types of elastic anomalies can coexist in the same superlattice and that they are correlated with structural anomalies. The exact way in which the elastic properties are affected by changes in the structure is under investigation at the present time.

STRUCTURAL CHARACTERIZATION

Given the strong correlation between the structural and elastic properties of superlattices, it is of the utmost importance to quantitatively characterize their structure. The structural properties of interest include both the lattice spacing changes resulting from interface strains and the amount of structural disorder (i.e. layer thickness fluctuations, interface disorder, interdiffusion, and dislocations). The two techniques most often applied to this problem are Transmission Electron Microscopy (TEM) and X-ray diffraction.

If the sample can be thinned to ≈100Å either perpendicular or parallel to the layers, TEM images may be obtained which give an indication of grain size or qualitative features

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of the layers. In some cases, lattice image pictures on the atomic scale are obtained which allow the study of local defects such as stacking faults or dislocations parallel and perpendicular to the layers [1,2]. TEM is limited by the extensive preparation required to thin the specimen which may change the sample and thus produce uncontrolled artifacts. The TEM image is the result of an average along the path of the election (< 100 Å) which requires modeling of sample thickness, focusing conditions, and structure for a quantitative structural determination. TEM also does not have the resolution to determine small changes in the lattice spacing and may be insensitive to the contrast of materials close in atomic number. Of course, quantitative TEM can be performed by a combination of quantitative intensity measurements combined with structural modeling.

The technique that is most commonly applied to determine both the lattice strains and structural disorder in superlattices is X-ray diffraction in the reflection geometry [3]. It is non-destructive and can provide structural information on the atomic scale. Unfortunately, the information that can be determined directly from the superlattice peak positions is limited to averaged lattice constants of the constituent layers. The low angle part of the spectrum (~ <15°) results from scattering by the compositional modulation of the layers and is not sensitive to their crystal structure. In principle, the low angle diffraction spectra gives directly the Fourier transform of the compositional profile, but disorder, multiple reflections, refraction effects, and surface reflections limits the information obtainable from the Fourier transform of the spectra. The low angle diffraction spectra can be modeled by a recursive application of optical theories [4]. The high angle part of the spectrum is dependent on both the compositional and structural modulation of the layers. The high angle superlattice peak positions are located about the expected Bragg positions of the constituents and are commonly indexed by the relation [5]:

$$\frac{2\sin\theta}{\lambda_{x\text{-}ray}} = \frac{1}{\bar{d}} \pm \frac{n}{\Lambda} \qquad (1)$$

where Λ is the modulation wavelength, $\bar{d}$ is the average lattice spacing of the superlattice, and n an integer determining the order of the superlattice peak. The only quantities that can be determined from the peak positions are $\bar{d}$ and Λ. Because $\bar{d}$ is a model independent parameter, it is a commonly used structural parameter to correlate with elastic anomalies. In a number of lattice mismatched metallic superlattices, $\bar{d}$ expands (~1-2%) with decreasing Λ [6].

The determination of the structural parameters of the individual constituent layers requires modeling of the superlattice structure to compare calculated with measured intensities. By adjusting structural parameters of the model to fit to the measured diffraction spectrum, it is possible to obtain the structure of the constituent layers. This type of structural characterization is commonly used in X-ray and neutron diffraction from bulk, powder samples using the Rietveld refinement procedure [7].

There is a considerable amount of work in the literature discussing modeling of superlattice structures. The models can, in general, be separated into two categories: relative intensity and disorder calculations. The relative intensity calculations are based on the step model [8] where the relative peak intensities are proportional to the square of the structure factor of a single bilayer. The step model has been refined to include compositional and strain profiles [9-12]. Most of the disorder models are based on the original work of Hendricks and Teller [13] assuming random sequencing of layers. These models have included random cumulative fluctuations in the modulation wavelength, layer thickness, and interface distances but have been limited to structures without composition or strain profiles within the layer [14-17].

Only recently has a general theoretical formalism been developed which combines composition or strain profiles with structural disorder [18, 19]. This theoretical formalism

has been incorporated into a general refinement procedure to fit the entire spectrum, determining both the average bilayer structure and statistical deviations from this average. The details of the refinement procedure and the accuracy of the results are discussed elsewhere [18,19]. The results of a refinement on a sputtered Mo/Ni superlattice are shown in Fig. 1. The open circles are the measured spectrum and the thin line is calculated assuming a perfect superlattice with bulk lattice parameters for the Mo and Ni layers. There are two major discrepancies: (i) the measured line widths are considerably broader than the instrumental resolution and (ii) the $\bar{d}$ peak position of the measured sample is at a lower angle than the calculated spectrum. The first results from the disorder in the superlattice and the second indicates that some lattice spacing within the unit cell is expanded over the expected value. The thick line is the result of a refinement procedure [18,19] including both lattice strains, layer thickness variations, and interface disorder. The refined spectrum quantitatively reproduces the measured spectrum over three orders of magnitude in intensity. A number of checks have been performed in order to assure that the type of refinement method described here gives meaningful results. In all cases, it was shown that this refinement is capable of providing quantitative agreement with independently measured parameters.

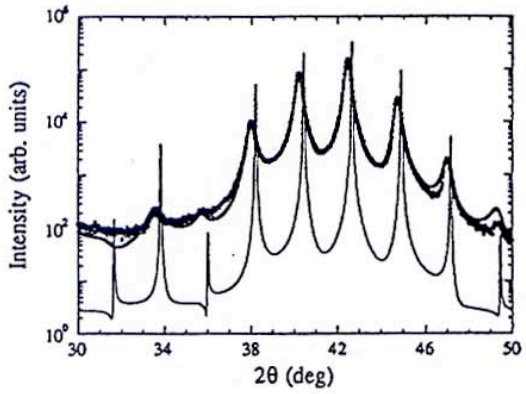

Figure 1: Measured X-ray diffraction spectrum of a $[Mo(20Å)/Ni(22Å)]_{130}$ superlattice (circles) and calculated spectra (thin line) assuming no structural disorder, bulk Mo and Ni lattice spacing, and integer number of atomic planes. Thick line is the result of the structural refinement procedure described in Refs. 18 and 19.

Refined values for the average lattice spacings (i.e. layer thickness divided by the number of atomic planes) for a series of Mo/Ni and Nb/Cu superlattices of equal layer thicknesses are shown in Fig. 2. Both systems, which consist of bcc(110) planes stacked on fcc(111) planes, have a large lattice mismatch, and show a ~1-2% expansion in $\bar{d}$ with

decreasing Λ. In both cases, the fcc layer (Ni,Cu) is found to expand with decreasing Λ whereas the bcc layer (Mo,Nb) is nearly Λ independent. The size of the expansion determined from the refinement is consistent with the measured expansion in $\bar{d}$. The majority of the expansion residing in the Ni layer is in agreement with earlier conclusions [5]. The origins of the lattice changes are not understood and are presently under study.

Information about the in-plane structure can be obtained by transmission or grazing incidence X-ray diffraction. Transmission studies are accomplished by thinning or removing the substrate and passing the x-ray beam through the superlattice. If the layers have different in-plane structures or are sufficiently lattice mismatched, the lattice spacing of the individual layers can be resolved. In-plane studies on epitaxial [20] and polycrystalline [21] films have claimed significant in-plane lattice strains which are associated with coherency strains and substrate interactions, [21] and related to the out-of-plane expansions by Poisson expansion.

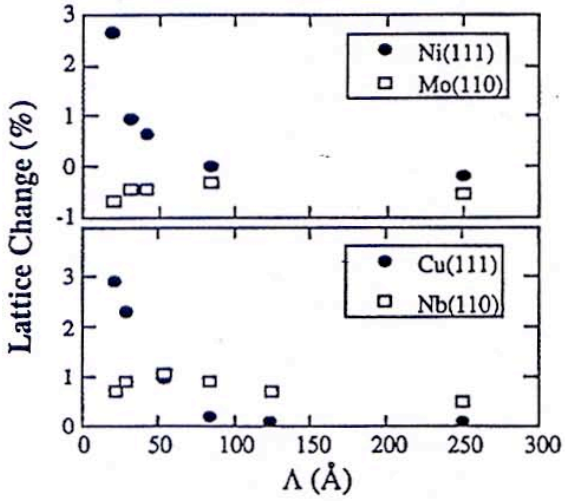

Figure 2: Refined values for average lattice constants for a series of Mo/Ni and Nb/Cu superlattices. Lattice constants are given as percentage change from the bulk values. The Nb/Cu results are for the same series of samples described in Fig. 3.

A number of techniques that probe the local atomic environment have also been applied to these problems. Extended X-ray absorption fine structure (EXAFS) [22] and X-ray photoelectron diffraction (XPD) [23] are element specific techniques which determine the nearest neighbor atomic distances and symmetry. Local magnetic environment of isotopes placed in different locations of the sample have been probed by Mössbauer spectroscopy using the fact that the hyperfine field, isomer shift, and quadrupole moments are sensitive to the nearest neighbor environment [24]. Similar information can be obtained

using Nuclear Magnetic Resonance (NMR), where the interface structure is related to the local magnetic properties [25,26]. Both Mössbauer and NMR are limited to specific elements and require, in many cases, modeling of the measured spectra.

Metallic superlattices for which a number of different structural characterization techniques have been applied to the same samples include Ag/Mn [27] and Co/Au [28]. In Ag/Mn the in-plane spacings determined from reflection high energy electron diffraction (RHEED) during growth [27] showed that the Mn lattice was matched to the Ag layers. The out-of-plane spacings were in quantitative agreement from XPD [27], EXAFS [22], TEM [2], and X-ray refinement [18] determinations and showed a thickness dependent bct structure for the Mn. These results are also in agreement with XPD studies of Ag/Mn/Ag trilayers [23]. In MBE grown lattice mismatched Co/Au superlattices [28] , RHEED [28], TEM [28], X-ray diffraction [20,28,29], and EXAFS [20] studies determined epitaxial orientations and lattice strains both in-plane and out-of-plane for both layers. Unfortunately, this type of detailed structural characterization has not be performed on a superlattice system in which the elastic anomalies have been reported. A detailed structural and elastic characterization of a series of sputtered Nb/Cu superlattices is in progress [30].

MECHANICAL PROPERTIES

A microscopic understanding of the mechanical properties of superlattices not only requires a knowledge of the atomic positions, but also their coupling. A combined study of mechanical and structural properties may provide fundamental solutions to questions regarding the origins of the anomalous elastic properties of superlattices ("supermodulus" effect). Over the past decade during which studies on superlattice properties have flourished, experimental difficulties have precluded a determination of the underlying causes for the supermodulus effect [31]. It is now only, after a period of conflicting reports, that various techniques are providing consistent experimental results for different elastic properties (see Fig. 3). Moreover, it is only recently that sophisticated structural measurements, together with the progress in mechanical measurement techniques provide the means of relating the elastic properties to theoretical calculations.

Nb/Cu is one of the best studied superlattice systems in which the softening of its shear elastic constant C_{44} is well established [31,32]. This softening is strongly correlated with an expansion as large as 1.7% in the average perpendicular lattice spacing, $\bar{d}$ (Fig. 3a). The shear velocity υ (Fig. 3b) extracted from Brillouin scattering determines C_{44} through $\upsilon=\beta(C_{44}/\rho)^{1/2}$, where ρ is the density, and β is a constant which is only weakly dependent on C_{11}, C_{33}, and C_{13}. Similar to Nb/Cu, other immiscible bcc(110)/fcc(111) superlattices (i.e. Mo/Ni and V/Ni) also exhibit shear softenings that strongly correlate with expansions in $\bar{d}$ [31].

Two new elastic measurements techniques have been applied for the first time to a superlattice system. The first technique determines the biaxial modulus from the strain dependence of the membrane modes of the film [32] and show (Fig. 3c) a stiffening of ~15% for Nb/Cu [32]. The second technique determines the flexural velocity υ' (Fig. 3d) from the time-of-flight of the first symmetric Lamb mode generated by a pulsed laser beam [34] and is related to the wavelength independent flexural modulus F_L through $F_L=\rho\upsilon'^2$. The measurements presented here are the most comprehensive study of any superlattice which exhibits elastic anomalies. The distinct feature of these measurements is that different anomalies with different behaviors may coexist in the same material. These differences, however, are not surprising since, at least in principle, the measured moduli are independent from each other.

The solid squares shown in Fig. 3 represent calculations using C_{11}, C_{12}, C_{13}, C_{33}, and C_{44} determined [35] from independent Brillouin scattering measurements of a Nb/Cu superlattice. Within an uncertainty of 13% in the biaxial modulus, and considerably smaller uncertainties in C_{44} and F_L the agreement with the present results is quite good, so it seems that present techniques have overcome the previous experimental difficulties.

The original reports in fcc(111)/fcc(111) noble-metal/transition-metal structures claimed large enhancements in the biaxial, Young, and flexural moduli of Cu/Ni [36,37], Cu/Pd [38], Ag/Pd [39], and Au/Ni [38] superlattices. Many of these systems have now been remeasured using new techniques which are less susceptible to experimental artifacts. A recent time-of-flight measurement of laser-generated and interferometrically detected ultrasonic signals claims no anomalies for the flexural, shear, biaxial and Young moduli in Cu/Ni and Cu/Pd superlattices (in the range of $17<\Lambda<40$Å) [10,40,41]. Also no shear anomalies were reported by Brillouin scattering, and by using a refined uniaxial tension test no anomalies in the Young moduli were observed [42]. In the case of Ag/Pd a novel Brillouin scattering technique has been used which measures the velocity of the longitudinal guided modes[43]. Enhancements in both the longitudinal C_{11} (~ 15%) and shear C_{55} (~ 50%) elastic constants were observed as the modulation wavelength was decreased below $\Lambda = 60$Å. The same technique showed a ~20% softening for C_{44} in Cu/Pd [35] in which Ref. 41 had found no anomaly. Overall, the new measurements seem to agree that previously reported anomalies are either nonexistent or are substantially smaller in magnitude. These studies also imply that for fcc(111)/fcc(111) superlattices the mechanical properties cannot be related to simple structural features and so sophisticated structural studies are imperative.

Some clues on the connection between structure and mechanical properties may be obtained from studies of a material that undergoes a structural phase transition. Since it is known that Fe can exist in a variety of epitaxial phases (determined by growth conditions), Fe/Cu superlattices [44] provide a unique system in which the correlation of elastic anomalies with structural changes can be studied. Fig. 4a, shows $\bar{d}$ for different relative thickness ratios of Fe/Cu. The samples with ratios 3:1 and 1:1 show only slight changes in $\bar{d}$ as a function of Λ, whereas the samples with the ratios 1:2 and 1:3 show little change down to the thickness at which a bcc α-Fe to fcc γ-Fe phase transition occurs (i.e. bcc(110)/fcc(111) to fcc(111)/fcc(111)). At this thickness the structural phase transition is signaled by an expansion in which the average lattice spacing becomes almost equal to the bulk Cu lattice parameter. Fig.4b shows the surface phonon velocity υ, measured by Brillouin scattering. As Λ is decreased υ decreases for all samples; similar to the behavior of other immiscible bcc/fcc superlattices. The most striking result here occurs for the samples with the nominal thickness ratios t_{Fe}:t_{Cu} 1:2 and 1:3 for which a minimum develops coinciding with the Λ at which the α-Fe to γ-Fe transition takes place. Furthermore, as both $\bar{d}$'s increase the surface phonon velocities increase with decreasing Λ. This behavior is opposite to that found for Nb/Cu and implies that it is not possible to assign shear softenings (stiffening) solely to decrease (increase) of the perpendicular lattice spacing. The example of Fe/Cu shows again that simple structural features cannot be used in classifying the elastic anomalies and consequently precise, quantitative structural measurements are needed.

Recent studies show that layering has more drastic effects on the plastic properties than the elastic moduli. A number of superlattices have been found which at small wavelengths exceed the hardness of their own constituents [33,45]. The hardness of a material is determined by both its elastic and plastic behaviors. Since the elastic anomalies are not as large as previously thought the effects of elasticity may be too small to explain

the large enhancements observed in the hardness of superlattices. Therefore, the influence of layering on plastic properties should be carefully examined.

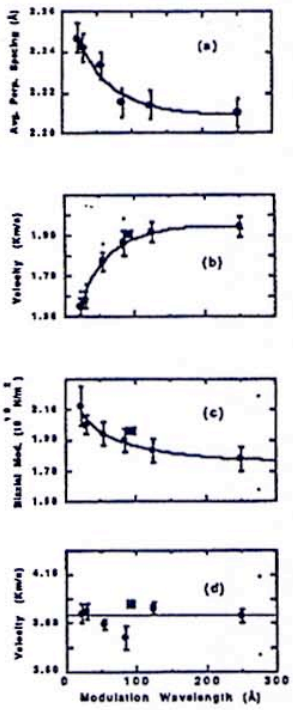

Figure 3:Structural and elastic properties of Nb /Cu superlattices vs. modulation wavelength; (a) average perpendicular lattice spacing, (b) surface wave velocity, (c) biaxial modulus, and (d) symmetric Lamb mode velocity. The solid squares are from Ref. 35 which determined C_{11}, C_{12}, C_{13}, C_{33} and C_{44} values for a Λ = 91 Å sample. The solid lines are guides to the eye (after reference 33).

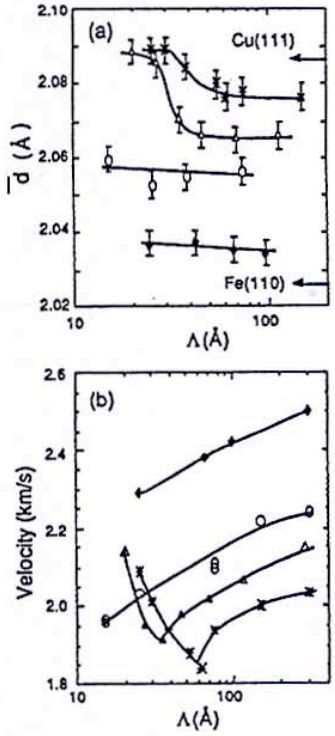

Figure 4: Structural and elastic properties of Fe/Cu superlattices vs. modulation wavelength; (a) average perpendicular lattice spacing, (b) surface wave velocity. The symbols indicate the following Fe/Cu thickness ratios: ◆ 3:1, O 1:1, Δ 1:2 and * 1:3. The solid lines are guides to the eye. (after reference 44).

It is well known that by placing a dislocation close to an interface the resistance to its movement toward the interface will be increased by repulsive forces of its own image. Based on this fact, Koehler [46] first proposed forming strengthened composites by stacking alternating layers of high and low elastic-constant materials with thicknesses smaller (<100 atomic layers) than those in which dislocation generating mechanisms (such as Frank-Read sources) can operate. Koehler showed that in these materials (i.e. Cu/Ni, Ta/W, Pt/Ir, etc.) a much larger force than in ordinary materials is needed, to drive a dislocation. Another mechanism has also been proposed which is analogous to grain boundary hardening, found in polycrystalline materials [47]. In this mechanism, the superlattice interfaces act as pinning sites for dislocations. The change in hardness given by a semi-empirical relation (Hall-Petch formula), is inversely proportional to the square root of the superlattice layer thickness (or the grain size).

Nano-indentation tests on Cu/Ni, Nb/Cu and Mo/Ni samples have shown that their hardnesses are anomalous at small modulation wavelengths. Although for Cu/Ni no elastic anomalies were found, its hardness increased (to a maximum of ~60% above its homogeneous mixture) and similar to polycrystalline materials obeyed the Hall-Petch relation [45]. In contrast to Cu/Ni, the enhancements for both Nb/Cu [32] and Mo/Ni [48] showed no dependence on modulation wavelength (100% enhancement for Nb/Cu and 300% for Mo/Ni over Ni; the hardness of Mo was not measured), favoring a mechanism similar to that suggested by Koehler. The two distinct behaviors of hardness could be related to the differences in the interfaces. The interfaces in Cu/Ni (fcc/fcc) are diffuse, whereas in Nb/Cu and Mo/Ni (bcc/fcc) their profiles are chemically sharp containing discrete steps. Hopefully, further experiments should help clarify questions regarding this issue.

We thank our collaborators in this field, over the years. Many of the ideas presented here have evolved from extensive discussions on these and related matters.

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