

Breakdown of Poisson's effect in Nb/Cu superlattices

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The individual lattice spacings in Nb/Cu superlattices are determined from a crystallographic study using transmission and reflection x-ray diffraction. The Cu layers show a small in-plane expansion which increases to $0.6 \pm 0.1\%$ as the modulation wavelength is reduced to 22 \AA . Arguments based solely on Poisson's effect predict that the strains perpendicular to the layers must be smaller and of opposite sign; contrary to this expectation a large perpendicular expansion ($4.2 \pm 0.7\%$) is measured. This finding brings into question the validity of models that rely only on interfacial forces to derive the perpendicular strains by using the bulk Poisson's ratio.

I. INTRODUCTION

Many superlattices exhibit properties that, based on bulk properties of their constituents, are anomalous.¹ These anomalies occur at small modulation wavelengths where the interfaces are expected to have considerable influence. The anomalous properties include changes in the elastic properties at small modulation wavelengths,² dimensional transitions in superconducting superlattices,³ and anomalous magnetotransport in magnetic superlattices.⁴ Many of these anomalous properties are thought to originate from changes in the physical and chemical structure of the constituents of the superlattice. The complexity of superlattices, however, has precluded complete structural determinations and their structures are often inferred from some structural information combination with assumptions regarding the nature of their interfaces, chemical compositions, and disorder.

Because superlattices are fabricated on substrates, there are restrictions on the scattering geometries which can be used. It is standard to obtain information about the crystallographic structure perpendicular to the surface, but nontrivial to obtain data on the in-plane atomic spacing. These limitations can be overcome by scattering at grazing incidence or in transmission geometry by sufficiently thinning or removing the substrate. These techniques have been applied in a few cases to lattice mismatched superlattices,^{5–9} and the results have shown small in-plane strains consistent with in-plane coherency.

Another limitation in the structural characterization of superlattices is that the diffraction spectra are, in general, a complex average of the constituent layers. In the standard reflection geometry the peak positions are determined only by the average lattice spacing of the constituents and the modulation wavelength.⁶ To overcome this limitation some authors have determined the average lat-

tice spacing as a function of relative composition and then extrapolated to each single constituent.¹⁰ Recent work on the modeling and fitting of x-ray spectra have succeeded in extracting more information including the perpendicular lattice spacing of each constituent.^{11–15}

Superlattices in which the measured in-plane strains have been compared with the out-of-plane strains estimated from comparisons with model calculations, have found that one of the constituents appeared to expand in all directions.^{8–10,13,15} This result is surprising if the driving forces for structural anomalies in superlattices are due only to interfacial in-plane coherency effects since with this model, perpendicular expansions which are larger and of the same sign as the in-plane strains would involve a violation of Poisson's ratio. On the other hand, since similar volume expansions have also been previously reported in epitaxial materials of only a few monolayers thick,^{16–18} it may not be surprising to find similar effects in superlattices.

In the relatively short history of metallic multilayers Nb/Cu has been one of the best studied systems. Since the first report on this system¹⁹ its superconducting^{20,21} and elastic^{22–24} properties have been subjects of extensive studies. The studies on structural properties of Nb/Cu have shown stackings of Nb(110) and Cu(111) layers which are incoherent in the plane forming grains with fiber texture ($\sim 50\text{--}100 \text{ \AA}$ in diameter) randomly oriented around their long axes.²⁵ Since there is only minimal interdiffusion (because of the small 4% solubility of Cu and Nb), Nb/Cu represent an ideal system for the study of fundamental relations between structural and elastic properties.

We present here an x-ray investigation of Nb/Cu superlattices which includes in-plane scattering as well as scattering from atomic planes at various angles from the superlattice normal. An analysis of our results based

only on peak positions and requiring no modeling or assumptions on interface structure, yields both in-plane and out-of-plane average lattice constants for each of the constituent layers. The out-of-plane strains thus obtained, are in agreement with values determined from structural refinement of reflection diffraction spectra. We show that Cu expands in all directions confirming that any model which relies *only* on interfacial strains and uses Poisson's ratio to evaluate the perpendicular strains is incorrect.

II. STRUCTURAL CHARACTERIZATION

The x-ray-diffraction investigation presented here involves scattering from unsupported Nb/Cu superlattices. Sample preparation and characterization with details of elastic measurements are described elsewhere.^{19,22} Briefly, equal thickness Nb and Cu layers were grown at a rate of 10 Å/s, at modulation wavelengths $\Lambda=22, 30, 55, 85, 125,$ and 250 Å, to total a thickness of $7 \mu\text{m}$ on (100) silicon substrates by dc magnetron sputtering. The structural measurements were made on a Rigaku θ - 2θ x-ray diffractometer sputtering. The structural measurements were made on a Rigaku θ - 2θ x-ray diffractometer employing Cu $K\alpha$ radiation. The measurements were performed both in transmission and reflection geometries. In transmission, wave vectors at angles of $\chi=90, 70.5, 60, 54.7,$ and 45° from the sample normal were probed. Well-resolved superlattice peaks were obtained from reflection spectra ($\chi=0^\circ$; see Fig. 1), indicating excellent crystallinity, texturing, and layering. The average perpendicular lattice spacing $\langle d \rangle$ and modulation wavelength Λ , which do not require modeling the superlat-

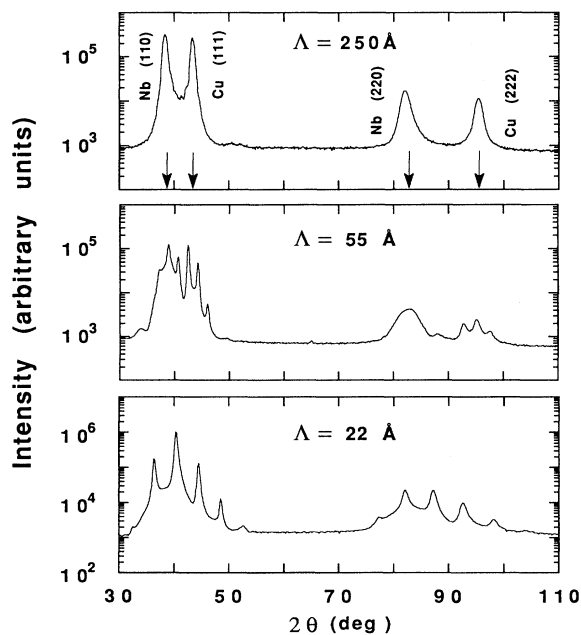


FIG. 1. θ - 2θ x-ray reflection spectra of Cu/Nb at $\Lambda=250, 55,$ and 22 Å with scattering angle $\chi=0^\circ$. The arrows indicate the peak positions corresponding to the bulk.

tice, were determined from the reflection spectra.²² As previously reported for Nb/Cu (Ref. 26) it was found that $\langle d \rangle$ increases as Λ is reduced.

Figure 2 shows typical θ - 2θ transmission scans for selected Λ 's at $\chi=90^\circ$ showing that Nb and Cu crystallites have their [111] and [110] axes perpendicular to the layers, and are randomly oriented in the plane of the film. In Fig. 2 it is clear that peak positions are changing with modulation wavelength. For example, the peaks corresponding to Cu{220} and Nb(112) move closer to each other, and become almost unresolved at $\Lambda=22$ Å. A general feature common to all the transmission spectra is a decrease in peak intensity with decreasing Λ , accompanied by a broadening of the peaks. The in-plane coherence length (ξ_{\parallel}), determined from the peak widths using Scherrer's equation, are almost equal for both layers and decrease from ~ 100 to ~ 45 Å with decreasing Λ . A comparison with the out-of-plane coherence lengths (ξ_{\perp} 's), obtained from reflection spectra ($\chi=0^\circ$), shows that the ξ_{\parallel} 's become substantially smaller than the ξ_{\perp} 's at wavelengths less than ~ 100 Å. In spite of the marked decrease in ξ_{\parallel} , there is a substantial increase in ξ_{\parallel}/Λ .

The transmission spectra were fitted to a superposition of Lorentzian functions. Angle-dependent terms (due to Lorentz polarization, absorption, and Debye-Waller factors) were ignored, since they constituted negligibly small corrections to the measured peak positions. In all cases excellent fits were obtained and the peak positions ob-

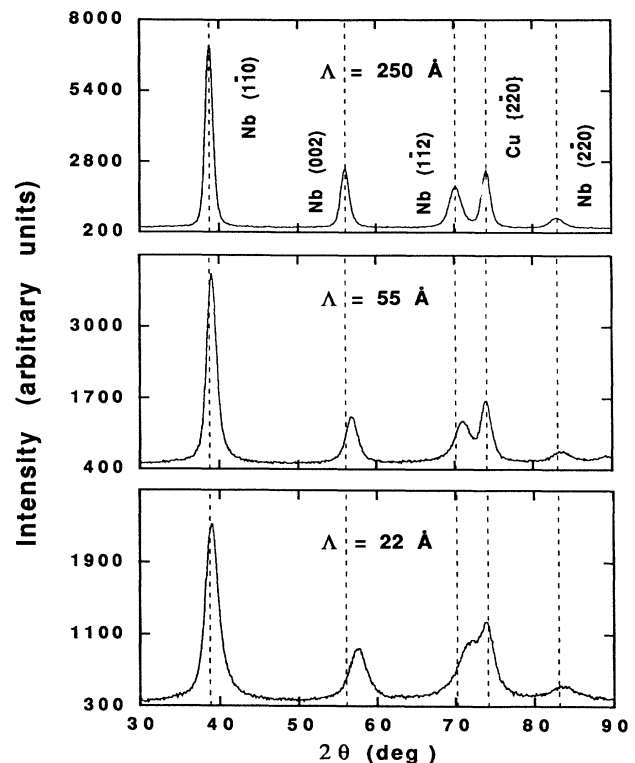


FIG. 2. θ - 2θ x-ray transmission spectra of Cu/Nb at $\Lambda=250, 55,$ and 22 Å with scattering angle $\chi=90^\circ$.

tained from independent scans agreed to within $\sim \pm 0.02^\circ$.

The in-plane spacings of Nb and Cu layers were determined directly from $\chi = 90^\circ$ scans, namely from the peaks labeled Nb(002), Nb(1 $\bar{1}2$), Nb(1 $\bar{1}0$), and Cu{2 $\bar{2}0$ } in Fig. 2. The out-of-plane spacings were determined from calculations based on geometric combinations of spacings along six independent directions obtained from peak positions at selected scattering angles. It is well known that if strains along six appropriate crystallographic directions are measured all six components of the strain tensor of a crystalline material can be determined.²⁷ In other words, if by x rays the lattice spacings along three principal directions and the angles between them are measured the lattice spacings along any direction can be determined. The calculations involve a system of linear equations whose coefficients are derived from the measured lattice strains.

The application of the above procedure to a superlattice involves the assumption that individual layers scatter incoherently along the layers. For the case of Nb(bcc)/Cu(fcc) this assumption is justified since individual layers are incoherent in the x - y plane.^{25,28} It should be noted that the spacings obtained are average spacings across each layer. Any attempt to derive more general expressions valid for nonuniform strains would involve modeling the interface; its structure and its defects. Further studies on these subjects and their implications are discussed elsewhere.²⁹ However, within accuracy of our measurements ($\pm 20\%$), we cannot distinguish any nonuniform strains and due to the sizable changes we report here, they do not change our conclusions significantly.

III. STRUCTURAL ANALYSIS

The Nb in-plane strains along [1 $\bar{1}0$] and [002] are shown in Fig. 3(a); both are contractions but they are anisotropic (i.e., 2.8% and 0.7% along [002] and [1 $\bar{1}0$], for the $\Lambda = 22$ Å superlattice, respectively). In principle, the [1 $\bar{1}0$] and the [002] directions in the strained Nb layers need no longer be orthogonal; if they were not, the [1 $\bar{1}2$] and the [$\bar{1}12$] directions in the basal plane would no longer be equivalent and a doublet should be observed in the spectra. Since we do not observe a doublet and the (1 $\bar{1}2$) peak does not appear to broaden more than the (1 $\bar{1}0$) and (002) peaks, we conclude that these latter two directions remain orthogonal. This is confirmed further by noting that since they are orthogonal a simple geometrical construction allows the strain along [1 $\bar{1}2$] to be calculated from the strains along [1 $\bar{1}0$] and [002]; the results are in excellent agreement with those obtained directly from the (1 $\bar{1}2$) peak in Fig. 2.

In discussing the out-of-plane structure it must again be noted that, in principle, the [110] growth axis of Nb is not constrained by symmetry to be orthogonal to the layers. Following the arguments given above, the lack of observable splitting in the off axis (200) and (101) Nb peaks (at $\chi = 45^\circ$ and 60° , respectively) implies that the [110] axis remains orthogonal to the plane. It is therefore possible to combine the in-plane strain with those measured along the $\langle 010 \rangle$ or $\langle 101 \rangle$ directions to find the strain

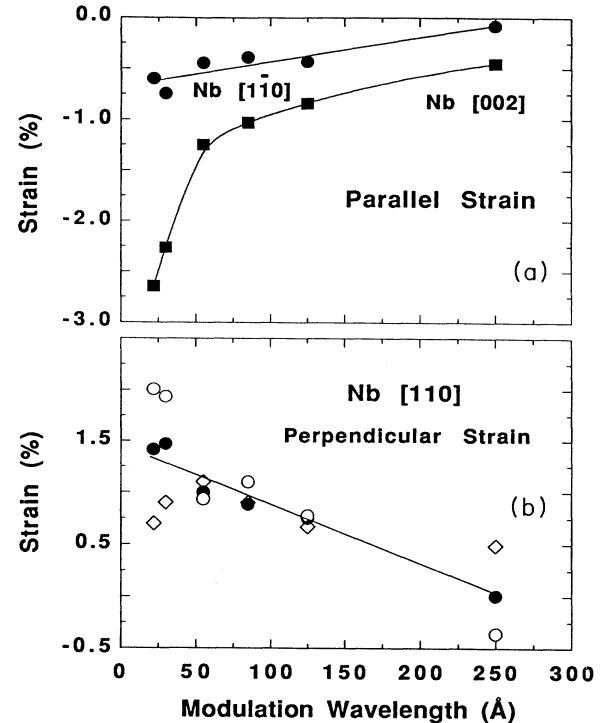


FIG. 3. Strains parallel and perpendicular to Cu/Nb layers. (a) Anisotropic parallel contraction of rectangular Nb(110) plane along Nb[1 $\bar{1}0$] and Nb[002] directions, and (b) perpendicular expansion along Nb[110], the solid (open) circles are obtained from Nb[1 $\bar{1}0$], Nb[100], and Nb[010] (Nb[101], Nb[011], Nb[1 $\bar{1}0$], and Nb[002]) spectra. The triangles are the results of the "structural refinement."

perpendicular to the layering. Assuming the angles between three Nb $\langle 100 \rangle$ directions and the strains along them are known, by using the spacing formula for a triclinic crystal,³⁰ we obtain (after imposing structural constraints discussed above) the following relation between the strains ϵ along [110] and [100] directions:

$$\epsilon_{[110]} = \epsilon_{[100]} + \frac{1}{2} \cos \beta, \quad (1)$$

where β is the angle between equivalent Nb[100] and Nb[010] directions. In terms of measured strains the following relation can be written for β :

$$\cos \beta = 2(\epsilon_{[100]} - \epsilon_{[110]}). \quad (2)$$

Our measured values for strains in Eq. (2) show that β decreases with decreasing Λ ($\sim 89.43^\circ$ at $\Lambda = 22$ Å), indicating a monoclinic distortion of the Nb lattice due to a nonuniform stretching of the Nb(001) plane. Combining Eqs. (1) and (2) yields the strain along Nb[110]; shown in Fig. 3(b) as solid circles. The open circles are results from an independent calculation based on strains measured along [101], [011], [1 $\bar{1}0$], and [002] directions, using similar equations to the ones discussed above. The good agreement obtained between these independent determinations provides an estimate for the accuracy of results. The perpendicular expansions can also be com-

pared with the results obtained from “structural refinement” or fitting¹¹ to spectra similar to those in Fig. 1 which are also plotted (open diamonds) in Fig. 3(b). The agreement between the results obtained by these two completely independent methods reinforces the reliability of both techniques.

We return now to the analysis of the Cu. The in-plane Cu $\{2\bar{2}0\}$ peaks arise from reflections from three sets of planes. These planes may not be equivalent because the adjoining Nb layers do not have threefold symmetry. Since we observe neither a splitting nor a larger broadening of this peak, we conclude that all three sets of planes maintain equal spacings. The strain in Cu $\langle 2\bar{2}0 \rangle$ is plotted in Fig. 4(a), and shows an increasing expansion with decreasing wavelength; reaching 0.6% at 22 Å (open squares). The out-of-plane data from scattering angles $\chi=54.7^\circ$ and 70.5° , corresponding to reflections from Cu $\{200\}$ and Cu $\{1\bar{1}1\}$ planes, respectively, can be again used to show that within experimental error the $[111]$ axis remains orthogonal to the layers. Since under these conditions Cu $\langle 200 \rangle$ directions remain equivalent, in terms of these directions three primitive vectors of equal lengths and subtending equal angles α (with one another) can be defined to describe the distortions of the Cu lattice. The relation between α and the strains ϵ measured along the in-plane $\langle 2\bar{2}0 \rangle$ and out-of-plane $\langle 200 \rangle$ directions can be written as follows:

$$\cos\alpha = 2(\epsilon_{\langle 200 \rangle} - \epsilon_{\langle 2\bar{2}0 \rangle}) . \quad (3)$$

Based on this equation our values of measured strains imply a trigonal stretching along the Cu $[111]$ body diagonal, with α decreasing by $\sim 1.65^\circ$ at $\Lambda=22$ Å. The strain caused by this distortion can be determined by writing the spacing formula for the general triclinic crystal³⁰ and imposing the symmetries discussed above,

$$\epsilon_{\langle 111 \rangle} = \epsilon_{\langle 200 \rangle} + \cos\alpha . \quad (4)$$

The open circles in Fig. 4(b) show the data obtained from the equation above. In order to check the internal consistency of our results, an alternative set of primitive vectors in terms of three equivalent Cu $\langle 1\bar{1}1 \rangle$ directions can be chosen to calculate both parallel and perpendicular strains. An equation similar to Eq. (3) can be written as follows:

$$\cos\alpha' = -\frac{1}{3} + \frac{8}{3}(\epsilon_{\langle 1\bar{1}1 \rangle} - \epsilon_{\langle 2\bar{2}0 \rangle}) , \quad (5)$$

where α' is the angle that Cu $\langle 1\bar{1}1 \rangle$ directions make with each other. We find that α' decreases $\sim 0.95^\circ$ at $\Lambda=22$ Å; this decrease is consistent with the trigonal distortion described above. This can be shown easily by writing an equation similar to Eq. (4) above,

$$\epsilon_{\langle 111 \rangle} = -\frac{1}{2} + \frac{3}{2}(1 + 2\cos\alpha')(1 + 2\epsilon_{\langle 1\bar{1}1 \rangle}) . \quad (6)$$

By combining all four equations and eliminating the angular terms, we obtain a system of two equations and two unknowns that we solve in terms of strains measured along $\langle 1\bar{1}1 \rangle$ and $\langle 100 \rangle$ directions. The calculated strains for Cu $\langle 2\bar{2}0 \rangle$ are plotted in Fig. 4(a) (solid squares), and are in agreement with strains directly measured from $\chi=90^\circ$ spectra (open squares). The solid circles in Fig. 4(b) correspond to the calculated Cu $[111]$ strain. Also shown in Fig. 4(b) are the results (open triangles) obtained from the “structural refinement” method.¹² All three measurements are consistent and show that there is a large perpendicular expansion in the Cu layers.

IV. DISCUSSION

If, as is often assumed, the anomalous properties of superlattices are determined only by coherency effects at the interfaces, then the perpendicular strains should result from standard elasticity theory. Since the perpendicular components of stress σ_{zz} must vanish throughout the superlattice (i.e., there are no external forces applied to the superlattice), the equilibrium condition for ϵ_{zz} in each layer should be

$$\epsilon_{zz} = -\frac{C_{13}\epsilon_{xx} + C_{23}\epsilon_{yy}}{C_{33}} , \quad (7)$$

where ϵ_{xx} and ϵ_{yy} are the in-plane components of strain and C_{13} , C_{23} , and C_{33} are the appropriate elastic constants (all positive definite). If both ϵ_{xx} and ϵ_{yy} are tensile (compressive), then the resulting ϵ_{zz} will be compressive (tensile) consistent with the expected behavior based on Poisson's effect. The elastic constants (C_{ij} 's) of Cu and Nb layers calculated based on their crystalline C_{ij} 's for

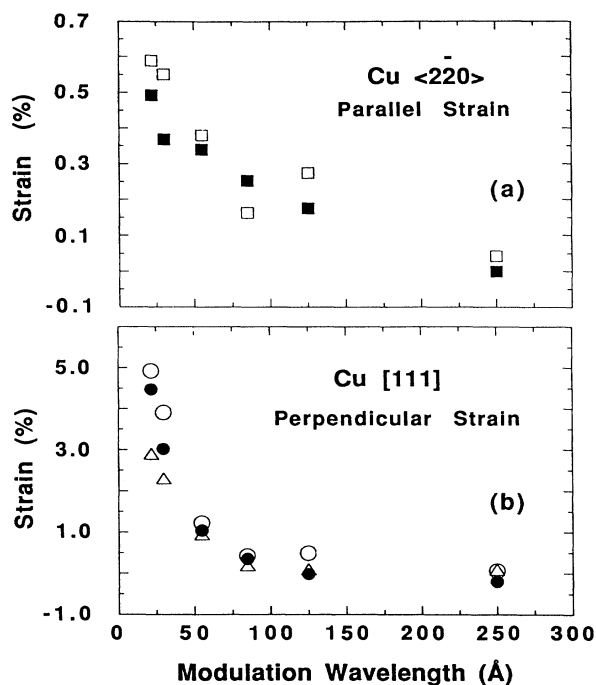


FIG. 4. Strains parallel and perpendicular to Cu/Nb layers. (a) Isotropic parallel expansion of hexagonal Cu(111) plane along Cu $\langle 2\bar{2}0 \rangle$, the closed squares are calculated as described in the text and (b) perpendicular expansion along Cu $[111]$; the triangles are the results of the “structural refinement,” the open (solid) circles correspond to calculations described in the text.

textured layers that are c oriented along Cu[111] and Nb[110] (Ref. 31) are listed in Table I. Since the Cu layers are under a small (0.6%) biaxial strain the listed constants are applicable. Further justification for using these values comes from a pseudopotential calculation³² where under similar strains, changes less than $\sim 10\%$ in C_{ij} 's of Cu were reported. Based on these elastic constants (see Table I) a perpendicular strain of -0.4% is expected for Cu, whereas experimentally an expansion of $4.2 \pm 0.7\%$ is measured. It is clear here that standard elasticity theory is in conflict with our experiment, both in sign and in magnitude, clearly showing the inappropriateness of using arguments based only on Poisson's ratio. Regarding Nb it is more difficult to make a quantitative statement since its in-plane strains are large. To the best of our knowledge no calculation has determined the C_{ij} 's of Nb at large strains. However, if the C_{ij} 's in Table I are used the agreement with experiment is good.

In addition to the fact that the relation between perpendicular and parallel strains cannot be easily explained by standard elasticity theory, the relations between parallel strains are also equally complicated. The expected preferred epitaxy of Nb/Cu is in the Nishiyama-Wasserman (NW) (Ref. 33) orientation (i.e., Cu[1 $\bar{1}$ 0]||Nb[001]). As a consequence, highly anisotropic strains are expected, as observed experimentally. The Nb(110) plane shows an anisotropic contraction reaching $\sim 0.7\%$ along [1 $\bar{1}$ 0], and $\sim 2.8\%$ along the [001] directions, and interestingly the angles between the Nb[001] and Nb[1 $\bar{1}$ 0] directions do not change over the entire range of modulation wavelengths. On the other hand, the Cu(111) plane shows a modest, isotropic expansion of $\sim 0.6\%$.

Another interesting feature of in-plane strains relates to uniform expansion of Cu(111) implying that only equal-biaxial forces are present in the plane of the layers. But this observation is difficult to reconcile with a highly anisotropic expansion of Nb(110) unless a drastic elastic softening along Nb[001] and/or stiffening along Nb[1 $\bar{1}$ 0] is considered. It is noteworthy to mention that a number of superlattice systems (including Nb/Cu) have shown large changes in their elastic constants. For example, Nb/Cu has shown an anomalous softening in C_{44} (Ref. 23) and a stiffening of biaxial modulus,²² and Ag/Pd an enhancement in C_{11} and C_{55} elastic constants.³⁴ Obviously, many factors involving changes in elastic con-

stants, electronic structure of the layers,³⁵ and even influence of defects should be taken into account to arrive at a comprehensive solution for parallel and also perpendicular strains.

The small expansion of Cu and the large contraction of Nb in the superlattice plane, and the large expansion of both Cu and Nb perpendicular to the layers, pose challenging problems for all superlattice models. In particular, the validity of models that by relying only on interfacial forces (i.e., surface tension³⁶ and coherency strains³⁷ models) use Poisson's ratio to derive the perpendicular strains are questioned. These models predict that strains perpendicular to the layers should be smaller and of opposite sign to those in the plane. The behavior of Cu in our samples clearly contradicts this expectation. Recent coherence model arguments applied to Nb/Cu superlattices have assumed interfaces to be coherent and used the average perpendicular expansion of the superlattice (determined from x-ray reflection spectra) as input into the model, and found Cu expanded in all directions.³⁷ Given that observed in-plane lattice changes are much smaller than the lattice mismatch, the formation of a coherent interface can be ruled out. Another structural model is the grain boundary model,³⁸⁻⁴⁰ where the strains induced by disorder at interfaces are calculated from computer simulations. The disorder is produced by twist boundary interfaces where atoms are displaced from their ideal crystalline lattice sites. Although a detailed comparison with the grain boundary model is not yet possible (because it has not been extended to fcc/bcc superlattices), model calculations for fcc/fcc superlattices with either lattice mismatched or grain boundary interfaces, reproduce qualitatively the features of the elastic anomalies and also yield small in-plane strains and large perpendicular expansions⁴⁰ in qualitative agreement with the present results.

Another complication for structural models is the relative magnitude of the in-plane strains: if interfacial stresses were responsible, the compressive and tensile forces in each constituent would be equal. Because the layers of Nb and Cu have equal thicknesses and their elastic constants are not widely different, roughly equal strains but of opposite sign are expected in each constituent. The 2.8% and 0.7% contractions of Nb[002] and [1 $\bar{1}$ 0], and the 0.5% expansion of Cu<2 $\bar{2}$ 0> again are not consistent with this expectation.

The recently proposed electron transfer⁴¹ model which has predicted an isotropic expansion of each constituent is also clearly at odds with the experimental data. The electronic explanation based on zone folding^{42,43} appears to be general enough to be consistent with our observations, but has so far not provided quantitative values for elastic anomalies or lattice changes. A recent electronic structure calculation³⁵ has found an electronic energy increase for Cu and a decrease for Nb, with increasing interfacial electronic energies for Cu and decreasing for Nb. A complete comparison with our structural measurements, however, requires specific information regarding individual spacings and further calculations are required for this purpose.

An important problem but a difficult one to account

TABLE I. Elastic constants of Cu and Nb in a rotated coordinate system (about z) where for Cu; z ||[111], y ||[1 $\bar{1}$ 0], and x ||[11 $\bar{2}$], and for Nb; z ||[110], y ||[001], and x ||[1 $\bar{1}$ 0]. The data are in GPa units.

C_{ij}	Nb	Cu
C_{11}	219	221
C_{22}	246	221
C_{33}	219	238
C_{12}	134	105
C_{13}	161	87.5
C_{23}	134	87.5

for is the possibility of interdiffusion. For example, the expansion of Cu due to electronic effects may also enhance the inclusion of Nb impurities. Based on Vegard's law of solid solutions an expansion of Cu is expected. If this expansion were more pronounced in out-of-plane direction it would lead to the breakdown of Poisson's effect. But given Cu's small in-plane and comparatively large out-of-plane expansions, we must note, however, that these anomalous expansions may not be entirely due to Nb impurities alone. The ~4% volume expansion of Cu layers would involve an ~10% interdiffusion of Nb atoms (based on Vegard's law). An interdiffusion of this size is not supported by x-ray refinement measurements, and given the immiscibility of these materials seems unreasonable.

In conclusion, the strain profiles of individual layers of the Nb(bcc)/Cu(fcc) superlattice were measured as a

function of modulation wavelength. Different x-ray-diffraction techniques consistently find perpendicular expansions for both layers. Most importantly our measurements show that in Cu layers, an anomalously large perpendicular expansion coexists with an in-plane expansion, a fact which cannot be accounted for by standard elasticity.

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