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Relationship between structural properties and elastic anomalies in Ag/Ni superlattices

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Abstract. The connection between the microstructure of sputter-deposited Ag/Ni superlattices and their elastic response has been investigated in order to give evidence of the role of interface effects in determining the physical properties of this kind of structure. X-ray and transmission electron microscopy (TEM) experiments have shown that the Ag/Ni interfaces are semicoherent even at very low periods; in addition, there are an appreciable number of structural imperfections, such as stacking faults, twins and atomic disorder at the interfaces. The softening of the shear effective elastic constant c_{44} , observed at low periods by means of Brillouin scattering experiments, can be simulated by assuming the presence of a modified layer at each interface, $2/2$ atomic planes thick, characterized by a reduced value of the shear effective elastic constant. A comparison between the set of experimental data relating to Ag/Ni superlattices and existing theoretical models suggests that elastic anomalies can be attributed to the presence of atomic disorder at the interfaces, rather than to coherency strains, surface tension stresses or electronic effects.

1. Introduction

The investigation of the elastic properties of metallic multilayers and superlattices has shown that it is common to find deviations and anomalies with respect to predictions based on continuum elasticity, within the effective-medium approximation [1]. In particular, Brillouin spectroscopy studies have given evidence of a marked decrease of the shear effective elastic constant c_{44} as the period decreases in a number of superlattice systems whose constituents are immiscible, such as Nb/Cu [2], Mo/Ni [3], V/Ni [4], Mo/Ta [5], and Ag/Ni [6]. In some other systems, whose constituents can form solid solutions, no systematic dependence of the elastic constants on the period has been found (Cu/Ni [7, 8], Cu/Pd [8] and Cu/Co [9]), or an enhancement of the elastic constants has been measured (Ag/Pd [10], Ta/Al [11]). In coincidence with the elastic anomalies, structural investigations performed by means of x-ray diffraction have shown that in most systems the average lattice parameter perpendicular to the interfaces undergoes an expansion, typically of 2%. A number of theoretical models have been proposed, which attribute the elastic anomalies to different physical mechanisms, such as electronic effects [12], coherency strains [13], surface tension stresses at the interfaces [14], deformations induced by a difference in the Fermi energy between the constituents [15] and the presence of disorder at the interfaces [16]. However, discrimination among the above models could not be made, since the amount and the quality of the structural information available from experiments is not sufficient to determine selectively the physical mechanism responsible for the elastic anomalies; it turns out that a satisfactory understanding of the

elastic response of metallic superlattices in terms of their structural properties is still an open problem.

In previous research, the elastic properties of Ag/Ni superlattices, both supported and free standing, have been extensively investigated by use of Brillouin light-scattering and conventional static techniques [6, 17–19]. This superlattice system is particularly interesting for studying the connection between structural and elastic properties, because of the high lattice mismatch existing between Ag and Ni ($\cong 15\%$) and their complete immiscibility. It has been found that the effective shear elastic constant c_{44} depends strongly on the bilayer period: it undergoes a softening of about 40% when the period decreases from 18 to 2 nm; in this same interval the elastic constant c_{33} exhibits a reduction of about 13%, while c_{11} remains substantially unchanged, as does the macroscopic Young's modulus. Annealing the specimens causes a complete recovery of the elastic softening, due to the destruction of the stratified structure and the formation of three-dimensional grains of pure Ag and Ni.

The aim of this paper is to analyse in some detail the structural properties of Ag/Ni superlattices and to investigate the connections with the elastic anomalies observed previously. Much attention is focused on the role of interface effects in determining the elastic response of this kind of structure. Theoretical models proposed in the literature are critically reviewed in light of the experimental data outlined here.

2. X-ray diffraction

The Ag/Ni superlattices analysed have been deposited by DC sputtering on glass substrates at the Centre d'Études Nucleaires de Grenoble (France); details of their preparation and growth conditions can be found elsewhere [20]. The specimens are equiplanar (i.e. with an equal number of atomic planes of Ag and Ni in a period) and the deposition temperature was either 300 K or 100 K. In the first case the superlattices remain adherent to the glass substrate, while in the second case they detach spontaneously when the gas is admitted into the sputtering chamber, after completion of the deposition. In a previous paper [20], Rodmacq presented an x-ray characterization of the Ag/Ni superlattices we are concerned with. He found that the superlattices are polycrystalline, with a well defined (111) texture perpendicular to the layers; no difference between superlattices deposited at different temperatures was found at large periods; however, for periods below 4/4 atomic planes the stratified structure is preserved only for the specimens deposited at 100 K (in those deposited at room temperature there is evidence of the formation of three-dimensional grains of pure Ag and Ni). The spectra were simulated assuming the superlattices to be ideal (step interfaces) while no attempt to reproduce the experimental linewidth was made; similarly, no attempt to determine the lattice parameters from each spectrum was made.

In the present research, we have performed a further x-ray investigation, in order to obtain more quantitative information on the microscopic properties of these superlattices. A θ - 2θ standard diffractometer, which is described in detail elsewhere [21], has been used for the analysis of five specimens deposited at 300 K (still adherent to their substrates), using the Cu $K\alpha$ radiation ($\lambda = 0.15418$ nm). In agreement with previous x-ray investigations of superlattice systems whose constituents are immiscible and present a large lattice mismatch [22], we have observed that the lineshape of these spectra is markedly affected by the presence of interfacial disorder. Following Locquet *et al* [23] the presence of random imperfections at every interface (such as misfit dislocations), or random thickness errors leading to a non-integer number of atomic planes per layer is simulated assuming that the distance at the interfaces between unlike atoms fluctuates around the average value

$d = \frac{1}{2}(d_{\text{Ag}} + d_{\text{Ni}})$, according to a continuous Gaussian distribution of width σ (d_{Ag} and d_{Ni} are the interplanar spacings along the growth direction, whose values for the bulk materials are 0.235 86 nm and 0.203 41 nm, respectively). The intensity scattered by a superlattice consisting of N bilayers can then be written as

$$I(q) = N[A^2 + B^2 + 2AB \exp(-q^2\sigma^2/4) \cos(qp/2)] + 2 \sum_{m=1}^{N-1} (N-m) \{ (A^2 + B^2) \times \exp(-2mq^2\sigma^2/4) \cos(2mqp/2) + AB \exp[-(2m+1)q^2\sigma^2/4] \cos(2m+1)qp/2 + AB \exp[-(2m-1)q^2\sigma^2/4] \cos(2m-1)qp/2 \} \quad (1)$$

where $|q| = 4\pi \sin \theta / \lambda$ is the exchanged wavevector, $A = f_{\text{Ag}} \sin(n_{\text{Ag}}qd_{\text{Ag}}/2) / \sin(qd_{\text{Ag}}/2)$ and $B = f_{\text{Ni}} \sin(n_{\text{Ni}}qd_{\text{Ni}}/2) / \sin(qd_{\text{Ni}}/2)$ are the amplitude factors, $p = (n_{\text{Ag}} - 1)d_{\text{Ag}} + (n_{\text{Ni}} - 1)d_{\text{Ni}} + 2d$ is the superlattice period, and f_{Ag} , f_{Ni} are the scattering factors [24].

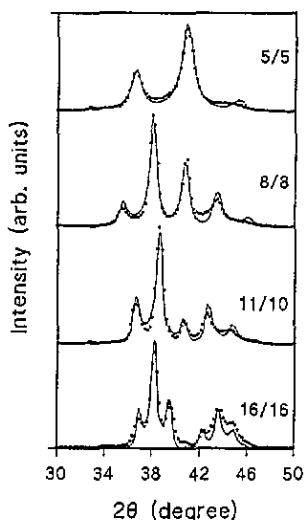


Figure 1. Experimental (points) and calculated (full curves) x-ray θ - 2θ diffraction spectra relating to Ag/Ni superlattices, obtained using the Cu K α radiation ($\lambda = 0.154$ 18 nm). For each spectrum, the number of atomic planes of Ag and of Ni is indicated.

The calculated full line shape is fitted to the measured diffraction spectra, taking the interplanar spacings d_{Ag} and d_{Ni} , the number of atomic planes per layer n_{Ag} and n_{Ni} and the fluctuation amplitude σ as free parameters. In the calculation, the angular dependence of the scattering factors is taken into account, as well as the corrections for the polarization, absorption and Debye-Waller factors. A comparison between the experimental x-ray θ - 2θ spectra (points) and those calculated using the above model (curves) is shown in figure 1. The behaviour of the average interplanar (111) distance d as a function of the period is reported in figure 2: in accordance with [20], a slight expansion ($\cong 0.7\%$) is observed for periods below 16/16 atomic planes per layer. We notice that this expansion is appreciably lower than that observed in a number of previously investigated metallic superlattices; in addition, the simulation shows that the above expansion results, for all the specimens, from the simultaneous but unequal expansions of both Ag (about 0.1%) and Ni (about 1.4%). The behaviour of the parameter σ/d , which is a measure of the degree of interfacial disorder, is shown in figure 3. We notice that the value of σ/d at low periods is of the same order

of magnitude as the lattice mismatch ($\cong 0.15$), thus suggesting that the interfacial disorder, responsible for the observed linewidth, is induced by the lattice mismatch. In addition, the value of σ/d increases roughly linearly with the period, indicating that the amount of disorder grows as the residual degree of coherency vanishes, due to the progressive appearing of misfit dislocations, as discussed in detail in section 4.

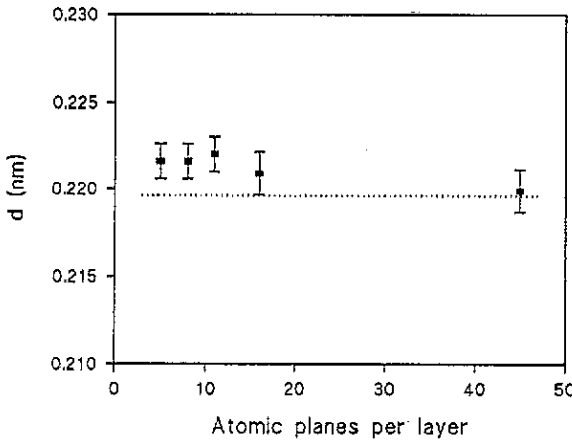


Figure 2. Values of the average interplanar distance d as a function of the period (expressed by the number of atomic planes of Ag and Ni per elemental layers), obtained from the x-ray diffraction spectra. The dotted line is the value expected for undistorted layers, obtained from the bulk values of the interplanar distances d_{Ag} and d_{Ni} .

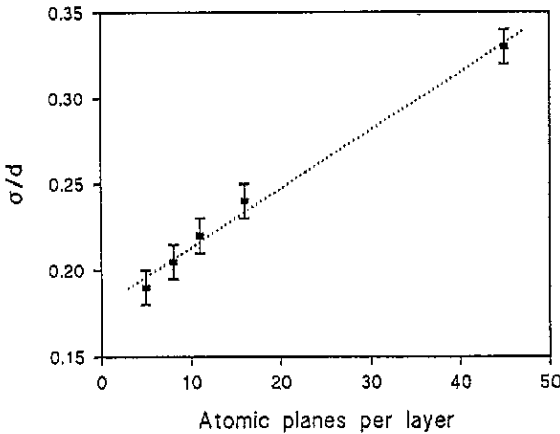


Figure 3. Dependence of the parameter σ/d on the superlattice period (expressed by the number of atomic planes of Ag and Ni per elemental layer), obtained from the x-ray diffraction spectra.

3. Electron microscope analysis

Transmission electron microscopy has been used to analyse the microstructure of four unsupported specimens, deposited at 100 K, with 3/3, 5/5, 16/16 and 40/40 atomic planes per layer, respectively. They have been prepared for TEM by Xe ion-beam milling at liquid-nitrogen temperature; both in-plane and cross section observations were carried out with a Jeol 4000 FX electron microscope, operated at 400 kV.

Figure 4 shows selected-area diffraction (SAD) patterns from the 5/5 and the 40/40 plane specimens. These pictures are similar to those obtained by Yuansheng and Simon [25] in a previous investigation of Ag/Ni superlattices; the strong (111) texture is confirmed, together with the anomalous presence of the extra reflections (422)/3 Ag and (111) of both materials. The former, whose intensity is found to increase with the period of the multilayer, corresponds to the (100) reflection of an HCP packing of hard spheres of the same size; it originates either at the interface between adjacent layers, where truncated FCC cells are present, or at planar defects such as stacking faults or twin boundaries, which locally generate a hexagonal symmetry [26]. The latter, whose intensity decreases with the superlattice period, can be explained by the streaks associated with (111) reflections belonging to the first-order Laue zone (FOLZ). This is demonstrated in figure 5, where an SAD pattern from the 5/5 specimen, tilted by 60° from the surface normal, shows that the forbidden (111) reflections belong to the FOLZ.

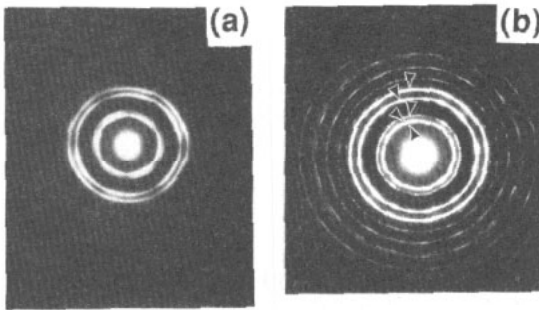


Figure 4. SAD from the 5/5 (a) and 40/40 (b) specimens. The arrows in figure 4(b) mark the following diffraction rings, starting from the inner ones: Ag (422)/3, Ag (111), Ni (111), Ag (220) and Ni (220).

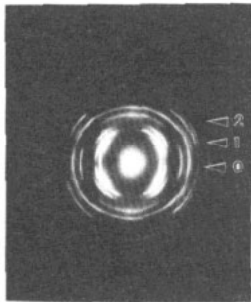


Figure 5. SAD from the 5/5 specimen tilted by 60° from the surface normal. The arrows mark the [111] Laue zones.

Dark-field observations show that all the specimens consist of columnar grains crossing several layers and eventually extending from the top to the bottom of the multilayer. For periods up to 16/16 atomic planes, the columns are less than 10 nm wide. Adjacent columns exhibit a slight misorientation around the common [111] axis, so that bright areas, about $0.5 \mu\text{m}$ wide, can be observed in the dark-field images obtained with electrons scattered in an angular width of about 5 mrad of the (220) reflection ring (figure 6(a)). In contrast, the 40/40 specimen has larger columns up to 100 nm wide, with random in-plane orientation (figure 6(b)). On reducing the size of the impinging electron beam, one can observe diffraction from a single columnar grain for large periodicities and from a small number of adjacent columns for lower ones; the rings of figure 4 collapse, in this case, to rather

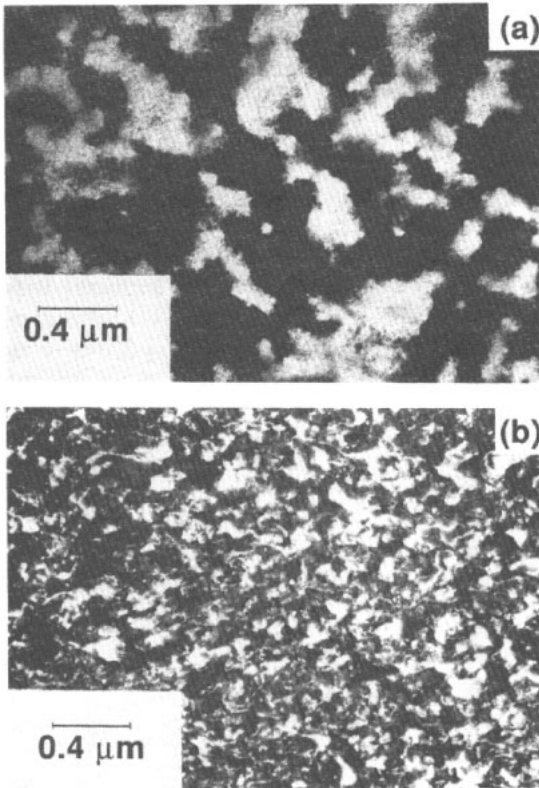


Figure 6. (220) dark-field image of the 5/5 (a) and 40/40 (b) specimens.

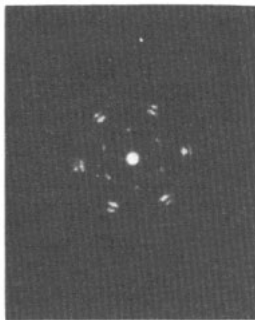


Figure 7. Microdiffraction pattern from the 5/5 specimen (see figure 4(b) for indexing).

well defined spots, as shown in figure 7. It is to be noticed that the forbidden (111) reflections make an angle of 30° with the (220) reflections of both metals which, pointing the same direction, indicate that Ag and Ni grow one over the other preserving the in-plane orientation. The Ni and Ag(220) lattice spacings (parallel to the interfaces) have been measured from the plane-view diffraction patterns. It has been found that, consistent with the observations of [25], they exhibit an appreciable dependence on the period, as seen in figure 8; an expansion of d_{220}^{Ni} of about 1.5% is seen, corresponding to a similar contraction of d_{220}^{Ag} ; however, the values of the lattice spacings are very close to those of bulk Ag and Ni, even in the specimen with only 3/3 atomic planes. This indicates that the interfaces are semicoherent with the lattice mismatch relaxed by interfacial dislocations, in agreement with previous HREM studies [27].

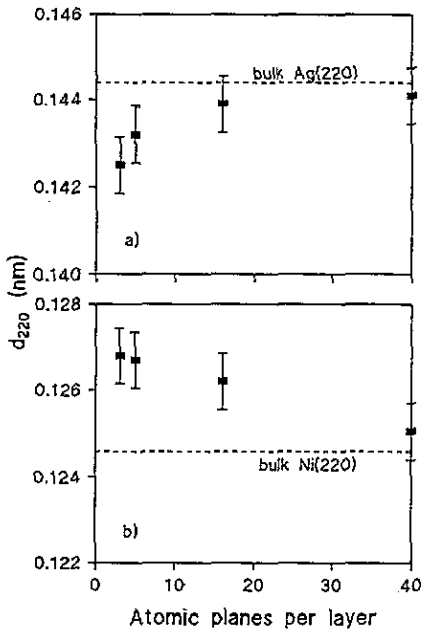


Figure 8. Behaviour of the lattice parameter parallel to the interfaces d_{220} for both Ag (a) and Ni (b), as a function of the superlattice period, determined from TEM diffraction experiments.

Further information about the microstructure of the specimens has been obtained from cross-sectional observations. Relevant diffraction patterns are seen in figure 9: the effect of the artificial periodicity is visible as a set of fringes, particularly evident at low periodicities (figure 9(a)), modulating only the reflections from planes normal to the growth direction (111). From analysis of figure 9(a) it can be argued that in low-period superlattices (e.g. 5/5) the [111] axis exhibits a spread of about 20° around the growth direction, so that reflections not normal to the surface are transformed in elongated streaks. The combined effect of [111] growth axis misorientation and reduction of the optically coherent zones due to the artificial periodicity is strong enough to excite the (111) reflections belonging to the FOLZ, in a plane-view observation. It is also interesting to note that in the [110] average zone axis extra spots can be observed due to the presence of twins about the growth direction (figure 9). In the 40/40 specimen, the columns are large enough to obtain a diffraction pattern from a single grain (figure 9(b)); streaks parallel to the growth directions are observed for all the reflections, except those from planes parallel to the multilayer surface, i.e. parallel to (111). This suggests that the displacement vector of the corresponding planar defects lies in these planes, in agreement with the observation of a large density of twins in the structure. Figure 10 shows the bright-field image of the 40/40 specimen, in cross section; the stratified structure is visible and it appears to be of good quality. Twins are also seen in this picture; twin boundaries make a small angle with the interlayer surface so that they do not lie on (111) planes, despite the fact that the reciprocal lattice streaks are observed parallel to the growth direction. From these cross-section observations one can also achieve a deeper insight into the presence of forbidden reflections in the plane-view diffraction patterns. The intensity of the $\text{Ag}(422)/3$ reflection appears to increase with the period, as a result of the growth of structural disorder. As for the (111) forbidden reflections, two regions can be distinguished, as follows:

- at low periods they originate from the misorientation of the columnar grains, combined with the extension of reciprocal lattice nodes, due to the small artificial periodicity; and
- at large periods, the effect is mainly due to the high density of planar defects.

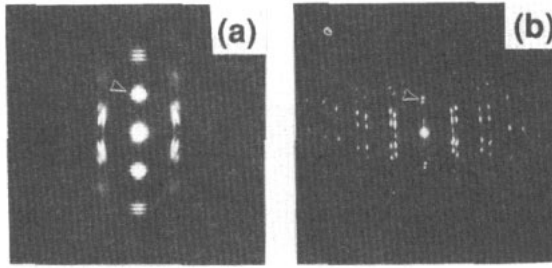


Figure 9. Electron diffraction pattern from the 5/5 (a) and 40/40 (b) cross-sectional specimens in the [110] orientation. The arrows mark the (111)Ag and Ni reflections.

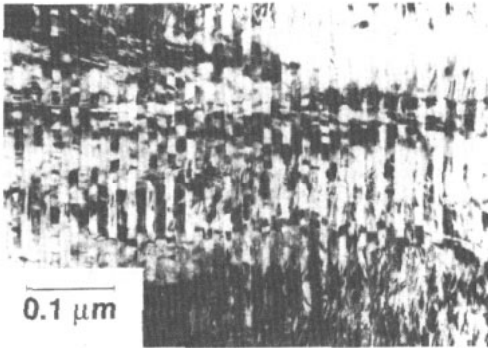


Figure 10. Bright-field image of the 40/40 cross-sectional specimen.

4. Elastic anomalies: the role of the interfaces

On referring to the experimental data reported in [6], we will now analyse the role of interface effects in determining the elastic response of Ag/Ni superlattices. In order to reproduce the observed dependence of the shear effective elastic constant c_{44} on the superlattice period, taking into account the presence of semicoherent interfaces and interfacial disorder, we use the simple model introduced by Clemens and Eesley [28]. In this approach, one assumes that at each interface there is a modified layer with effective elastic constants c_{ij}^{int} , so that the superlattice consists of the periodic repetition of three different elemental layers: Ag, Ni and the interface layer. Following Grimsditch and Nizzoli [29], the effective constants of the superlattice can be expressed in terms of those of the constitutive layers and their thicknesses D_{Ag} , D_{Ni} and D_{int} . In particular, for the shear elastic constant one obtains the following expression:

$$c_{44} = p(D_{Ag}/c_{44}^{Ag} + D_{Ni}/c_{44}^{Ni} + D_{int}/c_{44}^{int})^{-1}.$$

We have used the above expression to reproduce the behaviour of c_{44} observed experimentally. The values of c_{44}^{Ag} and c_{44}^{Ni} are assumed to be those measured on thick Ag and Ni films [6], while the elastic constant c_{44}^{int} and the relative thickness D_{int} are taken as free parameters. The result of the fit procedure to the experimental points is shown in figure 11: the best agreement with experiment has been found by assuming the interface layer to be made up of two atomic layers of Ag and two of Ni, with a modified elastic constant $c_{44}^{int} = 16.8$ GPa. The fairly good agreement between the calculated curve and the experimental points in figure 11 indicates that, in spite of its simple formulation, the

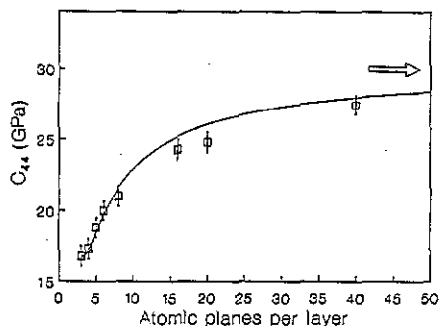


Figure 11. Experimental values (squares) of the shear effective elastic constant of Ag/Ni superlattices, determined from Brillouin light scattering. The full curve is calculated assuming the presence, at each interface, of a modified layer 2/2 atomic planes thick.

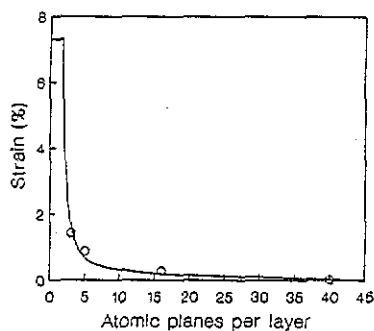


Figure 12. Comparison between the Ag strain parallel to the interfaces measured by means of TEM experiments (circles) and calculated according to the model of [32] (curve).

assumption of a modified interface layer is suitable to describe the measured dependence of the elastic response on the period of the structure.

At this point one could wonder whether a variation of the relative thickness between Ag and Ni elemental layers would significantly affect the response of this structure; this would indicate that the softening observed comes not only from interface effects but also from some other effect occurring inside one of the constituents. In fact, dealing with Mo/Ni superlattices, other authors have hypothesized that the elastic anomalies in this structure depend on some effect occurring in the Ni layers [30]. To clarify this point, we have again used Brillouin spectroscopy to characterize an Ag/Ni superlattice, 1 μm thick, in which the Ag elemental layers have a thickness double that of Ni (1.2 nm against 0.6 nm). In this case we have found that the Rayleigh wave velocity is 1270 m s^{-1} , corresponding to an effective elastic constant $c_{44} = 17.2$ GPa. Significantly, this value is consistent, within the experimental error, with that obtained using the model described above, i.e. assuming again an interface layer 2/2 atomic planes thick, with $c_{44}^{\text{int}} = 16.8$. We then conclude that the softening observed at low periods can be attributed to effects originating at the interfaces, while it does not appreciably depend on the ratio between the thicknesses of the constitutive Ag and Ni layers.

5. Discussion

We will now briefly discuss the connections between the structural and the elastic properties outlined above, in light of the theoretical models proposed in the past to explain the behaviour of metallic superlattices. The first important property which is to be analysed is the semicoherency of Ag/Ni interfaces. It is well known that the degree of coherency at the superlattice interfaces depends on a number of parameters, such as the natural lattice

misfit between the two constituents, the relative thicknesses of the layers and their elastic constants. In particular, it can be defined a critical value p_c of the period, so that for periods lower than p_c the interfaces are coherent and the layers are strained, in order to assume the same value of the interatomic distance; on the other hand, for periods larger than p_c , the strain is progressively relaxed through the creation of misfit dislocations, while the layers become undistorted. We have then used the model proposed by Van der Merwe and Jesser [31] to calculate the critical period and the residual coherency strain at the interface for a superlattice characterized by a lattice mismatch of 14.7% (Ag/Ni). Figure 12 shows a comparison between the calculated strain parallel to the interfaces and the values obtained from the experimental points of figure 8. It can be seen that for $p \geq p_c \cong 0.9$ nm the expected strain decreases drastically, as a consequence of the transition from coherent to semicoherent interfaces; even in the specimen with only 3/3 atomic planes ($p \cong 1.3$ nm), the experimental point accounts for a residual strain at the interface of about 1.5%, i.e. ten times lower than the natural lattice misfit; for higher periods the strain further decreases, up to vanishing for the 40/40 specimen. This decrease of the residual strain can be interpreted in terms of the progressive creation of misfit dislocations at the interfaces, whose presence is also accounted for by the increase of the parameter σ/d with period in x-ray spectra (see figure 3). On the basis of the curve of figure 12 one can also understand why it was found experimentally [20] that it is impossible to grow superlattices with 2/2 atomic planes per layer; in this case, in fact, the period is about 0.88 nm, i.e. lower than p_c , and the layers should undergo very large deformations.

With respect to the physical mechanisms responsible for the elastic anomalies, we can exclude any relevant role of coherency strains at the interfaces, since, as shown above, Ag/Ni superlattices are substantially non-coherent for every period analysed; in addition, if we compare the deformations of the constitutive layers perpendicular and parallel to the interfaces, we can argue that the deformations of the constitutive layers do not obey predictions based on the Poisson effect. In fact, TEM observations have given evidence of a contraction of Ag and a simultaneous expansion of Ni parallel to the interfaces (figure 8); as a consequence, one should expect that Ni contracts perpendicularly to the interfaces, but this is contrary to the x-ray observed expansion of Ni (section 2); this behaviour is similar to the recently observed breakdown of the Poisson effect in Nb/Cu superlattices [32]. Therefore, it appears that the approach of [13], based on the role of coherency strains, is not able to explain the observed dependence of the elastic constants on the period. Similarly, the theoretical model based on surface tension stresses at the interfaces [14] is not consistent with our data, since it predicts a contraction of the lattice parameters parallel to the interfaces for both the constituents and this is inconsistent with the observed expansion of d_{220}^{Ni} (figure 8). Moreover, the phenomenological model based on electron transfer between adjacent layers [15] and on the Murnaghan equation of state, which has been successfully used in the past [1] to connect the expansion perpendicular to the interfaces to the variation of c_{44} , seems not adequate for the Ag/Ni system; from the observed 0.7% expansion (perpendicular to the interfaces) it would predict a softening of c_{44} three times lower than the one actually observed. Finally, the electronic model which attributes the elastic anomalies to a critical contact between the Fermi surface and the artificial Brillouin zone [12] predicts a hardening of the elastic response, rather than the softening observed. The only theoretical approach which seems to be consistent with our experimental data is that of Jaszczak *et al* [16], which attributes the softening of the elastic constants to atomic disorder at the interfaces; molecular dynamics simulations show that, because of the non-coherency of the structure, the positions of atoms close to the interface are not well defined, so that when the period decreases there is a slight expansion perpendicular to the interfaces, while the elastic constant

c_{44} undergoes a drastic reduction. This model therefore gives a physical foundation to the assumption of a modified interface layer, used to reproduce the experimental points of c_{44} (figure 11); the elastic anomalies are due to *intrinsic* interface effects and not to a simple modification of the bulk elastic response, due to dimensional changes occurring in the layered structure. As a consequence, the elastic anomaly is predicted to become more and more intense for superlattices with a large lattice mismatch and semicoherent interfaces, such as the Ag/Ni system. We point out that the presence of atomic disorder at the interfaces is consistent not only with TEM evidence of semicoherent interfaces, but also with the increase of the parameter σ/d in x-ray spectra and with the noticeable enhancement of the electrical resistivity at low periods, because of electron scattering at the interfaces [33]. As a final remark, we note that the elastic response at large periods approaches that expected for an ideal superlattice, although TEM experiments give evidence of a growing density of planar defects with increasing superlattice period; this suggests that the influence of stacking faults, twins, etc on the elastic response is negligible, with respect to the influence of the interface effects.

In conclusion, the connections between the structural and elastic properties of Ag/Ni superlattices have been investigated, in order to give evidence for the role of interface effects in determining their physical characteristics. It has been found that the elastic softening measured by Brillouin spectroscopy experiments can be explained by assuming the existence of a modified interface layer, whose elastic constants are substantially reduced with respect to those of the constitutive materials. The physical mechanism responsible for this softening is the presence of atomic disorder at the interfaces, which are semicoherent even at very low periods, due to the high lattice mismatch existing between Ag and Ni.

Acknowledgments

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