NANOCONTACT MEASUREMENTS IN SUPERLATTICES

T. BAUMANN, J. B. PETHICA*
Brown Boveri Research Center, 5405 Baden, Switzerland

M. GRIMSDITCH AND IVAN K. SCHULLER
Materials Science Division, Argonne National Laboratory,** Argonne, IL 60439

ABSTRACT

Many metallic superlattices are known to exhibit dramatic anomalous elastic properties as a function of modulation wavelength. All measurements to date measure either a shear modulus or a longitudinal modulus in the superlattice plane. Here we present a method which also probes longitudinal elastic behavior perpendicular to the layers using a nano-hardness tester. In this method, a diamond tip is indented into the material and both the indentation depth and applied force are constantly monitored during loading and unloading. The elastic properties are extracted from the unloading part of the force versus displacement curve. Contrary to the anomaly found in a shear modulus, no anomaly in Young's modulus was found in the present study on Mo/Ni superlattices.

INTRODUCTION

Over the past few years the mechanical properties of superlattices have been investigated by both mechanical and light scattering techniques. In most of these investigations the elastic properties, contrary to theoretical expectations, depend on the superlattice modulation wavelength (Λ) [1]. The elastic properties that are generally measured are either shear moduli, or longitudinal moduli _in_ the plane of the film, but so far there have been no elastic moduli measurements related to a direction along the superlattice normal. The reason for this lack of information on these systems is their size: superlattices are usually grown by evaporation, sputtering, or Molecular Beam Epitaxy (MBE) techniques and hence the thicknesses which can typically be attained are 1 micron or less. It has therefore been necessary to resort to unconventional techniques [1] to determine their elastic properties but none of these techniques (e.g., bulge tester, Brillouin scattering, vibrating reed) has yielded information of a property perpendicular to the layers.

EXPERIMENT

Here we report our results using a "nano-indenter" (which will be briefly described) on Mo/Ni superlattices. We have chosen to study this system since it has been studied using Brillouin scattering [2] and shows a substantial softening (~30%) in a shear elastic modulus as the modulation wavelength is reduced from ~50 nm to ~2 nm. The samples have also been characterized using x-rays [2] which show that the average lattice constant expands when Λ is reduced. This lattice expansion has been used to explain the elastic anomaly by performing a molecular dynamics calculation [3]. A simpler but far less rigorous approach, based on Murnaghan's equation of

Mat. Res. Soc. Symp. Proc. Vol. 77. © 1987 Materials Research Society

state, has recently been used to relate the elastic anomalies observed by Brillouin scattering in Mo/Ni, Nb/Cu and Au/Cr to the observed changes in lattice spacings [4,5].

Detailed descriptions of the "nano-hardness tester" or "nano-indenter" instrument are given in Refs. [6,7,8]. It consists of a diamond tip in the form of a pyramid on which known variable forces can be applied. The position of the tip is also monitored very accurately so that when the tip is brought into contact with a surface a force versus displacement curve can be obtained. Figure 1 shows such a plot obtained from a Ni film; the three sets of points correspond to three runs in which the tip penetrated 150, 300 and 450 nm into the material. The slowly rising portions correspond to the loading portion of the cycle in which the load force increases as the contact area between tip and sample increases. The steep descending portions are the unloading portions of the cycle; it is from the slope of the initial linear portion of the steep unloading regions (where the contact area is assumed to remain constant) that information on the elastic properties is obtained, e.g., a perfectly vertical decrease would imply zero elasticity and a retracing of the loading curve would imply perfect elastic behavior. Since the initial elastic unloading is linear, the contact area initially remains constant. By extrapolating this line to zero load, the purely plastic indent depth is found, and knowing the indenter shape, the radius a of a contact area at full load can be calculated. For the initial punch-like unloading then, Young's modulus E can be calculated from

$$\frac{\partial P}{\partial h} = \text{slope} = \frac{2Ea}{1-\nu^2} , \tag{1}$$

where ν is Poisson's ratio (see [6-9] for details). Elastic compression of the indenter (which is a small effect) is not allowed for here, but this does not affect the conclusion of this paper.

Thus, for an isotropic material the combination of elastic constants that is determined in such an experiment is expected to be closely related to Young's modulus (E). In our case, since the Mo/Ni superlattices have cylindrical (≡ hexagonal) symmetry, the combination of C_{ij}'s being measured may be more complicated. We shall not deal with this aspect here and refer to the measured elastic modulus as E. Figure 2 shows the measured values of E (dots) for a number Mo/Ni superlattices. Since the value of E does depend slightly on the depth to which the tip is introduced into the material, the results presented are normalized to a penetration of 200 nm. The results presented in Fig. 2 are for samples deposited on silicon. Measurements on films deposited on mica substrates gave highly variable, and generally too low, values for the modulus; this was probably due to defoliation of the mica, the resulting gaps between the layers giving anomalously low contributions to the apparent compressibility of the film on the surface. No such problems were seen for silicon substrates. In Fig. 2 we also present the measured hardness as determined from the plastic region of plots similar to those in Fig. 1 (triangles) (see [6]). The error bars are based on the reproducibility of the results. The hardness of our Mo/Ni samples agrees with that of a sputtered Ni film but is about 3 times larger than the hardness measured on a polycrystalline Ni sample. This difference between the two Ni samples can be understood on the basis of the effective "work hardening" of the sputtered film.

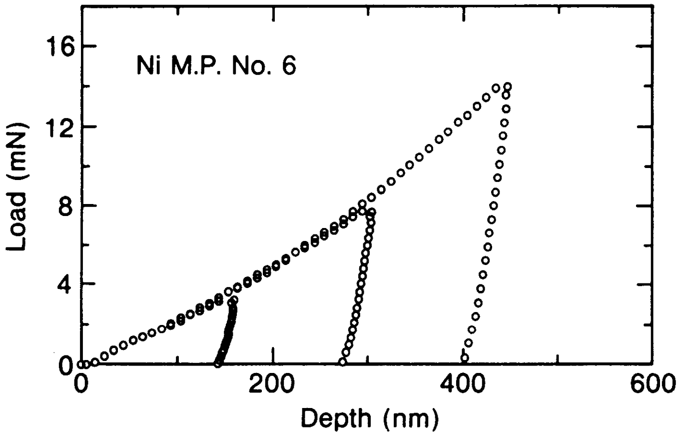

Fig. 1. Loading and unloading curves for a Ni film using the nanoindenter

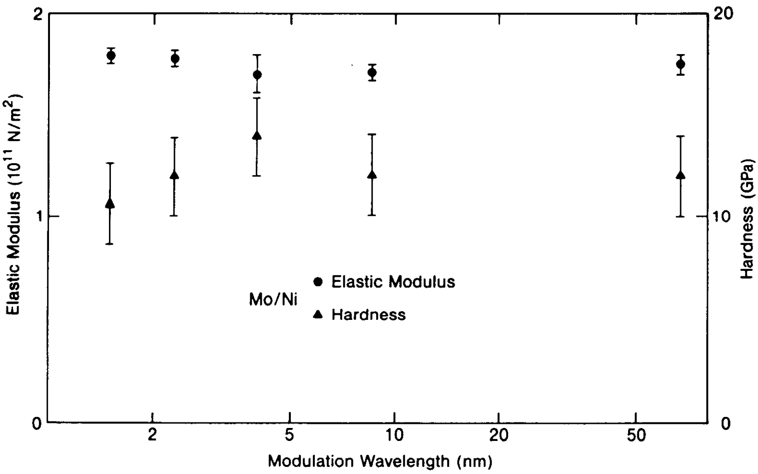

Fig. 2. Elastic modulus (dots) and Hardness (triangles) for Mo/Ni superlattices.

DISCUSSION

Within experimental accuracy, there is no effect of the superlattice modulation on either the modulus E or the hardness. This is a rather surprising result when compared with the ~30% change observed in a shear modulus [2] and the expectations, based on Murnaghan's equation of state, that the lattice expansion softens all elastic constants in a similar fashion [4,5]. At this point it is hard to determine conclusively the reason for the present results.

(i) If the moduli and hardness perpendicular to the layers really do not change the simple explanation based on Murnaghan's equation of state [4,5] to explain the Brillouin results will have to be abandoned. However, the success of this simple theory to explain all measurements to date and the fact that it favorably compares to the more complicated molecular dynamics calculations implies that it's breakdown is unlikely.

(ii) It is possible that the particular combination of elastic constants entering into our measured E change in such a manner that their contributions cancel (this seems an unlikely possibility).

(iii) The most likely explanation is that the uniformity of E and of the hardness might be due to the intermixing of the superlattice layers produced by the plastic deformation. In this case for each modulation wavelength the material "pushing" on the tip will be both Ni and Mo, or an alloy so that it averages out to roughly the same value in each case. Alternatively, the plastic deformation changes the structure and modulus of the material beneath the tip. However, since the contribution to the elastic strain comes from a region larger than the plastically deformed zone, this cannot be the entire explanation. The solution to this problem is to avoid plastic deformation. This is not easy on the nanometer scale, most nominally rounded indenters being sufficiently uneven to give some local plasticity. Very recent work [10] has shown that purely elastic deformation occurs at sufficiently small loads. Further testing of multilayer materials is therefore planned using small loads.

REFERENCES

[*] On leave from Cavendish Laboratory, Madingley Road, Cambridge CB 30H3, England.

[**] Work performed under the auspices of the U.S. Department of Energy, BES-Materials Sciences, under Contract #W-31-109-ENG-38 and the Office of Naval Research contract N00014-83-F-0031.

[1] For a review see, I. K. Schuller, IEEE 1985 Ultrasonics Symposium, edited by B. R. McAvoy (IEEE, New York), p. 1093.

[2] M. R. Khan, C. S. L. Chun, G. P. Felcher, M. Grimsditch, A. Kueny, C. M. Falco and I. K. Schuller, Phys. Rev. B 27, 7186 (1983).

[3] I. K. Schuller and A. Rahman, Phys. Rev. Lett. 50, 1377 (1983).

[4] P. Bisanti, M. Brodsky, G. Felcher, M. Grimsditch and L. R. Sill (in press).

[5] M. Grimsditch (in these proceedings).

[6] J. B. Pethica, R. Hutchings and W. C. Oliver, Phil. Mag. A 48, 593 (1983).

[7] W. C. Oliver, MRS Bulletin, September/October 1986, p. 15.

[8] M. F. Doerner and W. D. Nix, J. Mat. Research, July/August 1986, p. 601.

[9] J. L. Loubet, J. M. Georges and G. Meille, in "Microindentation Techniques in Materials Science and Engineering', eds. P. B. Blau and B. Lawn, ASTM Philadelphia 1985, pp. 72-89.

[10] W. C. Oliver and J. R. Pettica, to be published and W. C. Oliver paper GP15 this meeting.