FINAL REPORT FOR

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"Mechanical Properties of Interfaces and Superlattices"

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Mechanical Properties of Interfaces and Superlattices

This proposal was dedicated to the study of mechanical properties of interfaces and superlattices. These properties were correlated with growth techniques and extensive structural characterization. The principal goal of this proposal was to ascertain whether structural or electronic effects control the mechanical properties of interfaces and superlattices.

Molecular Beam Epitaxy (MBE) and Sputtering was used to prepare single interfaces and multilayers. *In-situ* structural characterization was acco.nplished mainly using High Energy Electron Diffraction (HEED). Post preparation Microcleavage Transmission Electron Microscopy (MTEM), together with temperature dependent x-ray diffraction was performed to characterize the structure of the sample. The main successes during the last funding period of this proposal includes the simultaneous study of a variety of elastic constants of Cu/Nb, Fe/Cr and W/Ni superlattices together with detailed structural studies, the measurement of the elastic constants of GaAs/AlAs superlattices and the development of a vibrating drum method for measurements of the biaxial modulus of thin films. In addition we have correlated the structural changes found in metallic superlattices with theoretical calculations.

I RESEARCH UNDER PREVIOUS FUNDING PERIOD

The research performed during the last period of this proposal was based on a variety of developments accomplished during the first funding period of this proposal. The main emphasis was to study the relationship between elastic properties and structure.[1] This was accomplished by a simultaneous study of a variety of elastic constants together with detailed structural measurements, performed on the same samples. These measurements were extended to a variety of metal/metal and semiconductor/semiconductor superlattices. From the technical point of view the vibrating membrane method, we developed in the last period, was perfected and applied to a variety of systems of interest.

a. Technical Developments

We have finished testing and construction of the vibrating membrane elastometer which allows reliable measurements of the biaxial modulus of thin films.[2] To measure the elastic properties of the thin film, this is stretched over a circular knife-edge support to form a drum head geometry. By exciting the film with a coil and detecting the induced vibrations with an electrode the normal modes of vibration of the drum can be measured with high accuracy. The film's tension is obtained from the frequency of its membrane modes, and its strain by using an optical technique.[3] As a check for the correctness of this method a number of films composed of different elements (Ta, Ni, Cu, Nb and Ag) prepared by a variety of techniques (rolling, electrodeposition, sputtering and evaporation) were tested. For instance, Fig. 1 shows the strain versus the square of the membrane resonant frequency (υ^2), which is proportional to the tension for Nb. The slope of the curve gives directly the biaxial modulus. The results obtained from these measurements are within ~5% of known bulk values,[4] and thus represent the most reliable method for measurement of biaxial moduli in thin films.

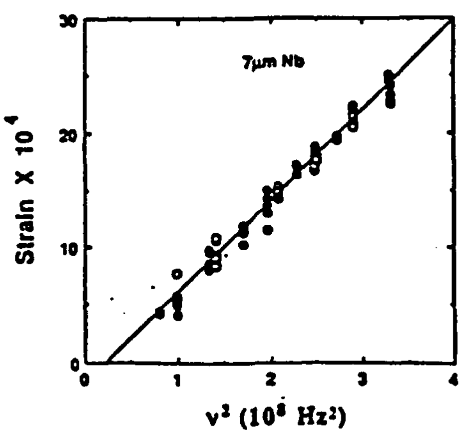

Fig. 1 Strain vs υ^2 of a sputtered [110]-oriented niobium film. Tension is reduced (increased) for filled (unfilled) circles. For filled circles Δ (strain)/$\Delta\ \upsilon^2 = 8.135 \times 10^4\ s^2$ and $Y_B = 0.164$ TPa.

One important component in these type of measurements is understanding the limitations of the measurement method. We have performed a combined theoretical experimental study in order to understand the two limits for modeling vibrating thin films; as a plate or as a membrane.[5] We have identified the sources of experimental uncertainties, developed the mathematical formalism for treating films as vibrating membranes and presented methods for checking the self-consistency of the results. One interesting basic question was to identify the mechanism that generates the vibration in the films in our experimental apparatus. The theoretical calculations and experimental results imply that the drive mechanism for the vibrations arises from eddy currents produced by the drive coil. The frequency dependence and static magnetic field dependence is in agreement with theoretical ideas. For instance, Fig. 2 shows that the static field dependence of the vibration frequency of an Al film is linear, in accordance with theoretical expectations.

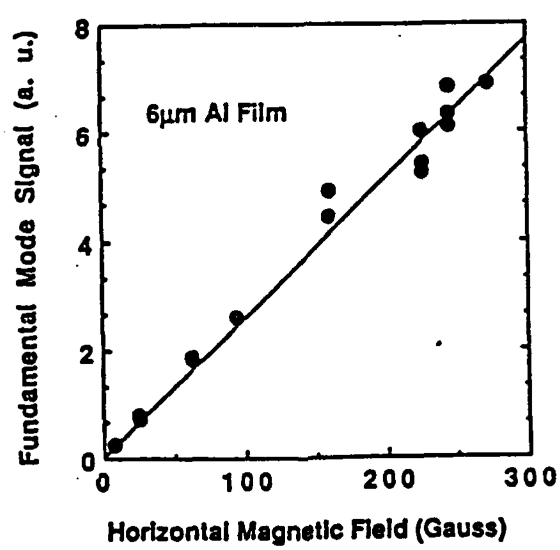

Fig. 2 Signal amplitude vs horizontal B field intensity for a 6 μm aluminum film

b. Correlation Between Structure and Elastic Constants

One of the main aims of this work has been to ascertain the connection between the microscopic structure and elastic constants. In many superlattice systems it is found that the microscopic structure changes considerably with decreasing layer thickness. Although in some superlattices correlations were found between particular lattice parameters and some elastic constants (such as the shear modulus) the exact mechanism for this connection was not clarified. The main reasons for this was that a complete crystallographic study[6] was never attempted together with the simultaneous determination of a variety of elastic constants. We have addressed this issue in a variety of ways, either by studying the detailed structure and several elastic moduli in the same set of Nb/Cu films or by studying the moduli of a system (Fe/Cu) which undergoes a structural phase transition.

A study of the mechanical properties of Cu/Nb superlattices shows that for modulation wavelengths below ~ 50 Å the biaxial modulus increases by ~15%, the shear modulus decreases by ~30% and the flexural modulus does not change.[7] All these are correlated with changes in the average lattice parameters as shown in figure 3.

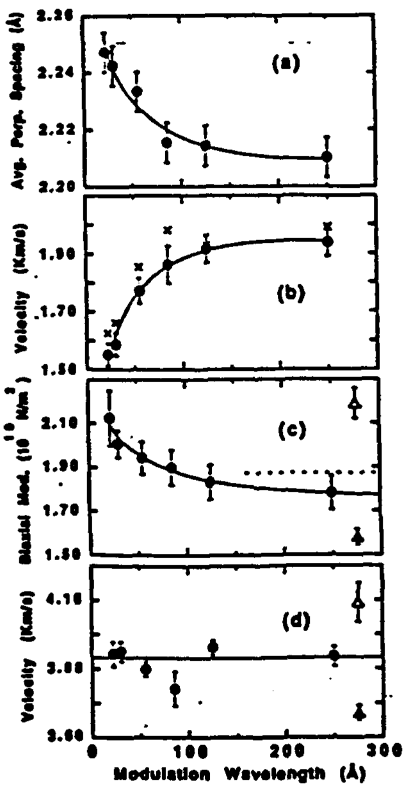

Fig. 3 Structural and elastic properties of Cu/Nb superlattices vs. modulation wavelength; (a) average perpendicular lattice spacing, (b) surface wave velocities in supported (crosses) and unsupported (full circles) superlattices, (c) biaxial modulus and (d) symmetric Lamb wave velocity. The solid lines are guides to the eye. The filled (unfilled) triangles represent the values for pure niobium (copper) films.

These observations show that anomalies with different signs of the elastic moduli can coexist in the same materials. Measurements performed on both supported and unsupported films show that the anomalous effects are not induced by the presence of the substrate. Quite interestingly a considerable increase is found in the hardness of the superlattice almost 50% above the value of either Cu or Nb as shown in figure 4. This shows that although no major effects are found in the elastic moduli, the hardness can be considerably enhanced and manipulated in an advantageous fashion.

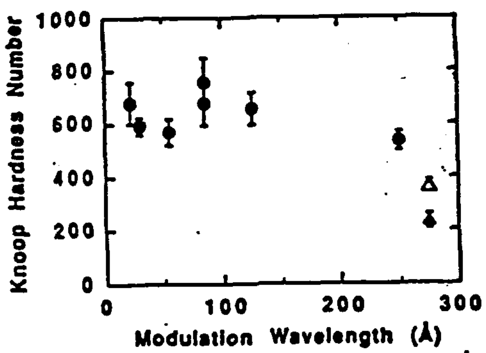

Fig. 4. Knoop microhardness vs modulation wavelength for Cu/Nb.

Fe/Cu superlattices offer a unique opportunity for studies connecting structure and mechanical properties. The reason for this is that Fe undergoes a structural phase transition as a function of layer thickness, in which bcc α-Fe transforms into fcc γ-Fe.[8-10] This structural phase transition in which the Fe layers become coherent with the Cu layers is signaled by clear cut changes in the elastic and magnetic properties. As an example figure 5 shows the surface phonon velocity on Fe/Cu superlattices. At large thicknesses, there is agreement between calculated (indicated by the arrows) and measured velocities. The calculated values are obtained using the bulk elastic constants of the constituents. As the superlattice wavelength decreases, the phonon wave velocity initially decreases. The most striking results are obtained from samples in which the Cu layer is much thicker than the Fe layer. In this case a minimum develops which coincides with the α-Fe to γ-Fe structural phase transition. This is also the point at which the Fe and Cu lattices become coherent in the plane of the film. Clearly structural changes affect strongly the elastic properties.

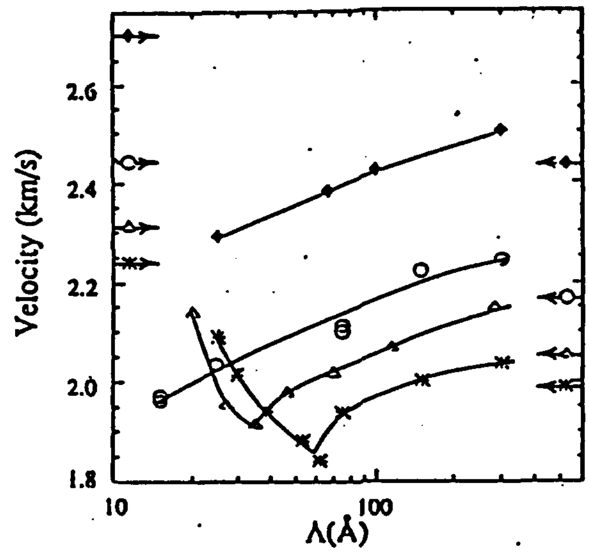

Fig. 5. Surface phonon velocity vs L for different ratios of t_{Fe}: t_{Cu}: ◆ 3:1, O 1:1, Δ 1:2 and * 1:3.

A superlattice of much applied importance is the semiconductor GaAs/AlAs.[11] Many of the transport properties of these superlattices have been studied for a large number of years, however to the best of our knowledge their elastic constants have not yet been determined.[11] We have performed a detailed study of the shear elastic moduli of GaAs/AlAs superlattices. These superlattices do not exhibit any major structural changes as a function of layer thickness and the shear elastic constant doesn't change much either. Figure 6 shows the various elastic constants determined in a large number of superlattices together with calculations based on the bulk moduli, also determined by us. Clearly, in these superlattices there is a good quantitative agreement between the experimental and calculated values without any unusual changes as a function of modulation wavelength.

In a series of detailed structural and elastic constant studies we have compared the shear elastic constant and the structure of W/Ni superlattices. Structural refinement of the reflection x-ray diffraction spectra[12] was used to determine the out-of-plane lattice spacings of the constituent layers. The Ni(111) lattice spacing expands ≈ 3% and the W(110) lattice spacing is constant with decreasing modulation wavelength Λ down to Λ≈30Å. Transmission x-ray diffraction was used to determine the in-plane structure. The W layers undergo an anisotropic

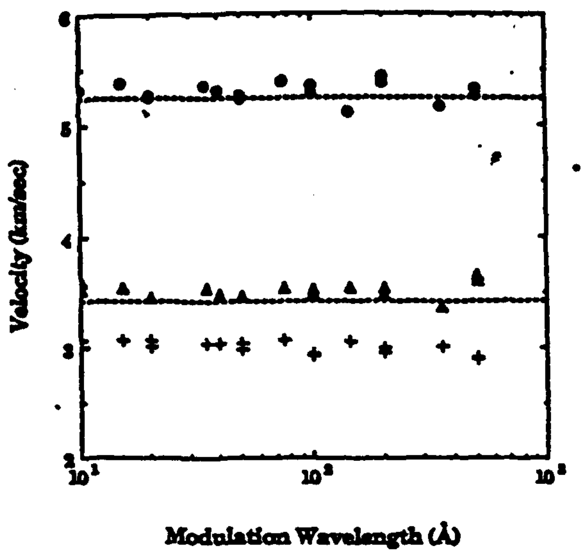

Fig. 6 Sound velocities in GaAs/AlAs superlattices as a function of modulation wavelength. The dots, triangles, and crosses correspond to longitudinal, transverse, and surface waves, respectively. The dashed lines are longitudinal and transverse sound velocities determined from the calculated elastic constants of the superlattices.

contraction in-plane with the [002] directions contracting ≈ 2% and the [1$\bar{1}$0] remaining constant with decreasing Λ. The Ni [2$\bar{2}$0] expands ≈ 1.5% with decreasing Λ. The Ni layer expands both in-plane and out-of-plane contradicting Poisson ratio arguments relating in-plane and out-of-plane strains. Below Λ ≈ 35 Å, the multilayers undergo a structural transition, in which both layers transform into a random close packed structure. The shear velocity decreases ≈ 22% with decreasing Λ down to the disorder transition and then is Λ independent. The results imply that there is a correlation between the origin of the elastic anomalies and amorphization.

To summarize all the studies done in the field, we have shown that the experimental fact that measured elastic and structural properties of superlattices are strongly correlated can be understood on the basis of a simple model based on the packing of hard spheres. The model is consistent with features of many models that have been proposed to explain the supermodulus effect, but contrary to previous explanations, it allows predictions for a given pair of constituents to be made. For an arbitrary pair of elements, it predicts the existence or non-existense of an elastic anomaly, and a rough estimate of its magnitude.

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APPENDIX I

Publications under the previous period of this grant

I. Papers Published

1. Biaxial Elastic Modulus of Metallic Films Determined from Vibrating Circular Membranes
   A. Fartash, Ivan K. Schuller, M. Grimsditch
   Appl. Phys. Lett. 55, 2614 (1989).

2. Light Scattering by Surface Acoustic Waves on Corrugated Metal Surfaces
   W.M. Robertson, M. Grimsditch, A.L. Moretti, R.G. Kaufman, G.R. Hulse,
   E. Fullerton and Ivan K. Schuller
   Phys. Rev. B. 41, 4986 (1990).

3. Structural and Elastic Property Changes in Ag/Co Superlattices Induced by
   High Energy Ion Irradiation
   E. Fullerton, Ivan K. Schuller, R. Bhadra, M. Grimsditch and S. Hues
   Mat. Sci. Engr. A-126, 19 (1990).

4. Elastic Properties of GaAl/AlAs Superlattices
   M. Grimsditch, R. Bhadra, Ivan K. Schuller, F. Chambers and G. Devane
   Phys. Rev. B. 42, 2923 (1990).

5. Elastic Anomalies in Superlattices
   Ivan K. Schuller, A. Fartash, and M. Grimsditch
   Mat. Res. Soc. Bull. XV, 33 (1990).

6. Vibrating Membrane Elastometer for Reliable Measurement of Mechanical Properties of
   Metallic Films
   A. Fartash, Ivan K. Schuller and M. Grimsditch
   Rev. Sci. Instrum. 62, 494 (1991).

7. Surface Phonons in Cu/Ni Superlattices
   J. Mattson, R. Bhadra, J. Ketterson, M. Brodsky and M. Grimsditch
   J. Appl. Phys. 67, 2873 (1990).

8. Evidence for the Supermodulus Effect and Enhanced Hardness in Metallic Superlattices
   A. Fartash, Eric E. Fullerton, Ivan K. Schuller, Sarah E. Bobbin, J.W. Wagner,
   R.C. Cammarata, S. Kumar and M. Grimsditch
   Phys. Rev. B. 44, 13 760 (1991).

9. Thin Film Modeling for Mechanical Measurements: Should Membranes be used or Plates?
   A. Fartash, Ivan K. Schuller and M. Grimsditch
   Jour. Appl. Phys. 71, 4244 (1992).

10. Effect of Structure on the Anomalous Mechanical Properties of Metallic Superlattices -
    INVITED PAPER
    Ivan K. Schuller, A. Fartash, Eric E. Fullerton and M. Grimsditch
    Mat. Res. Soc. Symp. Proc. 239, 499 (1992).

11. Connection Between Giant Magnetoresistance and Roughness in Sputtered Fe/Cr Superlattices - INVITED PAPER
David M. Kelly, Eric E. Fullerton, F.T. Parker, J. Guimpel, Y. Bruynseraede, Ivan K. Schuller
International Conference on the Physics of Transition Metals, Darmstadt, Germany, July 20-24, 1992, eds. P.M. Oppeneer, J. Kübler, Vol. I, (World Scientific, 1993), pg. 419.

12. Relationship Between Structural Phase Transitions and Elastic Anomalies in Metallic Superlattices
Eric E. Fullerton, Ivan K. Schuller, F.T. Parker III, Kathryn A. Svinarich, Gary L. Eesley, R. Bhadra, M. Grimsditch
J. Appl. Phys. 73, 7370 (1993).

13. X-ray Diffraction Characterization and Sound Velocity Measurements of W/Ni Multilayers
Eric E. Fullerton, Sudha Kumar, M. Grimsditch, David M. Kelly and Ivan K. Schuller
Phys. Rev. B. 48, 2560 (1993).

II. Book Chapter

1. Elastic and Structural Properties of Superlattices
M. Grimsditch and Ivan K. Schuller
Chapter in "Atomic Level Properties of Interface Materials"
Chapman and Hall, Publishers, ed. S. Yip and D. Wolf, 1990.

III. Papers Submitted (Not yet published)

1. Phenomenological Explanations of Elastic Anomalies in Superlattices
M. Grimsditch, Eric E. Fullerton and Ivan K. Schuller
Presented at 1993 Spring Meeting of the Materials Research Society
San Francisco, CA
April 12-16, 1993.

III. Invited Presentations at Topical or Scientific Technical Society Conferences

1. Metallic Superlattices: Structural and Elastic Properties
M. Grimsditch and Ivan K. Schuller
6th Latin American Symposium of Surface Physics (SLAFS-6)
Cusco, Peru
September, 1990.

IV. Contributed Talks

1. Kinetics of Supported Small Metallic Clusters
Sun M. Paik, Sihong Kim and Ivan K. Schuller
March 1992 Meeting of The American Physical Society
Indianapolis, Indiana
March 16-20, 1992.

APPENDIX II

Students and Postdoctoral Collaborators Supported Under this Grant

E.E. Fullerton was a graduate student who received his Ph.D. during in 1992 and was partially supported by this grant. D. Kelly is a graduate student who will finish his Ph.D. during the coming year. J. Guimpel and S.M. Paik were postdoctoral fellows supported by this grant.