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19. ABSTRACT (Continue on reverse if necessary and identify by block number) Raman and Brillouin scattering, infrared Fourier transform absorption spectroscopy and scanning tunneling electron microscopy have all been used in this program to investigate the elastic and structural properties of Langmuir-Blodgett (LB) films, a class of materials which hold considerable promise for many applications in electronics and optics. <i>next page</i>					
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18. (continued)
anomalous melting, Brillouin scattering, film elastic properties, molecular reorientation.

19. (continued)

→ Many new measurements and discoveries have been made. To summarize:

(1) Two-step melting of an LB film was observed for the first time by infrared spectroscopy. The onset of strong disorder in the hydrocarbon tail was found at temperatures lower than those at which the headgroup lattice melts.

(2) Thermal and UV polymerization of LB films was followed spectroscopically and found to lead to equivalent structures. Conditions for polymerization without lattice distortion were found.

(3) Scanning tunneling microscopy was used to image LB molecules on the surface for the first time.

(4) The elastic properties of a cadmium arachidate LB film were measured for the first time. Namely $c_{44} < 4.0 \times 10^8 \text{ N/m}^2$, $c_{66} < 4.5 \times 10^9 \text{ N/m}^2$, $c_{11} \approx 1.1 \times 10^{10} \text{ N/m}^2$, $c_{33} \approx 2.1 \times 10^{10} \text{ N/m}^2$, $c_{13} \approx 1.0 \times 10^{10} \text{ N/m}^2$, and $c_{12} > 3 \times 10^{10} \text{ N/m}^2$. The shear elastic constant c_{44} is quite small for a solid film. There is evidence for molecular orientation in the depolarized spectra of the CdA films. Strong similarities in acoustic properties between these LB films and smectic B liquid crystals were found.

Final Report

1. INTRODUCTION

It is only in the last few years that the potential of Langmuir-Blodgett films for applications in electronics, electrooptics, energy conversion etc. has been demonstrated. Although optimal configurations or materials have not been achieved, effects and phenomena have been observed to prove feasibility. Their uniqueness arises from the facts (1), that the films can be fabricated one monolayer (20-40Å thick) at a time with very simple and reproducible techniques¹, and (2), that their properties can be engineered by adding functional side-groups at various positions along the back-bone chain². In fact it has been projected that molecular electronics, that is electronics based on devices of molecular dimensions, may be a reality by the year 2020². In fact, because of their potential for molecular engineering, the list of potential and demonstrated applications seems to be endless, limited only by the ingenuity of researchers in the field.

The most common type of LB films are composed of long chain molecules of the form $(CH_2)_n$, to which are attached the various molecular subgroups that allow the properties of the film to be designed for specific applications. Crosslinking with chemical bonds introduces increased structural strength. These films, fabricated one monolayer at a time by the Langmuir-Blodgett technique¹, have hydrophilic head groups attached to hydrophobic tails and they form structures with well-defined orientations when compressed together on a water surface. As a properly prepared substrate is successively dipped through the LB molecule-loaded water surface, successive monolayers are deposited to produce oriented films 1 - 1,000's of layers thick. This special fabrication technique should (and does) result in films with unique structural and elastic properties, i.e. they should be two-dimensional in character with weak inter-plane coupling and strong inter-molecular coupling inside a given plane.

As pointed out by Roberts² in his recent review article, there is a scarcity of information about the structural and elastic properties of LB films, despite the fact that for device applications, a material must have acceptable mechanical properties. During the duration of this contract we have made considerable progress in applying



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various techniques to the measurement of the structural and elastic properties of LB films.

The structural characterization work was performed at IBM San Jose. Raman scattering, Fourier transform infrared absorption spectroscopy (FTIR) and scanning tunneling electron microscopy (STM) have all been used to characterize the structure of the molecules in Langmuir-Blodgett films. The first two techniques have been used to determine the average orientation of the molecules, as well as the intramolecule vibration frequencies and force constants. STM provided a picture of the molecules right at a interface. The highlights are that with FTIR it was observed that the monolayer assemblies melt in a two step process³: first the tails become disordered and then at the bulk melting point the head groups melt. Polarized Raman spectra⁴ show that up to close to the bulk melting point there is little evidence for any conversion from the all trans configuration to any gauche bonds configurations. Disordering of the tails must be mainly by chain tilting. Further molecules with unsaturation can now be polymerized with little in-plane expansion⁵ by a mixture of the cis and trans isomers. This is important to prevent peeling or cracking on the substrate. These polymerized films exhibit enhanced stability and interesting structural changes from polymerization. The scanning tunneling microscopy observations⁶ were the first of a lipid bilayer and gave lattice spacings in close agreement with electron diffraction results⁷. This means that we can make organic films and measure their molecular orientation and packing, and most significantly see an image of them. Details of the various measurements are given in section 2.

The intermolecular forces which bond adjacent layers and maintain the individual molecules within each plane determine the mechanical integrity of the film, and are reflected directly in the response of the medium to applied stresses, that is in the elastic constants. Brillouin scattering was used to measure the elastic constants since it does not perturb the film. We have built a state-of-the-art Brillouin spectrometer and have performed the first Brillouin scattering measurements on LB films^{8,9}. The highlights are as follows: we have found very small values for the shear elastic constants c_{44} and c_{66} of cadmium arachidate, the classic LB molecule for which the most information is available. This explains the relative ease with which LB films are removed via the scotch tape test. We have also found that shear stresses applied parallel to the LB layers are strongly coupled to the reorientation of

individual cadmium arachidate molecules. These results have raised fundamental questions about the distinction between solid-phase LB films and liquid-phase smectic B liquid crystal media. In fact, De Gennes in his classic book¹⁰ on "The Physics of Liquid Crystals" speculates on a solid-phase version of smectic B liquid crystals which seems to describe many of the properties of LB films.

2. RESULTS

2.1 Structural Characterization of LB Films

In order to understand phase transitions in L-B film systems, the infrared spectra of L-B films were measured as a function of temperature. Surprisingly, a two step melting process in Langmuir-Blodgett monolayers was observed and for the first time we observed structural changes which occurred during heating, melting and annealing cycling. Prior to the melting point, a pretransitional disordering of the hydrocarbon tails was observed by changes in the spectrum. This was followed by a breakup of the head groups at the lattice melting point. When the temperature was cycled from room temperature to a few degrees below the melting point, the monolayer assembly was recovered. At higher temperature the lattice assumes a new structure and the monolayer assembly structure can not be recovered.

In order to prepare more stable films, the two isomers, octadecyl fumaric acid and octadecyl maleic acid ($C_{18}H_{37}OCOCH=CHCOOH$) were synthesized, and Langmuir-Blodgett films of their cadmium salts were prepared on silver, aluminum and KRS-5 substrates. The orientation of the molecules relative to the substrate surface and the lateral packing of the chains in the unit cell were studied by means of transmission and glancing angle reflection infrared spectroscopy. Polymerizations of these films by both UV radiation or thermal heating were followed spectroscopically as a function of the exposure time. By comparison of these films with films polymerized on a water surface, it was concluded that all three polymerization processes produce films with similar molecular structures. Now one isomer expanded on polymerization and the other contracted. A proper mixture gave a film with no expansion or contraction - an important attribute to prevent peeling or cracking and instability.

As an alternative to transferring a film and then polymerizing it on the substrate, we transferred a monolayer of a polymer, poly(octadecylmethacrylate), directly to a solid substrate. This comb-like polymer was transferred to a solid hydrophilic surface as either a single monolayer in a polar heads down and aliphatic tails up configuration, or as a multilayer structure by Z-type head-to-tail deposition. IR spectroscopy in transmission and reflection at grazing incidence, gave noticeably anisotropic spectra, indicative of an orientation of the methacrylate and hydrocarbon side chains relative to the surface. In particular, the C=O stretching vibration at 1742 cm^{-1} , which is usually the most intense in isotropic films of methacrylate polymers, showed a considerable anisotropic character, indicating that change in the dipole moment of the C=O bond is primarily parallel to the surface. Likewise other bands attributable to backbone and side chain vibrations have been identified and used to determine the orientation of the respective groups relative to the surface providing a molecular picture of the polymer topography. This approach of direct transfer broadens our ability to fabricate stable films and should find considerable utility. We need to identify the appropriate molecular parameters for effective production of molecular monolayers.

An image of a lipid layer was obtained at molecular resolution by scanning tunneling microscopy. This work was in collaboration with A. Bryant, D. Smith and C.F. Quate at Stanford University. This image was a top view image, consisting of a Langmuir-Blodgett film deposited as a double layer of cadmium arachidate on a graphite (001) surface. By rapidly scanning the tip parallel to the film plane and recording the current, a lateral resolution of better than one angstrom was achieved. The images showed the packing of the lipid molecules in the film plane and allowed for a direct verification of the order as well as the nature and distribution of defect sites. The measured molecular spacing of 4.9 angstroms was in agreement with electron diffraction data. This technique is expected to provide valuable information about thermally or light induced order-disorder transitions in ultrathin organic films, as well as about the molecular organization in composite films.

2.2 Measurement of Elastic Properties

Brillouin scattering has proven over the years to be a very powerful tool for measuring the elastic properties of a variety of media¹¹. It is especially applicable to investigating LB films because it is non-contact in nature. We have spent a great deal of time perfecting our Fabry-Perot interferometer. It is a multi-pass tandem device (two Fabry-Perots in series, swept in perfect synchronization) with the light passed back-and-forth through each interferometer a minimum of three times via corner cubes. All alignments etc. are stabilized to within 5 angstroms via capacitive feedback circuits.

We have performed the first experimental investigation of the elastic properties of L-B films. The Brillouin spectra of Langmuir-Blodgett films contain a rich variety of features, most of which we have been able to interpret. We find them to be highly anisotropic, with shear properties reminiscent of smectic B liquid crystal media¹², and we have observed depolarized spectra usually associated with molecular orientational fluctuations in a liquid. Strong coupling between the shear strains that slide the LB monolayers relative to each other and the reorientation of the LB molecules has been observed in some of the depolarized spectra. This result raises some questions concerning the liquid-like properties of these films. In addition, the large anisotropy leads to "bunching" of the guided acoustic waves.

The L-B films used were formed from cadmium salts of the fatty acid, arachidic acid, $\text{CH}_3(\text{CH}_2)_{18}\text{COOH}$, hereafter referred to as CdA. Film samples ranging in thickness from 11 up to 401 layers thick ($27.8 \text{ \AA}$ per layer as measured by x-ray diffraction) were fabricated on both molybdenum and BK-7 glass substrates using a Joyce-Loebel Langmuir trough. Standard substrate cleaning and deposition techniques were used. The first layer was deposited with the hydroxyl group attached to the substrate, and successive layers were deposited with alternating orientations, commonly referred to as "Y" type.

A typical Brillouin spectrum from the CdA samples is shown in Fig. 1. The polarized backscattering spectrum of a molybdenum substrate coated with 75 layers shows three distinctive features arising from at least three guided acoustic modes. The peak corresponding to the smallest frequency shift is due to the Rayleigh wave. The velocity is obtained from the frequency shift and the scattering geometry (which gives κ_p). The variation of the Brillouin frequency shifts versus film thickness is

illustrated in Fig. 2. In two separate experiments the elastic constants c_{33} , c_{44} , and c_{66} were measured, and the remaining constants c_{13} and c_{11} were obtained by a parameterized fit to an acoustical model of the L-B film.

The value for c_{33} was obtained by observing backscattering at near normal incidence from longitudinal phonons in a 301-layer sample coated onto a BK-7 glass substrate. The angle of incidence of the incident light was 14° , and the scattered light was collected normal to the surface. A frequency shift of 23.6 GHz was measured for the longitudinal phonons traveling along a direction 7° from the surface normal. The sound speed of the longitudinal phonons is 3980 m/sec using an index of refraction of $n_0 = 1.525$. We have measured the density of CdA films with Rutherford backscattering to be $1.32 (\pm 0.06) \text{ g/cm}^3$. Therefore $c_{33} \cong 2.1 \times 10^{10} \text{ N/m}^2$.

The values c_{44} and c_{66} were estimated by observing guided Love modes in the L-B film. "s" polarized light at an angle of incidence of 60° was directed onto a 301 layer L-B sample; the "p" polarized scattered light was collected along the surface normal. No guided Love modes were observed as shown in Fig. 3. Since there are no clear features larger than 3 GHz it is possible to place limits on the values of c_{44} and c_{66} . The scattering angle within the film is 146° , therefore

$$c_{66} \sin^2 \theta + c_{44} \cos^2 \theta < 3.6 \times 10^9 \text{ N/m}^2$$

where θ is the direction of the acoustic wave in the film. Here $\theta = 17^\circ$, therefore $c_{44} < 4 \times 10^8 \text{ N/m}^2$ and $c_{66} < 4.5 \times 10^9 \text{ N/m}^2$.

Using the values for c_{33} and c_{44} , the remaining elastic constants c_{11} and c_{13} can be obtained by a parameterized fit to a model of the Rayleigh wave thickness dispersion data. The parameterized model of the Rayleigh wave velocity was constructed by modeling the L-B film as a hexagonal film (symmetry axis normal to the surface) on an isotropic substrate. The calculated Rayleigh wave dispersion curve, as well as dispersion curves for the higher order guided wave modes, are shown in Fig. 4 for comparison with experiment. Note the "bunching" of the guided-wave modes at velocities near the Rayleigh wave velocity of the free substrate. It is a direct consequence of the large difference between the shear and

compressional elastic moduli, and is not obtained for isotropic films. It is scattering from two of these modes that gives rise to the remaining two peaks in Fig. 1.

No depolarized scattering was observed from Love waves; the spectrum is shown in Fig. 3. The long diffuse tails, which appear to be due to Brillouin features centered at or near zero frequency shift, may arise from molecular orientational fluctuations as in molecular liquids. The appearance of these features suggests that the individual long chain L-B molecules are undergoing orientational fluctuations inside the L-B lattice, leading to a large damping of the shear waves polarized parallel to the layers.

These results show that the elastic properties of these L-B films are highly anisotropic. The compressional constants are comparable to those obtained for oriented polymer films.¹⁴ In contrast, the resistance to shear strains is small, as evidenced by the small shear elastic constants, much smaller than those observed in polymers. These properties are similar to results reported previously for smectic B liquid crystals, raising interesting questions about the similarities and differences between L-B films and smectic B liquid crystal media. Similarly DeGennes has identified a solid form for smectic B liquid crystal media that appears to have similar properties to L-B films.¹⁶

In summary, the elastic constants of L-B deposited films of CdA have been measured for the first time by Brillouin spectroscopy: namely $c_{44} < 4.0 \times 10^8 \text{ N/m}^2$, $c_{66} < 4.5 \times 10^9 \text{ N/m}^2$, $c_{11} \cong 1.1 \times 10^{10} \text{ N/m}^2$, $c_{33} \cong 2.1 \times 10^{10} \text{ N/m}^2$, $c_{13} \cong 1.0 \times 10^{10} \text{ N/m}^2$, and $c_{12} > 3 \times 10^{10} \text{ N/m}^2$. The shear elastic constant c_{44} is quite small for a solid film. There is evidence for molecular reorientation in the depolarized spectra of the CdA films.

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Figure Captions

Fig. 1. Typical Brillouin spectrum of 75 layers of CdA coated onto a molybdenum substrate. The scattering geometry is illustrated in the upper right corner.

Fig. 2. Brillouin spectra of CdA film samples on molybdenum. The samples range in thickness from 11 to 401 monolayers thick.

Fig. 3. Depolarized scattering from a CdA sample 301 layers thick. No phonon modes were observed larger than 3 GHz.

Fig. 4. Comparison of Brillouin scattering measurements with Rayleigh and Sezawa dispersion curves for a hexagonal film with $c_{33} = 2.1 \times 10^{10} \text{ N/m}^2$, $c_{13} = 1.0 \times 10^{10} \text{ N/m}^2$, $c_{44} = 4.0 \times 10^{10} \text{ N/m}^2$, and $c_{12} = 3.0 \times 10^{10} \text{ N/m}^2$ deposited on a polycrystalline molybdenum substrate.

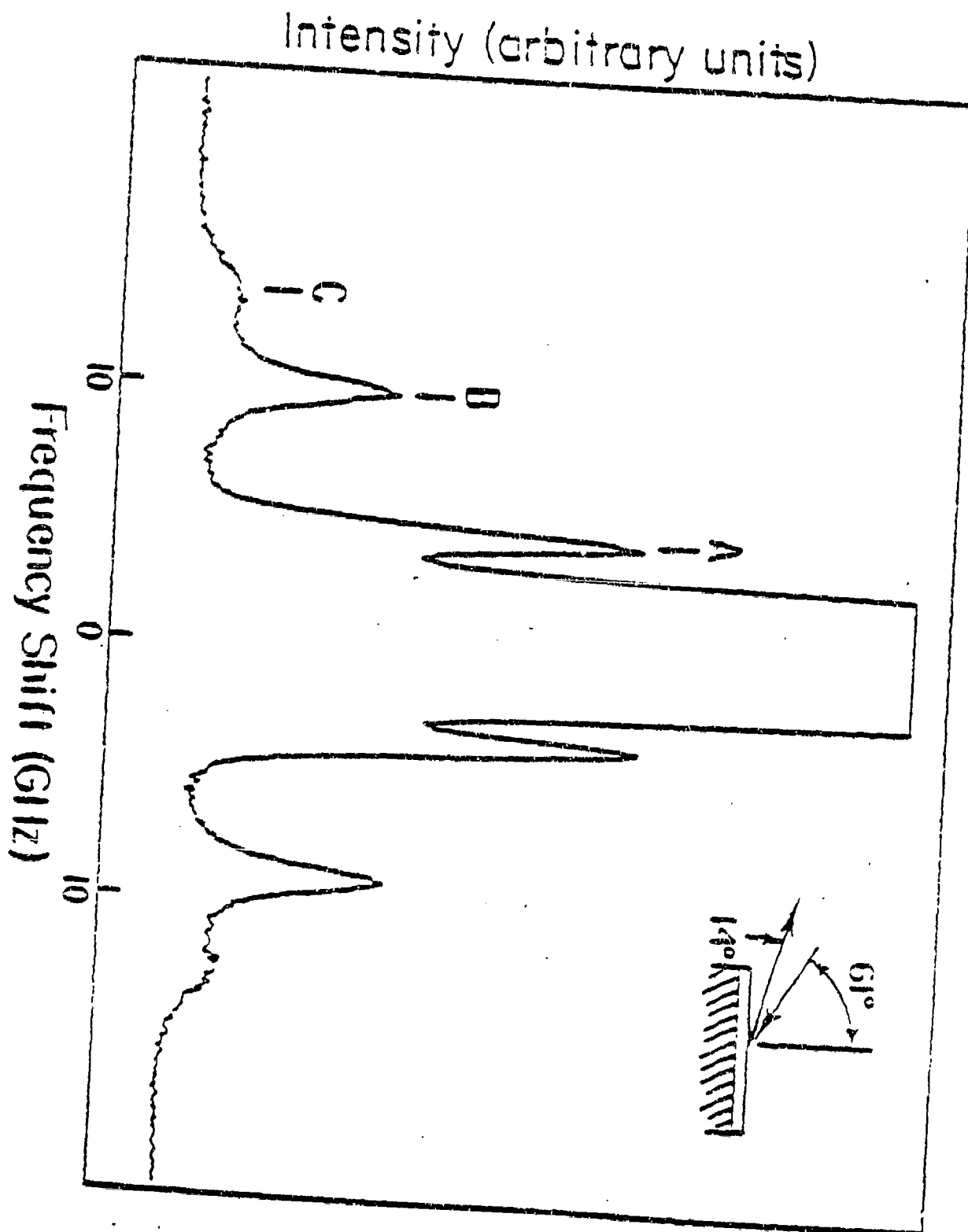


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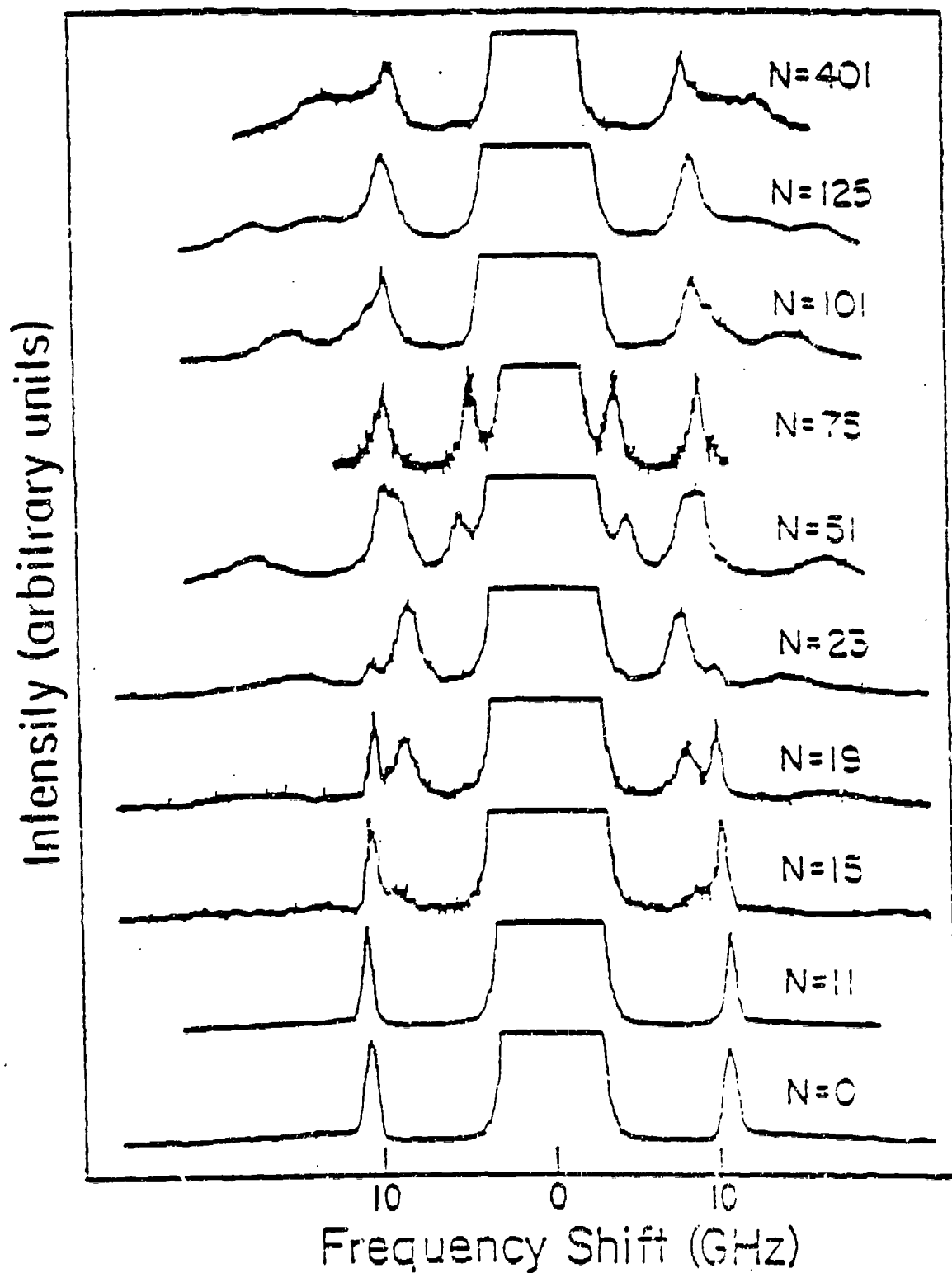


Fig. 2. Brillouin spectra of CdA film samples on molybdenum. The samples

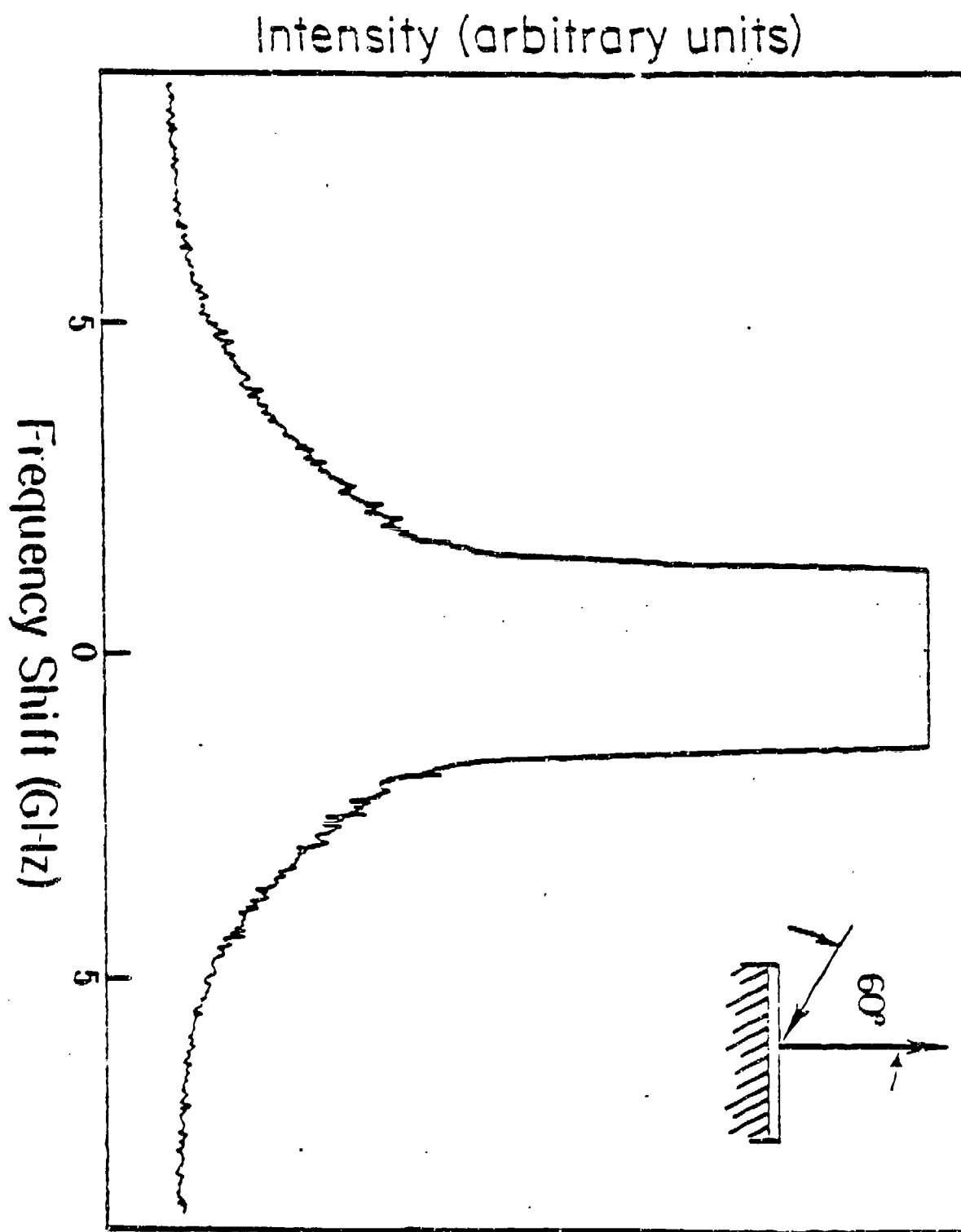


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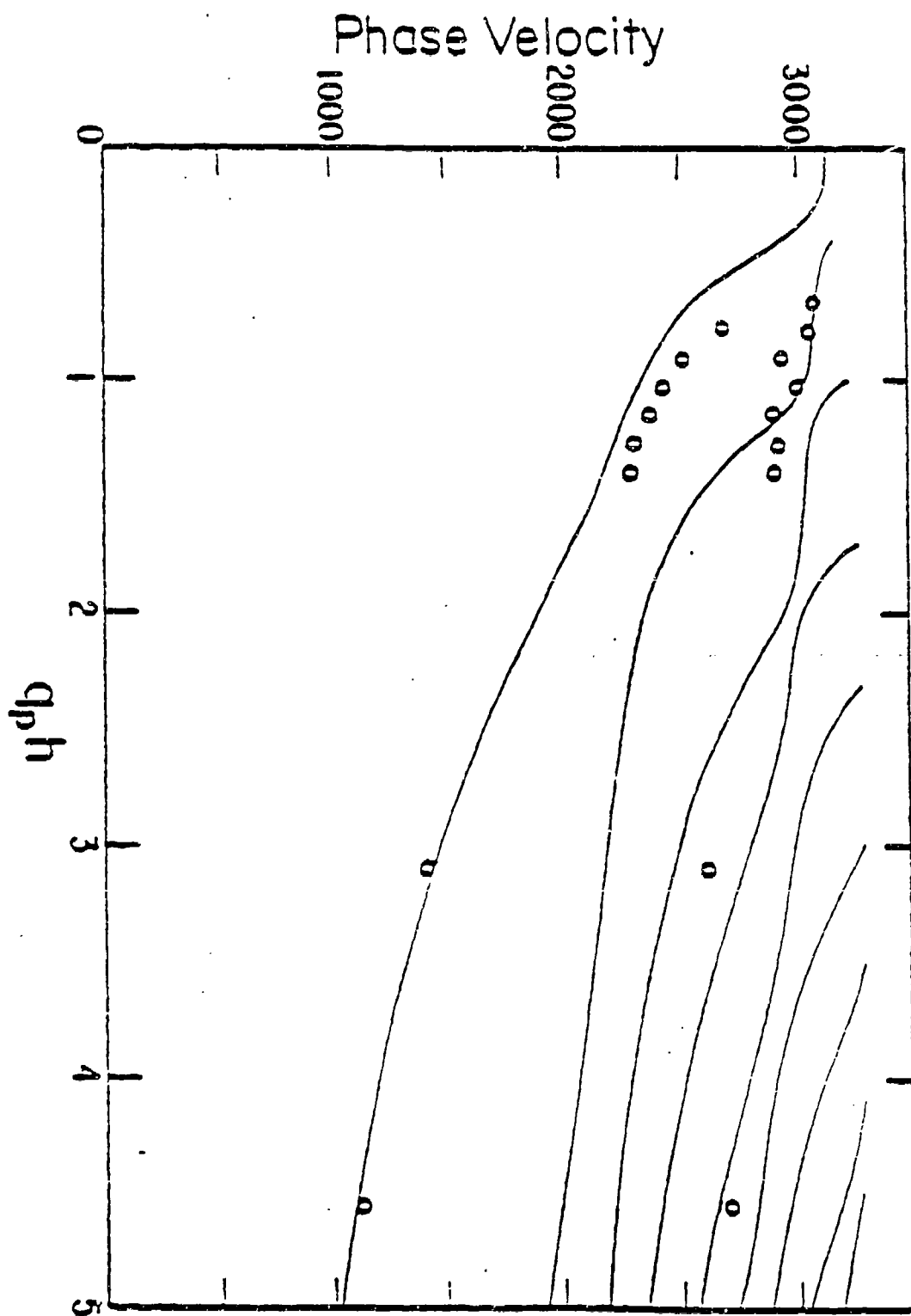


Fig. 4. Comparison of Brillouin scattering measurements with Rayleigh and Sezawa₁₀ dispersion curves for a hexagonal film with $c_{33} = 2.1 \times 10^{10} \text{ N/m}^2$, $c_{13} = 1.0 \times 10^{10} \text{ N/m}^2$, $c_{44} = 4.0 \times 10^{10} \text{ N/m}^2$, and $c_{12} = 3.0 \times 10^{10} \text{ N/m}^2$ deposited on a polycrystalline molybdenum substrate.