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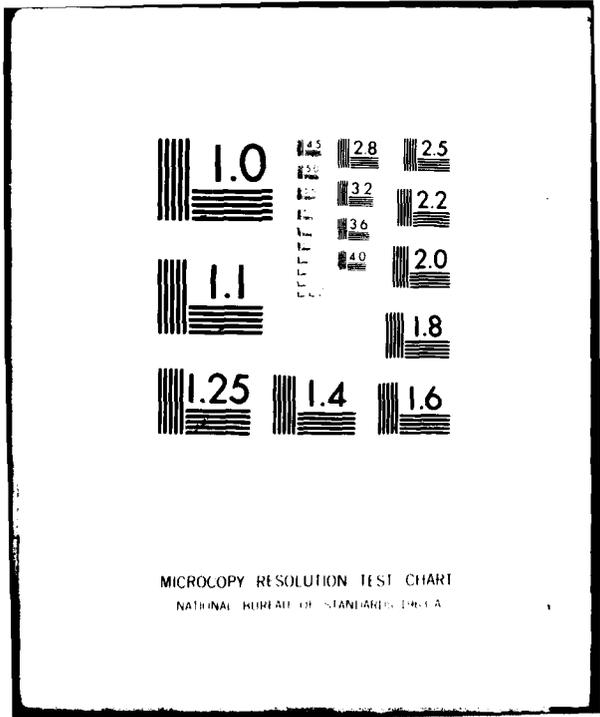
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Brillouin Scattering of Solithane 113 at High Pressure,

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Sept 11, 1980

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Abstract

Brillouin spectra from Solithane 113 have been obtained as a function of pressure at 26°C using a Fabry-Perot Interferometer. The modulus was derived from the sound velocity and was found to double as the pressure increased to 2.67 K bar. In addition the modulus was measured as a function of scattering angle in the range 64° to 161.45° . For both the pressure and the angle-dependent data it was found that the moduli were greater at gigahertz frequencies than at one megahertz, indicating frequency dispersion.

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Solithane 113 is a polyurethane which has been used as a potting compound for suspending solids and isolating them from thermal and mechanical shock originating in the surrounding environment. Questions have been raised as to its ability to resist cracking when the applied stress has high frequency components, in the gigahertz range. These questions have been prompted by recent work done on crack initiation and propagation theory.^{1,2} The theory suggests a possible relation between friability and the frequency spectrum of the elastic modulus of a material. An effort is being made therefore to determine the modulus of Solithane over as broad a frequency range as possible under a variety of experimental conditions. (e.g. vs. temperature and pressure) Here we report initial measurements of the longitudinal modulus, M , by Brillouin scattering at frequencies of ~ 10 GHz. These experiments were made over a range of frequencies and hydrostatic pressures. An increase in frequency in this range by a factor of 2 produced $\sim 20\%$ increase in the longitudinal modulus. An increase in hydrostatic pressure from 0-40000 psi resulted in a linear doubling of the modulus. Lower frequency data of Gupta³ show a similar dependence on pressure, although his moduli at 1 MHz are substantially ($\sim 25\%$) smaller than ours at 10 GHz.

We see therefore that the modulus of Solithane depends significantly on pressure, temperature, and frequency.

The increase of the modulus with frequency and with pressure indicate that the ability of Solithane to insulate included materials from high speed and/or high pressure stress is significantly less than for low frequency, low pressure stress. Brillouin scattering has been used to characterize many polymers.⁴ It is a technique in which light scattered at an angle θ from traveling thermal sound waves in a material is doppler shifted in frequency

by an amount proportional to the sound velocity of the thermal phonon. An incident beam of light with propagation vector $k_o = 2\pi/\lambda_o$ where λ_o is the incident wavelength, interacts with thermal phonons in a material of refractive index n , and is scattered at an angle θ with respect to the incident beam. The phonons which couple with the scattered light have a propagation vector $q = 2\pi/\Lambda$, where Λ is the sound wavelength.

$$q = 2k_o n \sin(\theta/2) = \frac{4\pi n}{\lambda_o} \sin(\theta/2) \quad (1)$$

The doppler frequency shift of the scattered light is f , and is related to the sound velocity v by

$$v = 2\pi f/q \quad (2)$$

Presuming that we are dealing with longitudinal (compressional) sound waves only and assuming that the sound attenuation is small we calculate the real part of the modulus⁵ by

$$M' = \rho v^2 \quad (3)$$

where ρ is the density. The imaginary part of the modulus has the form

$$M'' = \frac{2\rho\Gamma\pi}{q} \quad (4)$$

where Γ is the full width at half maximum height of the Brillouin peaks.

The index of refraction of Solithane as function of pressure is not known. We determined the room pressure index with an Abbe refractometer and extrapolated this value to high pressure with the Clausius-Mossotti relation

$$\rho = c \frac{n^2 - 1}{n^2 + 1} \quad (5)$$

where c is the proportionality constant. n is 1.5123 at room pressure and increases to 1.551 and 40000 psi according to this relationship.

Experimental Details

Solithane 113 pre polymer was manufactured by Thiokol Chemical Corporation and was prepared by California Institute of Technology in the form of transparent sheet with 6 mm thickness.⁶ The sample used in these experiments had a 50/50 resin to catalyst mixture. Solithane was cut by razor blade and polished to a smooth finish with emery cloth. For transparency it was inserted in an optical cell with index matching parafin oil. The cell was made large so that the flare spots due to elastic scattering of the laser beam as it entered and exited the cell were distant from the scattering volume. The container was mounted on a rotating optical holder for accurate angular measurement and excitation was achieved by a single frequency 514.5 nm Argon-ion laser.

The experimental arrangement is shown in figure 1. The optical axis of the sample-apertures-Fabry-Perot interferometer-lenses-detector was indicated by a He-Ne laser beam. Alignment was maintained using this reference and the scattering angles were measured by successively reflecting back on themselves from the sample cell entrance window the He-Ne beam and the Ar⁺ beam. Corrections to this angle were made for the refractive index of the sample and its index matching liquid. The scattering volume was first imaged on a pinhole to reduce stray light. The light was then re-collimated for the Fabry-Perot and detected by an EMI 9658 phototube with photon counting electronics. Data was stored in a multichannel analyzer.

The Fabry Perot was a Burleigh instrument with 2 inch diameter mirrors, scanned by a home built digital ramp and tuned with an automatic stabilizing servo. This maintained a typical finesse of 40.

For the high pressure experiment solithane was cut to fit the cavity of a 60,000 psi Nova cell. The pressure cell was a 4-window cross design which

permitted easy optical alignment. Pressure was generated by a 40,000 psi Enerpack hand pump and was measured by a 40,000 psi Aminco gauge. The cell was filled with silicon oil for pressure transmission and index matching. We found it necessary to use a separator to isolate the silicon fluid from the colored pump oil. The transparent silicon oil 2) minimized light loss, b) provided index matching, c) did not freeze or deteriorate at the highest working pressure, d) had a convenient viscosity and e) protected the Nova cell from water.

In order to calculate ρv^2 from equation 3 it was necessary to determine the pressure dependence of the density of solithane 113. An apparatus employing a piston in a capillary tube was designed for the Nova cell such that compression of the sample in the tube could be determined by telescopically observing the motion of the piston.

Results and Discussion

Brillouin frequency shifts for solithane were measured for 4 different angles: 64° , 90° , 118.4° , and 161.45° at one atmosphere and 26°C . The longitudinal modulus, calculated from equations 2 and 3, changed nonlinearly from 6.78×10^5 psi at 64° to 7.95×10^5 psi at 161.4° . These results are plotted in figure 2.

The longitudinal modulus of solithane as a function of pressure is shown in the upper curve of figure 3. Over the 40000 psi range of the experiment the modulus increased linearly to more than double its room pressure value. Similar linear relationships have been seen for other polymers.⁸ The 1 MHz data of Gupta is shown in the lower curve. The shift of the modulus to higher values at higher frequencies indicates a significant frequency dispersion in this quantity.

In summary, Brillouin scattering is the only method which easily provides measurements at gigahertz frequencies. We have demonstrated the utility of this technique to the study of the moduli of a commercial polyurethane (Solithane 113) as a function of stress rate and pressure. The twofold increase of the longitudinal modulus with pressure and its sharp increase with frequency indicate that the sample becomes more brittle and glassy in this frequency/pressure regime.

Future work will explore the temperature and pressure behavior of Solithane in the gigahertz frequency regime, with more detailed examination of frequency-temperature-pressure interrelationships. The effect of systematic variations in polymer composition and plasticization will also be determined.

Acknowledgements

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We also appreciate permission given by Dr. Y. Gupta to include his data.

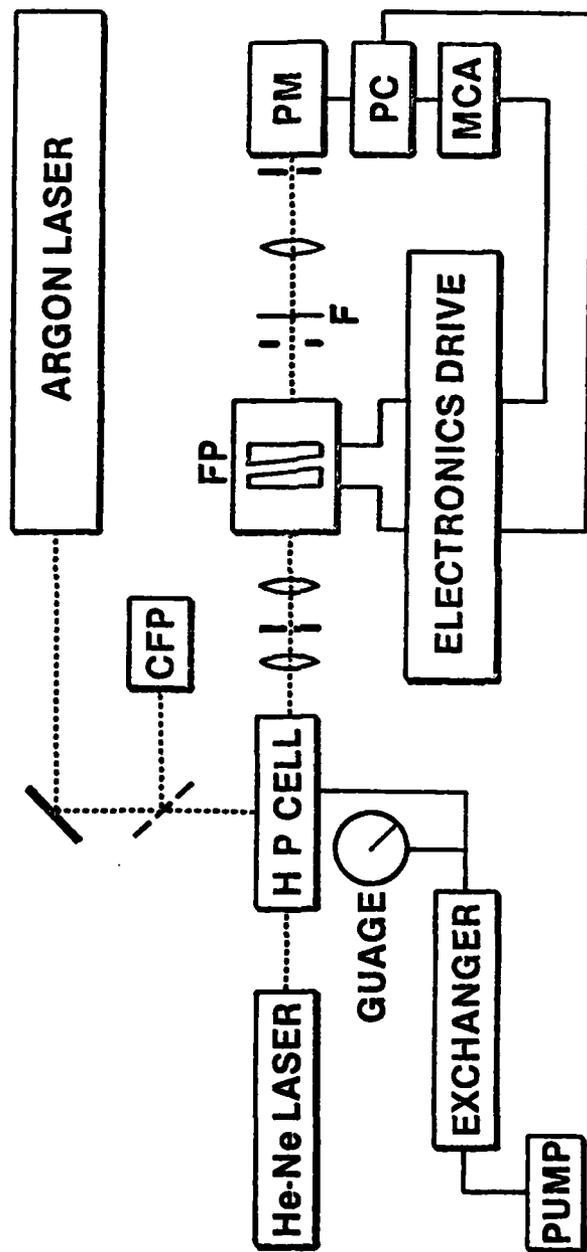


Fig. 1 - Brillouin scattering setup.

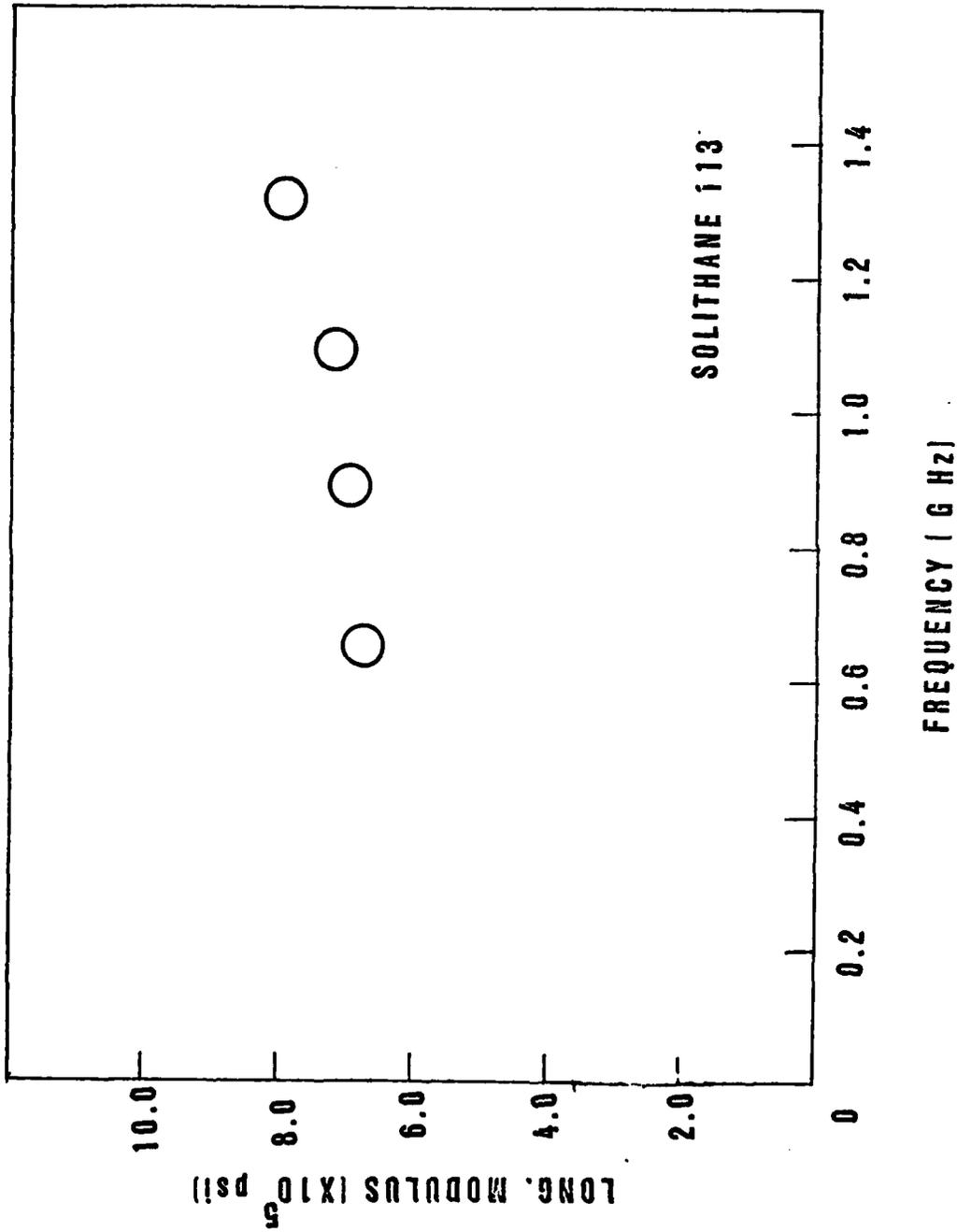


Fig. 2(a) - Longitudinal modulus of Solithane 113 as a function of frequency
the real modulus.

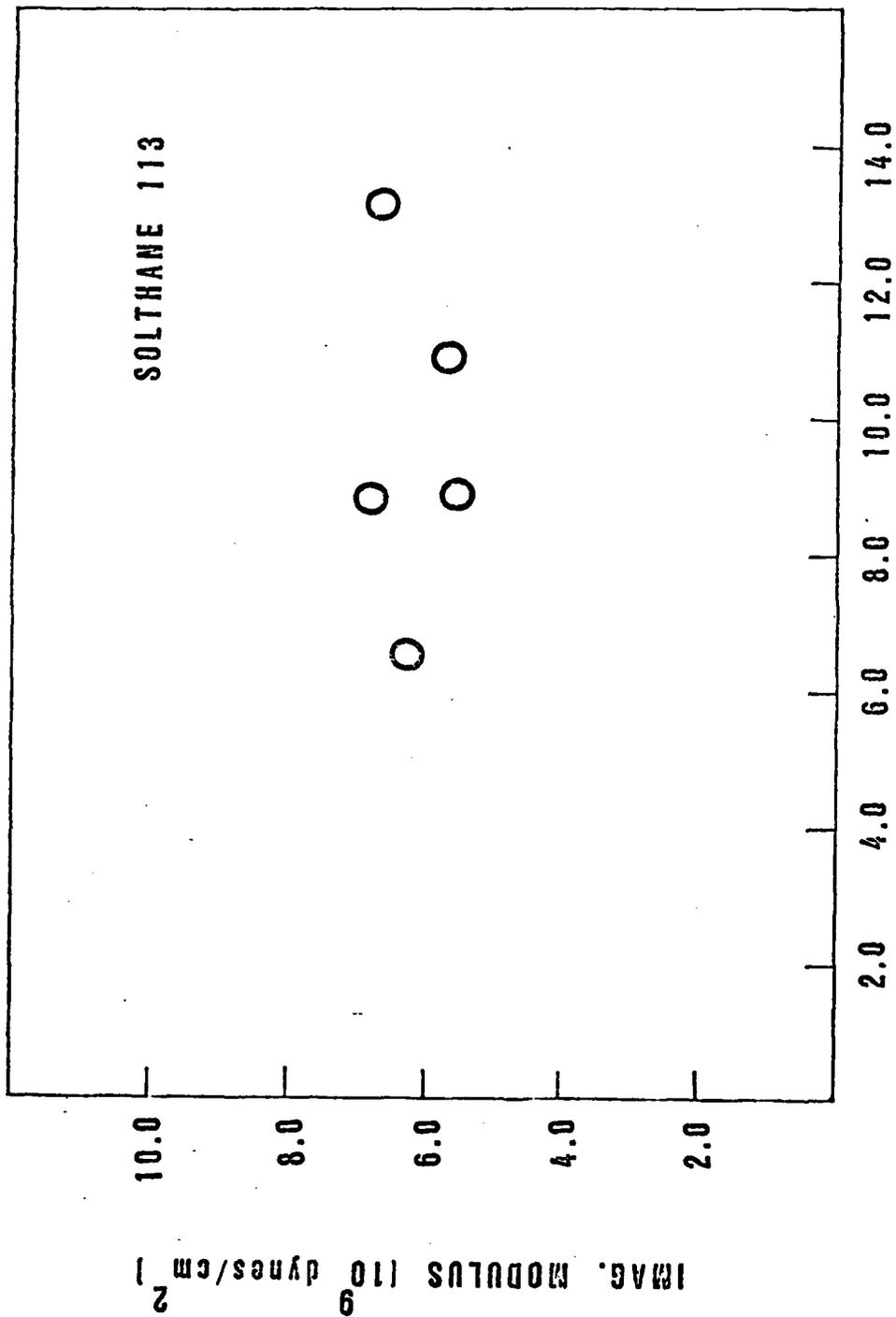
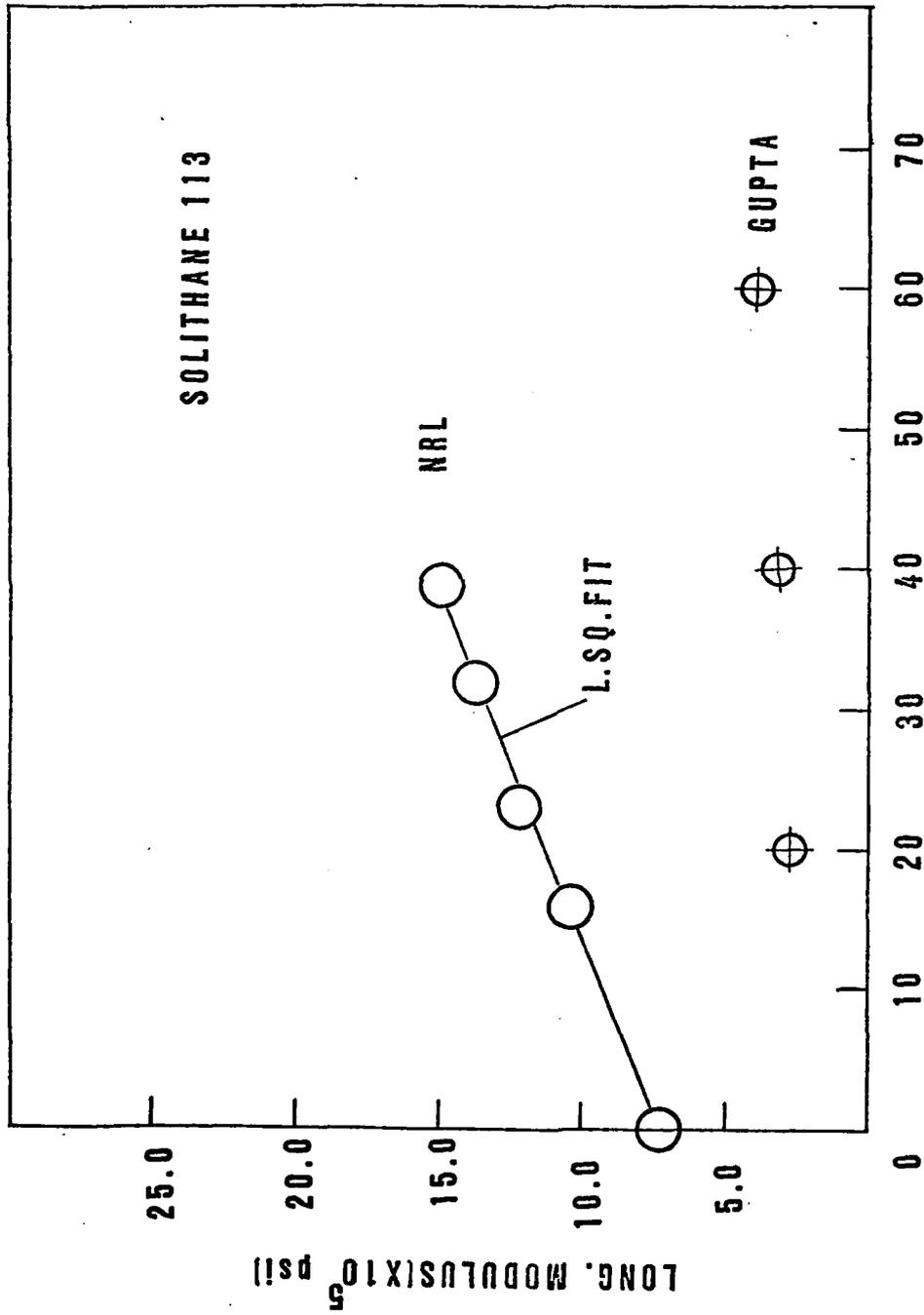


Fig. 2(b) - Longitudinal modulus of Solithane 113 as a function of frequency the imaginary modulus.



PRESSURE ($\times 10^3$ psi)

Fig. 3 - Moduli of Solithane 113 vs pressure. Upper curve:
real long modulus of Gupta at 1 MHz.

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