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ACOUSTIC MICROSCOPY AT CRYOGENIC TEMPERATURES

Annual Summary Report

1 July 1982 - 30 June 1983

Contract No. N00014-77-C-0412

G.L. Report No. 3628

September 1983

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1. The Cryogenic Microscope

In the past year, the cryogenic acoustic microscope has evolved into an imaging instrument capable of producing sub-500 $\stackrel{\circ}{\text{A}}$ resolution images of a variety of objects.

In addition to developing a reliable high resolution (sub-500 Å) imaging capability, we have proceeded on two fronts. First, a promising new thermal imaging technique has been tested which offers hope of sub-surface imaging with the cryogenic microscope. Secondly, we are continuing to raise the operational frequency of the microscope. As a follow-on to this work we are now planning to double the frequency to 8 GHz. The preliminary testing has been done and it now appears that this will be a straightforward matter since the acoustic transducers with our design have proven to be efficient at this frequency.

1. Imaging with 570 Å Acoustic Waves

Early in this reporting period we achieved great success operating the acoustic microscope at 4.2 GHz in superfluid helium at extremely low temperatures $(T \approx .1^{\circ}K)$. This was reported in our Status Report¹ and published in Applied Physics Letters.² The wavelength of sound in the helium for that experiment was 570 Å and the resolution was determined to be better than 500 Å.

Several images of integrated circuits were recorded, as included in Figs. 1 and 2, and the important features of the microscope are now becoming clear. The resolution of the acoustic microscope now exceeds the finest optical microscopes. Some comparison can be made with the scanning electron microscope (SEM). These comparisons have shown that although the SEM presently has a higher lateral resolution, the acoustic instrument has a depth of focus that is much less than that of the SEM. The result is that shallow changes in topography of a surface are more easily seen with the high resolution acoustic microscope than with an SEM.

- 1 -



FIGURE 1

- 2 --



Also, the nature of the SEM requires the object be conducting (or plated with a conductor). The acoustic microscope has no such requirement. The sample, of course, must be placed in a low temperature environment for the acoustic microscope, but this does not damage the specimen. In our dilution refrigerator design with the "top-loader" we can change the specimen in two hours. There is no requirement for warming the entire system to room temperature.

3. The Acoustic Microscope as a Phonon Detector

One advantage of the room temperature acoustic microscope has been the ability to image inside solid objects. However, with the cryogenic acoustic microscope, the contrast at the present operating frequency is from surface topography. The reason is as follows: The transmission of sound from the helium into a sample is governed by the mechanical impedance of the two materials. Because liquid helium has such a low density and sound speed, its impedance is much smaller than virtually all solids, and the reflection coefficient at the heliumsolid interface is practically unity.

There is an anomaly (Kapitza Anomaly) however, in the classical transmission of sound across a liquid helium-solid interface which becomes significant for frequencies near 50 GHz and increases with frequency, until the transmission reaches up to 50%, depending on the solid surface.

The above statements suggest that one way to image inside solid objects would be to use high frequency, high energy sound to penetrate and probe the sample. This possibility and its application to the acoustic microscope is sketched in Fig. 3. High energy thermal phonons, generated by a chrome thin film heater, shown in Fig. 3, can travel ballistically through a crystal (sapphire in this case) at the very low temperatures in consideration (\approx .1°K). The sample is mounted on the sapphire crystal. As the thermal phonons emerge into the liquid, they carry

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FIGURE 3

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with them information from within the sample about the scattering or absorption of some of these phonons. A large fraction of the thermal phonons are transmitted into the helium due to the Kapitza anomaly. Once in the helium, the thermal phonons scatter the coherent acoustic phonons which are used in the normal reflection instrument to form the image. The result is an image which contains information both from the reflection of acoustic phonons as well as the transmission of thermal phonons. The transmission information can be separated from the reflection image by comparing the images with the heater on and off.

Preliminary results of this concept are shown in Fig. 4. The experimental set-up is shown in Fig. 3. The sample was simply the surface of a sapphire crystal. The acoustic pulses and the thermal pulses were both approximately 20 ns. in length. The relative timing of the pulses was varied as the microscope "echo" was measured. The dip marked "L" is the interaction between the acoustic phonons and the "longitudinal" thermal phonons in the 2 mm sapphire slab. The "transverse" thermal phonons travel slower, convert to the longitudinal waves in the liquid at the surface, and cause the second bump in Fig. 4, labeled "T". The sapphire was aligned with the c-axis perpendicular to the surface so that the two transverse phonon modes were degenerate. The heater temperature was approximately 15°K.

Lateral resolution in the transmission technique is determined by the wavelength of the thermal phonons in the solid, the penetration depth of these phonons, and the size of the acoustic probe. A heater temperature of 2°K produces a large number of phonons with a wavelength less than 500 Å in most solids. We have used 2% Helium-3 in Helium-4 for our experimental fluid, and limited the thermal phonon propagation distance in the helium to less than 500 Å. Hence, with an acoustic probe approximately 500 Å across, we expect the transmission resolution to be less than 1000 Å.

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This technique of phonon detection with high spatial resolution may have important applications. The imaging of the variation of the superconducting energy gap in tunnel junctions is one exciting possibility. Imaging fluxons in type II superconductors is another possibility since the superconducting regions are known to be transparent to phonons with energy less than 2Δ , whereas the normal regions (fluxons) are not. Lastly, a spatial study of the Kapitza anomaly would be a new way to study this old and difficult problem.

4. Higher Resolution

A great deal of progress has been made toward raising the operating frequency of the microscope from 4.2 GHz to 8.0 GHz. Non-linear effects in the Helium-3 worsen as the frequency squared, and the noise temperature of the pre-amplifiers also worsens with frequency so that improvements to the system must be made if we are to operate at the higher frequency. Improving our understanding of the thin-film ZnO transducers, low-noise low temperature GasFet pre-amplifiers, as well as chirp systems have contributed to the effort.

We expect 8 GHz operation in the near future since the acoustic transducers have already passed our initial tests.

REFERENCES

- 1. Status Report, 1 July 1982 1 January 1983, G.L. Report No. 3541.
- J. S. Foster and D. Rugar, "High Resolution Acoustic Microscopy in Superfluid Helium," Appl. Phys. Lett. <u>42</u>, 869-871 (15 May, 1983).

FIGURE CAPTIONS

- Fig. 1 (a) Acoustic image of a transistor on a silicon integrated circuit. The aluminum lines are approximately 2 μm wide.
 - (b) Scanning electron micrograph of the same region. Arrows point to corresponding features on the two images. Scale bars are 2 $\mu m.$
- Fig. 2 High magnification image of an aluminum line and base contact of a transistor. Features resembling grain boundaries are visible. The scale bar is 1 um.
- Fig. 3 Illustration of the system for incorporating thermal phonons in the imaging system.
- Fig. 4 The system of Fig. 3 used as a detector of thermal phonons. (See text).

Miscellaneous Activities during Report Period

(1 July 1982 - 30 June 1983)

Publications

- J. E. Heiserman, "Cryogenic Acoustic Microscopy: the Search for Ultrahigh Resolution using Cryogenic Liquids," Physica 109 & 110B, 1978-89, North-Holland Publishing Company (1982).
- J. A. Hildebrand, D. Rugar & C. F. Quate, "Biological Acoustic Microscopy -Living Cells at 37°C and Fixed Cells in Cryogenic Liquids," Proc. Electron Microsc. Soc. Am. <u>40</u>, 174-77 (1982).
- D. Rugar, J. S. Foster & J. Heiserman, "Acoustic Microscopy at Temperatures Less than 0.2°K," in <u>Acoustical Imaging</u>, vol. 12, E. A. Ash and C. R. Hill, eds., 13-25, Plenum Press, New York (1982).
- J. S. Foster and D. Rugar, "High Resolution Acoustic Microscopy in Superfluid Helium," Appl. Phys. Lett. <u>42</u>, 869-71 (15 May 1983).

In Press

 D. Rugar, "Two Applications of Microwave Acoustics in Liquid Helium: High Resolution Microscopy and Direct Measurement of Phonon Dispersion."
 To be published by Springer-Verlag as a volume in their Series in Solid State Sciences.

Meetings and Invited Talks

- C. F. Quate Invited paper, 104th Meeting of the Acoustical Society of America, Orlando, Florida, November 8-12, 1982. Paper entitled "Acoustic Microscopy."
- D. Rugar Invited talk, Santa Clara Chapter of IEEE Sonics and Ultrasonics,
 January 1983. Talk entitled "Ultrahigh Resolution in the Acoustic Microscope."

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- C. F. Quate Invited talk, Golden Gate Metals and Welding Conference, San Francisco, California, February 9-11, 1983. Talk entitled "Microscopy via Acoustics and Photoacoustics."
- C. F. Quate Chairman of Session on "Thermal Waves and Non-Destructive Control". Third International Topical Meeting on Photoacoustic and Photothermal Spectroscopy, Paris, France, April 5-8, 1983.

Future Presentations

- D. Rugar Invited paper, Fourth International Conference on Phonon Scattering in Condensed Matter, Stuttgart, West Germany, August 22-26, 1983.
 Paper entitled "Two Applications of Microwave Acoustics in Liquid Helium: High Resolution Microscopy and Direct Measurement of Phonon Dispersion."
- D. Rugar Invited paper to be presented at the 1983 IEEE Ultrasonics Symposium, Atlanta, Georgia, October 31-November 2, 1983. Paper entitled "Recent Developments in High Resolution Acoustic Microscopy at Stanford."
- D. Rugar Invited paper (special session on Novel Application of Macrosonics) to be presented at the 106th Meeting of the Acoustical Society of America, San Diego, California, November 8-11, 1983. Paper entitled "New Applications of Nonlinear Acoustics at Microwave Frequencies."

Manuscripts in Progress

- D. Rugar, "Nonlinear Gaussian Focused Acoustic Beams," to be submitted to Journal of the Acoustical Society of America.
- D. Rugar, "Resolution Beyond the Diffraction Limit in the Acoustic Microscope A Nonlinear Effect," to be submitted to Journal of Applied Physics.
- J. S. Foster and D. Rugar, "Cryogenic Acoustic Microscopy," invited paper for special issue of IEEE Transactions on Sonics and Ultrasonics.