ACOUSTIC MICROSCOPY AT CRYOGENIC TEMPERATURES

Annual Summary Report

1 July 1980 - 30 June 1981

Contract No. N00014-77-C-0412

G.L. Report No. 3369
November 1981

Reproduction in whole or in part is permitted for any purpose of the United States Government.
ACOUSTIC MICROSCOPY AT CRYOGENIC TEMPERATURES

C.F. Quate
J. Heiserman

Edward L. Ginzton Laboratory
W.H. Hansen Laboratories of Physics
Stanford University, Stanford, CA 94305

Office of Naval Research
Physics Division Code 412
Arlington, Virginia 22217

November 1981

10

ACOUSTIC MICROSCOPY

1. REPORT NUMBER
2. GOVT ACCESSION NO.
3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle)
5. TYPE OF REPORT & PERIOD COVERED
   Annual Summary Report
   1 July 80 – 30 June 81
6. PREPARING ORG. REPORT NUMBER
   G.L. Report No. 3369
7. AUTHOR(S)
   C.F. Quate
   J. Heiserman
8. PERFORMING ORGANIZATION NAME AND ADDRESS
   Edward L. Ginzton Laboratory
   W.H. Hansen Laboratories of Physics
   Stanford University, Stanford, CA 94305
9. CONTROLLING OFFICE NAME AND ADDRESS
   Office of Naval Research
   Physics Division Code 412
   Arlington, Virginia 22217
10. MONITORING AGENCY NAME & ADDRESS (IF different from CONTROLLING OFFICE)
11. REPORT DATE
   November 1981
12. NUMBER OF PAGES
   10
13. SECURITY CLASS. (OF THIS REPORT)
   UNCLASSIFIED
14. SECURITY CLASS. (OF THIS ABSTRACT)
   UNCLASSIFIED
15. DECLASSIFICATION/DOWNGRADE
    SCHEDULE

16. DISTRIBUTION STATEMENT (OF THIS REPORT)
   "Approved for public release; distribution unlimited"

17. DISTRIBUTION STATEMENT (OF THE ABSTRACT ENTERED IN BLOCK 20, IF DIFFERENT FROM REPORT)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Cont. on reverse side if necessary and identify by block number)
   Acoustic microscopy
   Liquid argon
   Mechanical scanning
   Liquid nitrogen
   High frequency acoustic properties

20. ABSTRACT (Cont. on reverse side if necessary and identify by block number)
   This report summarizes our progress in a research program devoted to high resolution acousto-
   mic microscopy and cryogenic temperatures. During the past year we have studied the acoustic
   properties of two cryogenic fluids—liquid argon and liquid $^4$He below the temperature of
   0.5 K. In argon we have determined that nonlinear effects provide us with a dramatic increase
   in resolving power for focused acoustic beams. In liquid helium we have measured the attenuation
   versus temperature of sound at 1 GHz where the wavelength is less than 2500 $\AA$. The predicted
   behavior below 0.56 K where the attenuation varies as the fourth
power of temperature is confirmed for the first time at these small wavelengths. It gives us further confidence in our program to realize an instrument that will permit us to examine our specimens with an acoustic probe that is less than 1000 Å in diameter.
SUMMARY OF ANNUAL REPORT

Our principle goal in this research program is to explore the acoustic properties of liquid helium with wavelengths below 1000 Å. We hold the ultimate goal of using a scanned acoustic microscope to explore microscopic features of natural materials such as metals, ceramics, polymers and fabricated structures as used in macroelectronic devices. The present techniques available for studying materials and structures in the subminature world are limited to the electron microscope. It is insensitive to the elastic properties of the samples. Contrary to this, the acoustic microscope operating at room temperature has proven to be a suitable method for studying the elastic properties.

We hope to gain new insight into material properties -- grains and grain boundaries in metals -- inhomogeneties in ceramics or polymers -- and defects in microelectronic devices. It will be one of the major applications of helium -- a cryogenic fluid with unusual properties.

The year covered by this report produced several advances that bring us close to the realization of an operating instrument which will allow us to explore the microscopic region that we are so interested in. We have, also, advanced our knowledge of the properties of liquid helium at temperature below 1ºK as explored with sound waves near 1 GHz where the wavelength is less than 2500 Å.

One major event during the year was the Rank Foundation Conference in London in September 1980. The entire conference was focused on the principle of mechanical scanned microscopy. Major areas of microscopy were treated. It now turns out microscopy with optical, x-rays and acoustics use this principle...
to generate images with valuable information. Our work was a major component of the conference. The results are now completely published by Academic Press.¹ We attach the title page and table of contents as an Appendix.

We have a dilution refrigerator capable of cooling the microscope to the neighborhood of 0.15°K that will now dissipate 0.2 milliwatts of power. This will take care of the power dissipated by our mechanical scanning circuits.

The attractive feature of liquid helium at this temperature is very low attenuation for acoustic waves. It is also attractive because the wave velocity is 0.24 x 10⁻⁵ cm/sec -- a factor of eight below water. We have in this program succeeded in putting together a form of acoustic interferometer which has permitted us to measure the acoustic attenuation at the low temperature less than 1°K. That work confirms the fact that the attenuation decreases as the fourth power of the temperature. At that temperature (where we propose to operate) the attenuation is reduced to the point where it is no longer a limitation on the operation frequency. This is in contrast to room temperature instruments where the basic limitation is the intrinsic attenuation of the liquids.

Little experimental work has been done in liquid helium since the low impedance of the liquid presents a major obstacle. The impedance of helium is 0.03 -- about 50 times lower than that of water. If we are to exploit the marvelous properties that are found there we must deal with the problem of coupling acoustic energy from a high impedance crystal to the low impedance liquid. We have used the approach of matching layers since it is well known that a layer of material one quarter wavelength in thickness will serve to eliminate reflections provided that the impedance of the layer can be made equal to the geometric mean of the high crystal impedance and the low liquid.

impedance. This is no easy task since the optimum impedance of the layer, near 1.0 mech-ohm, is lower than any that is readily available in solids. It has required a major effort to find the solution but we think it has now been done. The work was adequately described in our 1st annual report (G.L. Report No. 7149, July 1980).

During the interval covered by this report we have made the decision to use single carbon layers rather than the tungsten-carbon layer. A double layer would permit more efficient transmission of energy through the interface but we would be limited to a rather narrow band of frequencies. Many carbon layers have now been fabricated on various surfaces. We think we understand the process rather well. Of major interest to this program is our newly acquired ability to deposit the carbon on the curved surfaces of the acoustic lens. We have mounted the coated lenses in helium and tested their focusing properties by translating a flat reflector through the focal plane. The reflected signal rises to a maximum and then decreases as the reflector is moved continuously through the focal region. This confirms our belief that the principles developed at room temperature for acoustic microscopy can now be translated over to the cryogenic instrument.

The component that remains to be examined is the system for mechanical scanning. We have preliminary indications that the system used in liquid argon (and nitrogen) can be adapted to helium. It will be part of the program during the first quarter of the next annual period.

One distinguished result with cryogenic fluids during this interval has been the discovery by Rugar -- Hunt Fellow for 1981 -- that the resolution can be improved by pushing the amplitude of the sound waves into the region of nonlinear behavior. The evidence for improved resolution has been demonstrated in the images as described previously.
As we now understand the phenomena it comes about as a result of the generation of second harmonic near the focal region of our spherically converging waves. These second harmonic waves are generated on spherical wavefronts and they converge to a waist (at the focus) that is narrower than that achieved by the fundamental wave. The theory has not been fully developed but it will be a component of the work for the next annual period.

Much of cryogenic microscopy have been developed through extensive work in liquid argon and nitrogen. It is easier to work there and there we have enjoyed considerable success. The images in nitrogen and argon are superior to optical images. We feel that they are merely a prologue to the work that is ahead of us in liquid helium below 1ºK.
PUBLICATIONS


APPENDIX

London, England
September 1980.
CONTENTS

Contributors ix
Preface ix

Fundamentals of Scanning Systems 1
G. S. Kino

SCANNING ACOUSTIC MICROSCOPY

Microwaves, Acoustics and Scanning Microscopy 23
C. F. Quade

Gas Medium Acoustic Microscopy 57
H. K. Wickramasinghe and C. R. Pettit

Cryogenic Acoustic Microscopy 71
J. Heismann

Acoustic Microscopy: Imaging Microelectronic Circuits with Liquid Metals 97
J. Azzal

Mechanically Scanned Acoustic Microscope composed of Plane and Concave Transducers for Transmission Mode 119
H. Chubaact

Metrology and Imaging in the Acoustic Microscope 127
R. D. Weglein

Spectroscopic Study of Defects in Thick Specimen Using Transmission Scanning Acoustic Microscopy 137
J. K. Wang, C. S. Tsai and C. C. Lee

Characterisation of Acoustic Microscopes for Non-destructive Testing 149
B. Nongaillard, J. M. Rouvaen, R. Torguet and E. Bridoux
Contents

Defect Detection for Microelectronics by
Acoustic Microscopy
R.L. Hollis and R.V. Hamner 155

SCANNING OPTICAL MICROSCOPY

Theory and Principles of Optical Scanning
Microscopy
W.T. Welkord 165

Developments in High Resolution Confocal
Scanning Light Microscopy
G.J. Brakenhoff, J.S. Binnerts and
C.L. Woldringh 183

Imaging Modes of Scanning Optical Microscopy
C.J.R. Sheppard 201

Scanning Optical Microscopy of Semiconductor
Devices
T. Wilson, J.S. Garnaway and C.J.R. Sheppard 227

Resolution of Near-Field Optical Fibre
Refractive Index Profiling Methods
W.J. Stewart 233

A CCD Linear Array Based Scanning System for
Rapid Analysis of Biomedical Material on
Standard Microscope Slides
A.S.J. Farrow 241

SCANNING PHOTOACOUSTIC MICROSCOPY

Scanning Photo-Acoustic Microscopy
Y.H. Wong 247

Photoacoustic Microscopy at Low Modulation
Frequencies
M.V. Luukkala 273

Thermal-Wave Imaging and Microscopy
A. Rosenowitg 291

Electron-Acoustic Microscopy
G.S. Cargill 319

Photothermal Radiometry for Spatial Mapping
of Spectral and Material Properties
P.E. Nordal and S.O. Kanstad 331
Contents

The Optacoustie and Photothermal Microscope: The Instrument and its Applications
G. Bense 341

The Mirage Effect in Photothermal Imaging
D. Fournier and A.C. Borcara 347

Photoacoustic Imaging of Compositional Variations in Hg$_{1-x}$Cd$_x$Te Semiconductors
J.F. McClelland, R.N. Kruseley and J.L. Schmit 353

SCANNING SOFT X-RAY MICROSCOPY

The Scanning X-Ray Microscope - Potential Realizations and Applications
E. Spiller 365

Imaging and Scanning Soft X-Ray Microscopy with Zone Plates
G. Schmahl, D. Rudolph and B. Niemann 393

A Scanning UltraSoft X-Ray Microscope with Multilayer Coated Reflection Optics: First Test with Synchrotron Radiation Around 60 eV Photon Energy
R.-P. Haslibeh 413

Photoelectron Detection of X-Ray Images for Contact Microscopy and Microanalysis
F. Pollock 435

X-Ray and Particle Microscopy using Fresnel Zone Plates
N.M. Cagliio 443

Scanning Soft X-Ray Microscopy - First Tests with Synchrotron Radiation
H. Rarback, J. Kenney, J. Kirs and X.-G. Xie 449

Subject Index 457