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19 ABSTRACT (Continue on reverse if necessary and identify by block number) The project is aimed at developing a strategy for generating coherent sound waves in solids and liquids at very high frequencies, i.e., above 2000 GHz. Sound waves at these frequencies are important probes for investigating the physical properties of solids and liquids. Our approach is directed toward the use of periodic structures in piezoelectric films of zinc oxide. These multilayer films with alternate layers of piezoelectric active material will serve as efficient transducers if the period of the alternate layers are equal to the wavelength of sound. We have been successful in fabricating multilayer transducers that work at 16, 32 and 96 GHz. We forecast their use in a number of applications such as the cryogenic acoustic microscope.					
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MILLIMETER-WAVE ACOUSTIC TRANSDUCERS

Annual Summary Report

June 1, 1987 - September 30, 1988

Office of Naval Research

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Principal Investigator:

C. F. Quate

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August 1988

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Our primary objective in this program is to develop strategies for generating sound waves in solids and fluids at very high microwave frequencies. If we use superfluid helium as the operating fluid we are, in principle, able to work with frequencies much higher than 10 GHz. Sound waves at these frequencies are extraordinarily useful for probing the physical properties of solids and liquid helium. These waves are fundamental to several areas in physical acoustics.

Our main thrust has been to exploit the piezoelectric properties of Zinc Oxide (ZnO) in the form of thin films. For most applications the films are approximately one-quarter wavelength in thickness mounted on a sapphire substrate with an intervening layer of gold. Designs of this type suffer from two difficulties if we try to operate in the frequency range above 10 GHz. First, the layer thickness decreases and this introduces the problem of shorting arising from pinholes penetrating the thin layers of piezoelectric material. Second, the electrical impedance of the transducers decreases to the point where it is difficult to couple energy into this system without resorting to heavy tuning. This narrows the operating bandwidth and diminishes the utility of the system.

Our strategy for overcoming both of these problems is to use multilayer films in the transducers. We use alternating layers of active and inactive ZnO. A periodic structure of this type will generate strong sound waves when a uniform rf electric field is placed across the transducer stack with a frequency such that the wavelength of sound in the layered material is equal to the period of the layers.

The multilayer films have been proposed in the literature, but it is only at the very high frequencies of interest that they are compatible with multiple thin film deposition techniques. At the lower frequencies described in previous proposals the individual layers ( $\lambda/2$ ) have a thickness that is too large to be fabricated with known technology. For frequencies in the GHz range, the individual layers are

sub-microns in thickness and it is practical to lay down ten or more layers on substrates of sapphire.

In our approach we use a layer of ZnO with the c-axis normal to the substrate (as in conventional transducers) for the active layer. For the inactive layer we again use ZnO, but tilt the c-axis away from the direction of the normal. This approach, which is unique to our work, allows us to use the same material for the entire stack. This avoids problems of strain build-up that would occur if we use a different non-piezoelectric material, such as aluminum, for the inactive layer.

Efficient transducers of this type will be basic to a number of investigations. These include: the high resolution cryogenic acoustic microscope; the tunneling and force microscopes; and the probing of thin metallic films. The cryogenic acoustic microscope has been operated at Stanford with a frequency of 8 GHz and a resolving power of 200 Å. Investigation of the cryogenic instrument is being carried out at Stanford, at the Aerospace Laboratory in El Segundo, and at the Olympus Research Laboratory in Japan. At McGill University in Canada, the multilayer films are being studied by the group associated with Professor G. Farnell. These probes have been proposed for use with the tunneling and force microscopes both in Stanford and in West Germany by K. Dransfeld at the University of Konstanz. Hadimioglu, working at Stanford with A. Kapitulnik, is developing techniques for using the high frequency sound waves to investigate the physical properties of thin metal films. We are confident that high frequency sound waves reflected from these films will allow us to study transitions in films with a thickness of several hundred Angstroms.

In the annual period covered by this report, we have concentrated much of the effort on the improvement of the quality of the films of Zinc Oxide and on a study of the film properties and the structure of the multilayer stack. We have also incorporated them into lens assemblies at 8, 16 and 32 GHz. These assemblies

consist of a sapphire rod with a spherical lens ground in the surface opposite to the multilayer piezo-transducers. We have also expended some effort on the scanning components that are needed to implement the scanning instruments that we envision. Several lens assemblies at 16 GHz have been transferred to Dr. M. Muha at the Aerospace Laboratory for incorporation in their cryogenic acoustic microscope.

Our work on improved quality for the piezoelectric films was done by carefully reviewing the procedures for depositing the Zinc Oxide. We have studied such parameters, the rates of deposition, the temperature of deposition and the flow of oxygen to the system. This study has allowed us to deposit high quality films on a variety of substrates with a piezoelectric coefficient that approaches the value for single crystals.

Our work on the study of film properties has been carried out with the TEM, the SEM, and the Auger spectrometer. With the TEM, we deposit the film on silicon substrates and thin the silicon to the point where the electron beam can penetrate the structure. With the SEM, we fracture the devices in such a way that we can view the layers "edge-on". The studies with the Auger spectrometer can be done with films deposited directly on gold films.

We have learned that the stoichiometry of the material is good throughout the entire structure with as many as 25 layers. We know that the first two layers are "oxygen-rich" and thus undoubtedly diminishing the piezoelectric coupling in this region. We find from studies with the SEM that the transition region from the inactive tilted layer to the active normal layer is abrupt as desired. We believe that the transition from the active normal layer to the inactive tilted layer is gradual. This could reduce the strength of the periodic structure and account for the discrepancy between the numerical calculations and the measured data.

During this annual period we made presentations of this work at the 1987 IEEE Ultrasonics Symposium.

The significant visits during this interval were made to El Segundo, California (Aerospace Laboratory), to Wetzlar, West Germany (Leitz Company) and to Japan (Ultrasonics Workshop at Sendai, and a visit to Hitachi and the Olympus Research Laboratory near Tokyo).

The people at the Aerospace Laboratory are constructing a cryogenic acoustic microscope for frequencies above 10 GHz for the purpose of examining electronic integrated circuits. We have established an informal working relationship with this group wherein we exchange information in that they are helping us with the components related to stepping motors and we are helping them with the piezo-scanners and the acoustic lens assemblies.

At Leitz, in West Germany, we spent time with M. Hoppe, who is in charge of the R&D on their acoustic instruments. They have found a strong interest in this instrument throughout the world and they are proceeding with advanced versions of the commercial instrument. They find that they can be used to monitor residual stresses in aluminum films as deposited in silicon. This is an example where acoustic microscopy proves to be a unique microscopic tool for studying problems that cannot be examined with other methods. In Japan, at both Hitachi and Olympus, we encountered similar findings where they were building a library of acoustic micrographs that revealed information unique to acoustics. At Hitachi, for example, they find that the acoustic instrument is useful for studying the surfaces of ceramic materials such as Silicon Nitride ceramics proposed for internal combustion engines. At Olympus, in addition to the work on the commercial instrument at room temperature, they are doing research on cryogenic instruments. They have a system operating at 1.5 GHz at 77 K in liquid nitrogen. Their resolution is equivalent to a water-based system working at 3 GHz. The people at Olympus are

now in the process of moving their cryogenic instrument into liquid helium in order to further improve the resolution.

In Sendai, Japan, we attended a Workshop on Acoustic Microscopy at Tohoku University. This meeting was organized by Professor N. Chubachi and Professor J. Kushibiki. It was an international gathering with most of the people working in this field. Our notes on the meeting reveal the progress that is being made by the important contributors to acoustic microscopy. The notes include a summary of the significant reports. We include them here for the sake of completeness.

Various people contributed their ideas concerning the present state of acoustic radiation as a form of imaging where the applications were unique. Applications that were difficult to investigate with other forms of radiation, particularly in the field of Microscopy. In this section we will include a summary of the main points of the Workshop as separated by the person reporting.

1. Dr. M. Hoppe at Leitz, West Germany

Major applications of the Scanning Acoustic Microscope (SAM) are emerging in the field of composites and copolymer materials. In the polymer materials it is possible to determine the distribution on the various components and thereby characterize the quality of the material. With paint, it is also possible to study the distribution of the particles of pigment throughout the layer of paint and thereby judge the quality of the product.

Leitz has studied the distribution of magnetic particles in magnetic tapes -- material that is opaque to optical waves. This is an example of studying 'black' material in a way that is not available with optical probes.

In another sector, Leitz has demonstrated that the instrument can be used to detect the residual photoresist that remains in the via's that are part of the integrated



circuits patterns and that are incompletely processed. It is a problem that is difficult to handle with other forms of microscopes.

2. Dr. R.S. Gilmore at General Electric, New York

At GE, they find that the acoustic instrument is useful for controlling processes. Two examples were cited: (1) Paint can be analyzed with the acoustic instrument by studying the distribution of particles in the layers and using this as a control for the entire process. (2) In the production of plastic the acoustic instrument is used to examine the samples as they are being manufactured and this information is sufficient to allow them to control the process. In other fields, Gilmore cites the utility of acoustics for examining pressure welds of various kinds.

3. Dr. R. Moore at Exxon, Louisiana

Moore has found that acoustic waves as focused in the microscope is useful for the study of coke. It is conventional practice to use optical reflectivity as a means of characterizing the carbon content of coal and coke. The problem is that the reflectivity is very small -- after all, these materials are black and they don't reflect much light. The reflectivity for acoustic waves is thirty times larger and this points to the utility of the SAM for examining these materials.

4. Drs. H. Kanda and I. Ishikawa at Hitachi, Japan

The work at Hitachi points to the utility of acoustic microscopy in the study of subsurface damage that results from grinding processes. They cite two examples: (1) The study of silicon surfaces that have been ground and polished to a mirror-like finish, and (2) the surface of silicon nitride as finished by various techniques. With the polished silicon they find that it is possible to reveal the subsurface damage by varying the acoustic frequency from 200 MHz to 600 MHz. The velocity variations in the damaged layer is sufficient to produce an image. In the silicon nitride material they show that the surface condition as revealed with acoustic imaging can vary in ways that are significant when this material is used for

such applications where sliding friction is important, i.e., in engines where pistons slide against the wall of the cylinder.

5. Dr. K. Keraki at Olympus, Japan

One important example from the work at Olympus is the study of packaged IC devices. The package often consists of a black polymer material which serves to protect the device from light, but at the same time this precludes the use of light for inspecting the leads that connect the device to the outside world. Furthermore, they include small glass particles in the black material to control the thermal properties of the package. These particles scatter sound and make it difficult to examine these structures with acoustic waves. The people at Olympus have found that this can be circumvented by using rather low frequencies of the order of 30 MHz in their microscope. The resulting images are dramatic and reveal the structure of internal leads in sufficient detail to determine the integrity of the overall package.

6. Professor J. Kushibiki at Tohoku University, Japan

It is clear from the presentations at this Workshop that the line focus beam pioneered at Tohoku University is a superior technique for characterizing the properties of materials, particularly anisotropic materials, over rather large areas. It was demonstrated that complete wafers can be examined and characterized by this technique.

7. Drs. A. Kulik and G. Gremaud, Institut de Genie Atomique, Switzerland

They told us that acoustic microscopy could be used in general areas of Physics and they urged the audience to consider problems in this category. In particular, they outlined their system for studying internal friction and detailed how the scanning microscope could be used to provide new and useful information in this area.

8. Professor J. Attal at the Universite des Sciences et Techniques du Languedoc, France

He pointed out that the acoustic properties of mercury, namely, the high acoustic impedance, made this an ideal coupling fluid for imaging deep inside solid materials. He demonstrated his point with silicon wafers where he showed images of the 'back side' of the wafer. With his system, one can image from the back side of the wafer where there are no circuits and leave the active side filled with devices untouched.

9. Dr. J. Bereiter-Hahn at Johann Wolfgang Goethe University, West Germany

He showed with a film that biological cells in the living state could be studied in great detail with the acoustic microscope. It is our opinion that this indicates that this instrument will be most useful in this field.

10. Professor M. Tanaka at Tohoku University, Japan

Tanaka is studying sections of tissues with the acoustic microscope to elucidate the elastic properties on a fine scale. This will not only enable him to obtain new information on the tissue sections themselves, but also help him and his colleagues to improve their understanding of the images they record with the low frequencies used to study the internal structure of the human body.

11. Dr. J.K. Zieniuk at IPPT-PAN, Poland

He revealed a new type of scanning capacitive probe and it was suggested that this too might in some way be coupled to acoustic waves to serve a new and useful purpose.

In summary, we conclude that Acoustic Microscopy is entering a stage where applications, unique and important to this form of radiation, are readily identified. These range from composite structures to coatings and surfaces on through subsurface damage to biological structures. The commercial instruments are now becoming available in increasing numbers and this should accelerate the

process of identifying new problems that can only be solved with this form of  
microscopy.

Attached is our Publications/Patents/Presentations/Honors Report for 1 October,  
1987 through 30 September, 1988.

June 1988

OFFICE OF NAVAL RESEARCH  
PUBLICATIONS / PATENTS / PRESENTATIONS / HONORS REPORT  
FOR  
1 OCTOBER 1987 through 30 SEPTEMBER 1988

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CONTRACT NO0014 - 87-K-0447

R&T NO. 4126945

TITLE OF CONTRACT: Millimeter-Wave Acoustic Transducers

NAME(S) OF PRINCIPAL INVESTIGATOR(S) C. F. Quate

NAME OF ORGANIZATION: Stanford University

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June 1988

PAPERS SUBMITTED TO REFEREED JOURNALS  
(Not yet published)

"Piezodriven Scanner for Cryogenic Applications"  
Lloyd J. La Comb, Jr. and C. F. Quate  
Submitted to Review of Scientific Instruments  
Partially supported by the National Science Foundation

(To be published in September 1988)

June 1988

PAPERS PUBLISHED IN REFEREED JOURNALS

NONE

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PAPERS PUBLISHED IN NON-REFEREED JOURNALS

NONE



June 1988

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June 1988

INVITED PRESENTATIONS AT TOPICAL OR  
SCIENTIFIC/TECHNICAL SOCIETY CONFERENCES

- C.F. Quate, "Surface Tunneling Microscopy", Fourth Annual Science & Technology Seminar 'Advances in Catalytic Technologies', Catalytica, San Jose, California, October 26, 1987 (supported by Catalytica)
- C.F. Quate, "Molecular Imaging with the STM and the AFM", International Symposium on Surface Interactions, Neve Ilan, Jerusalem Hills, Israel, March 13-18, 1988 (supported by Conference organizers, unrestricted funds & Dean's Office, Humanities & Sciences, Stanford)
- C.F. Quate, "Acoustic Microscopy - Historical Aspects", International Research Conference on Ultrasonic Micro-Spectroscopy for Material Characterization (UMS), Sendai, Japan, June 6-8, 1988 (supported by Conference organizers and unrestricted funds)
- C.F. Quate, "Imaging with the STM and the AFM", International Conference on Solid State Devices and Materials (SSDM), Tokyo, Japan, August 24-26, 1988 (supported by Conference organizers and unrestricted funds)

CONTRIBUTED PRESENTATIONS AT TOPICAL OR  
SCIENTIFIC/TECHNICAL SOCIETY CONFERENCES

- B. Hadimioglu, "Multilayer ZnO Acoustic Transducers at Millimeter-Wave Frequencies", 1987 IEEE Ultrasonics Symposium, Denver, Colorado, October 14-16, 1987 (supported by NSF and AFOSR), also authored by L.J. La Comb, Jr., L.C. Goddard, B.T. Khuri-Yakub and C.F. Quate
- L.J. La Comb, Jr., "Interface Structure of Multilayer ZnO Acoustic Transducers", 1988 IEEE Ultrasonics Symposium, Chicago, Illinois, October 3-5, 1988 (to be supported by this Contract), also authored by B.T. Khuri-Yakub, C.F. Quate and B. Hadimioglu

HONORS/AWARDS/PRIZES

1988 IEEE Medal of Honor for "the invention of the Scanning Acoustic Microscope" received at the IEEE Annual Medals Presentation and Reception, Sheraton Boston Hotel, Boston, Massachusetts, May 9, 1988

June 1988

GRADUATE STUDENTS SUPPORTED UNDER  
CONTRACT FOR YEAR ENDING 30 SEPTEMBER 1988

Lloyd J. La Comb, Jr.

POSTDOCTORALS SUPPORTED UNDER  
CONTRACT FOR YEAR ENDING 30 SEPTEMBER 1988

Babur B. Hadimioglu (until January 1988) - he now  
serves in a consulting capacity at Stanford.