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# Laser Acoustic Microstructure Analysis at the Micron and Nanometer Length Scale

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# LASER ACOUSTIC MICROSTRUCTURE ANALYSIS AT THE MICRON AND NANOMETER LENGTH SCALE

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## ABSTRACT

Laser acoustic approaches to investigating the inteaction of elastic waves with microstructure in materials is presented that probe both the micorn and nanometer length scales. At the micron length scale, a full-field imaging approach is described that provides quantitative measurement of amplitude and phase of the out-of-plane acoustical motion at GHz frequencies. Specific lateral acoustic modes can be identified in addition to the primary thickness mode with spatial resolution sufficient to image wavelengths as small as 4.5 μm.

## INTRODUCTION

Conventional nondestructive evaluation methods for interrogating the microstructure of materials are limited to macroscopically large spatial averaging over many microstructural features. The result is that only empirical correlations can usually be made between these measurements and the actual state of a material's microstructure. However, we know from many metallurgical studies, that microstructural features, such as grain size, grain boundaries, development of dislocations and microcracking and the dynamics of these features change significantly with environmental factors, such as stress, corrosion and aging. Acoustic waves are often used as microstructural probes as these waves are attenuated and scattered from most features. At this time our understanding of the interaction of these waves with microstructural features is either empirical or based on mean field approaches using the averaging caused by the conventional measurement methods. Today, a new approach has become available that has the ability to extend acoustical measurements to the length and time scales of individual microstructures. This new approach is based on laser acoustics where optical means are use to both generate and detect acoustic waves. Due to the noncontacting and manipulation properties of optical approaches, one can now make measurements at ultrahigh frequencies (UHF) probing features of micron or even nanometer size on time scale of nanoseconds or even picoseconds. Our research goal is to extend these optical techniques to GHz frequencies and beyond for probing the interactions of surface acoustic waves directly with grain boundaries, dislocations and secondary phases with the intent of validating and quantifying our knowledge of microstructural properties through acoustic wave interactions. This paper describes our progress to date on a full-field imaging approach useable at the micron scale and the use of picosecond acoustics for the nanometer scale.

## MICRON LENGTH AND NANOSECOND TIME SCALE MEASUREMENTS

Laser ultrasonic techniques, using nanosecond pulses for thermoelastic generation have been widely proven as a versatile tool for measuring acoustic wave motion at surfaces up to very high frequencies (MHz – GHz). Anisotropy in materials within a single grain and the properties of dislocations and grain boundaries, dictate that measurements over the all planar directions need to be made in order to see all the waves scattered or directed in off-axis directions. A full-field imaging technique would greatly speed up the measurement process and could illuminate interesting phenomena quickly, such as mode and harmonic conversion.

A versatile method for imaging acoustic motion to fill the need described above has been developed at the INEEL that utilizes the photorefractive effect in optically nonlinear materials to perform adaptive interferometry.[1,2] Optical interference is developed within a photorefractive material with this technique and the output is an optical image whose intensity distribution is directly proportional to the surface vibration amplitude, for small ultrasonic displacements. Utilizing this approach, no post processing of the data recorded by a video camera is required to produce images of the surface vibration amplitude over large areas. Application of this approach to imaging of traveling wave motion in plates with millimeter length and microsecond time resolution has been previously described.[3,4,5] Here, we present results extending this measurement technique through a microscope for imaging wave motion on the micron length and nanosecond time scales suitable for investigating features comparable to the size of a single grain in metallic materials.[6]

The method of imaging employs interferometric processing of the entire image of a surface through a photorefractive dynamic hologram. Photorefractivity refers to that process where optical excitation and transport of charge carriers within select nonlinear optical materials produces a diffraction grating or hologram from the interference pattern developed inside the material. A spatial and temporal charge distribution results in the photorefractive material that reflects the phase information impressed onto an optical signal beam (e.g. by a vibrating surface). The INEEL method records the photorefractive grating produced at a fixed beat frequency between the phase modulated signal and reference beams. It can directly measure vibration amplitude and phase with a response proportional to the Bessel function of order one, providing a linear output for small amplitudes. The method accommodates rough surfaces and exhibits a flat frequency response.

Figure 1 shows the inphase and quadrature images of resonant motion in a thin film bulk AlN acoustic wave resonator (FBAR) provided by Agilent Technologies Inc. The primary thickness mode is easily observed as the major feature of the impedance curve shown. However, device operation is compromised by the presence of lateral modes in these piezoelectric plates that can only be inferred from the impedance. In contrast, full-field imaging easily observes the lateral modes providing quantitative measurement of the mode excitation amplitude and spatial distribution.

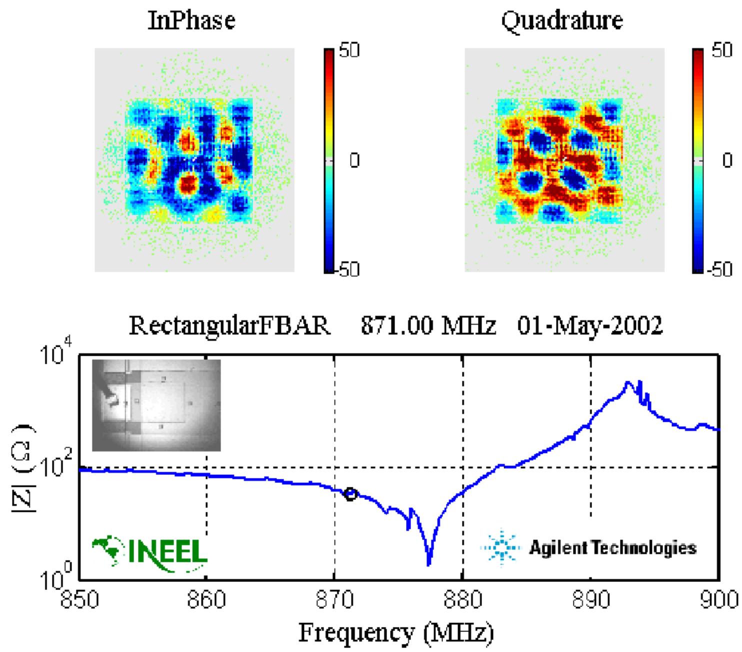


Figure 1. Inphase and quadrature images of a thin film bulk acoustic wave resonator near its first thickness mode with the electrical impedance. An image of the actual resonator plate is shown in the insert of the electrical impedance.

Figure 2 shows an image of acoustic motion at 862 MHz in the plate. Given the length and width of the plate (170 µm x 160 µm), the Fourier transformed image shows the smallest wavelength present at about 4.5 µm. The ultimate resolution of this imaging technique is believed to be the same as other optical methods, roughly a half wavelength of light. Work is in progress to determine the ultimate resolution of this acoustic imaging probe at the micron scale.

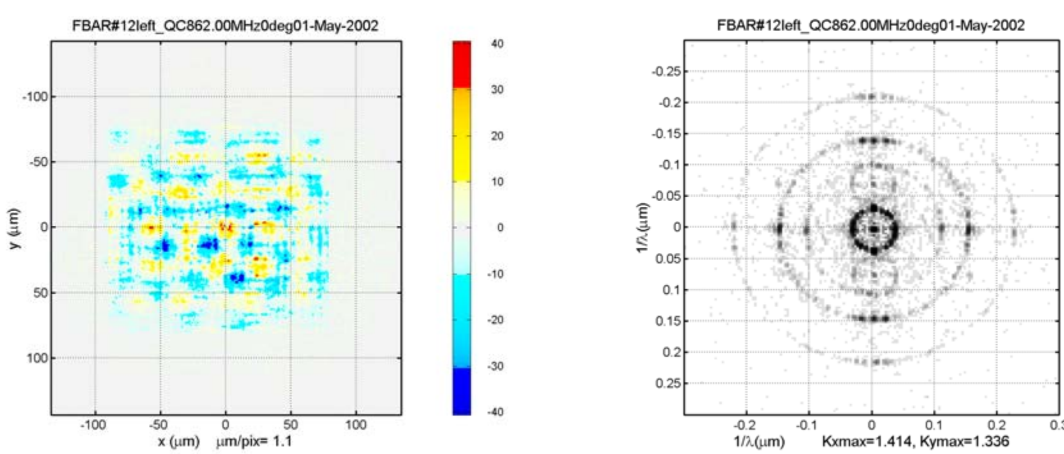


Figure 2: (left) Image of an acoustic mode at 862 MHz, (right) magnitude of the Fourier transform showing the presence of multimodes and the smallest spatial wavelength of about 4.5 µm.

## MEASUREMENTS AT THE NANOMETER AND PICOSECOND SCALE

To a large extent the way in which a structural material ages is a function of the motion and interaction of lattice imperfections such as dislocations. A dislocation refers to a localized rearrangement of the constitutive atoms of a crystalline material from that of a perfect lattice. Thus it seems natural to probe the properties of dislocations by dynamic modulation of the lattice atoms, i.e. phonons. Indeed, characterizing microstructural features using phonons has been actively pursued for over four decades. For instance, studies involving long wavelength acoustic phonons with frequencies in the MHz range (ultrasonic waves) have proven successful in predicting many properties of dislocations. However, the relatively large acoustic wavelength associated with MHz ultrasonic waves have limited investigation of the direct interaction of acoustic phonons with dislocations to idealized material systems, i.e. pure metals with large dislocation lengths. Complimentary techniques involving short wavelength thermal phonons have been successful in ascertaining properties of dislocations with sub-micron dimensions. However, these techniques involve mapping the intensity distribution of thermal phonons scattered by an array of dislocations and hence lack the ability to provide information regarding the phase of the scattered phonon field. In addition, as a result of the limited spatial resolution, most techniques used to characterize material microstructure are constrained to measuring microstructural properties that are averaged over large distances as compared to the length scales associated with individual microstructural features.

Our work involves exploiting the temporal and spatial resolution provided by picosecond acoustics to directly probe material microstructure. Generation of picosecond acoustical pulses entails irradiating a sample with a short, very intense laser pulse (pump beam) and subsequent conversion of the absorbed laser energy into acoustic phonons via a variety mechanisms[7] (thermoelastic, deformation potential, piezoelectric). Laser detection (probe beam) of the acoustic pulse is accomplished by either monitoring strain induced changes in the reflectivity or using interferometric techniques capable of resolving real and imaginary modulation of the refractive index as well as phase changes caused by surface displacement[8].

To date, the generation techniques associated with picosecond acoustical experiments have allowed only the generation and detection of longitudinal and shear waves that propagate into the bulk[9]. For this reason picosecond acoustics has typically been confined to the study of elastic properties of thin films. Unfortunately the microstructure of thin films is not characteristic of microstructure found in structural materials[10,11]. Thus investigation of microstructures features germane to structural materials requires applying picosecond techniques to other sample geometries. This has lead researchers to investigate new modalities for generating picosecond acoustics[12,13]. For instance, consider the generation of picosecond surface acoustic waves (SAW's). Picosecond SAW's are appealing because they can be used to study near surface properties of bulk material. The bandwidth of a SAW is related to the acoustic transit time across the lateral dimension of the absorbed laser light, roughly speaking the laser spot size at the sample surface. Thus, conventional methods are limited to generation of nanosecond SAW's with wavelengths in the μm range[14]. However creative manipulation of the generation process can be achieved through spatial modulation of the refractive index at the sample surface. Along these lines we have investigated generating picosecond SAW's by using electron beam lithography to modulate the surface absorption characteristics and hence the acoustic wavelength on a sub-μm scale.

The two material systems studied consisted of 5μm long bars periodically deposited on (100) Si, Fig. 3, and (100) GaAs substrates. For the Si sample, the bar axis coincided with the [001] direction and for the GaAs sample the bar axis coincided with the [011] direction. The Rayleigh velocities, determined from the elastic constants, for the unloaded Si and GaAs samples described above are 4.92 nm/ps and 2.72 nm/ps respectively. The bar height, h, was approximately 50 nm and the periodicity, p, was 220 nm. The metallization duty cycle, η, was approximately 50% and 70% for the Si and GaAs substrates respectively, Fig. 4. The experimental setup is shown in Fig. 4. The pump and probe pulse trains are derived from the same femtosecond Ti:sapphire laser (Coherent Mira 900) with ~ 100fs pulse duration and a 76MHz repetition rate. The 800nm output was used for the probe beam while the second harmonic at 400 nm was used for the pump beam. The pump beam was modulated at 1 MHz to facilitate lock detection. The pump and probe beams were combined using a dichroic beam splitter to allow both beams to pass through the same 50x microscope objective producing a 2 μm spot at the sample surface.

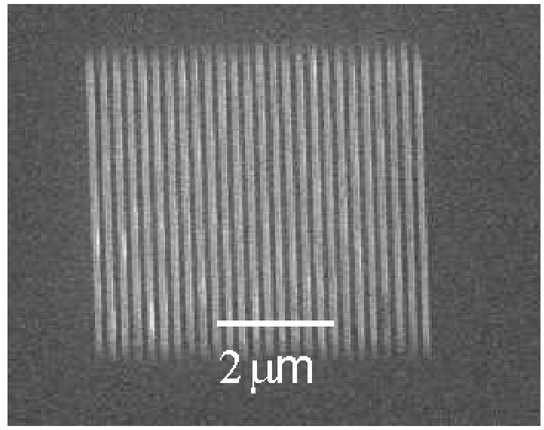


Figure 3. SEM image of regular array of aluminum targets on a Si substrate.

Transient reflectivity measurements of surface acoustic waves produced in this fashion on silicon and GaAS substrates are shown in Fig. 5. The center frequency for the Si and GaAs samples is 23 GHz and 13 GHz respectively. These frequencies correspond closely to the frequency predicted using the Rayleigh velocity of the substrate material, $\upsilon=V_R/p$ (22.4 GHz and 12.4 GHz for Si and GaAs respectively).[15] However, as the ratio of the bar height to the grating period, h/p, necessarily becomes large for high frequency gratings, mechanical loading by the grating can greatly alter the SAW velocity from that of a pristine substrate. For instance, Koskela *et al*.[16] have demonstrated suppression of bulk wave radiation in $LiNO_3$ by slowing the leaky-SAW velocity below the slow surface skimming shear wave velocity using heavy mechanical loading provided by a Au grating. For $h/\lambda=.025$, a 10% reduction in velocity was found.

In order to qualitatively explore the influence of mechanical loading on the SAW velocity; consider a perturbation theory first presented by Auld.[17] The theoretical development involves expanding the interface stress (interface between the substrate and bar material) in a Taylor series about the strip height. Retaining only first order terms, the change in phase velocity can be expressed as, $\Delta V/V=F_v(h/p)\eta$, where $F_v$ is the first order mechanical scattering coefficient. In order to place the present study in context to previous efforts,[12,13,16] the theoretical changes in velocity caused by gold as well as aluminum bars will be

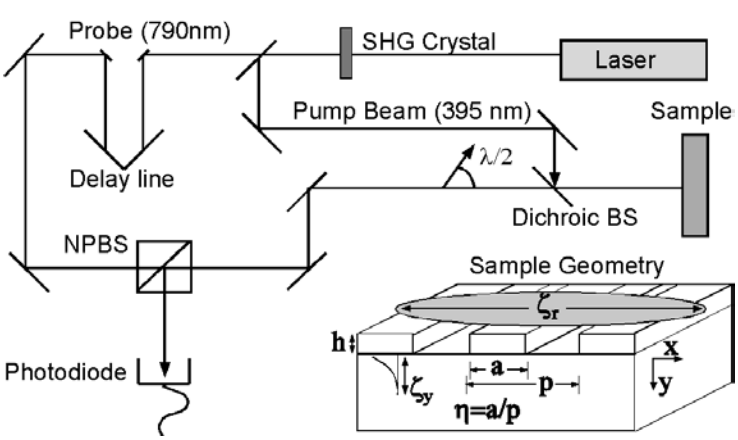


Figure 4. Experimental setup and sample geometry.

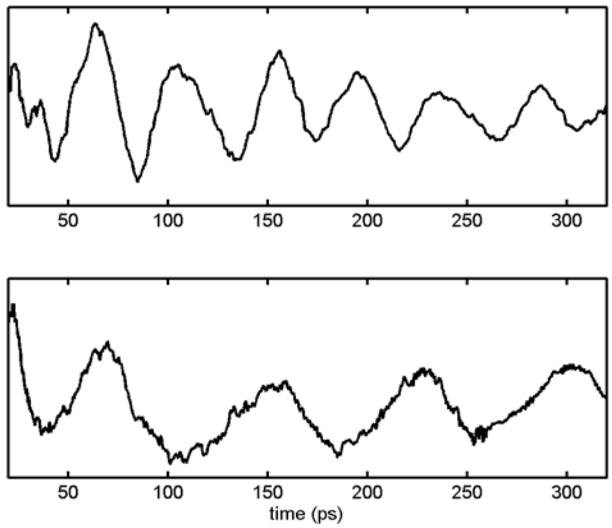


Figure 5. Transient reflectivity measurements for Silicon (top) and for GaAs (bottom) substrates.

presented. For the GaAs system, (100) plane and [011] propagation direction, $\Delta V/V|_{Al}$=0.24(h/$\lambda$)$\eta$ and $\Delta V/V|_{Au}$=-5.11(h/$\lambda$)$\eta$. Piezoelectric coupling for this system is small and the effect of piezoelectric shorting on the velocity was neglected. For the Si system, (100) plane and [010] propagation direction, $\Delta V/V|_{Al}$=-1.26(h/$\lambda$)$\eta$ and $\Delta V/V|_{Au}$=-13.9 (h/$\lambda$)$\eta$. These results illustrate that mechanical loading for the Al/GaAs and Al/Si systems only slightly influence the SAW velocity and hence the center frequency, in contrast to systems using gold as the grating material. Indeed, the results presented in this paper in addition to previous efforts[13] verify that for an aluminum grating on a Si substrate the effect of mechanical loading on the SAW propagation velocity is small.

The signal to noise ratio is larger for the Si sample than for the GaAs sample. This is most likely related to the ability to generate a thermal gradient in directions parallel to the surface. The lateral thermal gradient to a large extent is governed by the lateral modulation of the energy density of the absorbed pump light. For a pump wavelength of 400 nm, the optical skin depth for Si and GaAs is 82 nm and 15 nm respectively. The skin depth for the aluminum grating ~ 10 nm, so it is expected that the Si system will more efficiently generate SAW's at a frequency corresponding to the grating period than the GaAs sample.

## CONCLUSIONS

An imaging optical lock-in acoustic wave measurement method has been described that can be effectively utilized at GHz frequencies and micron length scales. Measurements on a thin film bulk acoustic resonator show both bulk thickness and lateral acoustic modes. Spatial resolution at the micron level was illustrated by imaging a particular mode wavelength of 4.5 µm. Coupled with detailed modeling of anisotropic elastic properties of materials, imaging provides a powerful technique for microstructure measurement and analysis at the micron scale. An approach for extending current capabilities to include spatial and temporal resolution on the nanometer and picosecond scale was outlined. This approach involved interrogating material microstructure with picosecond SAW's. Initial experimental efforts entailed the production of SAW's in Si and GaAs. The method utilized electron beam lithography to modulating the optical reflectivity and hence the lateral thermal gradients at the sample surface. The center frequencies were determined to be 22 GHz and 13 GHz for the Si and GaAs sample respectively. These frequencies correspond closely to the frequencies predicted using the SAW velocity of the substrate materials, confirming the that for both the Al/GaAs and Al/Si sample, the effect of mass loading on the propagation velocity is small.

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