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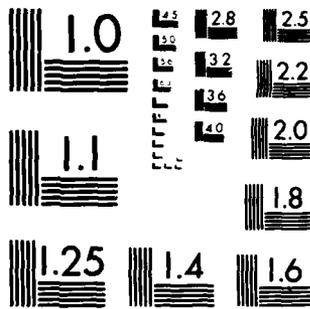
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# Research Report

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APPLICATION FOR THIN-FILM ULTRASONIC MEASUREMENTS

A. C. Tam

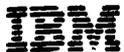
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**PULSED-LASER GENERATION OF ULTRA-SHORT ACOUSTIC PULSES:  
APPLICATION FOR THIN-FILM ULTRASONIC MEASUREMENTS**

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**ABSTRACT:** We demonstrate opto-acoustic generation and detection of acoustic pulses of 1 nanosecond duration in condensed matter. Such opto-acoustic pulses are at least one order of magnitude shorter than those previously reported. These narrow acoustic pulses find new application for thin-film pulse measurements, which are not possible with conventional transducer techniques. Round trip echo times are measured to 1% accuracy for stainless steel films of 12  $\mu\text{m}$  thickness. This provides a new high accuracy thickness measurement in a pulsed mode. Ultrasonic attenuation for the narrow acoustic pulses are very large and are also measured.

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It is generally well known that pulsed lasers of short duration  $\tau_L$  can be used to produce (via the opto-acoustic effect) acoustic pulses<sup>1</sup> of short duration  $\tau_a$ . However, the shortest detected  $\tau_a$  reported in the literature is<sup>2</sup> about 10 nsec, and other reported opto-acoustic (OA) pulses are longer despite the use of picosecond lasers.<sup>3,4</sup> A cause of large OA pulse width is the finite risetime of the acoustic detector. However, even with the use of a detector with negligible risetime, Sullivan and Tam<sup>5</sup> has shown that  $\tau_a$  is still limited by an acoustic transit time  $\tau_t$  defined as the acoustic propagation time across the OA source of length  $\ell$  in the direction of observation. The investigation of Sullivan and Tam<sup>5</sup> indicates that

$$\tau_a \approx [\tau_L^2 + \tau_t^2]^{1/2}. \quad (1)$$

Thus, even with the use of ultrafast detectors, short  $\tau_a$  can only be obtained with short  $\tau_L$  and  $\tau_t$ . For optical excitation of a highly opaque solid (or liquid),  $\ell$  is equal to the optical attenuation length for an end-on measurement (*i.e.*, normal to the solid surface); in this case, if  $\ell \approx 10^{-5}$  cm (for highly opaque sample), and  $c$ =velocity of sound  $\approx 10^6$  cm/sec,  $\tau_t \approx 10^{-11}$  sec is obtained, which means that  $\tau_a$  on the order of 10 picoseconds can be achieved in principle for sufficiently short  $\tau_L$ . In practice, factors like large attenuation, detector rise time, surface roughness, and so on, may make the detection of very narrow acoustic pulses difficult.

This paper describes a first attempt in the generation of ultra-short acoustic pulses in solids, with  $\tau_a \approx 1$  nsec, which is presently limited by the laser pulse duration, detector risetime and sample surface roughness. Such narrow acoustic pulses with highly reproducible lineshapes are ideally suitable for measuring the properties of thin films like thickness  $d$ , or acoustic velocity  $c$  and attenuation  $\alpha$ .  $c$  and  $\alpha$  are highly

correlated with physical properties of the film like porosity,<sup>6</sup> strain,<sup>7</sup> grain size,<sup>8</sup> microstructure,<sup>9</sup> and field distribution.<sup>10</sup> We demonstrate our technique on stainless steel films of thickness  $d$  as small as  $12\ \mu\text{m}$ , which is measured to 1% accuracy by our pulsed technique. In comparison, conventional ultrasonic pulsed echo techniques based on the use of transducers are not well suited<sup>11</sup> for  $d < 100\ \mu\text{m}$  because of the much longer ultrasonic pulse duration achievable.

Our experimental arrangement to demonstrate the generation and detection of OA pulses with  $\tau_a \approx 1\ \text{nsec}$  is indicated in Fig. 1. The pulsed excitation source is an atmospheric-pressure  $\text{N}_2$  laser (PRA model LN1000) producing laser pulses at  $337\ \text{nm}$  of duration  $\tau_L = 0.5\ \text{nsec}$ , and energy  $E = 1.5\ \text{mJ}$ . The laser beam is weakly focused to a spot size of about  $2\ \text{mm} \times 0.5\ \text{mm}$  on the sample, which is polished type 302 stainless steel film (Precision brand stainless steel shim from Precision Steel Warehouse, Inc., Downers Grove, Illinois) of thickness  $d$  ranging from  $12\ \mu\text{m}$  to  $260\ \mu\text{m}$ . The present technique is of course not limited to metal films; indeed we have made measurements on polymer films, which have much higher ultrasonic attenuation than stainless steel films. The OA pulse is detected "end-on" with the use of a transducer<sup>12,13</sup> coupled to the sample with a thin film of acoustic coupling material like water. The role of the acoustic coupling material is to fill the gaps (due to sample surface roughness of  $\sim 1\ \mu\text{m}$ ) at the contact interface between the sample and the transducer. The transducer, developed at Ginzton Laboratory, Stanford University, consists of an approximately  $5\ \mu\text{m}$  thick ZnO film between  $1000\ \text{\AA}$  thick gold electrodes; this whole structure was rf-sputtered on a polished surface of a single crystal sapphire buffer rod. The other end of the buffer rod has the shape of a truncated cone, with a flat polished surface of diameter about  $2\ \text{mm}$ . The transducer risetime  $\tau_r$  is  $\lesssim 1\ \text{nsec}$ , and our OA

pulse widths are partially limited by  $\tau_r$ . The transducer output is amplified by a 30 dB-gain preamp (Trontech model W1G2H, Neptune, New Jersey) with bandwidth 5-1000 MHz, and displayed on a 500 MHz oscilloscope.

The observed OA pulses for a stainless steel film of thickness  $14 \pm 2 \mu\text{m}$  (as measured by a micrometer) is shown in Fig. 2. Here, the first pulse A is due to the OA pulse that directly arrives at the ZnO sensor without undergoing any multiple reflections. The full width at half-maximum of the positive-going part of pulse A is about 0.8 sec, which is slightly larger than  $\tau_L = 0.5 \text{ nsec}$  ( $\tau_t$  is negligibly small here, on the order of  $10^{-11} \text{ sec}$ ). The exact shape of the pulse is also dependent on the response of the ZnO piezoelectric film. The time delay of A with respect to the laser firing is  $0.892 \mu\text{s}$  and is mainly due to the length of the sapphire buffer rod. Pulses B to I in Fig. 2 are due to multiple reflections at the surfaces of the sample, with the round trip time  $\tau_{RT}$  of the sample being given by the equal spacing between adjacent pulses. The value of  $\tau_{RT}$  is given in Table I. It is also clear from Fig. 2 that the pulses progressively becomes broader with more reflections, *i.e.*, more distance traversed in the sample. This is because of the phenomenon of frequency-dependent absorption, whereby higher Fourier frequency components are expected to be more strongly absorbed<sup>8,13,14</sup> in the steel sample. Fourier frequency decomposition of each of the pulses<sup>15</sup> shown in Fig. 2 should provide detailed information on the ultrasonic absorption spectrum in the hundreds of MHz range. Instead of performing this detailed ultrasonic absorption spectrum measurement, we have measured the mean effective attenuation coefficient  $\alpha$  (at the mean Fourier frequency) by plotting the heights of the pulses in Fig. 2 in a semilog plot with respect to time or propagation distance.. Diffraction effects are negligible in our data since the mean acoustic wavelength is  $\sim 20 \mu\text{m}$  and is much

smaller than the laser spot size of  $2 \text{ mm} \times 0.5 \text{ mm}$ . The resulting average attenuation  $\alpha$  at the average Fourier frequency is given in Table I.

The measurements for the nominally  $14 \text{ }\mu\text{m}$  stainless steel sample are repeated for four other thicknesses of the same material. Some results are shown in Fig. 3, and the observed round-trip time  $\tau_{RT}$  and the ultrasonic amplitude attenuation coefficient  $\alpha$  are also given in Table I.

From the measured thickness and  $\tau_{RT}$  of the thickest sample shown in Table I, the ultrasonic velocity  $c$  in the stainless steel material (rolled and polished type 302 at  $23^\circ\text{C}$ ) is found to be  $5.93(6) \times 10^5 \text{ cm/sec}$ . Using this velocity value for the other samples (made of the same material), a fitted thickness

$$d_{fit} = c \tau_{RT}/2 \quad (2)$$

can be obtained for each of the thinner samples. The results are shown in Table I. It is clear that for the thinner samples,  $d_{fit}$  is much more accurate than the directly measured thickness  $d$ , obtained by the use of a micrometer that can be read to  $\pm 2 \text{ }\mu\text{m}$ .

The effective attenuation coefficient  $\alpha$  obtained here by plotting the rate of decay of the multiple echoes is due to two causes, namely, the bulk absorption coefficient  $\alpha_b$  and the transmission factor  $s$  at the sample/transducer interface. For sufficiently small  $s$ , we have

$$\alpha = \alpha_b + (s/2d) . \quad (3)$$

Equation (3) is intuitively obvious, since  $\alpha$  is given by the effective amplitude decrease per unit path length, which is composed of the bulk loss, and the product of the number of arrivals of the sample/transducer interface to make up a unit path length and the

transmission factor out of the sample at each arrival. We found that Eq. (3) can fit most of the values of  $\alpha$  in Table I if we take  $\alpha_b = 20 \text{ cm}^{-1}$  and  $s = 0.05$ ; in this case, Eq. (3) gives the following values of  $\alpha$  in order of increasing thickness: 39.9, 29.8, 23.3, 22.0 and  $21.0 \text{ cm}^{-1}$ . These fit the effective attenuation coefficients for the four thicker samples to within 8% accuracy. However, the large difference for the thinnest sample (fitted  $\alpha = 39.9 \text{ cm}^{-1}$  versus observed  $\alpha = 73 \text{ cm}^{-1}$ ) is suggestive of a much larger value of  $\alpha_b$  for the thinnest sample.

In summary, we have made the first demonstration of the opto-acoustic generation and detection of 1 nsec acoustic pulses in condensed matter. These pulses are at least an order of magnitude narrower than those previously reported. The ultra-narrow OA pulses here are generated by  $N_2$  laser pulses of 0.5 nsec duration and detected with thin-film ZnO transducers. The ultra-narrow OA pulses are ideally suited for measuring thickness or acoustic velocity, and acoustic attenuation in thin films by observing the multiple echoes. Accuracy in thickness measurements of 1% is demonstrated for the  $12 \mu\text{m}$  thick stainless steel films once  $c$  is known; such high accuracies for thin films of  $10 \mu\text{m}$  thickness is previously impossible with pulsed transducer measurements. We also indicate the measurement of  $\alpha$ , which is known to be related to many important thin-film properties like grain size distributions and crystallinity. The measurement of position-dependent ultrasonic absorption by the present short-pulsed OA technique should provide a new imaging tool for characterizing thin film and locating defects. This is especially useful if noncontact techniques<sup>16,17</sup> with fast rise time can be developed to detect the short acoustic pulses on arrival at a sample surface, instead of using thin film transducer as done presently.

**ACKNOWLEDGMENTS**

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TABLE I

Summary of results for five samples  
of type 302 stainless steel films of different thickness.

Thickness $d$ by micrometer ( $\mu\text{m}$ )	Echo round-trip time $\tau_{\text{RT}}$ (nsec)	Ultrasonic velocity $c$ ( $10^5 \text{ cm/s}$ )	Fitted thickness $d_{\text{fit}}$ ( $\mu\text{m}$ )	Effective Ultrasonic attenuation $\alpha$ at about 300 MHz (neper/cm)
14(2)	4.24(2)	6.6 (9)	12.57(13)	73
26(2)	8.57(4)	6.1 (5)	25.4 (3)	31
76(2)	25.66(5)	5.9 (2)	76.1 (8)	23.0
128(2)	42.7 (1)	6.00(9)	126.6(13)	23.6
262(2)	88.4 (2)	5.93(6)	(reference)	19.7

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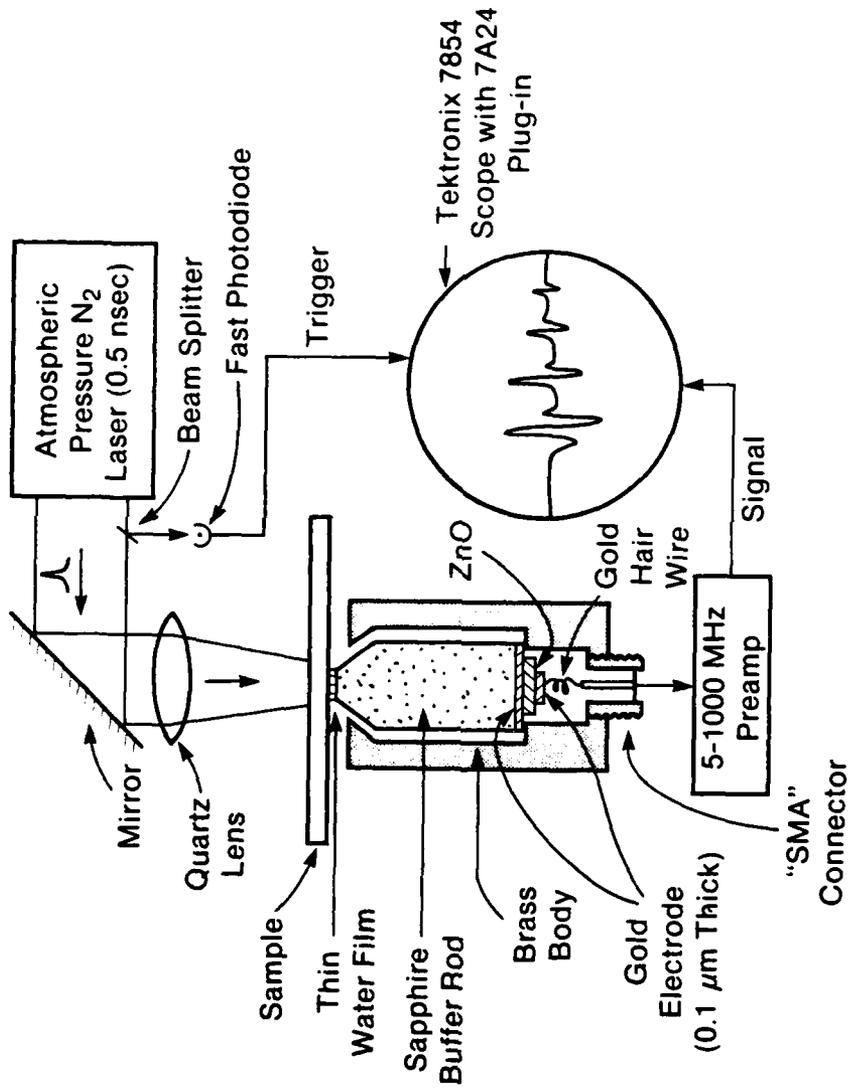


Figure 1. Experimental arrangement (not to scale) to demonstrate OA generation and detection of 1 nsec acoustic pulses in solids.

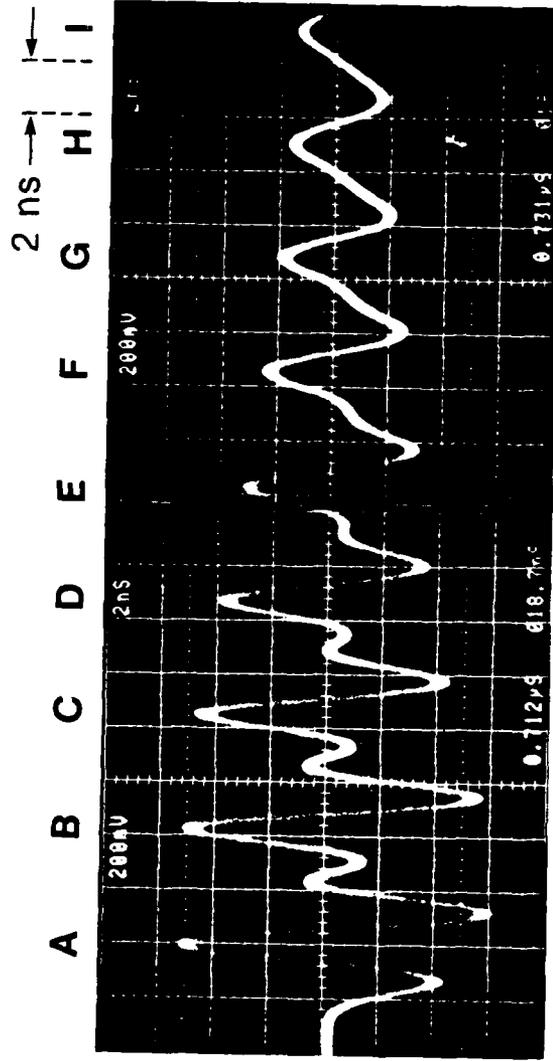


Figure 2. Observed laser-produced acoustic pulse that is multiply reflected in type 302 stainless steel film of thickness  $d (= 14 \mu\text{m})$  as measured by micrometer). The first nonreflected pulse A is delayed from the laser firing by  $0.892 \mu\text{sec}$ . Horizontal scale is 2 nsec/division.

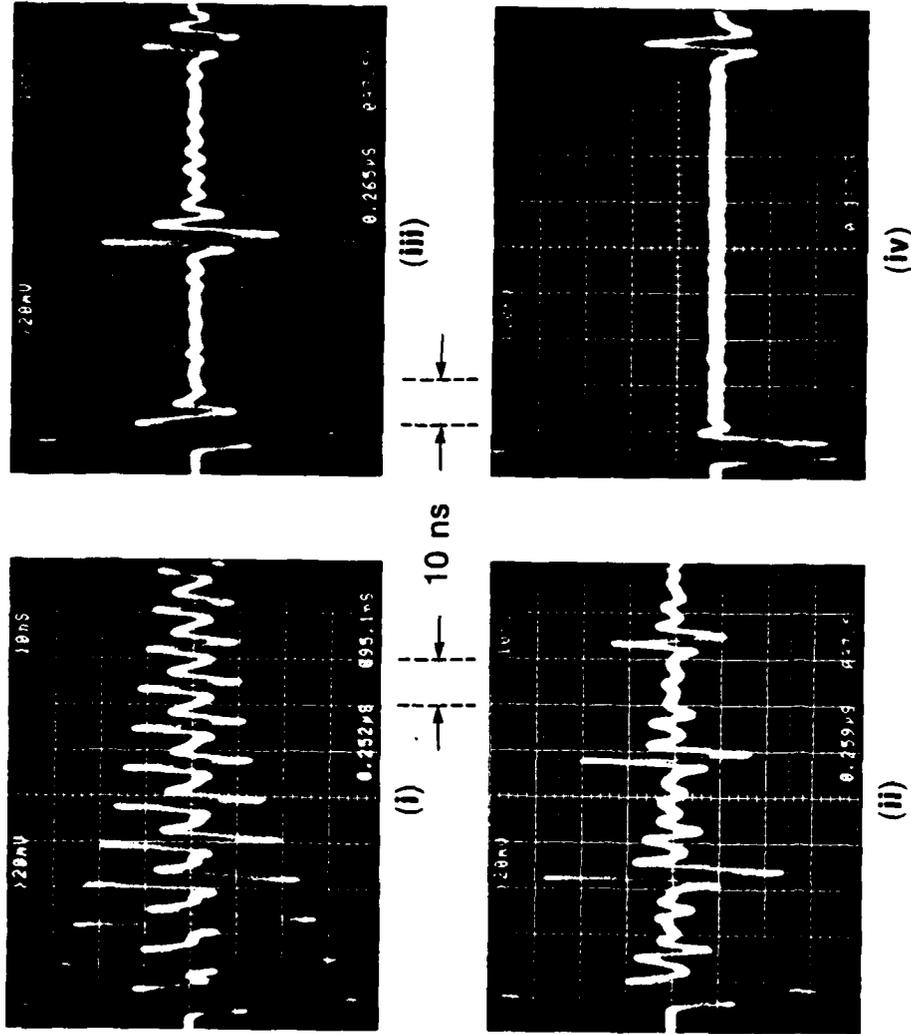


Figure 3. Observed laser-produced acoustic pulses for 302 stainless steel films of thickness  $d$  measured by micrometer. (i)  $d=26 \mu\text{m}$ ; (ii)  $d=76 \mu\text{m}$ ; (iii)  $d=128 \mu\text{m}$ ; (iv)  $d=262 \mu\text{m}$ . Horizontal scale is 10 nsec per division for all the scope pictures.

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