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Measuring Elastic Constants using Non-Contact Ultrasonic Techniques

R.S. Edwards, R. Perry, D. Cleanthous, D.J. Backhouse, I.J. Moore, A.R. Clough and D.I. Stone

Department of Physics, University of Warwick, Coventry CV4 7AL, UK

Abstract. The use of ultrasound for measuring elastic constants and phase transitions is well established. Standard measurements use piezoelectric transducers requiring couplant and contact with the sample. Recently, non-destructive testing (NDT) has seen an increase in the use of non-contact ultrasonic techniques, for example electromagnetic acoustic transducers (EMATs) and laser ultrasound, due to their many benefits. For measurements of single crystals over a range of temperatures non-contact techniques could also bring many benefits. These techniques do not require couplant, and hence do not suffer from breaking of the bond between transducer and sample during thermal cycling, and will potentially lead to a simpler and more adaptable measurement system with lower risk of sample damage. We present recent work adapting EMAT advances from NDT to measurements of single crystals at cryogenic temperatures and illustrate this with measurements of magnetic phase transitions in $Gd_{64}Sc_{36}$ using both contact and non-contact transducers. We discuss the measurement techniques implemented to overcome noise problems, and a digital pulse-echo-overlap technique, using data analysis in the frequency domain to measure the velocity.

Keywords: EMATs, elastic constants, phase transition
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INTRODUCTION

Elastic constants are one of the fundamental properties of materials, and a measurement of these can give information about the state of the material and identify phase transitions [1-9]. Ultrasonic measurements provide an inexpensive alternative to neutron scattering measurements as a probe of the elastic constants. Standard ultrasonic measurements of the elastic constants use piezoelectric transducers, requiring couplant, with measurements performed over a wide range of temperatures and magnetic fields. For these measurements an ultrasonic transducer generates longitudinal or shear waves in a sample, and the time between echoes is related to the velocity of sound within the material [2],

$$\Delta t = \frac{2L}{v} + \frac{\phi}{2\pi} \cdot \frac{1}{f} \tag{1}$$

where ν is the velocity, L is the sample thickness, f is the wave frequency and ϕ is the phase change on reflection. For a hexagonal close-packed crystal structure, such as in the Gd-Sc alloys, a longitudinal wave propagating along the c-axis will measure the C_{33} elastic constant, while a shear wave will give C_{44} [4,5,7,8];

$$C_{33} = \rho v_{long.}^2,\ C_{44} = \rho v_{shear}^2 . \tag{2}$$

Recently, non-contact methods of ultrasonic generation and detection have been developed for non-destructive testing (NDT), for example laser ultrasonics and electromagnetic acoustic transducers (EMATs) [10-12]. These also have potential for use in materials testing at cryogenic temperatures. We discuss here applications of EMAT techniques to measurements of phase transitions at cryogenic temperatures, introducing automated methods of digital data analysis. We illustrate these techniques with measurements on $Gd_{64}Sc_{36}$, an alloy which has been shown to be a simple helimagnet from its Neel temperature (around 140 K) to very low temperatures, with the turn angle locked in place by 30 K [7,8]. The single crystal was grown at the University of Birmingham and is cubic with dimensions of approximately 4 mm. We measure C_{33} as a function of temperature and magnetic field.

NON-CONTACT ULTRASONIC MEASUREMENTS

Figure 1(a) shows an experimental set-up for velocity measurements with a contact transducer such as quartz or PZT; a layer of couplant is required between the transducer and sample to allow transmission of the ultrasonic wave between transducer and sample [4]. The couplant must work over a wide range of temperatures, and repeated thermal cycling can damage the bond, reducing efficiency and ultimately leading to a loss of signal. In this case the experiment must be warmed to room temperature and the couplant replaced. Furthermore, the need for contact can lead to loading or contamination of the sample, leading to difficulties when measuring fragile materials.

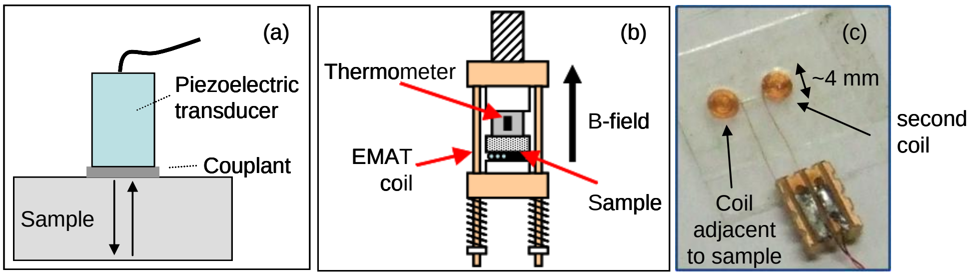

FIGURE 1. (a) Set-up for pulse-echo measurements using standard piezoelectric transducer; (b) EMAT for use at low temperatures with the sample c-axis and sound propagation parallel to the B-field; (c) differential coil EMAT (hand wound) – the magnetic field is oriented out of the page.

Non-contact ultrasonic techniques, for example EMATs, could help to remove some of these experimental difficulties when measuring electrically conducting and/or magnetic materials. An EMAT typically consists of a coil of wire plus a permanent magnet or an electromagnet. For generation of ultrasound a current is pulsed through the coil, and when held near an electrically conducting sample will induce a mirror current. The interaction of this current with the magnetic field leads, via the Lorentz force, to an ultrasonic pulse [10]. For magnetic samples the oscillating magnetic field from the current pulse again leads to a force, via magnetoelastic mechanisms. Through consideration of the magnetic field and coil configuration one can design EMATs to generate or detect desired waves. Figure 1(b) shows an EMAT developed for cryogenic measurements; again a coil is held close to a sample, with the magnetic field provided by the superconducting magnet used for changing material properties. Figure 1(c) shows a hand-wound differential coil wound using 0.08 mm diameter wire and used for both generation and detection at cryogenic temperatures. The sample is sat next to one coil, while the second coil sits in the same magnetic field and electrical environment. This allows removal of some of the electrical noise, and identification of effects which are due to the changes in the sample only as the temperature and magnetic field are varied.

COMPARISON OF CONTACT AND NON-CONTACT MEASUREMENTS

Measurements on $Gd_{64}Sc_{36}$ have been performed using a variety of experimental configurations; quartz or differential EMAT for both generation and detection, and quartz for generation with an EMAT for detection on the opposite side of the sample, working at 16.5 MHz. A typical set of echoes for EMAT generation and detection at 90 K with a magnetic field of 0.5 T applied along the c-axis is shown in figure 2(a). The EMAT currently sensitive to electrical noise from the superconducting magnet, and methods of noise reduction are being investigated. However, the echoes are clear and it is possible to measure the velocity from the time between echoes using digital analysis. The measurement of C_{33} at this temperature is shown in figure 2(b); a clear dip is observed at 1.67 T corresponding to a transition from simple helimagnet to alignment with the magnetic field. Shown also as a dashed line is the efficiency of the combined EMAT generation and detection, with the phase transition clearly measured as a significant increase in the efficiency [5]. Furthermore, the change in efficiency begins at around 0.5 T; at this temperature and magnetic field for a field applied along the a-axis we would expect a transition from helimagnetic to a fan phase [7,8]. Finally, figure 2(c) shows the behaviour of C_{33} over a range of temperatures and magnetic fields, highlighting the phase change.

$Gd_{64}Sc_{36}$ has been measured using both quartz techniques and EMATs. The measurement of the phase transition using C_{33}, attenuation and EMAT efficiency agrees very well with previously published measurements using quartz transducers, indicating the excellent potential of non-contact ultrasonic measurements of magnetic materials.

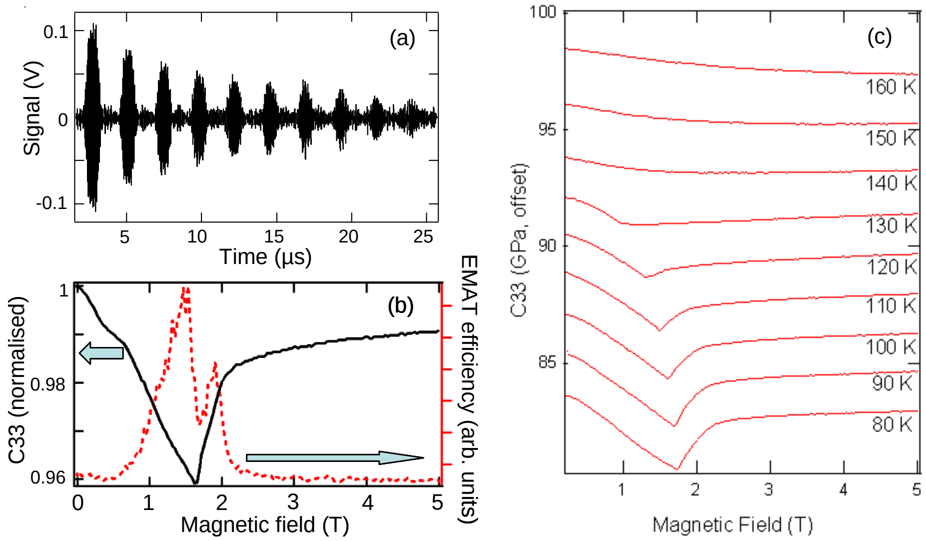

FIGURE 2. EMAT generation and detection of signals in $Gd_{64}Sc_{36}$ using a differential coil design. (a) EMAT signals at 90 K, 0.5 T. (b) measured C33 and EMAT efficiency at 90 K, (c) C33 at different temperatures and fields showing phase transition.

Digital Velocity Measurements

Pulse-echo overlap (PEO) was developed in the 1960s for measuring ultrasonic velocities [3,4]. This is now being replaced by digital methods, which allow higher accuracy and also enable data to be saved for later processing. We use cross correlation and frequency techniques to improve measurements and to remove some of the electrical noise in these EMAT measurements. Cross correlation works by windowing one echo and scanning it through the echo pattern, with areas of overlap showing as a strong signal. The advantage of this method is shown in figure 3, where the previously noisy signal becomes easy to analyse for Δt following processing.

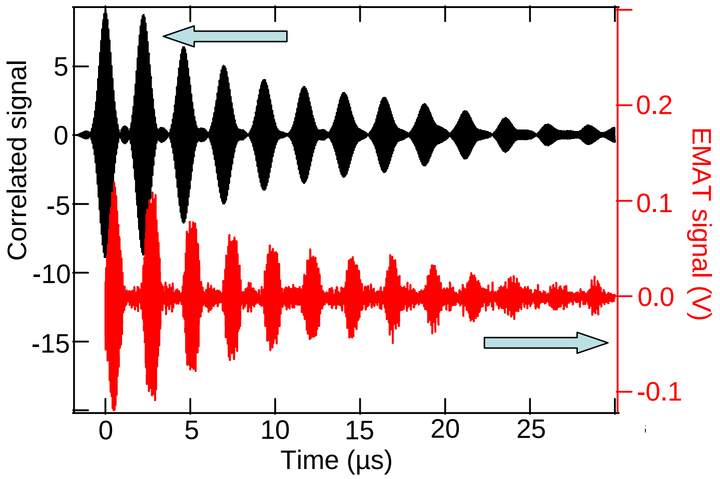

FIGURE 3. EMAT generation and detection at 0.5 T and 90 K (red). The black trace shows the cross correlated signal.

Another analysis method showing promise is the use of fast Fourier transforms (FFTs) to analyse the frequency content of the ultrasonic pulses [13]. An example of this is shown for two highly attenuated signals in figure 4, measured at room temperature and close to the phase transition. An FFT of a single echo will show the frequency content of the generated signal, and will be a narrowband signal around 16.5 MHz (green trace in figure 4(b)). However, an FFT of the entire wave pattern will show a convolution between this frequency and the frequency corresponding to the spacing in time between each echo [13]. This leads to the peaked patterns in figure 4(b) corresponding to the red and black time traces in 4(a). As the time between echoes changes the frequency spacing between the peaks will also change. The velocity of the waves can be calculated using the inverse of the frequency spacing between the peaks and the path travelled (twice the sample thickness); in the example in figure 4, the time between echoes calculated from the frequency spacing for room temperature measurements is 2.424 µs, whereas at 159 K the time between echoes is 2.487 µs, however, more accurate result requires zero-padding of the data. This can be used as an accurate measure of the velocity in samples where the electrical noise is high and signal amplitude low.

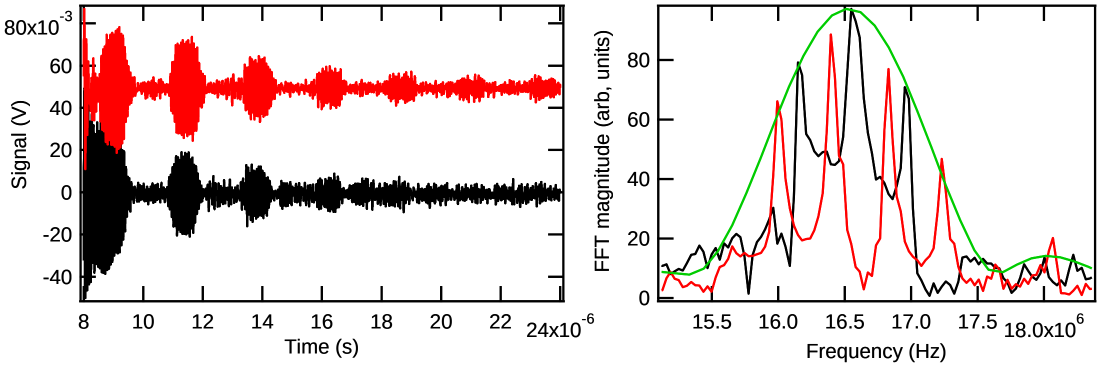

FIGURE 4. Quartz generation and EMAT detection. (a) red is at room temperature, black is at 159 K, at 1. (b) FFTs. Green shows the frequency content of a single pulse, red and black show the FFT of the full traces.

CONCLUSIONS

We have shown that electromagnetic techniques show promise for non-contact generation and detection of ultrasound in electrically conducting and/or magnetic single crystals. The resulting ultrasonic pulses can then be used to measure elastic constants and hence phase transitions, alongside measurements of the wave attenuation and the change in efficiency of the EMAT. A prototype system is in place, and we are investigating methods of noise reduction to offer a practical alternative to contact ultrasonic techniques for use on fragile samples.

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