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Characterization of Metallic and Non-Metallic Materials by Scanning Acoustic Microscopy

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Abstract

Scanning Acoustic Microscope (SAM) uses focused high frequency ultrasound to image and characterise the structural details of materials. It is used for microstructural characterisation, failure analysis, estimation of fracture toughness of ceramics and nondestructive evaluation. It has potential applications for elastic property characterisation, internal stress measurements and biological applications. A sophisticated SAM system has been procured first time in India, from M/s. KSI Germany during March 2007 and installed successfully at NDED. The operating frequency range of the system is 673 – 1023 MHz which corresponds to a spatial resolution of 1 µm. In a systematic study undertaken using SAM, to optimize the specimen preparation techniques for various materials have been optimized and surface and subsurface high resolution acoustic images of heat treated and sensitized AISI type 304 LN stainless steel and granites have been obtained and correlated.

1.0 Introduction

It is well known that, it is the microstructure of the material that controls the bulk properties. These bulk properties are mainly elastic properties. Many a time, optical and electron optic micrographs will not be adequate since they will not provide the information on the elastic properties of the constituents of the microstructure. Scanning acoustic microscope (SAM) will precisely meet this requirement. In 1949 Sokolov [1] put forward the concept of Acoustic Microscopy. Later concept was developed by Lemons and Quate [2] in the year 1974 (Stanford university). Acoustic microscopy (AM) is a recently developed method for characterization of microstructure. AM is used to reveal density, morphological and microelastic property variations in the image domain. Acoustic/ultrasonic imaging is the important tool for materials characterization. AM is analogous to optical reflection or transmission microscopy and it do not require any rigorous sample preparation such as etching, polishing, sectioning etc., as in traditional microscopy.

Using SAM, one can measure the acoustic properties of microscopic features like grain boundaries, second phases such as precipitates and inclusions and correlate them with bulk material properties. SAM can also be used for studying surface and sub-surface imaging of defects, sensitized microstructures, grain clusters, texture and strain fields, recrystallisation, phase change/ transformation, wave velocity (longitudinal and Rayleigh) in miniature specimens and elastic constants in micro specimens. Understanding SAM images needs considerable expertise on the expected acoustic contrast.

There are several techniques for acoustic microscopy, of which the Scanning Acoustic Microscopy (SAM) is unique in its image quality and resolution. By considering the application of SAM in materials characterization, attempt has been made to explain the basic principle, working and the different applications of SAM.

2.0 Working Principle of SAM

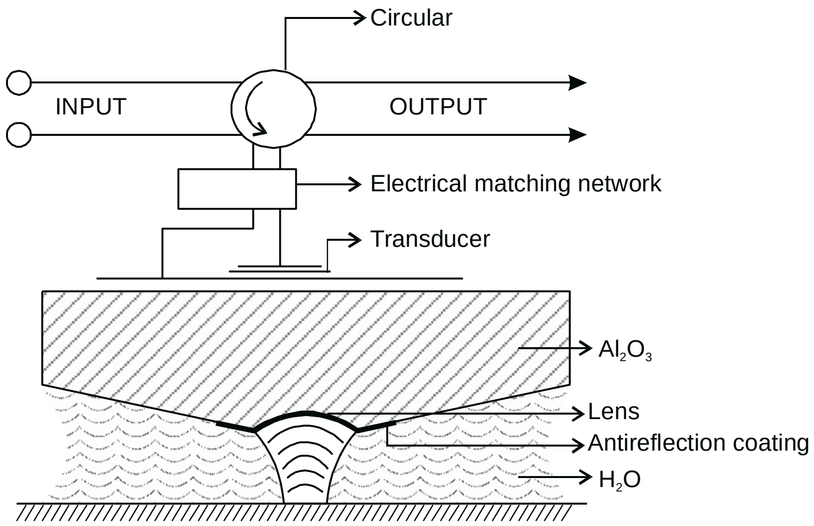


Fig.1 Basic lens geometry for reflection acoustic microscopy

The heart of the acoustic microscopy is the sapphire lens. At the back surface of the sapphire lens, a piezoelectric transducer is fixed. A short RF pulse is applied to the piezoelectric transducer, resulting in the propagation of acoustic pulse down the sapphire rod. The acoustic pulse is focused into the coupling liquid by the sapphire lens as shown in Fig.1. The focused acoustic pulse gets reflected back from the object, which is to be imaged.

In acoustic microscope, there are two modes of operation. In the first mode of operation namely reflection mode, the imaging object is placed at the focus of the sapphire lens. Therefore, the reflected acoustic waves return along the incident paths. Further, the reflected waves are converted into an electrical pulse by the transducer. The strength (amplitude) of the reflected pulse depends on the object being investigated. Thus, the amplitude is measured and is used to modulate the brightness of the display. In order to get the image of the object, the lens is scanned in raster pattern over the specimen. In the second mode i.e., the transmission mode, acoustic waves are transmitted through the object under study. The strength of the emerging waves on the other side of the object is used to study the object nature. In the reflection mode, the microscope is used to image the subsurface features, while in the second mode it is used to study the interior of thick specimens. The reflection mode is more popular than transmission mode. Due to the advancement in the field of electronics, the instrument has now possessed a high degree of sophistication. The commonly available range of resolution is 500 μm to 20 nm. The operating frequency range of SAM is 50 MHz to 1 GHz.

2.1 Acoustical Material Signature: V(z) Curve

In order to characterize these materials one has to measure the Surface Acoustic Wave (SAW) speed in the materials. The SAM is based on the principle of variation of amplitude and phase with the distance between the acoustic microscope lens and the specimen, generally called acoustical material signature or V(z) effect. The SAW is measured from the V(z) curve i.e., voltage versus defocus distance z, by moving the microscope lens vertically normal to the reflecting surface. The reflected signal voltages are recorded and it lies between a series of maxima and minima as the lens-specimen distance is varied. The observed maxima and minima are due to the phase difference between the central ray and the nonspecularly reflected critical ray that varies with the lens-specimen distance as shown in Fig.2.

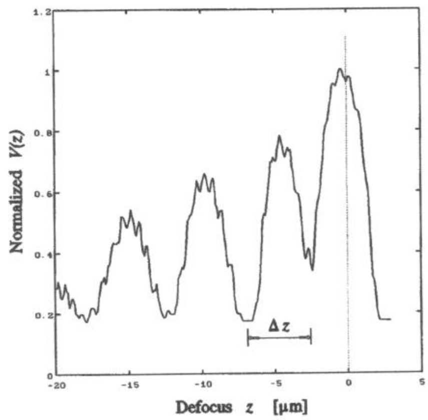


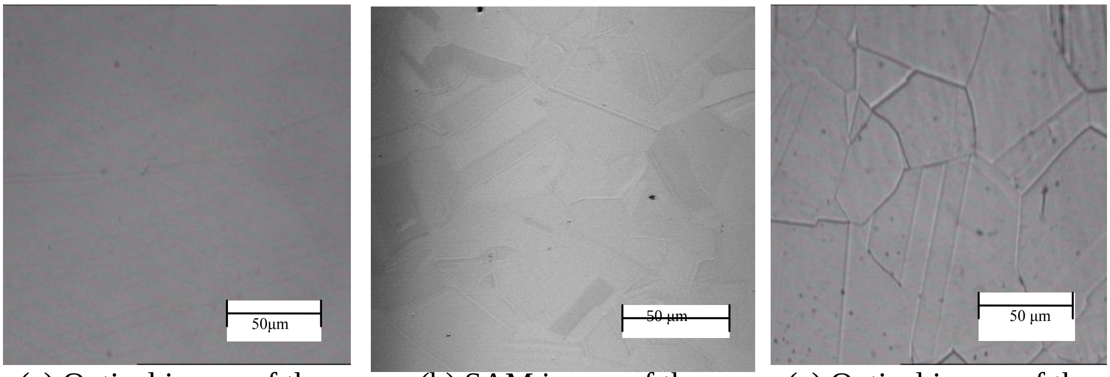
Fig. 2 Variation of voltage versus the defocus distance z

The technological development finds wide application for the NDE community for characterization of both isotropic and anisotropic materials. The development of the line focus acoustic lens finds application to study the anisotropic materials through acoustic microscopy. The materials, which are characterised through acoustic microscopy are AISI type 316 LN and a non-metallic material granite.

3.0 Experimental Studies

3.1 AISI Type 304 LN

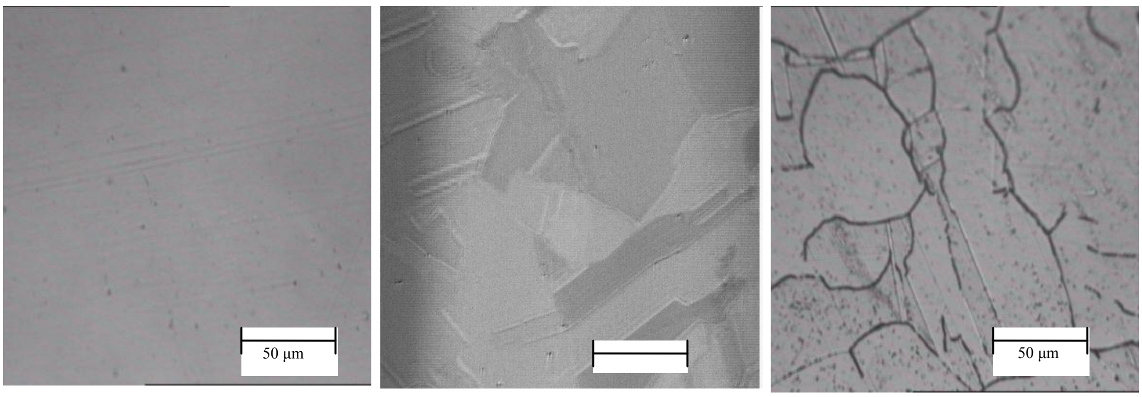
Sensitisation, in the strict sense, means ‘sensitivity, of a material to intergranular corrosion (IGC). The phenomenon of sensitization is of great practical significance because of thermal exposures during welding, fabrication, heat treatment etc. produce the metallurgical condition susceptible to intergranular attack. Austenitic stainless steel, such as AISI type 304 LN is candidate material for fast breeder reactor applications. This steel is used as fuel pin material under the 20% cold worked condition. However, the material gets sensitized when it is slowly cooled through the temperature range 450 – 850 °C or isothermally treated in the above range [3-5]. Sensitization brings down the mechanical, creep and corrosion properties of the components made out of this steel. Hence systematic studies mainly using metallographic techniques are usually followed for monitoring sensitization. However, metallographic techniques are time consuming and destructive. Moreover, metallographic techniques do not provide depth information. Hence SAM studies have been undertaken for the evaluation sensitized properties of AISI type 304 LN.



(a) Optical image of the polished sample (b) SAM image of the polished sample (c) Optical image of the etched sample.

Fig.3. Images of the not sensitized 304LN sample

Figure 3(a) shows the optical image of the polished surface of the not sensitized sample. Figure 3(b) shows the SAM image obtained by raster scanning at 850 MHz mid frequency and figure 3(c) represents the optical image of the etched AISI 304 LN not sensitized sample. Figure 4(a) shows the optical image of the polished sensitized sample. Figure 4(b) shows the SAM image obtained by raster scanning at 850 MHz mid frequency of the sensitized sample. Figure 4(c) represents the optical image of the etched AISI 304 LN sensitized sample about the temperature 700°C for 100 hrs. The SAM images reveal the broadening of the grain boundaries in sensitized sample, which is due to the formation of the chromium carbide precipitation along the grain boundaries.



50 µm

(a) Optical image of the sensitized sample (b) SAM image of the sensitized sample (c) Optical image of the etched sensitized sample.

Fig.4. Images of the sensitized 304 LN sample.

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Figure (5) show the sub-surface SAM images obtained for the same mid band frequency 850 MHz but having the defocusing at Z = 10.3μm. The grain boundaries are distinctly different due to the effect of sensitization. In the sensitized microstructure broadening of the grain boundaries take place due to the effect of stress relaxation by the depletion of chromium to the gain boundaries and the formation of chromium carbides at the boundaries. The strain created at the grain boundaries are seen as fringes.

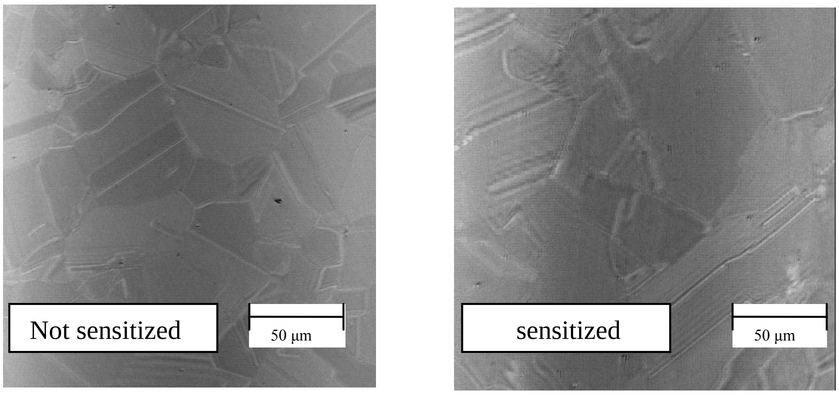


Fig.5. SAM Image defocused at Z= 10.3μm of the not sensitized and sensitized AISI 304LN

3.2 SAM Studies on Hampi Stone

The Vithala temple complex with its 56 musical pillars is indeed a splendid monument at Hampi in Karnataka. The roof is supported by huge pillars made of granite, about 15 feet height, each consisting of central pillar surrounded by detached shafts, all cut from one single block of stone. A sample of the granite stone from the broken musical pillar is taken for study of microstructure using scanning acoustic microscope. Granite is one of the hardest types of igneous rock. Granite is sometimes called "monumental stone," as many monuments are made from it. Because of the hard composition of granite, it is one of the most difficult stones to carve.

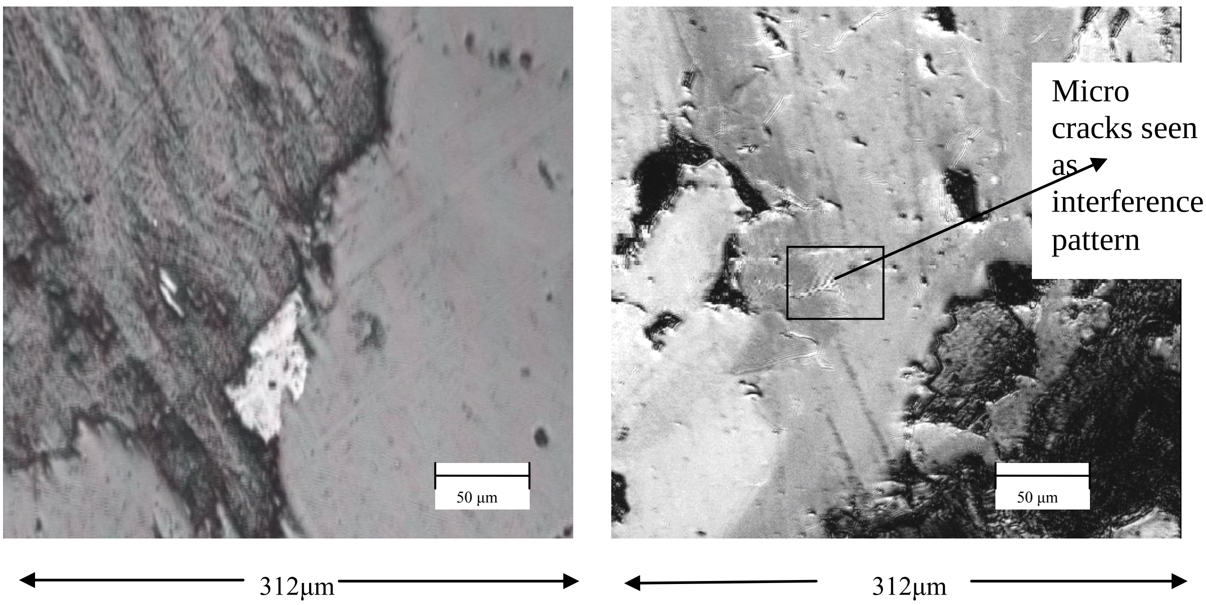
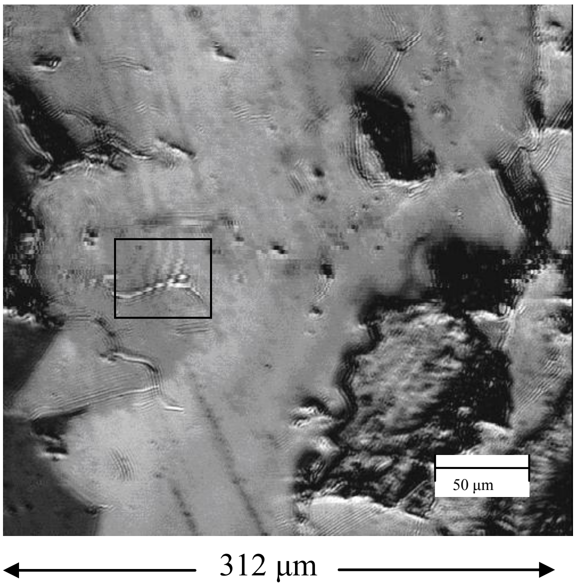


Fig.6. Optical image of Hampi stone

Fig.7. SAM image at surface (Z=0)



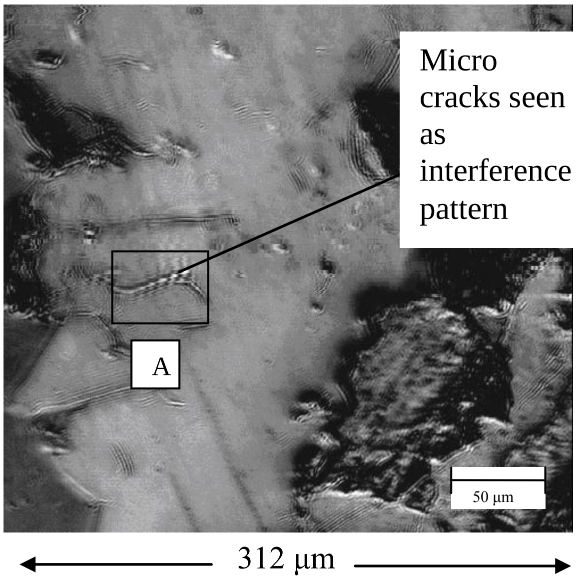


Fig.8. Image defocused at Z= 4µm

Fig.9. Image defocussed at Z=8µm

Figure 6 shows the optical image of the Hampi stone sample. Figure 7 shows the acoustic image at 850 MHz acquired using SAM. As high frequency ultrasonics have high sensitivity to surface cracks as compared to other techniques like dye penetration test, replica technique and optical microscopy. The micro cracks on the surface are enclosed in a rectangular box. The reflected ultrasonic waves interfere with the incident wave resulting in interference pattern around the crack region.

These interference patterns are more prominent at the subsurface, defocused at 4 µm (Fig.8) and 8 µm (Fig.9). These microcracks are not present in the optical image and detected using SAM proves to be a versatile technique for flaw detection at or near surface. The V(z) response of the granite stone is shown in the Fig. 10 at the frequency 851 MHz. The V(z) measurement is done at the region (A), as shown in Fig.9. From the V(z) curve the velocity of leaky surface acoustic wave is 3679m/s. The V(z) curve has a characteristic response that is dependent on the elastic properties of the reflecting surface.

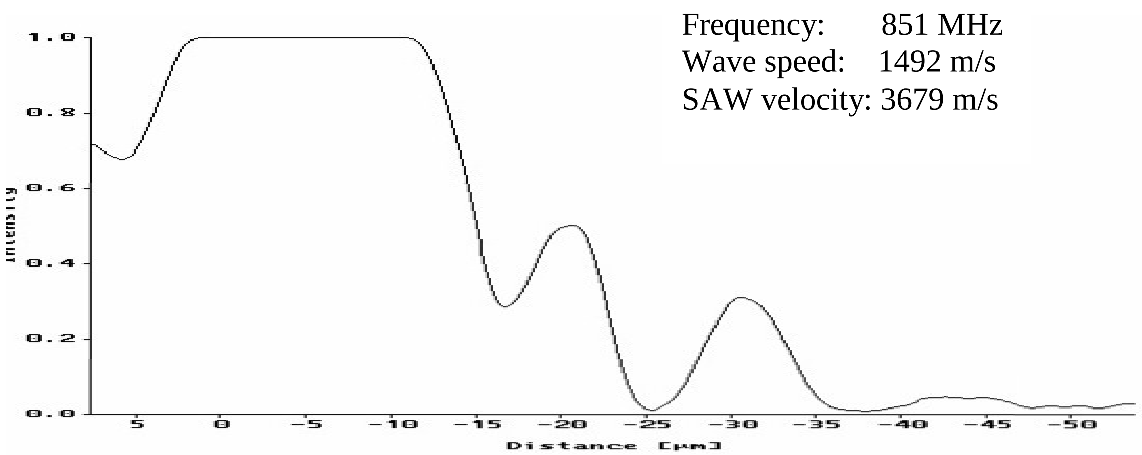


Fig.10. Typical experimental V(z)curve obtained Hampi granite stone

4.0 Conclusion

Scanning Acoustic Microscope is a non-destructive analytic tool using ultrasonic waves. In the acoustic signature mode (quantitative mode), the elastic parameters is evaluated. In the imaging mode (qualitative mode), the grain structure is revealed due to variation in the acoustic impedance across the matrix without etching. The contrast in the image is from the variation in elastic constant contributing from the different phases in the matrix. Assess to the subsurface defects, such as crack, inclusions, voids etc is possible, which makes it a versatile technique in non-destructive testing. In case of sensitized AISI type 304 LN, grain boundaries are more distinctly revealed by SAM images both at surface and sub-surfaces. In case of Hampi granite specimens, micro cracks which cannot be optically detected are revealed as interference fringes through SAM images.

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