Nondestructive Characterisation of Microstructures in Titanium Modified 20% Cold Worked 316 Type Austenitic Stainless Steel using Photoacoustic Spectroscopy

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Abstract. Photoacoustic (PA) technique is applied for the first time as a non-destructive tool to characterize microstructures in Ti modified and cold worked 316 type austenitic stainless steel. PA measurements were carried out on samples under solution annealed, cold worked and heat treated conditions. Results indicate that there is a striking difference in the photoacoustic signal patterns in case of solution annealed and cold worked samples whereas there is only a change in the amplitude of the photoacoustic spectrum observed in case of cold worked and heat treated sample. From the measured photoacoustic amplitudes, thermal diffusivity and conductivity were calculated. Results of the PA measurements correlate well with the ultrasonic velocity measurements made earlier on the similar cold worked and heat treated Ti modified stainless steel samples.

Introduction

Photoacoustic spectroscopy (PAS) is a method of obtaining the absorption spectrum of a substance by detecting and deconvoluting the periodic thermal changes in the samples in the form of the acoustic signal generated by micropressure changes in the gas surrounding the substance in a closed chamber. The main advantages of this spectroscopic technique are (i) most samples can be directly measured without treatment regardless of the state and shape, (ii) samples with poor optical characteristics of transmittance or reflection can be measured, (iii) sensitivity of measurement is enhanced when the intensity of the light sources is increased and (iv) depth wise information can be obtained.

The photoacoustic (PA) technique has been used as non-destructive and non-invasive tool for the evaluation of materials [1]. PAS in the form of photoacoustic microscopy (PAM) requires minimal or no sample preparation [2]. It has the ability to look at opaque and scattering samples and has the capability to detect the defects on the surface of thin film. PAM is a fast expanding field of research owing to its potential applications in thin film technology, medical diagnosis, semiconductor industries, etc. [2]. There is limited information on the application of photoacoustic spectroscopy as a non-destructive evaluation of microstructures in metallic materials.

We have demonstrated the potentials of PA measurements to characterise microstructures of a polycrystalline material under solution annealed, cold worked and heat treated conditions.

2. PA Technique

In this technique, a sample is placed inside a specially designed cell called photoacoustic cell, containing a suitable gas and a sensitive microphone. The sample is then illuminated with chopped radiation as shown in Fig. 1. Light absorbed by the sample is converted in part into heat by non-radiative deexcitation processes within the sample. The resulting periodic heat flow from the sample to the surrounding gas creates pressure fluctuations in the cell, which is detected by the microphone as an acoustic signal which is phase coherent at the chopping frequency. The resulting photoacoustic signal is directly related to the amount of light absorbed by the sample [3].

The information from this technique is limited to thermal diffusion length of the sample. However, by restricting the light modulation frequencies to low values, one can extend the thermal diffusion length to several hundred micrometers in samples with high thermal conductivity. Even though the technique is limited to physically thin samples, it finds special applications in non-destructive testing of thin samples and thick films [4].

PA measurements are carried out in two modes viz. [5]

(i) Depth profile analysis: Here measurements are done by keeping the wavelength fixed and changing the chopping frequencies.

(ii) Wave length scanning: i.e. by keeping the chopping frequency fixed, the wavelength of the incident radiation is changed.

3. PA Spectrometer

The present PA spectrometer consists of a 400W Xe-lamp (Jobin Yvon) as the light source. The design of PA cell is very critical to get a good signal to noise ratio. The sample is placed in the PA cell and the mike is placed very close to the sample. To get the modulated light, a mechanical chopper (Model PAR 650) is used with the source. The PA signal from the sample is fed to a lock in amplifier (Model Perkin Elmer 7225 DSP). The light is allowed to fall on the sample through a monochromator (Model Triax 180, Jobin Yvon) as shown in Fig. 1, when wavelength dependence is intended. The output of the amplifier is connected to analog or digital CRO whichever is available. We have used 5 MHz (Systronics) analog CRO (5MHz bandwidth, 1 MΩ input impedance).

4. Studies on Alloy D9

The material used in the present investigation is 15Cr-15Ni-2.2Mo-Ti modified austenitic stainless steel type 316 often called as alloy D9. This alloy is an important structural material for the Fast Breeder Reactor. This alloy is being used in the 20% cold worked condition for the specified reactor components.

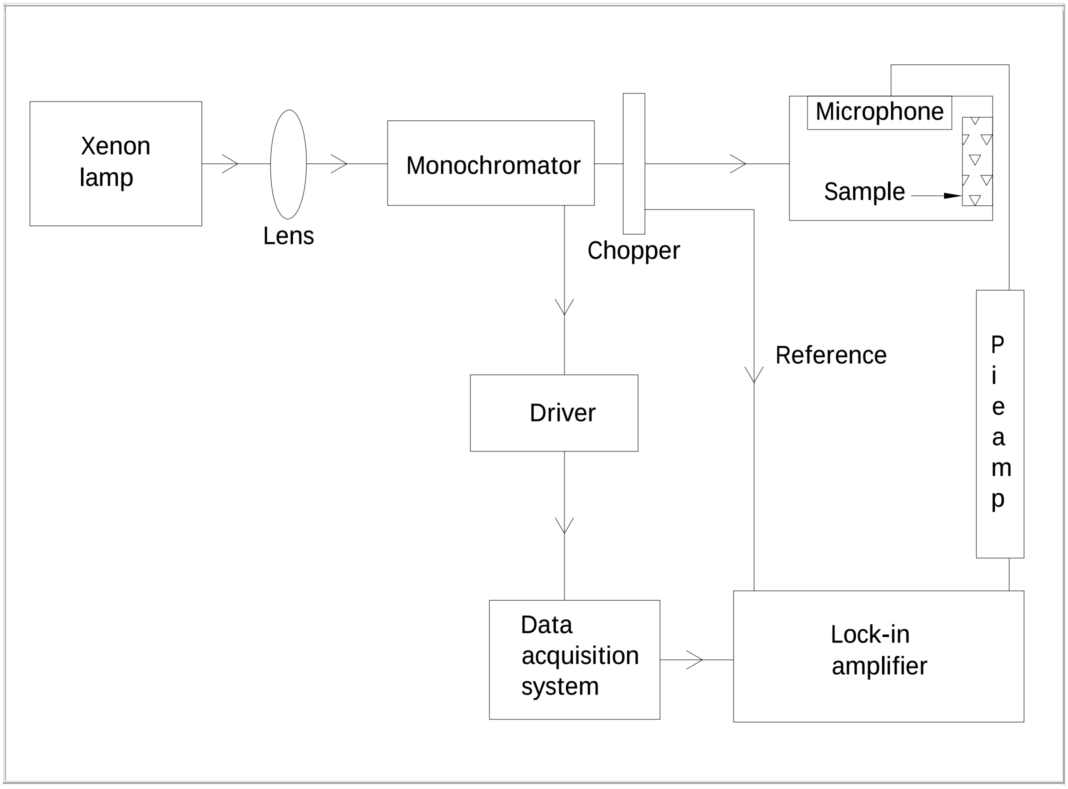

Fig. 1. Experimental arrangement for photoacoustic measurements

5.1 Sample Preparation and Metallography

Half a metre rod of alloy D9 having 12 mm in diameter was obtained and given solution annealing at 1343 K for ½ hr in vacuum furnace. It was then cut into two parts. Out of these, one rod was deformed in tension in an INSTRON tensile testing machine to impart prior cold work of 20%. Then cold worked rod was again cut into two pieces. Finally two 20% cold worked samples were obtained and out of which one sample was heat treated at 1073 K for 5 hrs duration. From each rod (under solution annealed, SA + cold worked and SA + CW + heat treated conditions), less than 1 mm thickness samples were obtained by special cutting arrangement (diamond wheel cutting) for PA measurements. 10 mm thick specimens were also obtained from each rod especially for ultrasonic velocity measurements.

Sample 1 - Solution annealed (SA),
Sample 2 - SA+20% cold worked (CW) and
Sample 3 - SA+ CW + Heat Treated for 5 hr at 1073 K.

Metallography was carried out on all samples for revealing the microstructural changes taken place due to 20% cold working and heat treatment. Figure 2 show the photomicrograph of samples 1 to 3, respectively.

4.2 PA Measurements

For obtaining PA spectrum of samples depth profile analysis has been used. Samples were positioned at the PA cell one after another and the corresponding PA spectra obtained are shown in Fig. 3. The thicknesses of the samples were kept less than 1 mm, so that the theory of PA for thin samples can be straight away applied. Typical PA spectra of the three samples are shown in Fig.3.

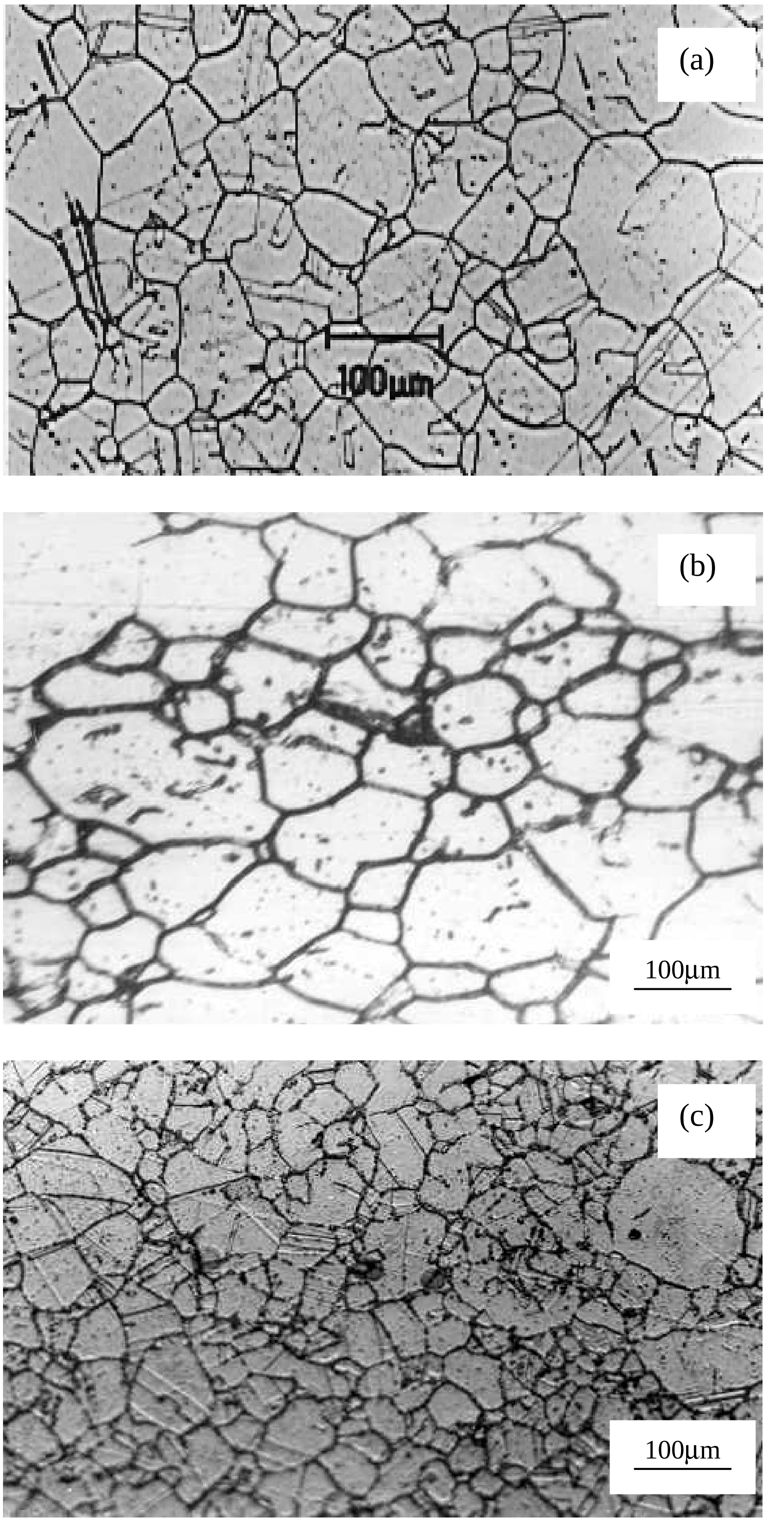

Fig. 2: Photomicrographs of (a) solution annealed, (b) SA + cold worked and (c) SA+ cold worked + heat treated at 1073 K for 5 hrs samples.

The thermal parameters, thermal diffusivity (α) and thermal conductivity (k) are calculated from these measurements using the following relations

$$\alpha = f_c\, l^2$$

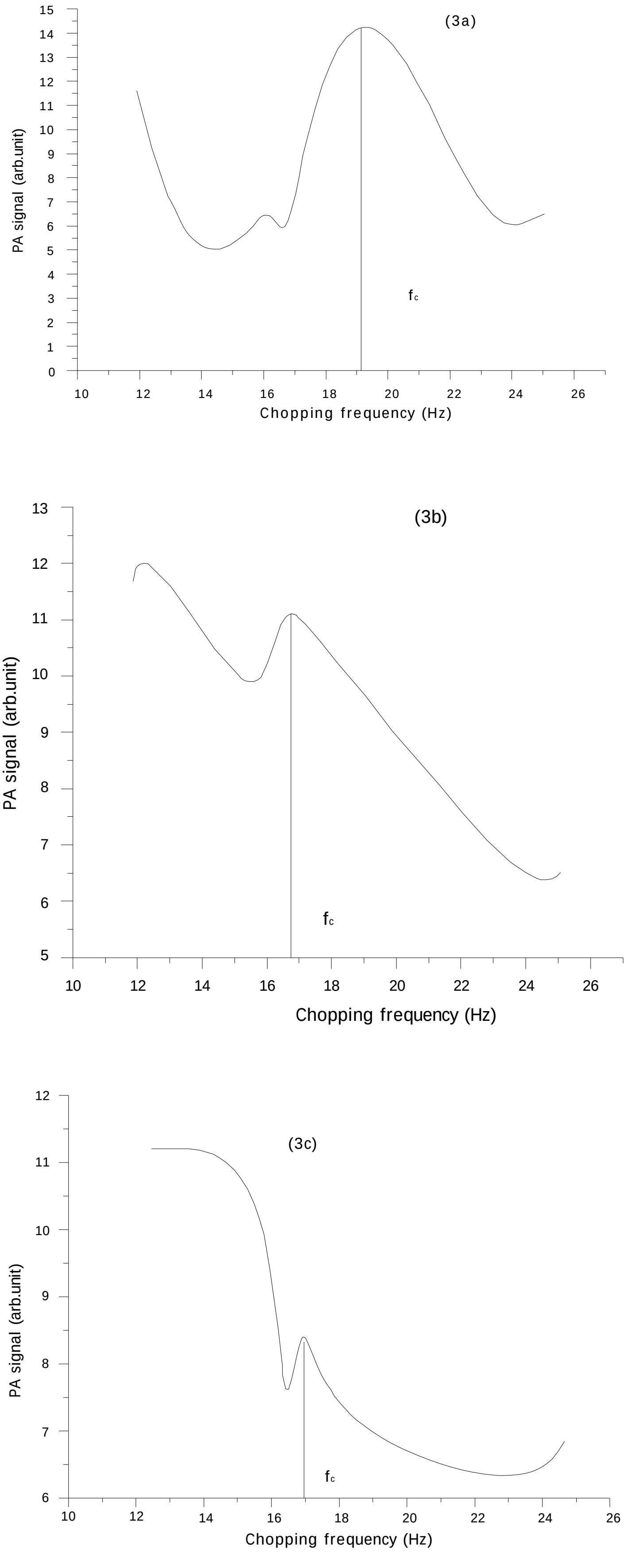

Fig. 3: Photoacoustic spectra of samples 1- 3 of solution annealed (3a), solution annealed + 20% cold worked (3b) and SA + CW + 5 hr heat treated at 1073 K (3c).

where f_c is the characteristic frequency at which the transition takes place in this alloy, and l is the thickness of the sample. Then, thermal conductivity is deduced from thermal diffusivity as

$$\alpha = \frac{k}{\rho c}$$

where ρ is the density and c is the specific heat of the sample.

4.3 Ultrasonic Velocity Measurements

Ultrasonic velocity measurements have been performed using 10 MHz normal beam broad band longitudinal wave transducer. The transducer is a piezoelectric ceramic contact type having a 10 mm diameter piezoelectric disc. Ultrasonic velocity is usually determined by measuring the time taken for the ultrasonic waves to travel the thickness of the material between the parallel faces. A precision of measurement of $\Delta V/V_0 = 10^{-3}$ is required for any meaningful application of ultrasonic velocity for non-destructive material characterisation. PC based experimental arrangement and LabView programming software has been adopted in the present study [6]. Provision has been made in the software to select the appropriate echoes for the measurements of transit time to an accuracy of ± 0.2 nano sec using cross correlation module and then displaying the velocity value with a resolution better than ± 0.01m/sec.

These ultrasonic values and thermal parameters are given in Table 1 for the three different samples of D9 alloy [6].

Table 1: Ultrasonic longitudinal velocities, thermal diffusivity and thermal conductivity of D9 alloys from photoacoustic measurements

S. No.	D9 alloy	V (ms^{-1})	Thermal diffusivity (cm^2s^{-1})	Thermal conductivity ($cm^{-1} K^{-1}$)
1	Solution annealed (SA)	5791	0.031	0.126
2	SA+20% cold worked (CW)	5905	0.022	0.088
3	SA+20% CW+ 5 hr treatment at 1073 K	5890	0.020	0.080

5. Results and Discussion

Figures 2a-c show the optical microstructures of the SA, SA + CW and SA + CW + Heat treated samples. Figure 2a shows the austenitic stress free grain boundaries along with marginal twins. Figure 2b shows the alignment of grains in the cold worked direction. In Fig. 2c, grain boundaries start disappearing while annealing twins are becoming visible. This shows recrystallisation is under progress and has attained the partially recrystallised state. Figure 3a-c show the PA amplitude spectra as function of chopping frequencies for samples 1-3. It is observed through PA spectrum that there is striking difference in the spectrum is seen between the samples 1 and 2 whereas with samples 2 and 3 there is only a difference in the PAS amplitudes. Correspondingly a shift in the characteristic frequency of the samples 1 and 2 is also observed. As confirmed by ultrasonic studies made earlier on the above samples, this striking difference in the PA spectrum indicates the deviation in the microstructures between samples 1 and 2 i.e. grain boundary elongation due to cold work (tensile pulling) or formation of texture. Reduction in the PA amplitude observed in the samples 2 & 3 indicates the microstructural reformation taken place due to heat treatment at 1073 for 5 hr. Table 1 illustrates the overall variations observed in the thermal diffusivity and the thermal conductivity parameters of all the D9 alloy samples considered in this study. Ultrasonic velocity shows an increasing trend from the solution annealed state to 20% cold worked state. After heat treatment at 1073 K for 5 hrs it shows a decreasing trend. On the other hand, thermal diffusivity and conductivity show a decreasing trends from the SA to heat treated state. This change in behaviour is attributed to the volume effect of the samples and type of measurements involved. Further studies with more number of samples prepared with intermediate temperatures are needed to understand and interpret the PA signal response with microstructural changes.

6.0 Summary and Conclusions

It is reported for the first time the application of PA measurements for microstructural characterisation on alloy D9 under solution annealed, SA+ cold worked and SA + CW + heat treated conditions. The results of the PA measurements are in close agreement with the ultrasonic velocity measurements made earlier. Observation of the PA spectrum indicates that there is distinct change in the pattern when the alloy is cold worked from the solution annealed state to the 20% cold worked condition. Photomicrographs of samples 1 and 2 are in support of this observation. In case of samples 2 and 3 there is only change in the amplitude of the PA spectrum observed and attributed to the reformation taking place due to 5 hr heat treatment at 1073K. It is concluded that PA measurements are quick and quantitative for the characterisation of microstructures in cold worked and heat treated alloy D9 and can act as a good complementary to ultrasonic velocity measurement technique in understanding the recrystallisation kinetics.

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