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In-situ measurement of Ti-6Al-4V grain size distribution using laser-ultrasonic technique

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ABSTRACT

A laser-ultrasonic system was established combining an 8 ns width pulsed laser for ultrasound generation and a two-wave mixing (TWM) interferometer for detection. Several Ti-6Al-4V samples were heat treated variously to get different grain sizes. Longitudinal wave back wall echoes were used to calculate the frequency dependent attenuation of ultrasound after the signals were denoised using wavelet transform. A model was built to calculate the log-normal distribution of grain size based on the correlation between the frequency dependent attenuation and the volumetric grain size distribution derived from electron backscattering diffraction data (EBSD) using Schwartz–Saltykov method.

Grain size is an important microstructure parameter of metallic materials affecting its mechanical [1], electrical and magnetic [2] properties directly. Grain size distribution also has a significant effect on the evaluation of microstructures [3–5]. Studies have shown that flow stress [6], yield stress [7], and hardness [8,9] depend not only on the mean grain size but also on the distribution. Ti-6Al-4V is a titanium alloy which has unusual properties relating to solution strengthening and grain refining strengthening [6,10,11]. Grain size distribution of Ti-6Al-4V can provide significant information that is of use in determining its material properties and can also assist in designing appropriate material processing parameters [12,13]. A survey of the literature [14–17] shows that, in the large majority of cases, grain size has a log-normal distribution as.

$$P(D) = \frac{1}{\sqrt{2\pi}\sigma D} \exp\left(-\frac{(\ln D - \ln \mu)^2}{2\sigma^2} \right), \quad (1)$$

where D is grain size, μ and σ denote expectation and deviation of grain size distribution respectively.

Traditional methods for metal grain size measurement including metallography and electron backscatter diffraction (EBSD) [18] are usually time consuming, and cannot meet the needs of in-situ testing which plays an important role in feedback control of material microstructure during material processing. And these two kinds of methods can only characterize the grain size in two dimension, which is actually only a section of the three dimensional grains. Therefore significant efforts have been focused on metal grain size measurement using rapid methods such as laser ultrasonics (LU) [19–27]. As a non-contact measurement technique, LU is also a very rapid method that can evaluate grain size with few laser pulses. In polycrystalline materials, the anisotropy between crystals causes scatter, and this has been deemed to be the main source of attenuation of ultrasound in such materials [28–32]. According to the relationship of wavelength λ and grain diameter D, three scattering mechanisms are commonly used [14–17,25–27,30–34].

$$\alpha(\lambda, D) = \begin{cases} C_R D^3 \lambda^{-4}, \lambda >> D (\text{Rayleigh}) \\ C_S D \lambda^{-2}, \lambda \cong D (\text{Stochastic}) \\ C_D / D, \lambda << D (\text{Diffusion}) \end{cases}, \quad (2)$$

where, C_R, C_S and C_D are material related constants. The power law relationship of wavelength, grain size and ultrasound attenuation changes in the transition zone between Rayleigh and Stochastic mechanisms [35–37]. When the grains are distributed near the transition zone, the main operative mechanism is the Rayleigh mechanisms. The wider the grain size distribution, the more the grains affect the attenuation according to the mechanism of the transition zone [35]. This fact is used in this study to find the way to calculate σ independently.

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Table 1
Chemistry composition of Ti-6Al-4V (in wt%).

V	AL	Fe	O	C	N	H	Y	Ti
3.9	6.22	0.3	0.2	0.08	0.05	0.015	0.005	Balance

Table 2
Heating temperature and holding time. No. 0 indicate original specimen.

No.	0	1	2	3	4
Temperature (°C)	–	850	950	950	1000
Time (h)	–	1	5	12	1

Table 3
Thickness of specimens after grinding.

No.	0	1	2	3	4
Thickness (mm)	3.175	3.034	2.769	2.722	3.084

The mathematical model was established using Ti-6Al-4V specimens (20 mm by 20 mm, thickness in 3.175 mm, chemical composition is shown in Table 1) for calculation of grain size distribution from ultrasound attenuation. Heat treatment processes were used to achieve different grain size distribution according to the characteristics of grain growth and phase transformation [38]. As shown in Table 2, specimens were heat to designed temperature at a speed of 5 °C/s and held for different durations in a Thermo Scientific Thermolyne FD1530M Muffle Furnace.

Since no protective gas was used during heat treatment, the specimens were ground with sandpapers as fine as 800 grit to remove the oxide layer before testing in laser ultrasonic system. The sample thickness after grinding (shown in Table 3) was measured using a digital micrometer. High frequency ultrasonic waves were generated and detected with pulsed (Q-switched Nd: YAG) laser and two wave mixing interferometer [39] respectively on each side of the samples, respectively. A pulsed laser beam with wavelength 1064 nm and pulse width 8 ns was focused to 2 mm in diameter on the sample surface which absorbs enough energy for material evaporation and generation of plasma. The ejection of plasma leads to a pulsed normal stress at sample surface. Longitudinal waves generated by the pulsed laser propagate along the thickness direction and reflect between both surfaces of Ti-6Al-4V plate. Each sample was detected using LU at least 20 times on different positions of the sample surface at room temperature (25 °C).

The detected signal was denoised using wavelet transform and further processed by subtracting the polynomial fitted baseline, as shown in (a) of Fig. 1. Fast Fourier Transformation was used combined with a Gaussian window to obtain the amplitude of each echo in the frequency domain. As shown in (b) of Fig. 1 the eight echoes have a central frequency around 25 MHz.

The contribution of ultrasound attenuation coming from diffraction should be removed since that has no relation to grain size [40,41]. In this study, the laser induced ultrasound comes from the normal stress source of 2 mm disc and spreads as spherical waves. Researches have shown that the ultrasound amplitude drops off as 1/z as waves spread [42,43]. Considering the attenuation caused by scattering and diffraction loss, the travelling longitudinal waves can be expressed as [44,45].

$$u(z,t) = \frac{A_0}{z}\exp(ik(z-\omega t)-\alpha z), \tag{3}$$

where A_0, z and α denotes the amplitude, travelling distance and

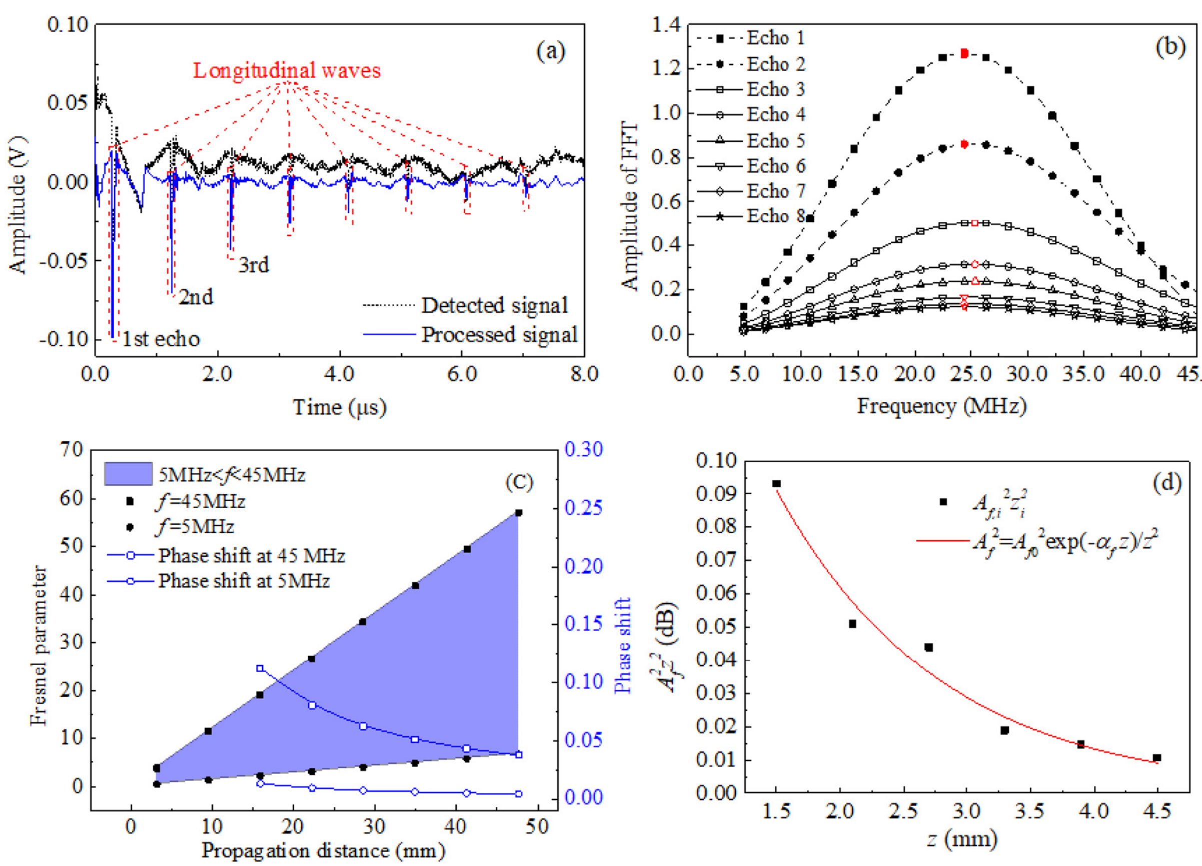

Fig. 1. Signal processing methods: (a) Ultrasound signal received by TWM interferometer; (b) FFT results of every longitudinal echo (central frequency is marked as red); (c) Diffraction effects on ultrasound amplitude spectrum at each echo (phase shift can be calculated at far field where spherical wave hypothesis is utilized); (d) attenuation rate fitted by multiple echoes. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)