46th Annual Review of Progress
in Quantitative Nondestructive Evaluation
QNDE2019
July 14-19, 2019, Portland, OR, USA

QNDE2019-1234

NONLINEAR ULTRASONIC TECHNIQUE FOR THE QUANTIFICATION OF DISLOCATION DENSITY IN ADDITIVE MATERIALS

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ABSTRACT

This research applies nonlinear ultrasonic techniques for the quantitative characterization of dislocation density in additive materials. The characterization is based on quantifying dislocation density resultant from the additive manufacturing process in order to increase the confidence in a printed part and help in a qualification process. Second harmonic generation techniques based on the transmission of Rayleigh surface waves are used to measure the acoustic nonlinear parameter, β, which is a proven indicator of dislocations, but has not been fully demonstrated for additively manufactured materials. Laser Powder Bed Fusion and Laser Engineered Net Shaping parts are compared with corresponding wrought stainless steel parts. An annealing heat treatment routine is used to reduce dislocation density in each part and evaluate the sensitivity of β to these changes. The acoustic nonlinearity parameter is found to be sensitive to the changes in dislocation density for the studied additively manufactured metals.

Keywords: Nonlinear ultrasonics, second harmonic generation, Additive Manufacturing

NOMENCLATURE

β	acoustic nonlinearity parameter
A_1	fundamental wave amplitude
A_2	second harmonic amplitude
x	propagation distance
κ	wavenumber

1. INTRODUCTION

Additive manufacturing (AM) can provide unique benefits over traditional manufacturing techniques. Prototypes and one-off parts can be quickly manufactured; complex geometries can be produced with tailored mechanical responses. These possibilities have generated interest in AM techniques in many industries, including energy and defense. However, adoption in critical scenarios has been slowed by the uncertainty associated with AM. Different print iterations of the same part can have varying mechanical properties depending on print rate, bulk material properties, and cooling rates.

High temperature gradients formed during the printing process can lead to increased dislocation density [1]. These dislocations can have an effect on the ductility, strength, electrical and optical properties of the finished part [2]. The potential performance variability limits the application of AM parts in highly critical scenarios. This research explores the viability of non-destructive evaluation (NDE) techniques to quantify the dislocation density within a printed part. This information could be used in a multi-physics approach to qualifying an AM part before it is used. A robust certification of printed parts is important to increase confidence in AM and its adoption into critical industries.

Nonlinear ultrasonic (NLU) techniques have been shown to be sensitive to microstructural changes, including dislocation density [3]. A sinusoidal wave is allowed to propagate through a material, and its interaction with the microstructural features causes the generation of waves at the second harmonic of the input. This second harmonic generation is quantified with the acoustic nonlinearity parameter β as derived by Herrmann et al. [4]

$$\beta \propto \frac{8A_2}{A_1^2 x \kappa} \qquad (1)$$

Equation 1 shows the proportionality of the relative acoustic nonlinearity parameter, β, to the ratio of the second harmonic, A_2, to the fundamental, A_1, squared. β is a relative parameter that assumes values of attenuation and diffraction to be constant for a sample. The attenuation assumption is checked with a corresponding set of linear ultrasonic attenuation measurements. The effectiveness of the proposed NLU techniques can also be evaluated in comparison to the linear ultrasonic attenuation measurements. The viability and effectiveness of NLU techniques for AM parts has not been fully explored.

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2. MATERIALS AND METHODS

Two different AM techniques are investigated in this study. A 316L AM specimen is made through Laser Powder Bed Fusion (LPBF) and a 304L AM specimen is made through Laser Engineered Net Shaping (LENS). In LPBF, a laser beam is used to fuse a small region of already deposited powdered material. Once an entire layer has been finished, the print bed will lower and a new layer of powdered bulk material is spread onto the print bed and the process is repeated [5]. LENS is a specific technology under the Directed Energy Deposition ASTM classification of AM. High-powered lasers are used to heat a substrate onto which a powdered bulk material is placed. The energy and material inputs are co-located allowing for complex shapes that benefit from spot-by-spot articulation [5].

As a comparison to each AM technique, a corresponding wrought specimen is also tested, resulting in a total of four specimens as listed in Table 1.

TABLE 1. MANUFACTURING TECHNIQUES AND METAL VARIANT

	Stainless Steel Grade	
	316L	**304L**
AM	316L LPBF	304L LENS
Wrought	316L Wrought	304L Wrought

The thinnest specimen measures at 1 cm; Rayleigh wave displacement is negligible beyond depths of 1.5λ [6]. With this experimental setup, wave displacement is negligible beyond 0.396 cm; therefore, reflections from the bottom surface of the specimen are ignored. All specimens are hand polished to a finish of 1500 grit to increase signal-to-noise ratio.

2.1 Heat Treatment

Dislocation density is changed in each of the specimens through a prescribed heat treatment regimen. Following the work by Smith et al. [7], this annealing profile leads to a decrease in dislocation density as predicted by the decrease in geometrically necessary dislocations (GNDs) [8]. For both AM and wrought 316L samples, gradual steps are taken in the heat treatment process to allow for finer gradation on the microstructural changes in relation to β measurements. The final heat treatment for each specimen is meant to cause a complete recrystallization in the material. Surface oxidation is minimized through the use of an inert atmospheric furnace and near-vacuum stainless steel foil bags. Following the heating, all specimens are air cooled at room temperature. Surface preparations of sanding are repeated following each heat treatment step.

TABLE 2. HEAT TREATMENT PROFILE

Sample	HT 1	HT 2	HT 3	HT 4
304L LENS	1325 K; 0.5 hr	1473 K; 2.5 hr	N/A	N/A
316L PBF	923 K; 0.5 hr	1223 K; 0.5 hr	1323 K; 0.5 hr	1473 K; 2.5 hr
316L Wrought	923 K; 0.5 hr	1223 K; 0.5 hr	1323 K; 0.5 hr	1473 K; 2.5 hr
304L Wrought	1325 K; 0.5 hr	1473 K; 2.5 hr	N/A	N/A

2.2 Rayleigh Wave Experimental Setup

The schematic seen in Fig. 1 illustrates the Rayleigh wave setup used in this study. A 2.1 MHz contact transducer is coupled to an acrylic wedge with a thin layer of a petroleum based oil. The transducer is connected to a function generator to produce a tone burst signal with a peak to peak voltage of 800 mV. 20 cycles are generated with a burst period of 20 ms. A high powered gated amplifier, the RITEC GA-2500A, is used to increase the signal-to-noise ratio. The wedge is then coupled to the top surface of the specimen. The entire setup is allowed to rest for 30 minutes prior to any measurements to reduce settling effects from the couplant and instrumentation startup.

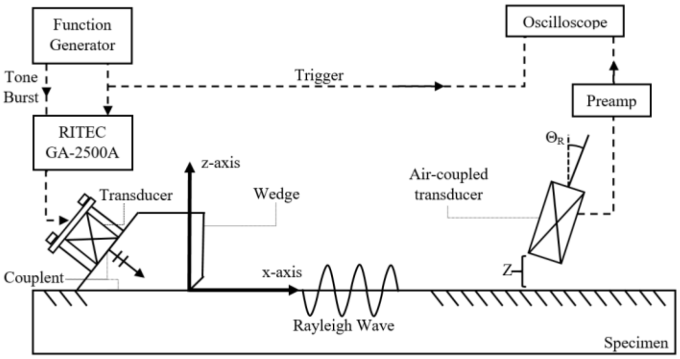

FIGURE 1: RAYLEIGH SURFACE WAVE NONLINEAR ULTRASONIC MEASUREMENT SETUP

A Rayleigh surface wave is generated and travels across the top surface of the specimen. Leaked Rayleigh waves are measured by an air coupled transducer placed 3 mm from the surface. This transducer is set in a moving stage that allows for three degrees of translation and one degree of rotation. A calibration routine is performed to find the path of maximum first harmonic amplitude propagation.

The signal is averaged 512 times and post amplified to improve signal-to-noise ratio. Transient effects of voltage overshoot at the beginning and ringing at the end of the signal are reduced by implementing a Hanning window [9]. The frequency response of the recorded signal shows a clear distinction between the fundamental harmonic and the second harmonic. These amplitudes are studied as a function of propagation distance. A relative value for β is found from the slope as follows from Equation 1.

3. RESULTS AND DISCUSSION

Rayleigh wave measurements are performed for each sample at each stage of the heat treatment plan. The results of these measurements before the recrystallization heat treatment are plotted in Fig. 2. β is normalized to the lowest value measured for each specimens, as only the relative differences between each specimen is of importance.

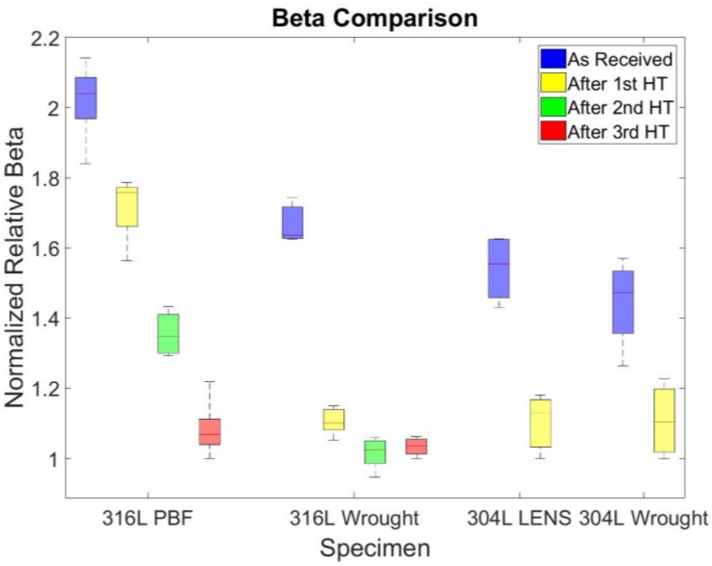

FIGURE 2: A COMPARITIVE BOXPLOT FOR NORMALIZED β VALUES. EACH BOXPLOT REPRESENTS FIVE NONLINEARITY MEASUREMENTS.

β is shown to decrease in each sample following heat treatment. This pattern matches the hypothesized behavior as it is predicted to be an increasing function of dislocation density [3]. In both AM specimens, the decrease in β is greater than their wrought counterpart; larger temperature gradients during the AM process contribute to a chaotic microstructural environment that can cause an increase in dislocation density [7].

The microstructural effects of the annealing heat treatment are inspected with microscopy and a hardness measurement at each stage of the heat treatment. Electron backscatter diffraction (EBSD) provides a measure of the GNDs which can be used to verify the purported changes in dislocation density from the heat treatment. Further corroboration of these changes are given by a Rockwell hardness measurement at each stage of the heat treatment cycle. Linear ultrasonic attenuation measurements show some sensitivity to the microstructural changes at high frequencies, but overlap between successive heat treatments may indicate a reduced sensitivity in comparison to the β measurements.

4. CONCLUSION

The nonlinear acoustic parameter shows high sensitivity to the predicted changes in dislocation density in additive manufactured materials. Similarly, the linear ultrasonic attenuation shows some sensitivity to these changes at high frequencies. The effectiveness of NLU for AM materials indicates the viability of its use in a qualification process following the printing process.

ACKNOWLEDGEMENTS

Financial support and materials for this research are provided by Sandia National Laboratories. Sandia National Laboratories is a multimission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International, Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA-0003525. SAND 2018-10721 C.

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