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GRAIN SIZE MEASUREMENTS USING ULTRASONIC BACKSCATTERING: ACCURACY AND PRECISION

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ABSTRACT

The grain size of alloys is one of the key material properties that controls mechanical strength. The grain size is carefully controlled during processing to create the desired mechanical properties and reliability in the final component. Traditional metallographic grain size measurements are destructive, time consuming and labor intensive. In addition, they only sample a small region of a component and cannot be performed on every component. Ultrasonic scattering measurements are excellent at probing metal microstructures and providing grain size if appropriate theories exist. We developed software that unifies the entire process including the data collection, backscattering coefficient calculation, theoretical predictions, grain size calculation, and visualization. We will report on the accuracy of the grain size measurements which was within the aircraft engine industry standard of ±1 ASTM # for all samples with grain size below ~50μm including "blind" samples, whose grain size we did not know. We will also report on the precision of the grain size measurements in equiaxed nickel alloy based on a systematic parametric study of the effects of various measurement and analysis parameters

Keywords: Ultrasound, scattering, grain size, materials characterization

1. INTRODUCTION

Nickel-based superalloys are used extensively in aerospace engines. As the engine manufacturers move to higher operating temperatures and the engines age, there is a need to characterize the grain size in these components both during manufacturing and operation. Novel ultrasonic scattering methods and software tools were developed to measure the grain size in dual microstructure disks and other nickel alloys. A first-of-its-kind "ultrasonic metallography software program" was created to measure the backscattering from metal alloys, predict the backscattering from existing theoretical formulations, and calculate the grain size. Furthermore, the ultrasonic scanning tank control, data acquisition, calculation of the measured ultrasonic attenuation and backscattered grain noise, theoretical prediction of the backscattered grain noise, and the optimization to calculate the grain size were integrated into this single software platform.

2. MATERIALS AND METHODS

When an elastic wave propagates through a metal, it is affected by the microstructure in many ways. Specifically, energy is absorbed as heat is generated by the movement of defects, such as dislocations and point defects. The energy can also be scattered in all directions because of the acoustic impedance mismatch at grain boundaries caused by the change in orientation of single crystals across the grain boundary. These energy-loss mechanisms attenuate the amplitude of the propagating wave. The portion of the energy that is scattered directly back to the transmitting transducer is called backscattering and can look like background "noise" during ultrasonic inspections. Because the backscattering from the grain boundaries resembles electronic noise, it is sometimes called backscattered grain noise. The grain morphology (size and shape) is the most dominant feature affecting attenuation and backscattering. In addition to the grain morphology, the attenuation and backscattering are also sensitive to many other microstructure properties, including the texture (long-range crystallographic orientation), residual stress, point defects, precipitates, and second phases. Whereas all of these parameters are important, for the purposes of this work, the focus was to use the backscattering from grain boundaries to measure the grain size in cubic nickel alloys (assuming they had equiaxed grains and no texture or secondary phases).

The theory describing the ultrasonic backscattering as a function of frequency was developed by Jim Rose [1]. The backscattering coefficient $\eta(\omega)$ is described by the equation:

$$\eta(\omega) = \left(\frac{\omega^2}{4\pi\rho V^4}\right)^2 \int d^3\left(\vec{r}-\vec{r}'\right)\left\langle \delta C_{3333}\left(\vec{r}\right)\delta C_{3333}\left(\vec{r}'\right)\right\rangle e^{2ik(\vec{r}-\vec{r}')\cdot\hat{k}}$$

To measure the grain size, the ultrasonic backscattering amplitude must be transformed to the backscattering coefficient predicted by the theory. To calculate the backscattering coefficient as a function of frequency from the measured backscattering amplitude in the time domain, capture a reference signal to help eliminate various measurement parameters including the transducer efficiency. This method has been described though many publications [2] and will not be described here due to space limitations.

TABLE 1. SINGLE CRYSTAL ELASTIC CONSTANTS USED FOR THEORETICAL CALCULATIONS OF BACKSCATTERING IN NICKEL ALLOYS [3].

Alloy	C_{11} (Gpa)	C_{12} (Gpa)	C_{44} (Gpa)	Anisotropy
IN718, Rene 88, Rene 104	244.37	137.66	101.96	1.91
Udimet	242.18	138.85	104.20	2.02
Waspaloy	267.74	141.38	103.71	1.64

2.1 Grain size calculation

The resultant backscattering coefficient as a function of frequency from the measurement is shown Figure 1 as blue points in with the theoretical prediction of the backscattering coefficient as a green line. To determine the grain size, the difference between the experimental and theoretical backscattering coefficients is minimized over the frequency "fit region" with the grain size as the adjustable parameter. In the case shown, the fit region was chosen by eye for the closest overlap of the experiment and theory within the bandwidth of the transducer. The grain size was determined when the difference was at a minimum, causing the fit to be "optimized." This example shows the results for IN718 in which the grain size was determined to be 18.5 μm (ASTM #8.6) using a least squares minimization method. Details of this process are published in reports by the authors [4]

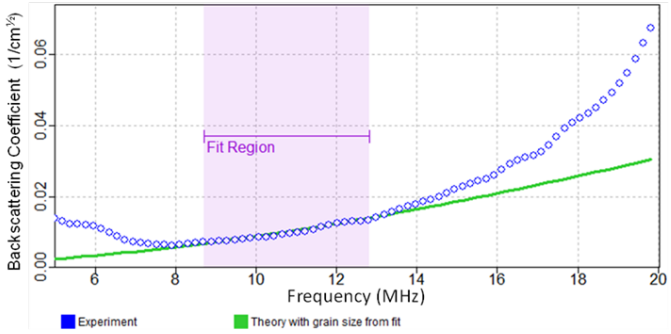

FIGURE 1: THE EXPERIMENT AND THEORETICAL BACKSCATTERING COEFFICIENT OVER THE BANDWIDTH OF THE TRANSDUCER. THE EXPERIMENTAL DATA IS IN BLUE POINTS AND THE THEORETICAL PREDICTION IS A GREEN LINE.

2.2 Validation

The grain size determined from the backscattering versus the grain size from metallography is shown in Figure 2. The two sets of dotted lines show the range of ±0.5 ASTM # and ±1 ASTM #. Currently, engine manufacturers accept specimens that have a range within ±1 ASTM # of specified grain size.

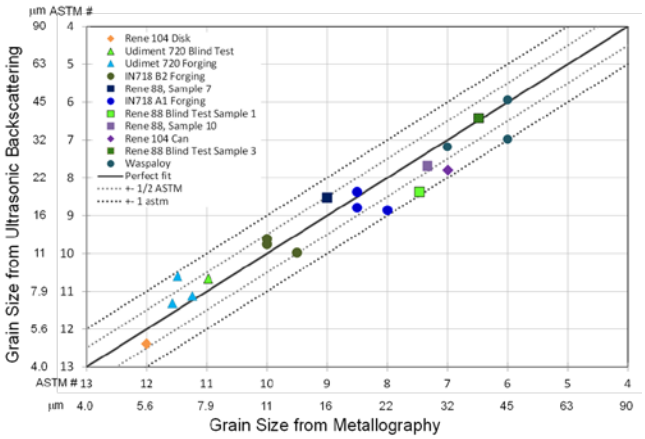

FIGURE 2. GRAIN SIZE FROM ULTRASONIC BACKSCATTERING MEASUREMENTS COMPARED TO DIRECT METALLOGRAPHY.

2.3 Parametric study

Accurately determining the grain size is an important step in realizing implementation of these measurements in production environments. Accuracy in grain-size measurement is largely controlled by how accurately the theory predicts backscattering. Another important step is to determine the precision and time needed for measurement and analysis processes.

For the data shown in Figure 2 great care was taken to ensure all data are collected with the highest precision possible. The process to set up backscattering measurements can be as long as an hour. For laboratory measurements, an hour per measurement is reasonable. However, a set-up time of approximately 5 minutes is expected/allowed in production environments. Therefore, the program included a parametric study to determine whether the precision of the setup and analysis could be relaxed to speed up the process while still keeping fidelity high enough to stay within ±1 ASTM # of the most precise measurement of grain size. In this parametric study we determined the effects of the 15 parameters below on the accuracy and precision of the grain size measurement

1. Repeatability 1 person
2. Reproducibility through round robin tests
3. Electronic averaging
4. Normalization of transducer
5. Sample orientation relative to scan axis
6. Water path for reference material
7. Transducer properties
    a. Band width
    b. Beam diameter
    c. Depth of field
    d. Focal length
    e. Frequency

f. Excitation pulse length and type
g. Frequency range within bandwidth of transducer

8. Length of time gate for backscattering calculation
9. Attenuation and absorption effects
10. Focal depth
11. Single crystal elastic constants
12. Depth dependence from single scan

3. RESULTS AND DISCUSSION

The results of the parametric study are shown in Table 2.

TABLE 2. RESULTS FROM THE PARAMETRIC STUDY

Parameter	Range studied	Grain size within ±1 ASTM # for range studied Range for grain size to be within ±1 ASTM #	Effects on grain size
Repeatability	1 user, 2 measurements	Yes	0.7 μm for 19 μm sized grains (4%) 0.1 ASTM # for ASTM # 8.5 sized grains (1%)
Reproducibility	4 users 3 different locations 3 different pulser receivers	Yes	±1.7 μm for 18.1 μm sized grains (9%) ±0.3 ASTM # for ASTM # 8.6 sized grains (3%)
Electronic averaging	1 to 256 waveforms	Yes	-1.7 μm to 3.9 μm for 18.1 μm sized grains (-9% to 22%) 0.3 ASTM # to -0.6 ASTM # for ASTM # 8.6 sized grains (3% to -7%)
Alignment of transducer to sample	±2°	Yes	-1.1 μm to 2.1 μm for 20.2 μm sized grains (-5% to 10%) 0.2 ASTM # to -0.3 ASTM # for ASTM # 8 sized grains (3% to -4%)
Alignment of transducer to reference sample	±2°	No Angle must be within -1.25° to 1.5° of normal for this sample and transducer	12.4 μm to 19.2 μm for 13.5 μm sized grains (92% to 142%) -1.9 ASTM # to -2.6 ASTM # for ASTM # 9.5 sized grains (-20% to -27%)
Sample orientation relative to scan axis (level of sample)	1.8° in one direction 2° in second direction	Yes	-2.0 μm to -4.5 μm for 14.9 μm sized grains (-1% to -30%) 0 ASTM # to 1 ASTM # for ASTM # 9.2 sized grains (0% to 11%)
Water path of reference signal	-1 cm to +4 cm from focused on back surface	Yes	-2.0 μm to -4.2 μm for 12.9 μm sized grains (-9% to -19%) 0.3 ASTM # to 0.6 ASTM # for ASTM # 8 sized grains (4% to 8%)
Ultrasonic frequency and transducer focal properties	Freq (MHz): 5, 10, 15 FL (cm) 17.1, 10.5, 10.7 F#: 8.9, 8.2, 8.4	Yes	2.3 μm to 5.6 μm for 12.9 μm sized grains (18% to 43%) 0.5 ASTM # to 1 ASTM # for ASTM # 9.6 sized grains (5% to 10%)
Frequency region for theoretical fitting	8.2 MHz to 17.5 MHz five 1.9 MHz long regions	Yes	-0.1 μm to 1.7 μm for 13.6 μm sized grains (-1% to 13%) 0 ASTM # to -0.3 ASTM # for ASTM # 9.4 sized grains (0% to -4%)
Length of the backscattering time gate	1 μs to 6 μs	Yes	-0.1 μm to 1.7 μm for 13.6 μm sized grains (-1% to 13%) 0 ASTM # to -0.3 ASTM # for ASTM # 9.4 sized grains (0% to -4%)
Attenuation	3 samples	Yes	-0.9 μm to -4.9 μm for 18 μm to 37 μm sized grains (-3% to - 15%) 0.1 to 0.4 ASTM # for ASTM # 6 to 8 sized grains (1% to 6%)
Focal depth	1 sample	Yes	1.3 μm for 14 μm sized grains (10%) -0.7 ASTM # for ASTM # 9.1 sized grains (-7%)
Single crystal elastic constants	C_{11}, -11.2% to 12% C_{12}, -27% to 29% C_{44}, -23%, to 25%	Yes	-4 μm to -7 μm for 18.7 μm sized grains (-1% to -30%) -0.9 ASTM ASTM # to 0.7 ASTM # for ASTM # 9.2 sized grains (-27% to 39%)
Depth dependence from a single scan	±1.6 cm from focal zone	No Analysis region must be within -0.2 cm to +0.7 cm of focal zone for this sample and transducer	-7 μm to 15 μm for 21 μm sized grains (-34% to -73%) -1.2 ASTM ASTM # to 1.6 ASTM # for ASTM # 8 sized grains (-16% to 20%)
Compounding effects	Limited analysis		Limited results not included in table

The ultrasonic metallography software has proven to be robust and relatively insensitive to alignment of the transducer with the sample and reference material. Specifically the transducer can be misaligned by ±2° or more when collecting backscattering data or as much as ±1° when collecting the reference signal. It is also insensitive to transducer properties, pulser/receiver, user, and can be used to measure grain size within ~1 cm of the focal zone of the transducer. The measured grain size from a round robin test on an IN718 specimen with multiple users, at different sites, using different ultrasonic pulser/receivers produced the same grain size within 0.4 ASTM# or 3 μm. These ranges are well within desired precision of grain size determination of ±1 ASTM # for most production facilities. During this project the ultrasonic metallography software also passed a blind test measuring the grain size in Udimet 720 and Rene 88 to within a range of 0.1 to 0.9 ASTM#.

4. CONCLUSION

Prior to this work, no effective NDE technology existed to measure grain size in a form that could be used by engineers or NDT practitioners. The unification of data acquisition, analysis, theory, grain size mapping and visualization is a first-of-a-kind measurement and calculation software to directly measure the grain size using ultrasonic backscattering. The results presented here provide the guidelines for implementation in production environments to allow users to measure the grain size from ultrasonic backscattering.

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