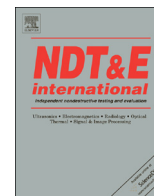




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# Detecting recrystallization in a single crystal Ni-base alloy using resonant ultrasound spectroscopy

L.H. Rettberg\*, B.R. Goodlet, T.M. Pollock

Materials Department, University of California, Bldg. 503, Rm. 1355, Santa Barbara, CA 93106-5050, United States

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## ABSTRACT

The use of resonant ultrasound spectroscopy (RUS) as a nondestructive evaluation (NDE) technique for Ni-base single crystal superalloys has been investigated. Manufacture of single crystal superalloys can be challenging due to the prevalence of defects induced during single crystal growth or subsequent processing. Common defects involve the presence of misoriented (non-single crystal) material that change the bulk elastic properties and, as a result, are detectable by RUS. To control the extent of misoriented material, recrystallization induced by shot peening the surface of the single crystal has been studied. RUS was then used to determine the presence and depth of misoriented material due to recrystallization. Recrystallization of shot peened cylindrical single crystal specimens occurred to a depth of 80  $\mu\text{m}$  and 178  $\mu\text{m}$  during subsequent heat treatments. Experimental average resonance frequency shifts of 1.835%  $\pm$  1.704% and 2.380%  $\pm$  2.910%, respectively, were measured over a frequency range from 20–200 kHz when compared to the baseline shot peened condition. Finite element (FE) models using the ABAQUS Lanczos Eigen frequency solver assessed the influence of recrystallization as a function of depth from the surface and showed good agreement with the measured resonance frequency shifts. For the greatest NDE sensitivity on production-scale turbine blades and other gas turbine components, a coupled RUS measurement and FE modeling approach is essential, and has the potential to improve single crystal processing approaches and manufacturing yields.

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## 1. Introduction

For several decades, single crystal Ni-base superalloys have been the material of choice for high pressure turbine blades in gas turbine engines [1,2]. More recently, they have been implemented in land-based combined cycle power generation gas turbine engines to achieve efficiencies over 60%. The most advanced Ni-base superalloys may contain ten alloying elements, including significant amounts of refractory elements, and possess excellent mechanical properties (creep and fatigue), oxidation and corrosion resistance. A critical development in the processing of Ni-base superalloys was the use of high thermal gradient casting to create directionally solidified turbine blades and, with the use of a grain selector/seed crystal, single crystals [2]. By removing high angle grain boundaries, single crystal superalloys can tolerate thermomechanical loading at temperatures in excess of 85% of their melting point. Without the requirement for grain boundary strengtheners, microsegregation and eutectic content in single crystal superalloys can be significantly reduced during heat treatments without causing incipient melting, improving fatigue life [3,2].

\* Corresponding author.

E-mail address: [rettberg@umail.ucsb.edu](mailto:rettberg@umail.ucsb.edu) (L.H. Rettberg).

A typical single crystal superalloy casting yield for aviation turbine blades may be as low as ~70% due to defects such as misorientation, high-angle boundaries and recrystallization, with defect criteria being defined by a variety of manufacturer specifications [2]. By using nondestructive evaluation (NDE) techniques, superalloy castings with defects can be detected and subsequently repaired or reverted. Standard NDE techniques include: fluoroscopic inspection, liquid penetrant inspection, radiographic inspection, and eddy current inspection [4,5]. Research described in this paper demonstrates the utility of a novel NDE framework which employs nondestructive resonant ultrasound spectroscopy (RUS) measurements, informed by finite element (FE) models to evaluate grain structure defects in single-crystal superalloy specimens. Considering the anisotropic elastic properties of Ni-base materials and their influence on the mechanical resonance of a 3D body, RUS is employed for rapid NDE of surface recrystallization, using forward FE models of resonance, validated by experiments.

### 1.1. Resonant ultrasound spectroscopy

In RUS, resonance modes are excited by a piezoelectric transducer(s) that provide a periodic displacement to the surface of the specimen. An elastic solid of any shape has normal modes and

natural frequencies. If driven by the transducer(s) at a natural frequency, the amplitude of oscillation of the normal mode is enhanced by the quality factor (Q) of the sample, enabling experimental measurement [6–10]. These amplified deflections are then recorded across a range of frequencies by additional piezoelectric transducers contacting the specimen to yield a broadband resonance spectrum when plotted as a function of the excitation frequency [6–10]. From peaks in the broadband measurement, resonance frequencies are deduced that are characteristic of the geometry and material properties of the specimen [6–10].

## 2. Elasticity considerations

Elastic waves excited in the specimen during RUS inspection are low-energy and generate very small sample deflections such that the assumption of linear elasticity is appropriate. The 3D constitutive law relating stresses ( $\sigma_{ij}$ ) and strains ( $\epsilon_{kl}$ ) is Hooke's law, given as:

$$\sigma_{ij} = C_{ijkl}\epsilon_{kl}, \quad (1)$$

where  $C_{ijkl}$  is the rank-four stiffness tensor.

Voigt shorthand maps the  $C_{ijkl}$  of the rank-four tensor to a 6-by-6 matrix ( $C_{ijkl} \rightarrow C_{pq}$ ). The two constituent phases of Ni-base superalloy single crystals,  $\gamma$ , and  $\gamma'$ , possess cubic crystal structures, affording the material cubic elastic symmetry. Cubic symmetry materials are fully defined by 3 independent stiffness values,  $C_{11}$ ,  $C_{12}$ , and  $C_{44}$  [8].

An important characteristic for RUS inspection of grain structure is the elastic anisotropy of the material, which is commonly defined for a cubic crystal by the Zener [11] anisotropy ratio:

$$A = \frac{2C_{44}}{C_{11} - C_{12}}. \quad (2)$$

Metals with low elastic anisotropy include Al and W with  $A_{Al} \approx 1.2$  and  $A_W \approx 1.0$ , while Ni, Fe, and Cu exhibit significant anisotropy with A values ranging from 2.4–3.2 [12]. Table 1 provides the stiffness values and calculated A for CMSX-4 [13], a Ni-base superalloy with similar composition and properties to the alloy employed in this study: René N5.

### 2.1. Engineering moduli of cubic single crystals

Single crystal Ni-base superalloy castings are typically solidified along the  $\langle 001 \rangle$  crystallographic direction since growth is preferred on the  $\{100\}$  family of planes, and the low modulus along this direction is favorable for strain-controlled fatigue [2]. The directionally-dependent Young's modulus ( $E_{[hkl]}$ ) relates normal stresses to normal strains as applied parallel to a crystallographic direction  $[hkl]$ , and is useful for comparisons to isotropic moduli. This constitutive behavior is in the same form as Eq. (1), whereby  $C_{ijkl}$  is replaced by  $E_{[hkl]}$  with the definition:

**Table 1**  
Single crystal stiffness values for CMSX-4, a 2nd generation single crystal Ni-base superalloy similar in composition to René N5, data from [13].

Stiffness	Value (units)
$C_{11}$	252 GPa
$C_{12}$	161 GPa
$C_{44}$	131 GPa
A	2.88 unitless

**Table 2**  
Directional elastic moduli for single-crystal specimens, calculated with data from [13].

Directional Moduli	Value (units)
$E_{[100]}$	126 GPa
$E_{[101]}$	231 GPa
$E_{[111]}$	320 GPa
$G_{[100]\langle\perp\rangle}$	131 GPa
$G_{(101)[\bar{1}\bar{1}0]}$	45.5 GPa
$G_{(110)[001]}$	131 GPa
$G_{[111]\langle\perp\rangle}$	58.1 GPa

$$E_{[hkl]} = \frac{C_{44}(C_{11} - C_{12})(C_{11} + 2C_{12})}{C_{44}(C_{11} + C_{12}) + (C_{11} + 2C_{12})\alpha_{[hkl]}}. \quad (3)$$

The direction cosine ( $J_{hkl}$ ) corresponds to the angle between the plane normal to the applied stress and the nearest  $\langle 100 \rangle$  crystallographic direction;  $J_{hkl}$  is zero along  $\langle 100 \rangle$ , maximum when  $J_{111} = 1/3$ , and median for  $J_{110} = 1/4$ . The anisotropy factor ( $\alpha$ ) is negative here and goes to zero as the material becomes isotropic ( $A \rightarrow 1$ ), defined as:  $\alpha = C_{11} - C_{12} - 2C_{44}$  [14,15]. From Eq. (3) it is clear that  $E_{[111]}$  is the highest directionally-dependent Young's modulus and  $E_{[100]}$  is the minimum modulus [13].

A directionally-dependent shear modulus ( $G_{(mno)[hkl]}$ ) can be calculated in a similar manner, where  $(mno)$  is the plane normal and  $[hkl]$  is the direction of shear on  $(mno)$ .  $G_{(mno)[hkl]}$  has rotational symmetry on  $\{100\}$  and  $\{111\}$  planes allowing for the direction of shear to be expressed as an arbitrary perpendicular direction ( $[\perp]$ ) contained in the plane, but loading on all other planes exhibit  $G_{(mno)[hkl]}$  that vary with the direction of shear.  $G_{(mno)[hkl]}$  and  $E_{[hkl]}$  are calculated for key directions using  $C_{pq}$  from Table 1 and are summarized in Table 2 for subsequent discussion.

### 2.2. Isotropic moduli for polycrystalline aggregates

When consisting of a large volume of randomly oriented grains, polycrystalline materials act as elastically isotropic bodies with no directional dependence of constitutive elastic behavior [16]. Through a series of FE models, Nygård's has demonstrated that the number of grains necessary for an isotropic response from an aggregate of cubic crystals depends on the anisotropy of the crystallites (A) and the preferred cut-off for an effectively isotropic response [17]. Considering the properties of René N5 specifically, an isotropic response would be expected from an aggregate volume containing 550 or more randomly oriented grains.

With isotropy requiring that  $A=1$  in Eq. (2), a degree of freedom is removed from the elastic body such that only two moduli fully define the response. The most common isotropic moduli being: Young's modulus (E), bulk modulus (K), shear modulus (G) or Poisson's ratio ( $\nu$ ) [12]; the specific pair of moduli selected is based on the context of their use. Isotropic moduli are determined through various averaging schemes (e.g. Voigt–Reuss–Hill [18], Hashin [19], Kröner [20], and Gairola–Kröner [21]), whereby all these schemes define K as:

$$K = \frac{C_{11} + 2C_{12}}{3}. \quad (4)$$

The first polycrystalline average for the shear modulus was devised by Voigt ( $G_{Vo}$ ) and assumed uniform strain across all grains to yield:

$$G_{Vo} = \frac{2C' + 3C_{44}}{5}, \quad (5)$$

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