# Temperature dependence of flexural and torsional elastic constants of two nickel-based superalloys

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A sonic resonance technique has been used to determine Young's and shear moduli of one directionally solidified polycrystalline alloy, Rene 80H, and one single crystal of alloy Rene N4 as functions of temperature from 300 to 1373 and 1298 K, respectively. The variations in the elastic constants of these two alloys are best represented by quadratic functions of temperature.

## 1. Introduction

Superalloys are attractive materials for applications where retention of mechanical properties at elevated temperatures is desirable [1]. The present work deals with the influence of temperature on the elastic constants of precipitation hardening alloys in the form of one directionally solidified polycrystalline (Rene 80H) specimen and one (Rene N4) nickel-based single crystal. An attempt is made to test whether the measured variations in the elastic constants of these two alloys follow models for the variations of elastic constants with temperature suggested by Wachtman *et al.* [2], Varshni [3] and Lakkad [4]. In addition, the temperature dependence of the elastic constants of the alloys is compared with that of MAR-M200 [5].

# 2. Materials and experimental procedure

#### 2.1. Materials

Single-crystal Rene N4, and directionally solidified polycrystalline Rene 80H are vacuum-induction melted and vacuum-cast nickel-based precipitation hardening alloys. Both alloys are composed of a face-centred cubic austenite ( $\gamma$ ) phase matrix, a coherent facecentred cubic precipitation ( $\gamma'$ ) phase, and other minor phases in the eutectic region with carbide phases ( $\gamma\gamma'$ ), if carbon is present. The  $\gamma$ -phase matrix is constituted of nickel in solid solution with cobalt, tungsten, tantalum, chromium, and iron.  $\gamma'$  phase is primarily Ni<sub>3</sub> (Al, Ti, Cb). The elements hafnium, carbon, boron and zirconium are added to modify the grainboundary properties. Grain-boundary modifying elements are present in Rene 80H and absent in Rene N4.

Table I gives nominal elemental and phase compositions of these two alloys. A detailed discussion of the role of various elements and phases in these types of superalloys is given by Dekker and Sims [1]. The specimens of Rene N4 and Rene 80H used in the present investigation were generously provided by General Electric Company, Cincinnati, Ohio. The rec-

tangular specimens of Rene N4 and Rene 80H used in the present work had dimensions of  $10.173 \,\mathrm{cm} \times$  $1.367 \,\mathrm{cm} \times 0.249 \,\mathrm{cm}$ . The deviation of the dimensions from the true rectangular form of a given specimen was less than 0.001 cm. The specimen of Rene N4 was a single crystal specimen with the  $\langle 100 \rangle$  axis parallel to its length within  $+1^{\circ}$ . Two specimens of Rene 80H were directionally solidified with predominent  $\langle 100 \rangle$  grain orientations in the longitudinal directions of the rectangular specimens and random grain orientations in the transverse directions of the specimens. The maximum deviation of grain orientations from  $\langle 100 \rangle$  was 15°. Approximately 50%, 30% and 20% of grains deviated between 0° and 5°. 5° and 10°, and 10° and 15° from the  $\langle 100 \rangle$  direction, respectively. The densities of Rene N4 and Rene 80H are 8.481  $\pm$  0.005 and 8.227  $\pm$  0.005 Mg m<sup>-3</sup>, respectively.

TABLE I Elemental (wt %) and phase (vol %) composition of Rene 80H and Rene N4  $\,$ 

Composition	Rene 80H	Rene N4	
Elements			
Cr	14.0	9.25	
Co	9.5	7.70	
Al	3.0	3.70	
Та	_	4.0	
Ti	4.8	4.2	
W	4.0	6.2	
Мо	4.0	1.5	
Cb	-	0.5	
Hſ	0.75	-	
С	0.17	-	
В	0.015	~	
Zr	0.01	-	
Ni	59.755	62.95	
Phases			
γ	38.0	37.6	
γ΄.	56.0	56.0	
γγ´	60	6.4	
Mc	∫ 0.0	-	



#### 2.2. Experimental procedure

Elastic constants were determined by using a sonic resonance technique [6]. Resonant frequency measurements were made on specimens of the superalloys suspended by either two carbon fibres or two chromel wires in a vacuum chamber. Temperature was measured with a chromel-alumel thermocouple with its hot junction located 1 mm away from the middle of the specimen or spot welded at one of the two nodal points from which the specimen was suspended. Temperature measurements have an uncertainty of  $\pm 1 \text{ K}$ . The resonant frequencies measured in different tests at a given temperature were not found to be significantly different from one another. A block diagram of the system used to perform the experiments is shown in Fig. 1. The values of Young's modulus, E(T), and shear modulus, G(T), at a given temperature, T, of a rectangular bar are calculated from its flexural,  $F_{\rm F}(T)$ , and torsional,  $F_{\rm G}(T)$ , resonant frequencies by means of the following relations [6], respectively

and

$$G(T) = 4L^{2}(T) \rho(T) F_{G}^{2}(T) R(T)$$
 (2)

 $E(T) = 0.946 \ 42 \ \rho \ (T) \ L^4(T) \ F_{\rm F}^2(T) \ C(T)/t^2(T)$ 

Where  $\rho(T)$  is the density at temperature T, L(T) and t(T) are the length and cross-sectional dimensions in the direction of flexural vibrations of the rectangular specimen; C(T) and R(T) are correction factors for determining Young's and shear moduli from the flexural and torsional resonances, respectively, of a rectangular bar [7, 8]. The magnitude of C depends on L, t, and Poisson's ratio, v. For the specimen geometry used in this work, the magnitude of C(T) varies between 1.004 and 1.005 for  $0 \le v \ge 0.5$ . Because the difference between these two values of C affects the computed value of Young's modulus negligibly, a value of 1.004 was arbitrarily chosen for all calculations. The magnitude of R is independent of temperature for an isotropic solid and is a function of the ratio of the width of the rectangular bar (1.347 cm) to its thickness (0.249 cm). The calculated value of R is 8.509. The Equations 1 and 2 may be rewritten either as

$$E(T) = 0.94642 \rho(T_0) L^4(T_0) F_{\rm E}^2(T) C / [t^2(T_0)(1 + \alpha \Delta T)]$$
(3)

Figure 1 A schematic drawing of experimental arrangement.

and

$$G(T) = 4R \rho(T_0) L^2(T_0) F_G^2(T) / (1 + \alpha \Delta T)$$
 (4)

or

$$E(T)/E(T_0) = F_E^2(T)/F_E^2(T_0)(1 + \alpha \Delta T)$$
 (5)

and

$$G(T)/G(T_0) = F_G^2(T)/F_G^2(T_0)(1 + \alpha \Delta T)$$
 (6)

In the above equations,  $\alpha$  is the coefficient of linear thermal expansion and  $\Delta T = T - T_0$ , where  $T_0$  is the initial temperature, i.e. room temperature  $300 \pm 2$  K. The values of  $\alpha$  for these two alloys have been obtained from General Electric Co. and are displayed in Table II. In the present investigation the resonant frequencies of flexural and torsional vibrations were reproducible to within 3 and 20 Hz, respectively, for all the experimental sets of data. Consideration of these errors, in addition to those errors arising from the measurement of density, thermal expansion coefficient, temperature, and orientation imply that the uncertainty in the values of Young's and shear constants are 1.5% and 1%, respectively.

#### 3. Results

(1)

#### 3.1. Rene N4

The ratios of Young's and shear constants calculated from their resonant frequencies at various temperatures are shown in Figs 2 and 3. Because the orientation of Rene N4 crystal's  $\langle 100 \rangle$  axis was parallel to the length of the rectangle, the Young's and shear constants measured in this work correspond to  $S_{11}^{-1}$ , and  $S_{44}^{-1}$  of the cubic Rene N4, respectively. The

TABLE II Linear thermal expansion coefficients, ( $\alpha$ ), of Rene N4 and Rene 80H

Temperature (K)	Thermal expansion coefficients $(10^{-5} \mathrm{K}^{-1})$		
	Rene N4	Rene 80H	
422	1.170	1.242	
589	1.260	1.260	
755	1.314	1.296	
922	1.350	1.368	
1089	1.440	1.458	
1255	1.560	1.620	
1366	1.602	1.764	



Figure 2 Ratio of Young's modulus plotted against temperature of  $\langle 100 \rangle$  single-crystal Rene N4. Quadratic fit given by Equation 12. (----) Tests: (+) 1, ( $\diamond$ ) 2, ( $\diamond$ ) 3, ( $\times$ ) 4.

relations between the elastic constants,  $C_{ij}$  and elastic compliances,  $S_{ij}$ , of a single crystal material with cubic symmetry are

Poissons's ratio (v) is given by

$$v = -S_{12}/S_{11} = C_{12}/(C_{11} + C_{12})$$
 (10)

$$S_{11} = (C_{11} + C_{12}) / [C_{11} + 2C_{12}) (C_{11} - C_{12})]$$
(7)  
$$S_{11} = (C_{11} + C_{12}) / [C_{11} + 2C_{12}) (C_{11} - C_{12})]$$
(7)

$$S_{44} = C_{44}^{-1} \tag{8}$$

and

$$S_{12} = -C_{12}/[(C_{11} + 2C_{12})(C_{11} - C_{12})]$$
 (9)

The values of  $S_{11}$  and  $S_{44}$  for Rene N4 at room temperature (298 ± 1 K) determined from the sonic resonance method are 7.645 × 10<sup>-3</sup> and 7.828 × 10<sup>-3</sup> GPa<sup>-1</sup>, respectively. The values of these constants determined from ultrasonic wave velocity measurements performed on single crystal specimens



Figure 3 Ratio of shear modulus plotted against temperature of  $\langle 100 \rangle$  single-crystal Rene N4. Quadratic fit given by Equation 13. (----) Tests: (+) 1, ( $\diamond$ ) 2, ( $\triangle$ ) 3, ( $\times$ ) 4.

of Rene N4 at room temperature [9] are 7.6553  $\times$  $10^{-3}$  and 7.8015  $\times$   $10^{-3}$  GPa<sup>-1</sup>. These two sets of values for  $S_{11}$  and  $S_{44}$  are obviously in very good agreement with one another and also within the errors of measurements. The variations in the values of flexural and shear moduli of Rene N4 as a function of temperature are shown in Figs 2 and 3. The variations of both moduli are nonlinear over the entire temperature range of measurements. This is not a unique situation because similar nonlinear dependence of the elastic constants on temperature has been observed in tungsten by Lowrie and Gonas [10] and by Brown and Armstrong [11]. The variations in the flexural and torsional moduli as shown in Figs 2 and 3 indicate that there is a sharp increase in the decrement of the values of these moduli around 1000 K. Such increase in the decrement of elastic constants with temperature has been reported by Marlowe and Wilder [12] in polycrystalline yttrium oxide, by Wachtman and Lam [13] in polycrystalline alumina, magnesia, thoria, mullite, silicon carbide, and nickel-bonded titanium carbides, by Lowrie and Gonas [10] in single-crystal tungsten, by Talmor *et al.* [14] in single-crystal niobium, and by Walker [15] in vanadium. Leibfried and Ludwig [16] have shown that the elastic constants of most pure metals and alloys decrease as  $T^4$  at low temperatures and as T in the classical limit above the Debye temperature, due to the anharmonic effects. The deviation from linearity at high temperatures is associated with higher order anharmonic effects and to the influence of vacancies whose concentration increases exponentially with temperature. In polycrystalline materials, the onset of faster decrease in elastic constants with temperature is found to be correlated with a rapid rise in internal friction. We do not have a collaborative evidence of either higher order anharmonic effects or rapid increase in internal friction. However, if grainboundary slip between y and y' phases is an important factor for the observed enhanced decrease in the elastic constants above 1000 K, then it is expected that the decrease in modulus should be dependent on the frequency of measurement. In other words, the values of elastic constants should be larger at higher frequencies. Table III presents the values of Young's modulus as a function of temperature measured under quasi-static loading [17], i.e. low frequencies and those measured in the present work. Table III shows that that values of Young's modulus for Rene N4 measured by the quasi-static method are not significantly different from those measured by the sonic resonance technique

over the entire range of temperature measurement. Thus the rapid decrease of Young's and shear modulus with temperature above 100 K could not be definitely attributed to grain-boundary slip in Rene N4.

The present set of measurements could not be carried out on other than  $\langle 100 \rangle$  orientations of Rene N4 bar as they were not available. Thus the measured moduli of Rene N4 as functions of temperature correspond to values of  $S_{11}$  and  $S_{44}$  of the cubic Rene N4 crystal. A single crystal with cubic symmetry requires knowledge of three independent elastic constants to describe its elastic deformation completely. In the present work, the value of missing constant  $S_{12}$  of Rene N4 as a function of temperature is determined from the measurements of  $S_{11}$  and  $S_{44}$  in the present work and of Young's modulus of Rene N4 crystal with orientations  $\langle 110 \rangle$ ,  $\langle 111 \rangle$ ,  $\langle 023 \rangle$ ,  $\langle 236 \rangle$ , and  $\langle 145 \rangle$  reported by Gabb *et al.* [17]. Values of  $S_{12}$  at 1033 and 1253 K were obtained by substituting the values of  $S_{11}$  and  $S_{44}$  determined in the present work and the values of  $S_{11}\langle l_1l_2l_3\rangle$  reported by Gabb *et al.* [17] in the following expression

$$S_{11} \langle l_1 l_2 l_3 \rangle = S_{11} - (2S_{11} - 2S_{12} - S_{44}) \\ \times (l_1^2 l_2^2 + l_3^2 l_1^2 + l_2^2 l_3^2)$$
(11)

where  $S_{11} \langle l_1 l_2 l_3 \rangle$  is the value of  $S_{11}$  in the  $\langle l_1 l_2 l_3 \rangle$ direction of a single cubic crystal and  $S_{11}$ ,  $S_{12}$ , and  $S_{44}$ are the elastic compliances of the crystal referred to principal orthogonal axes. The values of  $S_{12}$  at 1033 and 1253 K are the average values of  $S_{12}$  calculated from the measured values of  $S_{11}$  in the crystal orientation mentioned earlier. The values of  $S_{12}$  thus obtained are tabulated in Table III. The observed nonlinear variations in the elastic constants  $S_{11}^{-1}$  and  $S_{44}^{-1}$  as functions of temperature do not follow the models suggested by Wachtman et al. [2], Varshni [3], and Lakkad [4] probably due to higher order anharmonic effects. However, the observed variations in  $S_{11}^{-1}$ and  $S_{44}^{-1}$  are faithfully reproduced by the quadratic relations given below for temperature between 300 and 1298 K

$$S_{11}^{-1}(T) = S_{11}^{-1}(300) \times$$
  
1.01749 - 4.2430 × 10<sup>-5</sup>T - 1.143 × 10<sup>-7</sup>T<sup>2</sup>]  
(12)

$$S_{44}^{-1}(T) = S_{44}^{-1}(300) \times [1.05048 - 1.6284 \times 10^{-4} T - 7.5792 \times 10^{-8} T^{2}]$$
(13)

TABLE III Values of elastic compliances of single-crystal Rene N4 as a function of temperature

Elastic compliance (GPa) <sup>-1</sup>	Temperature (K)			
	298 K	1033 K	1253 K	
<u>S</u> 11				
Resonance*	$7.645 \times 10^{-3}$	$9.668 \times 10^{-3}$	$1.09897 \times 10^{-2}$	
Quasistatic <sup>†</sup>	$7.518 \times 10^{-3}$	$9.615 \times 10^{-3}$	$1.111 \times 10^{-2}$	
$S_{44}$ (Resonance)*	$7.828 \times 10^{-3}$	$9.768 \times 10^{-3}$	$1.0761 \times 10^{-2}$	
$S_{12}$ (Calculated) <sup>‡</sup>	$-2.921 \times 10^{-3}$	$-3.806 \times 10^{-3}$	$-4.383 \times 10^{-2}$	
Poisson's ratio	0.382	0.393	0.399	
*Present work.	<u></u>			

[

†[17].

<sup>‡</sup>See text, room temperature value of  $S_{12}$  was obtained from the ultrasonic wave velocity measurements given in [9].



Figure 4 Ratio of Young's modulus plotted against temperature of directionally solidified Rene 80H. Quadratic fit given by Equation 14. (----) Tests: (+) 1, ( $\diamond$ ) 2, ( $\diamond$ ) 3, (×) 4. ( $\nabla$ ) 5.

These quadratic relations for  $S_{11}^{-1}(T)$  and  $S_{44}^{-1}(T)$  are based on 79 observations for each. The standard errors of estimates are 0.8% and 0.4% for Equations 12 and 13, respectively.

Because the values of  $S_{12}$  are available only at 300, 1033 and 1253 K, no attempt was made to calculate a similar functional dependence of  $S_{12}$  on temperature.

#### 3.2. Rene 80H

Rene 80H is a directionally solidified polycrystalline superalloy. The fraction of grains which were orientated more than 15° away from  $\langle 100 \rangle$  was zero. The average grain-orientation angle calculated from the frequency distribution of grain orientations given in Section 2.1 is found to be 6°. Thus, Young's and shear moduli measured for Rene 80H should be identified as



Figure 5 Ratio of shear modulus plotted against temperature of directionally solidified Rene 80H. Quadratic fit given by Equation 15. (----) Tests: (+) 1, ( $\diamond$ ) 2, ( $\triangle$ ) 3, ( $\times$ ) 4. ( $\nabla$ ) 5.

 $S_{11}^{-1}$  and  $S_{44}^{-1}$ , respectively, of a single crystal of Rene 80H oriented 6° off  $\langle 100 \rangle$  axis. The values of Young's and shear moduli are determined to be 123.6 and 123.1 GPa, respectively, at 300 K. The ratios of Young's and shear modulus as functions of temperature are displayed in Figs 4 and 5, respectively. The dependence of flexural and torsion modulus on temperature for Rene 80H is similar to that of Rene N4. The variation in the values of these moduli with temperature is best represented by a quadratic function of temperature and are given by Equations 14 and 15, respectively.

$$E(T) = E(300) [1.01576 - 1.9280 \times 10^{-5}T - 2.1967 \times 10^{-7}T^{2}]$$
(14)

and

$$G(T) = G(300) [1.05548 - 1.8043 \times 10^{-4} T - 7.2100 \times 10^{-8} T^{2}]$$
(15)

where  $300 \le T \le 1373$  K. The quadratic relations for E(T) and G(T) are based on 265 and 201 observations, respectively. The standard errors of estimates for E(T) and G(T) are 0.9% and 0.8%, respectively. Because there are no published data pertaining to quasi-static measurements of Young's and shear moduli of Rene 80H as functions of temperature, it is difficult to comment upon the observed faster decrease in elastic constants with temperature.

#### 3.3. Comparison with MAR-M200

Finally, the observed variations in the elastic constants of Rene N4 and Rene 80N are not similar to those observed for polycrystalline super alloy MAR-M200 [5]. The observed variations of the elastic constants of MAR-M200 were bilinear, i.e. the slopes of Young's and shear moduli of MAR-M200 with temperature were found to change abruptly at 915 and 875 K, respectively [5]. The reason for the change appeared to be related to the elastic behaviour of the  $\gamma$  phase of MAR-M200. This conclusion was based on the measurements of Young's modulus of polycrystalline nickel-tungsten alloys containing 0.07% to 26.6% of tungsten by weight by Harringan and Nix [18]. However, no such breaks in slopes have been observed in the present work.

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