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### Ultrasonic Investigation of CMSX-6 Superalloy at Low and High Temperatures

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Abstract: The elastic constants  $C_{11}$ ,  $C_{44}$  and C' have been measured as a function of temperature in a single crystal of CMSX-6 alloy and from these data the bulk, Young's and shear moduli have been deduced, together with the anisotropy factor A and the elastic constant  $C_{12}$ . The temperature coefficient of the elastic constants  $C_{11}$  and  $C_{44}$  becomes gradually more negative on approaching the melting point of the alloy. This softening effect is interpreted in terms of a negative contribution to the elastic stiffness of the material arising from thermal vacancies within the disordered  $\gamma$  phase. The anisotropy factor A is found to fall in the typical range of fcc metals and the Cauchy relationship ( $C_{12} = C_{44}$ ) appears to be approximately fulfilled, suggesting that the lattice cohesion is associated with a field of central forces.

#### **1. INTRODUCTION**

The nickel-based single crystal superalloy CMSX-6 has been developed [1] for those high temperature applications where low density is a primary design requirement, as it is the case for aeroengine blades. The elastic anisotropy features of this and other superalloys are of great interest to design analysts, nonetheless, elastic constant data as a function of temperature are scarce or even not available at all in the open technical literature. Most of the elastic studies done so far have been devoted to the intermetallic compound Ni<sub>3</sub>Al [2-5], which is the strengthening phase ( $\gamma'$ ) of most of the Ni-based superalloys. To our knowledge data on the elastic constants of the CMSX-6 superalloy are not available in the literature, thus, the aim of the present investigation was that of measuring these physical quantities from liquid helium temperatures to near the melting point of the alloy ( $\cong$ 1560 K).

#### 2. EXPERIMENTAL

A single crystal in the form of a cylinder ( $\Phi$ = 1.36 cm l=14.1 cm) produced by Centro Sviluppo Materiali has been used in the present experiment; its nominal composition in weight was: 0.70 Ni, 0.10 Cr, 0.05 Co, 0.03 Mo, 0.02 Ta, 0.048 Al, 0.047 Ti, 0.001 Hf. The axis of the cilinder was superimposed to a <100> lattice direction.

For the high temperature measurements of  $C_{11}$  and  $C_{44}$  a technique was used, similar to that applied in [6] for investigations on refractory metals. A grove was machined at a distance h=1.41 cm from one end of the sample to a depth of about 0.3 cm and a quartz transducer was attached at the other end. The transducer was kept at room temperature, while the grooved part of the sample was inserted into a furnace and was heated in air up to temperatures of about 1500 K (see fig.1). A cromel-alumel thermocouple was spot-welded on the grooved part of the sample to measure the local temperature. Unfortunately, long single crystals were only grown with axis oriented along a <100> crystallographic direction, thus, only two elastic constants  $C_{11}$  and  $C_{44}$  could be measured at high temperatures with this technique.

The delay time  $\tau$  between the first two echoes of an ultrasonic pulse reflected at the cylinder basis and at the grove was measured at 10 MHz by the pulse-echo superposition technique [7]. As it is easily recognised the delay time only depends on length h of the groved portion of the sample, which is at an uniform temperature T, and on the ultrasonic velocity within the same sample region (v(T) =2h/\tau(T)).



Figure.1: Experimental sample arrangement

For low temperature measurements a small piece of the crystal was cut by means of a low speed diamond saw from the side of the cylinder, which had been kept cold during the high temperature measurements. This sample was machined to have two pairs of parallel and flat faces, respectively orthogonal to <100> and <110> crystallographic directions. The elastic constants were deduced from the well known formulas

$$C_{11} = \left[\rho v_L^2\right]_{[100]} \qquad \qquad C' = \left[\rho v_{T2}^2\right]_{[110]}^{[1\overline{10}]} = \frac{1}{2} \left(C_{11} - C_{12}\right)^{[1\overline{10}]} = \frac{$$

$$C_{44} = \left[\rho v_{T1}^{2}\right]_{[110]}^{[001]} \qquad or \qquad C_{44} = \left[\rho v_{T1}^{2}\right]_{[100]}$$

where  $\rho$  is the mass density, vL is the velocity of the longitudinal waves, vT1 and vT2 are the velocities of the shear waves and upper and lower indices represent the polarisation and propagation directions, respectively. The density  $\rho$  was measured with a hydrostatic balance and turned out to be equal to 7.98 g/cm3.

(1)

#### **3.RESULTS**

The measured elastic constants C',  $C_{11}$  and  $C_{44}$  are shown in figs. from 2 to 4. As it can be seen the elastic constants show a normal anharmonic behaviour at low and intermediate temperatures, as they decrease linearly with T between about 200 and 800 K, and as T<sup>4</sup> at low temperatures [8]. An interesting aspect of the high temperature data is the gradual decrease above 800 K of the temperature coefficient  $dC_{ii}/dT$ .

From the complete set of data obtained below room temperature the bulk modulus  $B=C_{11}-4C'/3$ , the anisotropy factor  $A=C_{44}/C'$  and the elastic constant  $C_{12}=C_{11}-2C'$  have been calculated and are reported in Table 1. Self-consistency of the data obtained at room temperature and below was checked by measuring the elastic constant  $C_{44}$  either by propagating longitudinal or transversal pulses according to relations (1). The practical moduli, Young's modulus E and shear modulus G, have also been deduced from the measured elastic constants by using well known relationships [9]. The values referring to <100>,<110> and <111> directions, from which data can be deduced for any other orientation [8], are listed in Table 1.



Figure.2: Dependence on temperature of the elastic constant  $C_{11}$ 



Figure.4: Dependence on temperature of the elastic constant C'



Figure.3: Dependence on temperature of the elastic constant C<sub>44</sub>



Figure.5: Difference between the values of the elastic constant  $C_{44}$  obtained from a linear extrapolation of low temperature data and the measured ones

#### 4. DISCUSSION

Although the composition of this superalloy is complex, its structure is simple, consisting of about 60 vol % coherent dispersion of  $\gamma'$  phase in the matrix of the nickel-aluminium solid solution ( $\gamma$  phase). In the present measurements the echo trains of the ultrasonic pulses were quite well defined, thus, indicating good coherency at  $\gamma/\gamma'$  interfaces and absence of incoherent precipitates.

The elastic constants of CMSX-6 superalloy at room temperature are quite close to those of pure Ni  $(C_{11}=2.51, C_{44}=1.23, C'=0.505, C_{12}=1.50, B=1.84 \text{ and } A=2.45)[4]$  and not much dissimilar from those of the pure  $\gamma'$  phase [4,5]. In particular, the value of the anisotropy factor coincides with that of pure Ni and falls in the middle of the range of values typical of fcc lattices (1.5 < A < 4) [10]. The Cauchy relation  $C_{21}/C_{44}=1$  approximately holds  $(C_{12}/C_{44}=1.15)$ , indicating that the chemical bond is dominated by central forces, as it is the case, to a lesser extent, of the pure  $\gamma'$  phase [5]. From these comparisons it appears that the outstanding mechanical properties of superalloys are not directly related to interatomic forces, but rather to specific features of lattice defects such as superdislocations and anti -phase boundaries.

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T(K)	C <sub>11</sub>	C44	C'	C12	В	A	E<100>	E<110>	E<111>	G<100>	G<110>	G<111>
30	2.407	1.286	0.488	1.431	1.757	2.637	1.339	2.333	3.101	1.286	0.707	0.615
50	2.406	1.285	0.487	1.432	1.757	2.638	1.338	2.332	3.100	1.285	0.707	0.614
70	2.405	1.284	0.486	1.432	1.757	2.641	1.335	2.329	3.097	1.284	0.705	0.613
90	2.402	1.281	0.485	1.432	1.755	2.642	1.332	2.324	3.091	1.281	0.704	0.612
110	2.397	1.277	0.483	1.431	1.753	2.643	1.327	2.317	3.082	1.277	0.701	0.609
130	2.393	1.272	0.481	1.430	1.751	2.642	1.323	2.309	3.071	1.272	0.698	0.607
150	2.388	1.266	0.480	1.429	1.749	2.640	1.319	2.300	3.060	1.266	0.696	0.605
170	2.382	1.261	0.478	1.426	1.745	2.638	1.314	2.292	3.049	1.261	0.693	0.603
190	2.378	1.256	0.476	1.426	1.743	2.638	1.309	2.284	3.039	1.256	0.691	0.600
210	2.372	1.251	0.474	1.423	1.739	2.637	1.305	2.276	3.027	1.251	0.688	0.598
230	2.366	1.245	0.473	1.420	1.735	2.635	1.300	2.267	3.015	1.245	0.685	0.596
250	2.357	1.240	0.471	1.416	1.730	2.633	1.295	2.258	3.002	1.240	0.683	0.594
270	2.351	1.234	0.469	1.413	1.726	2.630	1.290	2.249	2.989	1.234	0.680	0.591
290	2.345	1.228	0.467	1.410	1.722	2.628	1.286	2.240	2.976	1.228	0.677	0.589

Table 1 Measured and calculated elastic constants (in units of 10<sup>11</sup>Pa) below room temperature

As already noted, an interesting finding of the present investigation is the gradual decrease of  $dC_{ii}/dT$ , above 800 K. This effect is too large to be attributed solely to higher order anharmonic effects.

Solutioning of the  $\gamma'$  phase occurs at temperatures too high (T>1544) [1] to be considered as a possible source of the non-linear behaviour of C<sub>11</sub> and C<sub>44</sub> above 800 K. It is conceivable that this effect is due to thermal vacancies, whose concentration should exponentially increase with temperature, within both the  $\gamma$  and the  $\gamma'$  phase. This assumption, which is supported by the observation in  $\beta_2$ -NiAl of a decrease of C<sub>44</sub> and C<sub>11</sub> with the concentration structural Ni vacancies of [11]. Actually, the difference  $\Delta$ Cij between the extrapolated values of the lower temperature data and the measured ones display an exponential temperature dependence (see fig.5), which gives for the formation energy of vacancies EF a value of 0.75 eV. This figure is much lower than the EF of Ni vacancies within the  $\gamma'$  phase. We are not aware of determinations of EF within this phase, however, its value is expected to fall between those for pure Ni (1.39 eV) and Al (0.65 eV) [13]. The value of 0.75 eV is, then, compatible with this expectation. It is to be noted, however, that the size of the elastic constant changes (about 10 %) would imply concentrations of vacancies exceedingly high (10<sup>-2</sup>) [11], thus, suggesting that other contributions may come into play.

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