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High Temperature Mechanical Spectroscopy in Ordered $\text{Ni}_3(\text{Al}, \text{Ta})$

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Abstract: Mechanical loss measurements were performed on polycrystalline and single crystalline samples (of three different orientations) of a $\text{Ni}_3(\text{Al}, 1\% \text{Ta})$ alloy with the L_{12} ordered structure. Measurements were conducted in torsion pendula, on as-cast samples and samples predeformed either at 300 K or at 1300 K, and revealed the occurrence of two main phenomena: (a) The presence of a well-defined internal friction peak at a temperature of about 950 K for a frequency of 1 Hz. This relaxation peak has an activation energy of about 3 eV and its amplitude is strongly dependent on sample orientation and predeformation conditions. (b) An internal friction background, which amplitude at high temperatures (above 1100 K) mainly depends on strain amplitude and predeformation conditions. Both phenomena appear closely related to the presence of point defects, to the complex dissociation mode of superdislocations and to the change in dislocation mechanisms occurring at the peak temperature in flow stress (about 800 K) in relationship with a change in active slip systems.

1. INTRODUCTION

The mechanical properties of intermetallic compounds with the L_{12} ordered structure (e.g. Ni_3Al) have been the subject of extensive experimental and theoretical studies for the past three decades. The focus of this attention has been on the anomalous increase in the flow stress that is observed with increasing temperature under constant strain-rate deformation conditions (see for instance [1]). In addition to this anomalous flow stress behavior, the magnitude of the critical resolved shear stress (CRSS) in Ni_3Al single crystals is found to depend on both the orientation and the sign of the applied stress and therefore violates the Schmid's law for octahedral slip (see for instance [2,3]). The mechanical behavior of Ni_3Al compounds may be also strongly influenced by deviation from the stoichiometric composition [4] and by the presence of ternary elements [5].

The present work is aimed at providing new information about the rate controlling dislocation mechanisms for plastic flow in Ni_3Al ordered intermetallic compounds by performing mechanical loss measurements. These measurements were conducted, using torsion pendula, on as-cast and predeformed polycrystalline and single crystalline specimens. For the latter type of specimen three different crystallographic orientations have been investigated.

2. EXPERIMENTAL PROCEDURE

The material used in this study is a Ni_3Al alloy with the L_{12} ordered structure, of nominal composition $\text{Ni}_{75}\text{Al}_{24}\text{Ta}_1$, which was supplied by Professor D.P. Pope at the University of Pennsylvania. Polycrystalline samples are available, as well as single crystalline samples.

Parallelepipedic specimens were prepared for mechanical spectroscopy by using an electro-discharge machine. Single crystalline specimens were cut out along a $\langle 001 \rangle$ axis with $\langle 110 \rangle$ lateral faces, a $\langle 110 \rangle$ axis with $\langle 001 \rangle$ and $\langle 110 \rangle$ lateral faces and a $\langle 111 \rangle$ axis with $\langle 110 \rangle$ and $\langle 112 \rangle$ lateral faces. The specimens were then mechanically polished with successively finer grades of abrasive paper (up to $15\mu\text{m}$) for removing the electro-discharged layer from the surfaces.

Internal friction spectra were obtained as a function of temperature by performing mechanical loss measurements in a free decay torsion pendulum which operates at frequencies between 0.6 Hz and 2.5 Hz. The measurements were conducted between 300 K and 1225 K with heating/cooling rates of

2 K/min and with strain amplitudes ranging between 1×10^{-5} and 5×10^{-4} . Activation enthalpy values were determined by performing measurements in a torsion pendulum working with forced vibrations. In this case the strain amplitude was 1×10^{-5} and the imposed frequency varied from 10^{-5} Hz to 10 Hz.

Mechanical loss measurements were performed on as-cast specimens and on specimens that were in situ predeformed by torsion in the free decay pendulum at room temperature or at 1300 K. The amount of predeformation was of the order of 2.5 %.

3. RESULTS

3.1 Grain boundary effect

Figure 1 shows typical examples of internal friction spectra which were obtained as a function of temperature at large strain amplitude (5×10^{-4}) for the as-cast polycrystalline samples and for as-cast $\langle 111 \rangle$ -oriented single crystals. A well defined internal friction peak is observed for both types of specimens at a temperature of about 950 K for a frequency of 1 Hz. It can also be seen in Figure 1 that the amplitude of the internal friction background at low and intermediate temperatures, that is essentially below 800 K, is rather small (of the order of about 1.5×10^{-3}) and almost independent on temperature. On the contrary, at high temperatures (typically above 1050 K) and for both types of specimen, it exponentially increases with increasing temperature.

Figure 1: Internal friction spectra for as-cast polycrystalline and $\langle 111 \rangle$ single crystalline samples; frequency = 1 Hz, strain amplitude = 5×10^{-4} .

3.2 Orientation effect

Typical examples of internal friction spectra which were obtained as a function of temperature for the three different crystallographic orientations of single crystals are reported in Figure 2. These experiments were performed on as-cast samples with a strain amplitude of 5×10^{-4} and for a frequency of 1 Hz. It can be seen that the three spectra exhibit an internal friction peak at temperatures which are approximately 970 K for the $\langle 001 \rangle$ orientation and close to 950 K for the $\langle 110 \rangle$ and $\langle 111 \rangle$ orientations. It also clearly appears that the peak amplitude is strongly orientation dependent and has a maximum value for samples oriented along a $\langle 111 \rangle$ axis. This amplitude is lower for $\langle 110 \rangle$ samples and very small for $\langle 001 \rangle$ samples. At low and intermediate temperatures, the internal friction background is small and does not exhibit any significant dependence on sample orientation and temperature. In the high temperature range, it exponentially increases with increasing temperature and exhibits a clear orientation dependence since it is higher for $\langle 001 \rangle$ and $\langle 110 \rangle$ specimens than for $\langle 111 \rangle$ samples.

Figure 2: Internal friction spectra for as-cast $\langle 001 \rangle$, $\langle 110 \rangle$ and $\langle 111 \rangle$ single crystalline samples; frequency = 1 Hz, strain amplitude = 5×10^{-4} .

3.3 Strain amplitude effect

The effect of strain amplitude on the internal friction is illustrated in Figure 3. In this figure are plotted as a function of temperature the internal friction spectra that have been obtained for as-cast $\langle 110 \rangle$ single crystals by applying small and large strain amplitudes of 1×10^{-5} and 5×10^{-4} , respectively. As in the above experiments a frequency of 1 Hz was used for these measurements. Both spectra in Figure 3 have a similar shape but a higher internal friction is obtained for the 5×10^{-4} strain amplitude as compared with the 1×10^{-5} strain amplitude. However, after subtraction of the associated exponential damping backgrounds the two internal friction peaks have the same amplitude. This result demonstrates the absence of a strain dependence of the peak amplitude, while the amplitude of the high-temperature internal friction background (above 1100 K) appears to increase significantly with the strain amplitude.

Figure 3: Internal friction spectra for as-cast $\langle 110 \rangle$ single crystalline samples, using strain amplitudes of 1×10^{-5} and 5×10^{-4} ; frequency = 1 Hz.

3.4 Predeformation effect

Figure 4 shows internal friction spectra which have been obtained as a function of temperature for as-cast polycrystals, predeformed polycrystals at room temperature and polycrystals that were predeformed at high temperature (1300 K). The experimental conditions were a frequency of 1 Hz and a strain amplitude of 5×10^{-4} . It can be seen that predeformation at 1300 K induces a slight decrease in both the amplitude of the internal friction peak and the high-temperature background, while predeformation at 300 K leads to a strong enhancement of the internal friction background measured at high temperatures (above 1100 K), together with a modification of the right-hand side part of the internal friction peak.

Figure 4: Internal friction spectra for as-cast polycrystalline samples, polycrystalline samples predeformed at 300 K and polycrystalline samples predeformed at 1300 K; frequency = 1 Hz, strain amplitude = 5×10^{-4} .

3.5 Activation parameter measurements

The activation energy of the internal friction peaks was determined to be 2.97 ± 0.10 eV and 3.00 ± 0.05 eV for the polycrystalline samples and the $\langle 001 \rangle$ -oriented single crystalline specimens, respectively. The two peaks are enlarged with respect to a Debye peak by a width factor of about 2 and the related relaxation times are of the order of 10^{-15} s. The activation energy associated with the high-temperature internal friction background has a value of 1.00 ± 0.17 eV and the related activation volume that has been tentatively determined is of the order of about 60 ± 10 b³ (when measured in the 1200-1300 K temperature range) for both polycrystalline and $\langle 110 \rangle$ single crystalline samples, where $b = 0.254$ nm is the Burgers vector length of a superpartial dislocation ($b = \pm a/2 \langle 110 \rangle$).

4. DISCUSSION AND CONCLUSION

The well defined internal friction peak that was evidenced in this work for polycrystals (see Figure 1) should not be induced by grain boundaries since this peak is still present for single crystals, even if strongly reduced in amplitude in the case of the $\langle 001 \rangle$ -oriented single crystals (see Figure 2). It is worth

noting that extensive transmission electron microscopy (TEM) observations [6] have proved that the single crystals are totally grain boundaries and even subgrain boundaries free in the as-cast state and also after large deformation (20 % in compression) at high temperatures.

The similar activation energies that have been found for both polycrystalline and single crystalline internal friction peaks, as well as the characteristic features of the latter, suggest that these peaks are of the same type and could be interpreted as point defect relaxation peaks, certainly of the Zener type [7]. This assumption is supported by the strong dependence of the peak amplitude that is observed with sample orientation (see Figure 2) and by the absence of a dependence of the peak amplitude with strain amplitude (see Figure 3). The intense relaxation peak which was observed for polycrystals (Figure 1) could be induced by the re-orientation of elastic dipoles, while for single crystals an inappropriate orientation (such as $\langle 001 \rangle$ for instance) resulting in this case in an impossibility for dipoles to re-orientate may explain the drastic decrease in the peak amplitude. While the presence of Tantalum in our samples could lead for instance to the formation of Ta-Ta elastic dipoles, the similarities of the observed internal friction peak with the intense relaxation peak obtained by Chakib and Gadaud [8,9] at a temperature of 875 K for a frequency of 0.1 Hz in binary Ni_3Al polycrystalline alloys discard this possibility. Note that the latter authors also evidenced a second relaxation peak with a smaller amplitude at about 1050 K, again for a frequency of 0.1 Hz, the presence of which was attributed to diffusion of Nickel. This second internal friction peak was not observed in the present study.

Since various predeformations in torsion induce a significant decrease in the amplitude of the observed relaxation peaks (see Figure 4), it appears that they are also strongly related to deformation-induced dislocation mechanisms. A similar conclusion can be drawn for the high-temperature internal friction background whose high-temperature amplitude appears very sensitive to predeformation conditions.

When comparing the measured internal friction spectra with the anomalous flow stress behavior of $\text{Ni}_3(\text{Al}, 1\% \text{Ta})$, it appears that the onset of the relaxation peaks occurs at a temperature of about 800 K, for a frequency of 1 Hz, which is close to the peak temperature in flow stress. Slip trace analyses and TEM observations of the deformation substructures [6] have shown that the peak temperature in flow stress coincides with a change in the operating slip systems. While the main active slip system below the peak in flow stress is the primary octahedral slip system $\pm a[10\bar{1}](111)$, the active slip system above the peak is the primary cube slip system $\pm a[110](001)$. Corresponding dissociation modes of superdislocations ($\mathbf{b} = \pm a\langle 110 \rangle$) are also different [6]. Below the peak in flow stress, the edge superdislocations are dissociated on (111) in a glissile configuration into four Shockley partials ($\mathbf{b} = \pm a/6\langle 112 \rangle$) bounding an antiphase boundary (APB) and two complex stacking faults, while the screws appear sessile and dissociated as Kear-Wilsdorf locks [10]. Above the flow stress peak an opposite situation is observed: the edges are climb-dissociated in a sessile configuration as super-Lomer locks and the screws are dissociated on (001) into two superpartials ($\mathbf{b} = \pm a/2[110]$) bounding an APB. This change in rate controlling slip system may explain the orientation dependence of the internal friction background that is observed at high temperatures.

To conclude, we would like to emphasize that an APB is a chemical-type planar fault which is produced in ordered structures by slip of imperfect dislocations and could therefore favor trapping of elastic dipoles. The internal friction peaks observed in this study are certainly related to the presence of such point defects as well as to the presence of deformation-induced dislocations.

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