Deformation mechanisms of hard oriented NiAl single crystals

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Abstract. Hard oriented NiAl single crystals (<100> deformation axis) have been compressed at temperatures between 296K and 963K. At about 600K the yield stress changes from a plateau to a steep fall. Slip line investigations show that this coincides with a transition from {112}<111> to {110}<110> slip. At low temperatures slip of <111> dislocations is determined by the Peierls mechanism while in the plateau region the deformation mechanism is still not understood. For the deformation by {110}<110> slip the activation enthalpy measured supports the deformation mechanism proposed by Mills et al. [1] based on the diffusion-assisted motion of macro-kinks in <110> edge dislocations which are found to be decomposed into <100> edge dislocations. The slip transition temperature is discussed with regard to the brittle-to-ductile transition temperature of polycrystalline NiAl.

1. Introduction
NiAl is an intermetallic compound with a number of favourable properties that make it a promising candidate for high temperature, e.g. turbine engine applications. NiAl has a brittle-to-ductile transition temperature (BDTT) of about 600K for polycrystals and hard oriented single crystals (<100> deformation axis) [1, 2]. This corresponds to the temperature where besides primary slip on {110}<100> secondary slip changes from {112}<111> to {110}<110> [2 - 4]. Based on TEM investigations revealing a complicated core structure of dislocations with <110> Burgers vector, Mills et al. [1] proposed a model for dislocation motion on {110}<110> slip systems. It is the aim of the present work to analyze the strain rate and temperature dependence of secondary slip in NiAl above the BDTT and check whether the model by Mills et al. is appropriate in terms of activation parameters to explain the fall of yield stress of hard oriented single crystals at medium temperatures.

2. Experimental
The single crystal used in this work (16mm in diameter and 30mm long) exhibited a stoichiometric composition with a maximum deviation of ±1at.% and a <100> growth direction which was verified by Laue back reflection technique. Compression samples of 2.1×2.1mm² in cross section and 4mm in length were cut by spark-erosion and subsequently annealed at 1273K for 60h under vacuum (heating/cooling rate about 1K·min⁻¹). The {100} oriented side faces were ground using SiC paper of different grit size to remove the surface layer affected by the erosion process leading to the final sample size of 1.8×1.8×4mm³. The base and top surface were only slightly ground with fine SiC paper to remove the roughness caused by the erosion process, thus minimizing the change in orientation and planarity. Additionally, for slip line investigations two side faces per sample were electropolished three times for 10s with cooling in between using a solution of 10% perchloric acid and 90% ethanol at a temperature of 263K and a voltage of 20V.
All compression tests were carried out in an Instron testing system under vacuum with the deformation temperature being obtained by heating with a rate of 12K·min\(^{-1}\). Subsequently, a temperature dependent holding time of at least 1h was inserted to reach thermal equilibrium. The compression tests were performed with an initial strain rate of 3.6×10\(^{-5}\) s\(^{-1}\). To study the effect of strain rate, after reaching a plastic strain of approx. 1% the strain rate was doubled and then reset to the original value at a plastic strain of approx. 2% with the test being stopped at a plastic strain of approx. 3%. The accuracy of stress and strain is about 3%. The absolute accuracy of the temperature measurement is 10K.

To analyze the mechanical data it is assumed that above the BDTT the material obeys a power law constitutive relationship

\[ \dot{\varepsilon} = A\sigma^n \exp\left(-\frac{\Delta H}{kT}\right) \]  

between strain rate \( \dot{\varepsilon} \) and stress \( \sigma \) \([5]\) (\( k = \) Boltzmann constant), with the stress exponent \( n \) given by

\[ n = \frac{d\ln\sigma}{d\ln\dot{\varepsilon}} \]  

and the apparent activation enthalpy given by

\[ \Delta H = nk \frac{d\ln\dot{\varepsilon}}{d\ln(1/T)}. \]  

In order to investigate the slip lines and determine the activated slip systems two neighbouring surfaces of the deformed samples were inspected by optical microscopy.

3. Results and discussion

Figure 1 shows an optical micrograph of the surface of a sample deformed at room temperature (RT). The slip lines under an angle of 63.5° to the compression axis are caused by \{112\}<111\> slip. At low temperatures and high strain rates slip of <111> dislocations has been observed on \{110\}, too \([6]\). Figure 2 shows a sample deformed at 723K with horizontal slip lines which can be related to \{110\}<110\> slip as there are no slip lines under an angle of 45° on the neighbouring sample faces ruling out the possibility of \{110\}<111\> slip. The slip steps seen are produced by <110> screw dislocations. The Burgers vectors of <110> edge dislocations on \{110\} slip planes are parallel to the \{100\} side faces, thus they do not produce slip steps. In summary, the slip line investigations in the temperature range investigated show quite a sharp slip transition at about 600K.

The temperature dependence of the 0.2% yield stress is shown in figure 3. Similar curves have already been obtained by other researchers. The curves show a steep drop from a plateau at about 600K coinciding with the slip transition. Strain rate change tests show that there is practically no strain rate sensitivity in the plateau region, while at higher temperatures the mean stress exponents (average over a certain temperature interval) decrease from 4.3 to 3.7 (figure 4). Simultaneously, the apparent activation enthalpies increase from 1.5eV to 2.9eV.

It was the aim of the present study to analyze secondary slip in NiAl. Below about 600K the active secondary slip system is \{112\}<111\>. Deformation via \{112\}<111\> slip is temperature and strain rate dependent below room temperature. The activation volume at RT is about 20\(b^3\)\(_{<111>}\) \((\text{about } 3\ b^3\)\(_{<111>}\) at RT \([7]\) and below \([8]\)) which together with the very long Burgers vector (0.5nm) of the moving dislocations indicate that the most likely deformation mechanism is the Peierls mechanism.

Between RT and about 600K the deformation via \{112\}<111\> slip is neither temperature nor strain rate dependent. Frequently kinking takes place. According to Takeuchi \([9]\) the yield stress plateau may be explained by the Peierls mechanism of narrowly dissociated <111> screw dislocations (1.6\(b^3\)\(_{<111>}\)). However, this contradicts the strain rate insensitivity observed here as well as the compact core structure of <111> screws predicted by molecular static calculations using an embedded atom potential (≤ 1.2\(b^3\)\(_{<111>}\) \([10]\)).
Figure 1. Slip lines caused by \{112\}<111> slip at 296K after compression up to a true strain of 3.1% and corresponding schematic representation of one slip system (plane plus Burgers vector (arrow)).

Figure 2. Slip lines caused by \{110\}<110> slip at 723K after compression up to a true strain of 2.7% and corresponding schematic representation of one slip system (plane plus Burgers vector (arrow)).

Figure 3. 0.2% yield stress vs. temperature (literature data are taken from [2]).

Figure 4. Apparent activation enthalpies from a plot of logarithmic true stress vs. reciprocal temperature using the mean values given for the stress exponent $n$. 
Above about 600K the active secondary slip system is of the \{110\}<110> type. Mills et al. [1] proposed an interesting model for the movement of <110> dislocations in NiAl. According to TEM studies as well as atomistic calculations <110> edge dislocations tend to decompose into separate <100> edge dislocations with orthogonal Burgers vectors. This decomposition effectively locks the edge portions of <110> dislocations. Based on experimental evidence, it is suggested that motion of these dislocations is instead controlled by lateral movement of non-edge segments (macro-kinks). The model proposed requires the constriction of the decomposed edge segments involving the transfer of vacancies from one <100> dislocation to the other via pipe diffusion. The constitutive equation developed is in accordance with equation (1), with \( n = 3 \) and \( \Delta H = 1.5\text{eV} \) being the activation enthalpy for pipe diffusion (half of that for Ni lattice diffusion, 3.1eV [2]). These values compare quite well with those estimated in this work at about 700K (figure 4). In addition an activation volume of \( 2b^3 \) was obtained suggesting that the climb of <100> edge dislocations is the process limiting the deformation [11]. With increasing temperature the activation enthalpy observed approaches that of Ni lattice diffusion suggesting a change in diffusion mechanism. A quantitative comparison of stress or strain rate is difficult because the constant \( A \) depends on the height of the macro-kinks and the distance over which the <100> edge dislocations have decomposed, parameters which may be temperature and stress dependent.

It should be mentioned that another locking mechanism has been proposed for 35° <110> dislocations on \{110\} based on a TEM analysis of the dislocation core structure [12, 13]. Concerning the BDTT of polycrystalline NiAl [3] it is conspicuous that this temperature compares quite well with that of hard oriented NiAl single crystals [14], respectively the slip transition observed here. Moreover, the strain rate dependence of the BDTT of NiAl polycrystals yields an activation enthalpy of 1.3eV [15] indicating that the BDTT may also be determined by pipe diffusion mediated motion of macro kinks in decomposed <110> edge dislocations. As the ductility of a polycrystalline material requires the compatible deformation of its crystallites, secondary slip has to be activated in order to provide sufficient independent slip systems. Thus, below the BDTT the stress needed to fulfill this condition by activating \{112\}<111> slip systems is much higher than the fracture stress resulting in a brittle behaviour. Above the BDTT due to the onset of diffusional processes secondary slip changes to \{110\}<110> leading to a much lower stress needed for its activation. Hence, the requirements for strain compatibility in polycrystals that is ductility are fulfilled.

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