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Plasticity of the spinodally decomposing Ni-11.8 at.% Ti alloy

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ABSTRACT

The spinodal decomposition of supersaturated Ni - 11.8 at.% Ti single crystals (fcc structure) was studied during ageing at 853 and 1073 K. All the samples were homogenised at 1423 K and then waterquenched to room temperature prior to ageing. The quenched samples were found consisting of short-range ordered matrix and slightly L12 - ordered Ti-rich domains. During the first sixty hours of ageing at 853 K, a "metastable situation" was found, where the microstructure of the samples did not change very much. Between sixty and five hundred hours of ageing, concentration waves developed, which grew in amplitude and wavelength with increasing ageing time. Later on, coarsening started, leading to metastable γ' particles (L12 - structure), which began to become incoherent after 4000 h of ageing. The kinetics of Ni - 11.8 at.% Ti single crystals at 1073 K were investigated in order to study the phase transformation to the stable η - Ni₃Ti phase (D0₂₄ - structure). Not fully D0₂₄ - ordered nuclei with ABAC stacking sequences were observed. Furthermore, defective stacking sequences, derived from the ABAC stacking sequence, were found to occur frequently. A mechanism based on Shockley partial dislocations is suggested for the transformation of the fcc structure to the ABAC stacking sequence of the D0₂₄ - structure.

The mechanical properties of quenched and aged Ni - 11.8 at.% Ti single crystals were investigated by compressive, tensile and fatigue tests performed at room temperature. The quenched specimens showed planar glide in all types of tests, and even strain bursts, which are typical for short-range ordered single crystals, were recorded during fatigue tests. Superdislocations and dislocation pile-ups were observed by Transmission electron microscopy in quenched samples, which were deformed in compression. The samples aged at 853 K, which were consisting of a microstructure containing concentration waves, showed a very localised deformation behaviour. Instabilities were observed in compressive tests, and in tensile and fatigue tests a low ductility was noted. In all types of tests a very high yield strength was measured. It may be assumed, that these special mechanical properties are the result of the tendency to nucleate the stable η phase. The dislocation structure of samples consisting of a microstructure with concentration waves, was found to be dominated by regularly dissociated $a/2\langle 110 \rangle$ dislocations. This dissociation into Shockley partial dislocations takes place in the Ti-rich domains. The dislocations may be considered to act as microprobes which are very sensitive to the Ti concentration.

A model based on planar slip is presented, which describes the formation of defective microtwins, i.e. ABAC stacking sequences containing $a/2\langle 110 \rangle$ dislocation segments, out of regularly split, paired dislocations under an external stress in a microstructure consisting of concentration waves. These microtwins are the basic elements of the ABAC stacking sequence of the stable η phase.

Figure 1. **Full lines:** Samples deformed in uniaxial compression and aged for a) 62.5 b) 500 and c) 1000 hours at 853 K (measured at room temperature, crystal axis parallel to $[234]$, length-to-diameter ratio of the samples about 1.8). **Spotted lines:** Samples deformed in uniaxial compression and aged for d) 0 e) 62.5 f) 250 g) 500 h) 1000 and i) 2000 hours at 853 K (measured at room temperature, crystal axis parallel to $[124]$, length-to-diameter ratio of the samples about two).

INTRODUCTION

The decomposition of supersaturated Ni - 12 at.% Ti alloys has been investigated at a wide range of temperatures. It has been shown, that a metastable miscibility gap exists approximately underneath 1200 K. At ageing temperatures lower than about 853 K, the decomposition starts with the formation of periodic concentration waves followed by metastable, coherent γ' particles with L12 - structure. The composition of these metastable precipitates is stated to be lower than the nominal concentration of the L12 - structure of 25 at.% Ti [1,2]. The stable η phase with D0₂₄ - structure is formed, for example, at 873 K after forty days [3,4]. At higher temperatures (around 1073 K) the metastable γ' precipitates form fast; the phase transformation of parts of the matrix γ (fcc structure) and of previously formed metastable γ' particles to the stable η phase begins after about four hours. The phase transformation from γ to η phase was first investigated by Mihalisin and Decker [5]. These authors report, that the η phase, in some cases, nucleates at stacking faults in the γ matrix and that cold-work increases the rate of transformation to η by increasing the stacking fault density. Sass and Cohen [6] revealed the crystallographic nature of the stable η phase as a four-layer hexagonal, close-packed and ordered structure with stacking sequences ABAC.

EXPERIMENTAL

The Ni-11.8 at.% Ti alloy was prepared by vacuum induction melting of nickel (99.99%) and titanium (99.9%). Cylindrical rods were cast, and single crystals about 10 cm long and 5.5 mm in diameter were grown by electron-beam zone melting at a speed of about 0.5 mm/min. The operating pressure was always smaller than 10^{-5} mbar. The concentration gradient along the crystal axis was smaller than 0.05 at.%/cm. Single crystals with growth axes parallel to $[015]$, $[124]$, $[123]$ or $[234]$ were used for compressive, tensile and fatigue tests. Cylindrical

Figure 2. a) Cyclic hardening curve (A) from step tests of a quenched Ni - 11.8 at.% Ti single crystal (measured at room temperature, crystal axis parallel to $[1\bar{2}3]$). The plastic strain amplitude (B) is also displayed. b) Cyclic hardening curve (A) of a Ni - 11.8 at.% Ti single crystal quenched and aged for 500 hours at 853 K (measured at room temperature, crystal axis parallel to $[1\bar{2}3]$). The plastic strain amplitude (B) is also displayed.

rods 3 to 4 mm in diameter and 6 to 10 mm in length were cut by spark erosion. Cylindrical specimens with a length-to-diameter ratio between 1.8 and 3 were used for the uniaxial compressive tests. The samples were homogenised at 1423 K for 2 h in an evacuated quartz tube and then quenched into water. After a heat treatment at 853 K or 1073 K (in the case of the aged specimens) the samples were electropolished in a solution of 15 vol.% H_2SO_4 , 64 vol.% H_3PO_4 and water. In order to produce single crystalline tensile and fatigue samples, single crystalline cylinders about 3.5 mm in diameter and 10 mm in length were welded by butt welding (or soldering in the case of the quenched fatigue specimens) to cylindrical grips of Nimonic 75. The specimens were then homogenised, quenched and aged. After that, the specimens were ground with a corundum disk to final dimensions. The compressive tests were performed in a screw-driven machine, at a crosshead speed of 0.2 mm/min. The tensile tests were performed on a screw-driven machine with a constant strain rate of 0.0005 resp. 0.00005 s^{-1} . The fatigue tests were performed on a hydraulic machine. These tests took place under plastic strain control by applying a sinusoidal load cycle of 3 Hz and a zero mean strain. The plastic strain amplitude was kept constant by controlling the total strain amplitude on the specimen. Hysteresis loops were continuously monitored. The microhardness measurements were performed on a commercial Leitz Durimet 2 instrument. Thin foils for TEM samples were thinned mechanically, dimpled and electropolished in a solution of 20 vol % HNO_3 in methanol at 223 K using a twin-jet apparatus. TEM was performed on a Jeol 200CX microscope operating at 200 kV.

RESULTS

The microstructure of quenched Ni - 11.8 at.% Ti single crystal specimens, deformed in compression, is characterised by the presence of dislocation pile-ups and superdislocations, probably imposed by short-range order and long-range ordered domains. A moderate yield strength and a high ductility is measured in compressive (figure 1), tensile and fatigue tests of single crystalline specimens. Planar glide was observed in all these types of mechanical tests by surface investigations. In addition, strain bursts were recorded in fatigue tests (figure 2a), whose intensities were diminished by the presence of grain boundaries. This fatigue behaviour is common in short-range ordered alloys which show planar glide as Cu-Al or Ni-Cr [7,8]. The samples aged between 62.5 and 1000 hours show serrated flow in compressive tests. The serrated flow is strongly influenced by the length-to-diameter ratio and the orientation of the single crystals (figure 1). Surface investigations indicate planar glide and strongly localised

Figure 3. Weak-beam images of Ni - 11.8 at.% Ti single crystals taken with a Transmission electron microscope at 200 kV. a) Single crystal aged for 250 h at 853 K and deformed at room temperature in compression (crystal axis parallel to $[124]$). Paired dislocations dissociated periodically into four Shockley partial dislocations are visible in the glide plane (see small arrows). The zone axis is parallel to $[111]_{\text{fcc}}$ and the diffraction vector $g = [202]$ is parallel to the Burgers vectors of the undissociated parts of the dislocations. b) Single crystal aged for 500 hours at 853 K and deformed at room temperature in compression (crystal axis parallel to $[234]$). Accumulations of regularly dissociated dislocations are depicted. Line defects are marked with small arrows. The zone axis is parallel to $[001]_{\text{fcc}}$ and the diffraction vector is $g = [200]$.

deformation. The samples aged for 500 hours at 853 K show a very high yield strength in tensile and compressive tests, but also a low ductility in tensile and fatigue tests (figure 2b). Single crystals deformed in tension, failed after about 10 % plastic shear, if they were oriented for single glide (crystal axis parallel to $[123]$) and after about 3 % plastic shear in double glide orientation (crystal axis parallel to $[015]$). The microstructure of the specimens aged between 62.5 and 500 h at 853 K mainly consist of periodical concentration waves in $\langle 100 \rangle$ directions. The dislocation structure which developed during deformation in compressive tests, is dominated by regularly dissociated, paired dislocations whose periodicity correspond to the periodicity of the concentration waves of the microstructure. These dislocations act, therefore, as microprobes which are very sensitive to the Ti concentration (figures 3a,b). The observed dislocation type can be described as a superdislocation consisting of two identical $a/2\langle 110 \rangle$ dislocations regularly split into two $a/6\langle 112 \rangle$ Shockley partial dislocations (SPDs) each. The dissociation takes place on the $(111)_{\text{fcc}}$ glide planes in the Ti-rich domains, forming two complex stacking faults (CSFs) on both sides of an antiphase boundary (APB). In the regions where the dislocations split, i.e. in the Ti-rich domains, the width of the APB is small. The CSFs, however, are widely spread. This behaviour of the superdislocations can easily be explained, since the degree of $L1_2$ - ordering is probably increased in the Ti-rich region with respect to the Ti-poor domains and, therefore, also the APB energy. The CSF energy, however, is low in the Ti-enriched domains because of the tendency to transform to the hexagonal, stable phase. In polycrystals (grain diameter $d = 0.2$ mm), which were aged for 500 h at 853 K and deformed in fatigue, even pile-ups of regularly split dislocations were observed.

Between 1000 and 2000 h of ageing at 853 K, the yield strength of the specimens considerably decreases. The serrated flow effect, observed during compressive tests also decreases (figure 1), indicating a less localised deformation. The microstructure after 2000 h of ageing consists of γ' particles which are less regular distributed than the Ti-rich domains in the samples aged for 500 h at 853 K. Paired dislocations which cut the γ' particles during deformation are predominant in these samples. Therefore, the superdislocation type with regular splitting into SPDs must be favoured, if a regular distribution of Ti-rich obstacles is present with only small spacings among themselves and no sharp interfaces. Thus, it can be

Figure 4. a) Periodically dissociated screw dislocations with Burgers vectors $b = a/2[\bar{1}01]$ and equal line senses are accumulated in the glide planes by planar glide. One glide plane is shown. b) Every Shockley partial dislocation (SPD) reacts under an external stress with the neighbouring SPD in its glide plane forming $a/2[\bar{1}01]$ screw segments, which divide the complex stacking faults in different sections. c) Pairs of SPDs are operating on neighbouring $(111)_{\text{fcc}}$ planes and the following planes remain unchanged. Transformation blocks of four $(111)_{\text{fcc}}$ planes form a defective microtwin of the stable η phase, i.e. an ABAC stacking sequence containing $a/2[\bar{1}01]$ dislocation segments.

argued, that long-range interaction forces present in the microstructure consisting of concentration waves are responsible for the tendency to nucleate the stable η phase via the dissociation of $a/2\langle 110 \rangle$ dislocations in the Ti-rich domains. After 4000 h of ageing at 853 K the coherency limit of the η' particles is reached. The microhardness between 2000 and 4000 h of ageing, however, increases. Thus, cutting of η' particles by paired dislocations is presumed to remain the predominant deformation mechanism after 4000 h of ageing as in the samples aged for 2000 h at 853 K. However, a further ageing of the samples would probably lead to an Orowan mechanism and, consequently, to a second drop of the microhardness.

To determine the operating mechanism in the early stage of the η phase formation, the kinetics at 1073 K were studied. The nucleation of η plates was investigated after 4 and 24 h of ageing. The η nuclei were found to be not fully D0₂₄-ordered. Defective stacking sequences, derived from the ordinary ABAC stacking sequence of the η phase, and ledges were observed quite often.

DISCUSSION

A complete transformation of the fcc lattice to the ABAC stacking sequence of the η phase, maintaining full coherency with the nontransformed fcc lattice across densely packed planes, can be achieved by six Shockley partial dislocations (SPDs) operating on a unit of twelve $(111)_{\text{fcc}}$ planes. Pairs of SPDs are operating on neighbouring $(111)_{\text{fcc}}$ planes, and the following two $(111)_{\text{fcc}}$ remain unchanged [9]. In this way transformation blocks of four $(111)_{\text{fcc}}$ planes develop, which form a microtwin with an ABAC stacking sequence.

In figures 4a-c a model is presented, which describes the formation of defective microtwins, i.e. ABAC stacking sequences containing $a/2\langle 110 \rangle$ dislocation segments, out of regularly split, paired dislocations under an external stress in microstructures consisting of concentration waves. If periodically dissociated screw superdislocations, with Burgers vectors $b = a/2[\bar{1}01]$ and equal line senses, are accumulated in two neighbouring $(111)_{fcc}$ glide planes by planar glide under an external stress (as indicated in figure 4a) and if every Shockley partial dislocation (SPD) reacts under the back stress of the dislocation pile-ups with the neighbouring SPD in its glide plane (figure 4b), a highly localised deformation by forming small, defective microtwins could be produced (figure 4c). These microtwins are the basic elements of the ABAC stacking of the stable η phase. Since many $a/2[\bar{1}01]$ dislocation segments are produced by the reactions of neighbouring SPDs (figures 4a,b), a high dislocation density should be present in the reacting $(111)_{fcc}$ planes of the microtwins (figure 4c). Figure 3b shows accumulations of regularly dissociated dislocations of a sample aged for 500 h at 853 K and deformed in compression. Stacking faults and line defects are visible which might be the result of the presence of defective microtwins.

The described model predicts the immobilisation of the active slip bands by the induced phase transformation, if the value of the applied shear stress and the density of the regularly split dislocations is high enough in the glide planes after a certain amount of planar glide. Thus, the observed serrations in stress-strain curves of compressive tests could be related to the softening by shearing of the Ti-rich domains and the sudden immobilisation of the active slip planes by dislocation reactions leading to defective microtwins. After the immobilisation of a slip band, an increased shear stress is necessary to activate glide in other regions of the specimens. The early failure of samples aged for 500 h at 853 K in tensile and fatigue tests, however, might be the result of a) positive normal stresses produced during these types of mechanical tests, b) the very localised deformation behaviour of the specimens and c) the high local density of defects (dislocation segments in the model) produced by the formation of defective microtwins. The much earlier failure of single crystals oriented for double glide in comparison with single crystals oriented for single glide in tensile tests, can thus be explained by the increased formation of microcracks, since the local accumulation of defects is strongly enhanced by double glide following the model.

CONCLUSIONS

The special mechanical properties of samples with a microstructure containing concentration waves are the result of a) planar glide, b) very localised deformation and c) the tendency to nucleate the stable η phase by interactions of regularly dissociated dislocations. Defective structures derived from the ABAC stacking sequence of the stable η phase were also observed during nucleation of stable η plates at 1073 K.

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