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The high temperature decrease of the critical resolved shear stress in nickel-base superalloys

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Abstract

Nickel-base superalloys show a typical decrease in the critical resolved shear stress (CRSS) around 750–800°C. *In situ* tensile tests of the alloys NIMONIC 105 and NIMONIC PE 16 carried out in a high voltage transmission electron microscope revealed the onset of a new deformation mechanism at 780°C. Above 780°C the deformation is achieved by the viscous motion of pairs of identical γ-matrix dislocations with Burgers vector of type a/6$\langle 112 \rangle$ (a: lattice constant). The dislocations are dragging behind long intrinsic stacking faults and thus establish microtwins. Along the trace of the moving dislocations the γ'-particles are sheared. A crystallography/diffusion-based model is presented which explains the (i) unusual pairing of a/6$\langle 112 \rangle$ dislocations and (ii) the restriction of the viscous dislocation movement to high temperatures.

Keywords: Nickel-base superalloy SX; Critical resolved shear stress; Deformation twinning

1. Introduction

Single crystal superalloys are widely used as turbine blade material in aerospace and land-based gas turbines. Although great effort has been given to study their mechanical properties, there is still a fundamental problem, as the temperature characteristic of the critical resolved shear stress (CRSS) not completely understood: From ambient temperature up to around 750°C the CRSS is more or less constant and decreases sharply at higher temperatures (for NIMONIC 105 see [1], for MAR-M200 [2]). The superalloys NIMONIC 105 and NIMONIC PE 16 can be regarded as model systems for order hardening. They are hardened by spherical precipitates of the γ'-phase ($L1_2$ structure, an ordered fcc structure), which are embedded in the (fcc) γ-matrix. The alloys have a very low misfit between the γ-matrix and the γ'-particles. Both alloys have been investigated in *in situ* tensile tests [3,4] at room temperature. The results are in accordance with the well-accepted mechanism for deformation in alloys containing ordered precipitates [5,6]. Pairs of dislocations of the γ-matrix with Burgers vectors $b = a/2\ \langle 110 \rangle$ cut the γ'-particles. The first dislocation destroys the $L1_2$ structure in the γ'-particle and leaves behind an antiphase boundary (APB) while the second dislocation reestablishes the order again. Thus the APB exists only between the two dislocations of the pair inside of the γ'-particles.

This work is based on *in situ* high temperature tensile tests that were carried out for the alloys NIMONIC 105 and NIMONIC PE 16 in the temperature region around the decrease of the critical resolved shear stress and that were previously reported [7]. The main results of these tests are briefly summarized in the following: (i) a transition in behavior of the movement of dislocations was found—below 780°C and the movement was jerky, while above 780°C the dislocations moved in a viscous manner drawing stacking faults behind them. It could be observed that the γ'-particles were still governing the movement of the dislocations, by acting as pinning sites along dislocation line lengths. (ii) The transition is thermally stable, the specimens can be cycled thermally around 780°C and the typical behavior described in (i) is preserved. The high temperature movement of the dislocations is characterized by (iii) {111} glide planes, (iv) cutting of γ'-particles, (v) pairwise movement of dislocations of the γ-matrix with (vi) Burgers vectors of the same a/6$\langle 112 \rangle$ type drawing behind long stacking faults. (vii) Post mortem TEM

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investigations on *in situ* deformed specimens showed that, in the stable configuration without externally applied load, the dislocations lie in the γ-matrix [7].

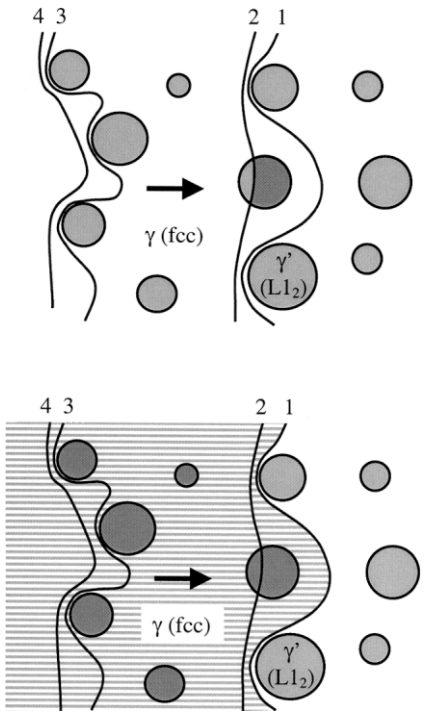

Fig. 1. Pairwise movement of dislocations through an area containing ordered γ'-particles. The typical planar faults are schematically drawn: (a) low temperature movement—pairs of a/2⟨110⟩ dislocations. No fault is produced in the γ-matrix. The movement of the first dislocation results in an APB in the γ'-particles, but the following dislocation reestablishes the order. (b) high temperature movement—pairs of a/6⟨112⟩ dislocations. Every dislocation produces a new stacking fault in the γ-matrix, complex stacking faults result in the γ'-particles. Subsequent pure glide processes do not annihilate existing faults.

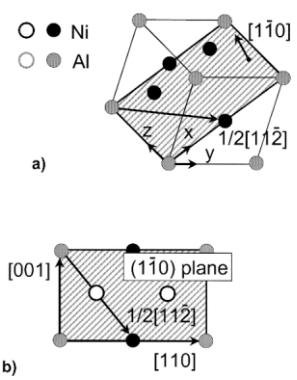

Fig. 2. The structure of Ni_3Al ($L1_2$). (a) The cubic cell with the Ni and Al atoms black and gray respectively. The normal of the (1$\bar{1}$0) plane and the [11$\bar{2}$] direction (Burgers vector) are indicated. (b) The view is perpendicular to the (1$\bar{1}$0) plane. Ni atoms above and below the (1$\bar{1}$0) plane are shown by black open circles.

2. Description of the problem and demands on a model

The high temperature behavior characterized under (iii)–(v) on the first glance is similar to the low temperature behavior, which could be directly observed during *in situ* tensile tests in the superalloys NIMONIC 105 and NIMONIC PE 16 [3,4]. The important observation, which is the key to understanding the high temperature behavior of both the superalloys in tensile tests, is given in (vi). While in low temperature tests the pairwise moving dislocations are of Burgers vectors a/2⟨110⟩, in the high temperature tests the dislocations have Burgers vectors of type a/6⟨112⟩ and draw long stacking faults behind them. The situations are schematically illustrated in Fig. 1a and b. It can be seen in Fig. 1a (low temperature movement) that an APB is left by dislocation 1 in one particle (dark gray), but the order is restored by dislocation 2. No fault is left in the γ-matrix. Fig. 1b corresponds to the high temperature movement: faults are left in the γ'-particles as well as in the γ-matrix because a Burgers vector of type a/6⟨112⟩ is neither a translation vector in the fcc structure nor in the $L1_2$ structure. The order is not simply restored by the following dislocations only, an additional process is needed.

A microscopic model should give reasons for the following features: (i) the decrease of the CRSS, which appears in the *in situ* experiments as the easy movement of dislocations around 780°C and the microstructural details listed above in (i) through (vii), especially the unusual pairing of identical a/6⟨112⟩ dislocations.

3. A crystallography/diffusion-based model

The γ'-phases in nickel-base superalloys contain several additional elements compared to the pure Ni_3Al $L1_2$ ordered intermetallic. For example, Co occupies the Ni sites and Ti can be found on the Al sites. For clarity, the following considerations will be carried out for the pure Ni_3Al structure, but they can be directly transformed into the general case of an $L1_2$ ordered structure. The structure of Ni_3Al is shown in Fig. 2a: the Ni atoms are black, the Al atoms gray. The coordinate system is given as it is used in the following: the (1$\bar{1}$0) plane is hatched. The view in Fig. 2b is perpendicular to the (1$\bar{1}$0) plane: the open black circles represent Ni atoms, which lie under and/or above the plane under consideration. Fig. 3a shows the crystal in the same projection like Fig. 2b, perpendicular to the (1$\bar{1}$0) plane, after the movement of a dislocation with Burgers vector a/6[11$\bar{2}$] on the ($\bar{1}\bar{1}\bar{1}$) plane (from upper left to lower right). A CSF (dotted line) is produced behind the dislocation: the plane of the CSF is ($\bar{1}\bar{1}\bar{1}$) and is thus perpendicular to the plane of observation. The CSF is energetically unfavorable because the Al atoms

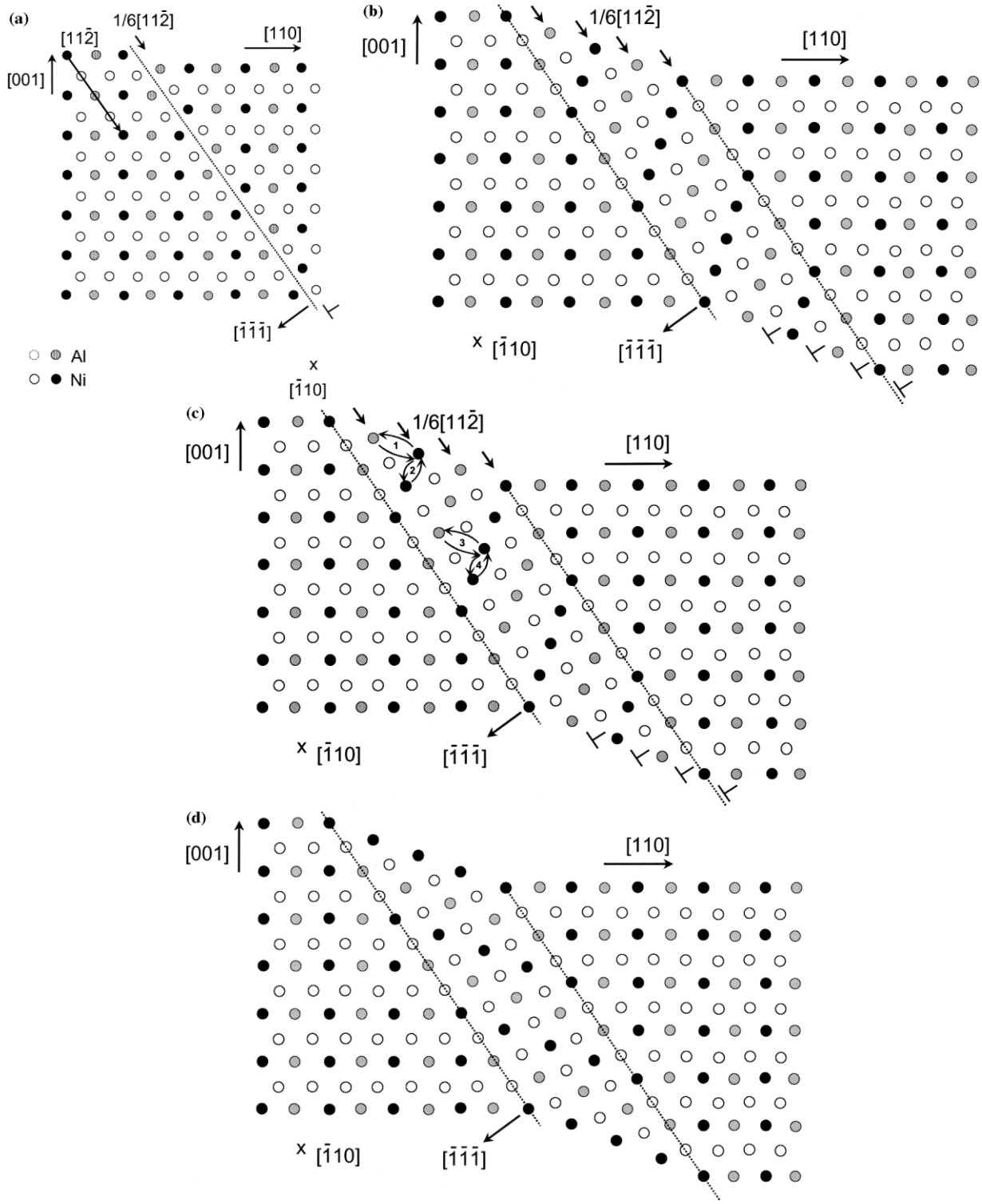

Fig. 3. (a) $(1\bar{1}0)$ plane in the $L1_2$ structure. An a/6$[11\bar{2}]$ dislocation moving on its $(\bar{1}\bar{1}\bar{1})$ glide plane has produced a complex stacking fault (CSF) along the dotted line, which is an intersection of the $(1\bar{1}0)$ and the $(\bar{1}\bar{1}\bar{1})$ plane. (b) Four moving dislocations have produced an energetically unfavorable pseudotwin between the two dotted lines. (c) A pair of double steps of diffusion is given, which reestablishes the order in the pseudotwin. It is pointed out, that for the operation of this mechanism two neighboring disordered planes of the pseudotwin are needed. (d) A microtwin in the $L1_2$ structure as it is produced by pairs of moving a/6$[11\bar{2}]$ dislocations with subsequent diffusion steps (c).

are too close to each other. A second a/6[11$\bar{2}$] dislocation cannot follow the first on the same ($\bar{1}\bar{1}\bar{1}$) plane for simple crystallographic reasons (Appendix). It is as-sumed, that it moves on a neighboring ($\bar{1}\bar{1}1$) plane. The third dislocation follows on the next ($\bar{1}\bar{1}\bar{1}$) plane and so on (Fig. 3b). This is the way how a microtwin could be established in an fcc structure (without order). In the $L1_2$ structure the corresponding result of the dislocation movement is a pseudotwin. This means that along the path of the dislocations the $L1_2$ structure is destroyed. The pseudotwin can be regarded as a stack of complex stacking faults that are energetically not favorable. A detailed discussion of the problems concerning twins/pseudotwins in ordered alloys is given by Christian and Laughlin [8].

In Fig. 3c a sequence of diffusion steps is indicated by bent arrows, which restore again the initial $L1_2$ structure and transform the pseudotwin into a true microtwin (Fig. 3d). It is remarkable that, for a given diffusion sequence, two neighboring moving dislocations are needed for operation. This explains the pairwise movement of the a/6$\langle$112$\rangle$ type dislocations. The dynamics of the movement can now be understood as follows: the dislocations are moving in pairs and produce CSFs behind them. As the CSFs have a high energy, they will be annihilated by a sequence of diffusion steps that take place just behind the moving dislocations. One half double-step of diffusion is needed per pair of the dislocations and per elementary crystal cell. It is plausible, that—under a certain stress—the dislocation movement can be "switched on" by raising the temperature, because of the strong temperature dependence of diffusion. On the other hand, the dependence on the applied stress at a constant temperature should be weak, as the influence of stress on diffusion is normally not strong. Fig. 4 shows the diffusion steps in the three-dimensional crystal. A closer inspection of the structure leads to a second diffusion sequence (dotted arrows), which has the same result as the one already presented, but with a shorter distance in the first jump. It cannot be decided at present which sequence is chosen by nature, but the second seems more probable because of the shorter jump width.

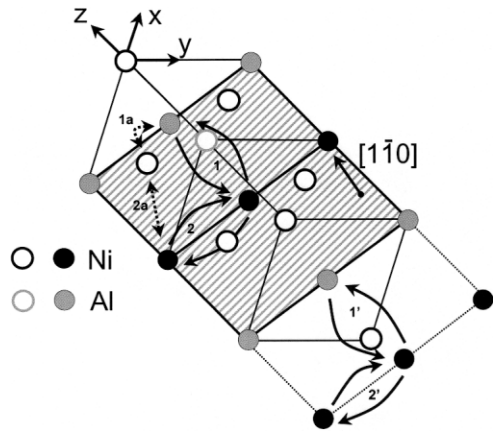

Fig. 4. Structure of the pseudotwin as created by dislocation movement (Fig. 3b). The diffusion steps from Fig. 3c are given by solid arrows (1,2) and (1′,2′). A similar sequence (1a,2a) is given by dotted arrows. The jump width (1a) is shorter than the corresponding width (1) and thus might be more probable.

4. Discussion

Deformation twins are reported in many fcc metals and ordered alloys [9]. Recently twins have been found in the superalloys SRR 99 and CMSX-4 after creep deformation in the vicinity of creep pores [10,11]. For the [110] tensile axis it was shown that the twins can form during creep tests at 750°C and 850 MPa at low strain levels [10]. Ardakani et al. [11] give no detailed mechanism for the twinning, but they presume that the (111) twins were formed as a result of pinning of an a/6[11$\bar{2}$] dislocation segment by the γ'-particles and by the movement of a/3[$\bar{1}$2$\bar{1}$] segments, which both originate from a/2[01$\bar{1}$] dislocations. Apart from the fact that there are considerable differences in microstructure of the NIMONIC alloys of the present study and the superalloys of the last generations, for example in composition, γ'-volume fraction and γ'-morphology, it is possible, that similar mechanisms operate in microtwin formation. But it should be emphasized that the mechanism proposed in the present study involves neither a/2$\langle$110$\rangle$ dislocations nor a/3$\langle$112$\rangle$ dislocations.

A central part of the above presented model is the assumption of certain diffusion steps that enable pairwise dislocation movement. Unfortunately no reliable data from tracer diffusion experiments exist for Al in Ni_3Al. The diffusion of ^{114}In in Ni_3Al, which is believed to behave similar to the diffusion of Al, is via the Ni sublattice by means of Ni vacancies [12]. If we assume a diffusion coefficient D of the order of $10^{-18}\,m^2\,s^{-1}$ for Ni in Ni_3Al, a jump width of $x =$ 0.5 nm ($= a_o\sqrt{2}$, a_o: lattice constant) gives—by the relation $x^2 = 4Dt$ – a time constant of roughly 0.06 s. In this time the dislocations can move two lattice spacings, the corresponding dislocation velocity is 13 $nm\,s^{-1}$. This is a reasonable value, which can be observed in the *in situ* experiments. This correspondence should not be overestimated because the diffusion process operates in the very neighborhood of moving dislocations, which cause strong lattice distortions. This will certainly influence the diffusion process and modify the data that are derived from material, which is normally dislocation free or with a low dislocation density, but always, of course, without moving dislocations.

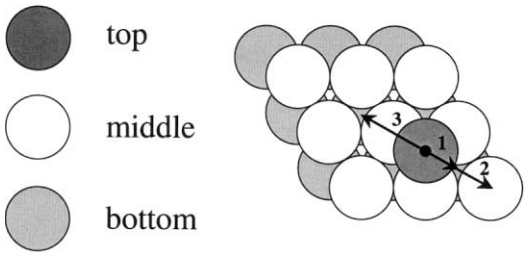

Fig. 5. Three close packed layers, {111} planes, in the fcc lattice (the top layer is represented for clarity by only one atom). A dislocation moving on the "middle" layer shifts the "top" layer in the directions given by arrows 1, 2, 3 according to the respective Burgers vectors $a/6\langle 112\rangle$, $a/6\langle 112\rangle$, $-a/3\langle 112\rangle$. The position reached by the combined shift of arrows $1+2$ is energetically unfavorable.

5. Summary

In the frame of a crystallography/diffusion-based model the pairwise movement of $a/6\langle 112\rangle$ dislocations in the superalloys NIMONIC PE 16 and NIMONIC 105 can be understood as a diffusion-controlled motion. This explains the decrease of the CRSS of the both alloys at around 780°C as the onset of the diffusion of the Ni vacancies, which enables the Al atoms to move. The microstructural features, as they could be resolved by the *in situ* experiments and by the post mortem TEM analysis of *in situ* specimens [1], are in accordance with the model. The main features are (i) the observation of {111} glide planes, (ii) identical $a/6\langle 112\rangle$ Burgers vectors for the whole pairwise moving group of dislocations, (iii) cutting of γ'-particles, (iv) long stacking faults behind the dislocations, and (v) the evolution of microtwins.

Appendix

In the low temperature dislocation movement two $a/2\langle 110\rangle$ dislocations of the γ-matrix move on the same {111} plane and cut the γ'-particles without leaving an APB inside of the γ'-particles. This is possible, because according to the relation (Eq. (1)) they can together transform into a complete dislocation of the $L1_2$ structure:

$$a/2\langle 110\rangle + a/2\langle 110\rangle \Rightarrow a\langle 110\rangle \quad (1)$$

The similar transformation (Eq. (2)),

$$a/6\langle 112\rangle + a/6\langle 112\rangle \Rightarrow a/3\langle 112\rangle \quad (2)$$

for the high temperature movement of the $a/6\langle 112\rangle$ dislocations, is not possible for crystallographic reasons: Two identical $a/6\langle 112\rangle$ dislocations cannot move behind each other on the same {111} plane. This is illustrated in Fig. 5, where the usual stacking sequence ABC for fcc crystals is given in a view perpendicular on a {111} plane. An $a/6\langle 112\rangle$ dislocation is assumed to move on the B layer (middle). This causes the shift of the top layer according to arrow 1. A following dislocation with the same Burgers vector leads to a further shift (arrow 2) into the non-equilibrium position.

The $a/3\langle 112\rangle$ dislocation, which is well known in $L1_2$ materials and superalloys [2], does not exist on the B layer. It is the dislocation with the reverse Burgers vector ($-a/3\langle 112\rangle$), which in theory could be found on the B layer. It causes a shift of the top layer according to arrow 3.

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