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Dynamic dislocation behaviour in the intermetallic compounds NiAl, TiAl and $MoSi_2$

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The dynamic behaviour of dislocations in NiAl, TiAl and $MoSi_2$ on 'easy' slip systems is studied by *in situ* straining experiments in a high-voltage electron microscope. At elevated temperatures, the dislocations are smoothly bent as in NiAl and TiAl or sometimes show superkinks as in $MoSi_2$, and they move in a viscous way. It is suggested that this dynamic behaviour as well as the flow stress anomaly are connected with the formation of atmospheres around the dislocations. A model is proposed assuming that the lowest energy configuration of a dislocation may require a certain number of antisite defects or other point defects in the dislocation core. This cloud of disordered structure may follow partly the moving dislocations to induce an additional friction, analogous to other diffusion controlled mechanisms. The view of atmospheres controlling the dislocation mobility in intermetallics at elevated temperatures is supported by measurements of the dependence of the strain rate sensitivity on the strain rate itself. © 1998 Elsevier Science Limited. All rights reserved

Key words: A. nickel aluminides: based on NiAl, titanium aluminides: based on TiAl, molybdenum silicides, B. plastic deformation mechanisms, D. defects: dislocations, F. electron microscopy.

1 INTRODUCTION

Many intermetallic alloys show a flow stress anomaly, i.e. an increasing flow stress in a range of increasing temperatures or, at least, a plateau (for a review Ref. 1). The flow stress anomaly seems to be quite a general phenomenon, which appears also in materials without an ordered crystal structure. It should be due to an intrinsic property of moving dislocations, and it is frequently accompanied by a characteristic temperature and strain rate dependence of the strain rate sensitivity as well as with serrated flow. The usual explanations are based on the core structure of superdislocations,[2] but the flow stress anomaly is observed also in cases where the deformation is mainly carried by ordinary dislocations as in γ TiAl.[3] Thus, the nature of the flow stress anomaly is still not well understood. The present paper compares the dynamic behaviour of simple dislocations, i.e. of dislocations with Burgers vectors of the type $\langle 100\rangle$ in NiAl, of $1/2\langle 110\rangle$ in γ TiAl, and of $1/2\langle 111\rangle$ in $MoSi_2$, observed by *in situ* straining experiments in a high-voltage electron microscope at elevated temperatures. In addition, the strain rate sensitivity is measured as a function of temperature and strain rate. A new model is proposed to interpret the experimental observations.

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2 EXPERIMENTAL

Microtensile specimens were prepared from NiAl single crystals with a $\langle 111\rangle$ or $\langle 221\rangle$ tensile axis, coarse-grained γ TiAl polycrystals, and $MoSi_2$ single crystals with a $\langle 201\rangle$ tensile axis. These specimens were deformed in a high-temperature straining stage[4] inside a high-voltage electron microsope operated at 1000 kV. The structures and the dynamic behaviour of dislocations were recorded on video tape. Experimental details and results are described for NiAl in Ref. 5 and for TiAl in Ref. 6. The orientations and deformation conditions resulted in a dominance of dislocations with the Burgers vectors mentioned in the Introduction.

Macroscopic compression tests were performed including strain rate cycling and stress relaxation tests in a wide range of strain rates to study the dependence of the strain rate sensitivity $r = \Delta\sigma/\Delta \ln \dot{\varepsilon}$ on the strain rate $\dot{\varepsilon}$. The NiAl crystals were deformed along $\langle 421 \rangle$ and the $MoSi_2$ crystals along $\langle 201 \rangle$. The stress relaxation curves were plotted as $\ln(-\dot{\sigma})$ versus σ. The inverse slope $\Delta\sigma/\Delta \ln(-\dot{\sigma})$ equals the strain rate sensitivity r. Values of r were taken at different stages along the relaxation curves, yielding r as a function of $-\dot{\sigma}$. Strain rates $\dot{\varepsilon}$ were calculated from $-\dot{\sigma}$ via the stiffness of the active part of the load train so that finally r is plotted versus $\log \dot{\varepsilon}$.

3 RESULTS OF *IN SITU* EXPERIMENTS

Figure 1 shows two stages of a video sequence of the motion of a dislocation with a $\langle 100 \rangle$ Burgers vector during *in situ* deformation of NiAl at 475°C. The angular shape of the dislocations is a consequence of the instability of the screw dislocations in the line tension model in anisotropic elasticity, as stated before.[5] The dislocations move in a smooth viscous way conserving their angular shape.

Ordinary dislocations with $1/2\langle 110 \rangle$ Burgers vectors, which dominate during the deformation of γ TiAl, are smoothly curved and move in a viscous way, too, as demonstrated in Fig. 2 for the *in situ* defomation at 660°C, i.e. in the range of the flow stress anomaly. During the first loading, however, the dislocations are formed in a very sudden event and move at very high velocities.

Figure 3(a) proves that in $MoSi_2$ deformed at 990°C, i.e. also in the range of the flow stress anomaly, resting dislocations with $1/2\langle 111 \rangle$ Burgers vectors on $\{110\}$ planes are arranged along $\langle 110 \rangle$ and $\langle 331 \rangle$ directions, i.e. they are not of screw character. Their motion appears either by shifting superkinks as in Fig. 3(b) or by taking a curved shape as in Fig. 3(c). After stopping in Fig. 3(d), the dislocation assumes again the oriented shape. In spite of these core effects, which were not observed for simple dislocations in NiAl and TiAl, the dislocations move in a smooth way, which seems to be a common feature of dislocation motion in intermetallic alloys at high temperatures. In all three materials, the dislocations move sometimes very quickly and stop near groups of other dislocations, showing that the dislocations may experience a low friction at high velocities and that long-range dislocation interactions are important, in addition to the processes controlling the dislocation mobility.

4 RESULTS OF MACROSCOPIC TESTS

The NiAl single crystals show a strong decrease of the flow stress of 220 MPa at room temperature down to 47 MPa at 400°C, but the same value still at 650°C. Figure 4 and the following figures present data on the dependence of the strain rate sensitivity r on the logarithm of the strain rate $\dot{\varepsilon}$. The data were obtained from stress relaxation tests as described in Section 2. The strain rate sensitivity r increases usually with increasing strain. To allow a comparison between different deformation conditions, within one figure data are plotted taken at approximately equal strains, i.e. 3% in Figs. 4 and 5 and less than 1% in Fig. 6. In NiAl, r increases

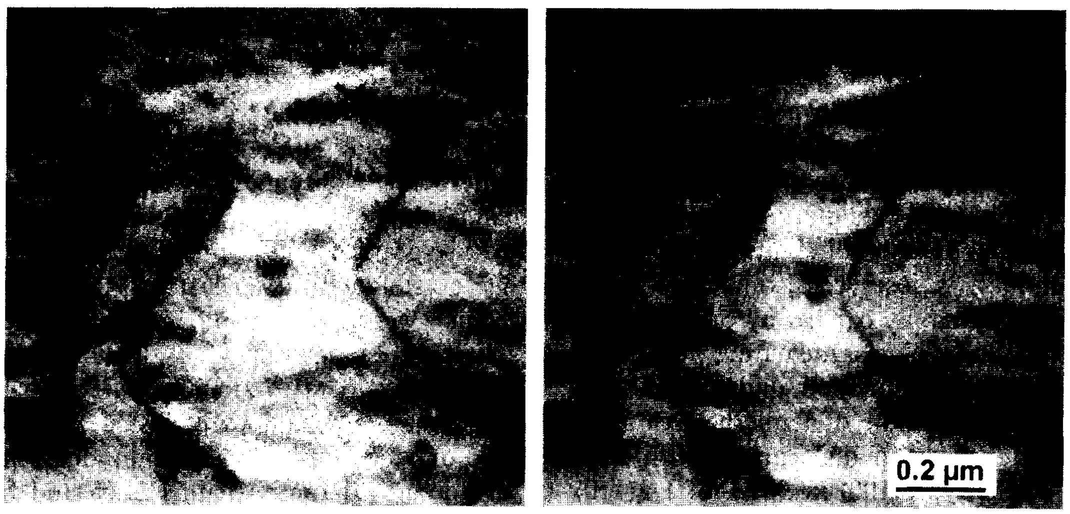

Fig. 1. Two stages of a video record of the viscous motion of a dislocation (marked by arrows) of $\langle 100 \rangle$ Burgers vector on a $\{001\}$ plane during *in situ* deformation of an NiAl single crystal at 475°C.

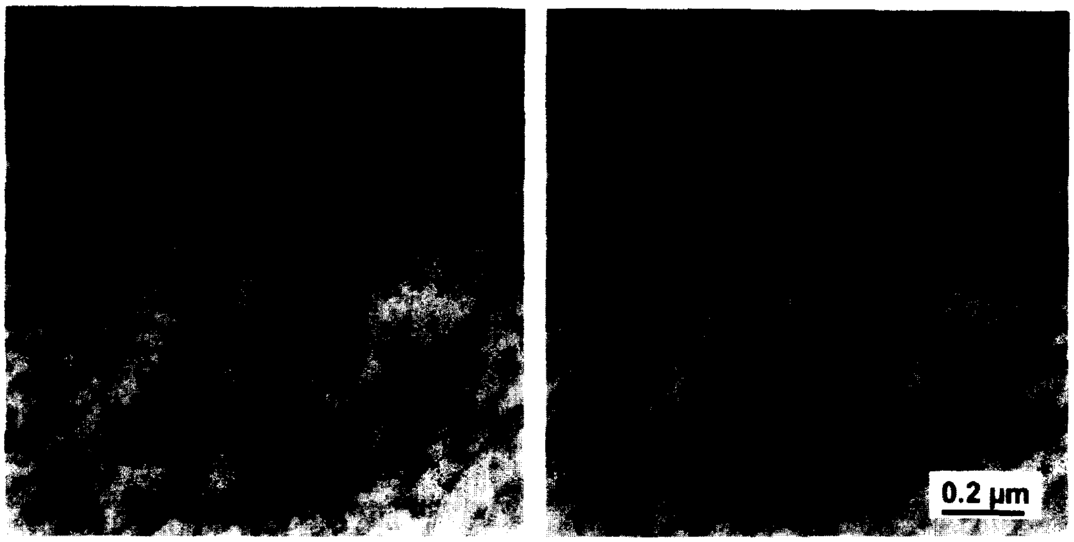

Fig. 2. Two stages of the viscous motion of dislocations of 1/2⟨110⟩ Burgers vector during *in situ* deformation of TiAl at 660°C.

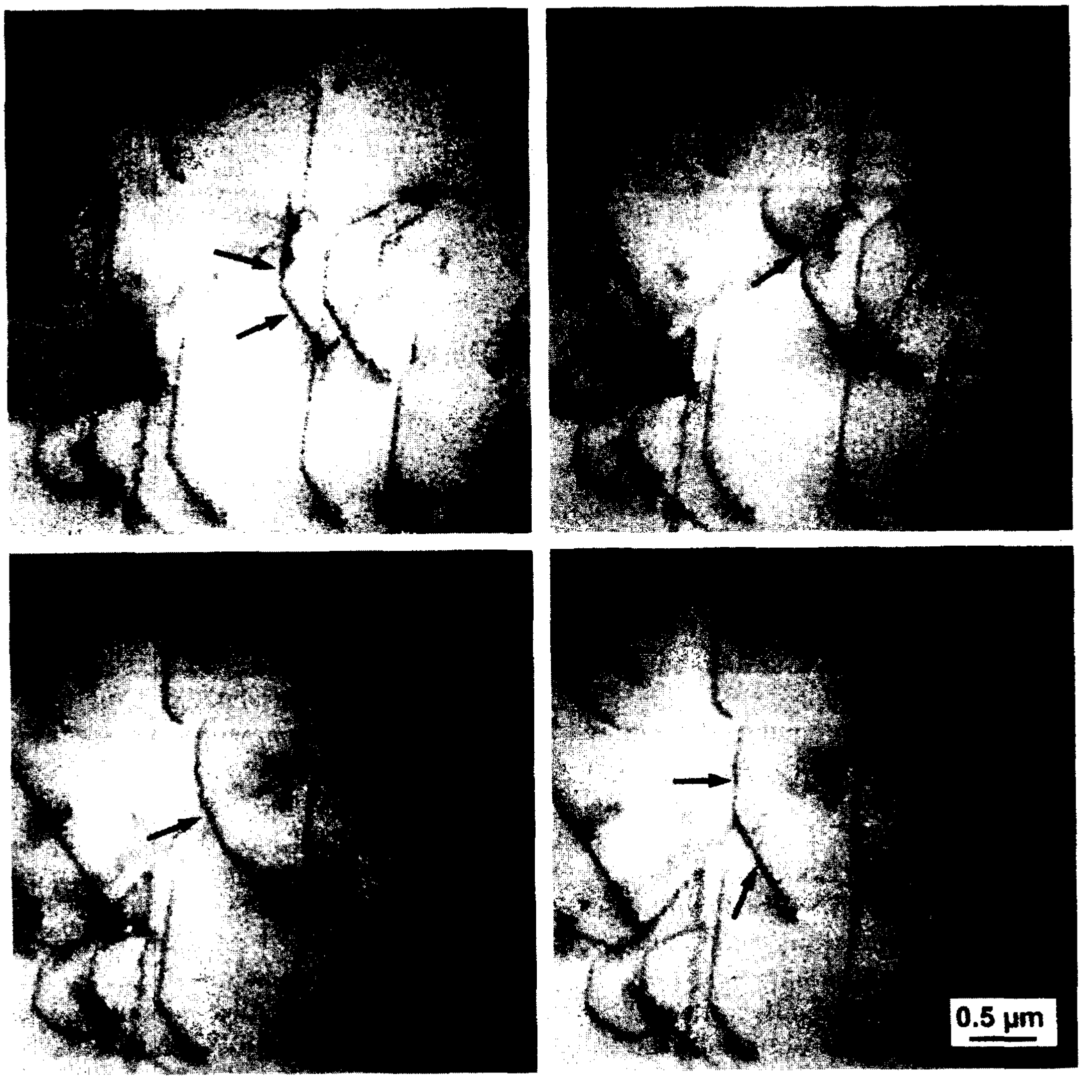

Fig. 3. Several stages of the motion of a dislocation of 1/2⟨111⟩ Burgers vector on a ⟨110⟩ plane during the *in situ* deformation of an $MoSi_2$ single crystal at 990°C. Segments in preferred orientations, a superkink and a bowed segment are marked by arrows.

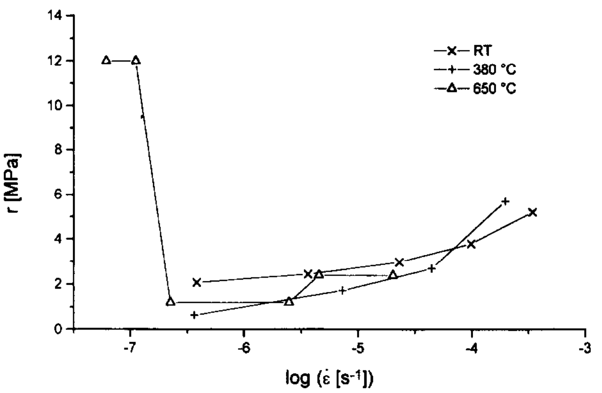

Fig. 4. Dependence of the strain rate sensitivity r on log $\dot{\varepsilon}$ for NiAl single crystals deformed along a ⟨421⟩ orientation at different temperatures. All data were taken from stress relaxation curves at a strain of about 3%.

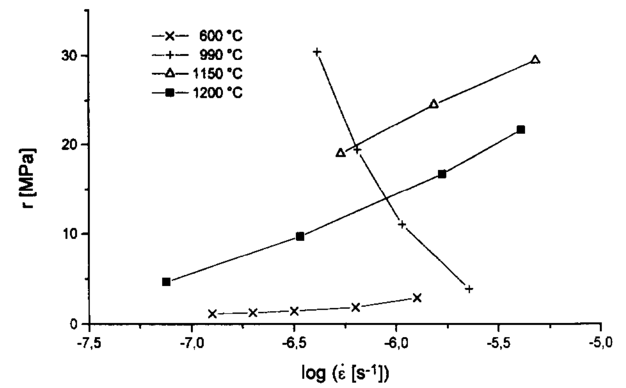

Fig. 6. Dependence of the strain rate sensitivity r on log $\dot{\varepsilon}$ for $MoSi_2$ single crystals deformed along a ⟨201⟩ orientation at different temperatures. All data were taken from stress relaxation curves at a strain of some tenths of a per cent.

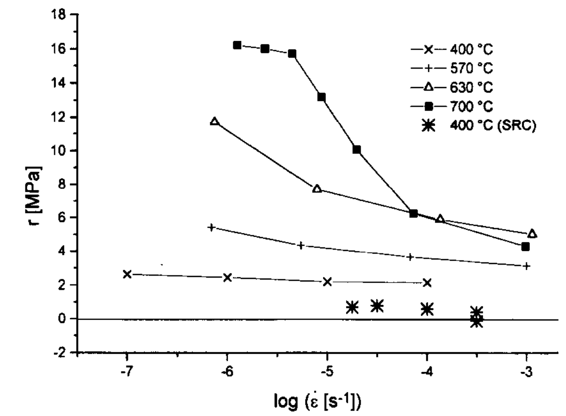

Fig. 5. Dependence of the strain rate sensitivity r on log $\dot{\varepsilon}$ for TiAl polycrystals deformed at different temperatures. Most data were taken from stress relaxation curves at about 3%. The data marked by asterisks originate from strain rate cycling tests.

with increasing log $\dot{\varepsilon}$, as expected of the thermally activated overcoming of obstacles to dislocation motion. In the whole temperature range, r is independent of temperature. At 650°C, a different process occurs at very low strain rates.

TiAl shows a flow stress anomaly as the flow stress remains constant at about 190 MPa through the whole temperature range between room temperature and 700°C. Figure 5 demonstrates that the strain rate sensitivity r exhibits an inverse dependence on log $\dot{\varepsilon}$. In accordance with this observation, r decreases with decreasing temperature. Strain rate cycling experiments (SRC) at 400°C show even a slightly negative strain rate sensitivity. This effect cannot be observed in stress relaxation tests.

$MoSi_2$ in ⟨201⟩ orientation displays a flow stress anomaly with the peak stress between about 950 and 1000°C as observed previously.[7] As plotted in Fig. 6, r shows a normal dependence on log $\dot{\varepsilon}$ at 600°C, a temperature in the range of the usual low temperature flow stress decrease. However, in the range of the flow stress anomaly at 990°C, r shows a strong inverse dependence on log $\dot{\varepsilon}$. At high temperatures, r behaves again in the normal way. The measurements on the different materials prove that there exists an intimate relation between the occurrence of a flow stress anomaly and the inverse rate dependence of r.

5 DISCUSSION

Thermally activated mechanisms controlling the deformation at room temperature, i.e. a jog mechanism in NiAl[5] and precipitation hardening in TiAl,[6] cease to operate in the range of elevated temperatures discussed in the present paper. Here, the dislocations move viscously in all materials studied, which should be connected with an additional contribution to the flow stress, not operative at low temperatures and perhaps responsible for the flow stress anomaly in the respective cases.

It has been suggested by a number of authors (see, e.g. Ref. 8 and Ref. 9) that the flow stress anomaly is connected with strain ageing, i.e. the formation of point defect atmospheres around the dislocations. In these papers, defects existing in the lattice are supposed to form these atmospheres: impurities, additions in low concentrations, or structural defects as vacancies or antisite defects as a consequence of a non-stoichiometric composition of the materials. The theory describes the interaction of theses defects with the dislocations (e.g. Ref. 10). Binding energies are calculated by determining the difference between the energy of a dislocation containing the defect in its core and the sum of the energies of the dislocation without the defect and the formation energy of the defect. In most cases positive or negative binding energies are obtained below 1 eV. However, in a particular case—an Ni antisite defect in the core of a dislocation along $\{01\bar{1}\}$ with a $\langle 111\rangle$ Burgers vector in NiAl—the dislocation reconstructs locally, and the binding energy turns out to be remarkably larger than the formation energy of the defect. This means that the energy of the dislocation including the defect in its core is lower than the energy of the dislocation without the defect.

In this paper, a new model is proposed. It considers that the latter observation need not be an exceptional case, but that generally in intermetallics the lowest energy state of a dislocation may be a configuration containing a certain concentration of point defects in the dislocation core, independent of other chemical or structural disorder of the material. In particular, the arrangement of atoms in the core of the dislocation may differ from the regular arrangement in the ordered structure. During motion, this low energy state can only follow the dislocation via diffusional processes inducing a frictional force, in close analogy to the formation of atmospheres of existing defects (for a review of the theory, see Ref. 11). The diffusion processes inside the dislocation core will then be of short-range character and may occur at much lower activation energies than the respective processes in the bulk, as observed in Ref. 9. Molecular dynamics simulations allowing for atomic mobility should be carried out to prove the possibility of the new mechanism. It is of intrinsic nature and suggests a viscous motion of the dislocations as observed by the *in situ* experiments. As discussed in Ref. 11 the frictional stress increases with increasing dislocation velocity, reaching a maximum, and finally decreases at even higher velocities. The low resistance to motion at high velocities may explain the fact that the dislocations sometimes move very fast, most clearly expressed in the instantaneous formation and motion of dislocations during the first loading of TiAl specimens in the *in situ* experiments. Under the assumption of a constant mobile dislocation density, the slope of the curve of the frictional stress versus the logarithm of the dislocation velocity equals the strain rate sensitivity r. It is high at low velocities or strain rates, goes to zero at the maximum of the frictional stress, and turns to negative values thereafter. Figures 5 and 6 prove this decrease of r with increasing log $\dot{\varepsilon}$ for TiAl and $MoSi_2$, just in the ranges of their flow stress anomaly, while the dependence is normal in Fig. 4 of NiAl, which does not show a flow stress anomaly. With increasing strain, the dislocation density increases, leading to lower dislocation velocities and correspondingly to higher values of r.

In conclusion, the viscous motion of dislocations observed during *in situ* straining experiments in an HVEM may be interpreted by the formation of atmospheres in the cores of moving dislocations. These atmospheres need not only consist of point defects already existing in the material, but they may be of intrinsic nature if the dislocations have a lower energy in a configuration comprising a certain concentration of point defects compared to their usual ordered state. The consequence of the action of atmospheres is an 'inverse' dependence of the strain rate sensitivity on the strain rate, as it is observed experimentally in the ranges of the flow stress anomaly. Of course, further experimental and theoretical studies are necessary to prove whether the proposed model acts in some of the intermetallic materials, or not.

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