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High-voltage electron microscope high-temperature *in situ* straining experiments to study dislocation dynamics in intermetallics and quasicrystals

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Summary

The dynamic behaviour of dislocations in several intermetallic alloys, studied by *in situ* straining experiments in a high-voltage electron microscope, is compared at room temperature and at high temperatures. In contrast to room temperature, the dislocations move viscously at high temperatures, which is explained by diffusion processes in the dislocation cores. In quasicrystals, the viscous dislocation motion can be interpreted by models on the cluster scale.

1. Introduction and experimental

The present paper reviews recent results of the author's research group on the dislocation motion during the plastic deformation of different materials. *In situ* straining experiments at temperatures up to 1150 °C were performed in a high-voltage electron microscope (HVTEM). Because it is difficult to produce temperatures above 1000 °C in a TEM specimen chamber by resistance heating, almost all *in situ* deformation studies published in the literature were carried out below 900 °C. The present double-tilting high-temperature straining stage (Messerschmidt & Bartsch, 1994) uses electron bombardment to heat the specimen grips, a method introduced into the design of TEM stages by Komatsu *et al.* (1977) which allows users to produce a high heating power within a small volume. All operations of the stage, i.e. the temperature control via thermocouples on both grips, the straining control and the force measurement, are fully computer-controlled, as outlined in Fig. 1. In order to avoid the destruction of sensitive digital equipment by possible discharges in the high-voltage circuits of the electron bombardment heating systems, the electric parts connected to the stage are galvanically separated from the analogue-digital and digital-analogue converters by opto-couplers in an input-output amplifier box. During the experiments, the motion of dislocations is directly observed and recorded on video tapes. This experimental set-up opens unique opportunities to study the microprocesses of current high-temperature materials including ceramics in the temperature range of their application. Because some of these materials are very brittle at room temperature, the preparation of the microtensile specimens as well as their handling requires particular care.

2. Results

The macroscopic plastic properties of crystalline materials are controlled by both the dynamic behaviour of individual dislocations and their mutual long-range elastic interactions. By means of the usual mechanical testing, only the joint action of both phenomena can be observed. Conventional transmission electron microscopy of deformed specimens reveals static dislocation structures, which give information on the dislocation interactions but tell mostly very little about the processes that control the dislocation dynamics. *In situ* deformation in a TEM is therefore almost the only way to study the dynamic behaviour of individual dislocations. These experiments allow a characterization of the configurations of dislocations under load, i.e. whether they are smoothly bent, bowed-out between obstacles or arranged along preferred crystallographic directions. They also show whether the dislocations move in an unstable or stable way and whether the motion is smooth and viscous or jerky. These dynamic properties allow us to draw conclusions on the mechanisms controlling the dislocation mobility.

In the intermetallic alloys NiAl (Messerschmidt *et al.*, 1997), NiAl-0.5at%Ta (Messerschmidt *et al.*, 1999a), Ti-52at%Al (Häussler *et al.*, 1999) and $MoSi_2$ (Guder *et al.*, 1999), the dislocation dynamics show a number of similarities (Messerschmidt *et al.*, 1999b). Near room temperature, the dislocation mobility is controlled by

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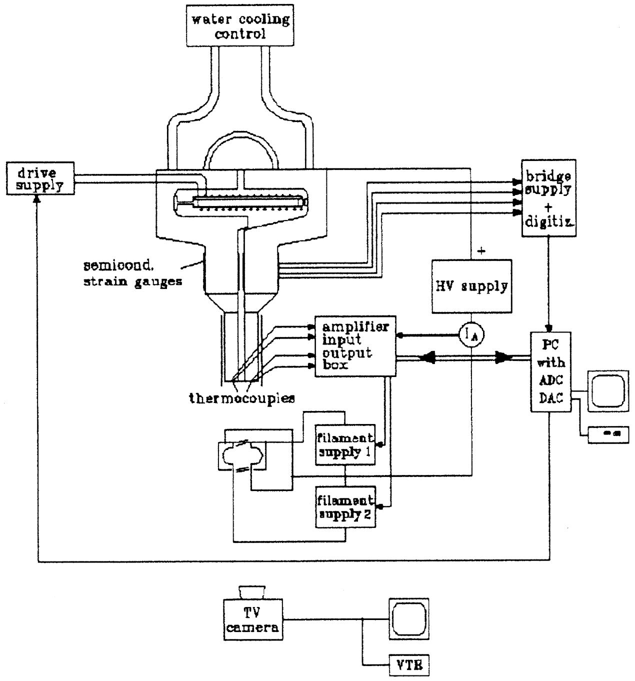

Fig. 1. Scheme of electric control of the high-temperature deformation stage after Messerschmidt & Bartsch (1994).

thermally activated processes such as overcoming localized obstacles, impeding by jogs or the Peierls mechanism. In the first two cases, the dislocations bow out between the obstacles and move in a jerky way. At high temperatures, the dislocations move in either an unstable or a viscous way. In most materials they are then smoothly bent. Successful *in situ* straining experiments on $MoSi_2$ single crystals, which are very brittle even at 1000 °C, showed that the dislocations are straight and orientated along crystallographic directions, as shown in Fig. 2. During their motion, the dislocations may form superkinks as in dislocations A2 and A3 in Fig. 2 at 12 s, or they may attain a smoothly bent shape as in dislocation A2 at 60 s. The similarities in the high-temperature behaviour of the dislocations in different materials are interpreted by diffusion processes in the dislocation cores. The details of these processes can differ greatly. Extrinsic point defects such as impurity atoms may form atmospheres around the dislocations. These atmospheres can also consist of intrinsic point defects such as vacancies or antisite defects in intermetallics. According to a new model, the configuration of lowest energy of a dislocation may require a certain concentration of point defects in the dislocation cores, which are not present in the undisturbed crystal (Messerschmidt *et al.*, 1998a; Messerschmidt *et al.*, 1999b). The atmospheres will partly move with the dislocations, causing an additional dragging force. The straight shape of dislocations in $MoSi_2$ may result from a climb dissociation

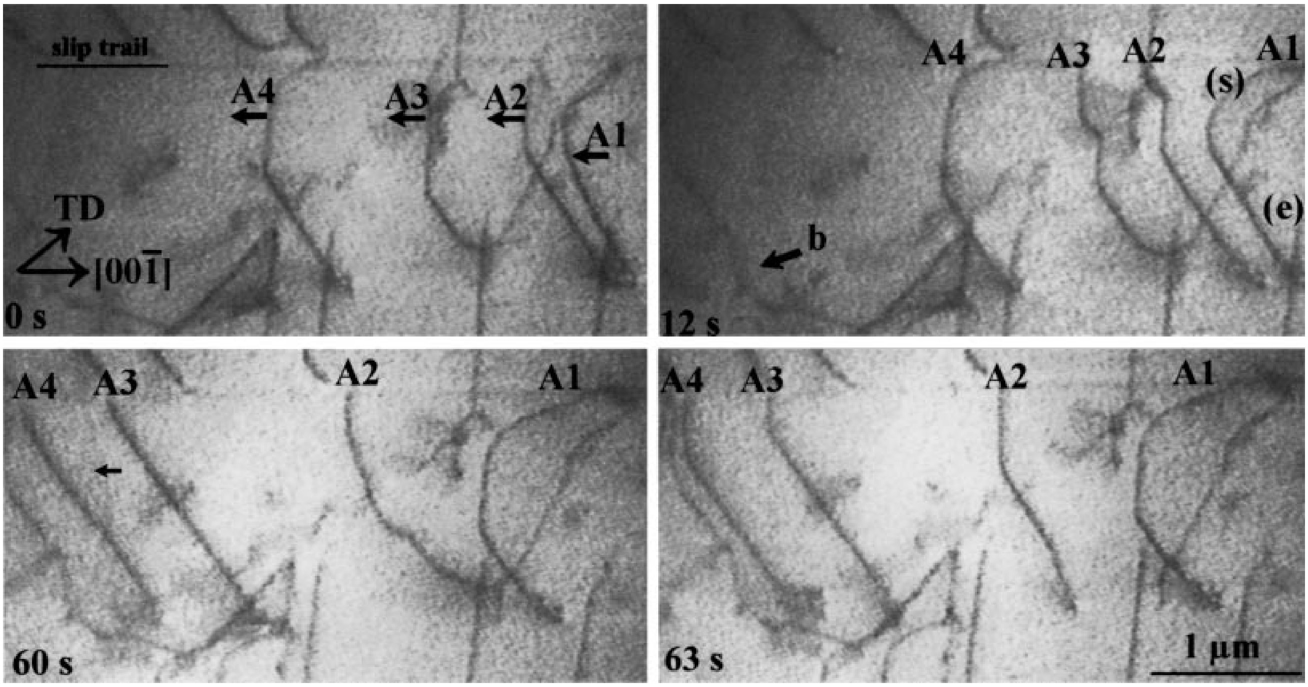

Fig. 2. Video sequence of the motion of dislocations A1 to A4 of $1/2[1\bar{1}1]$ Burgers vectors on $\{110\}$ planes during *in situ* deformation of a $MoSi_2$ single crystal in a high-voltage electron microscope at 1000 °C. TD: tensile direction. Arrows: direction of motion. b, Projection of direction of Burgers vectors. e, Edge component. s, Screw component. After Guder *et al.* (1999).

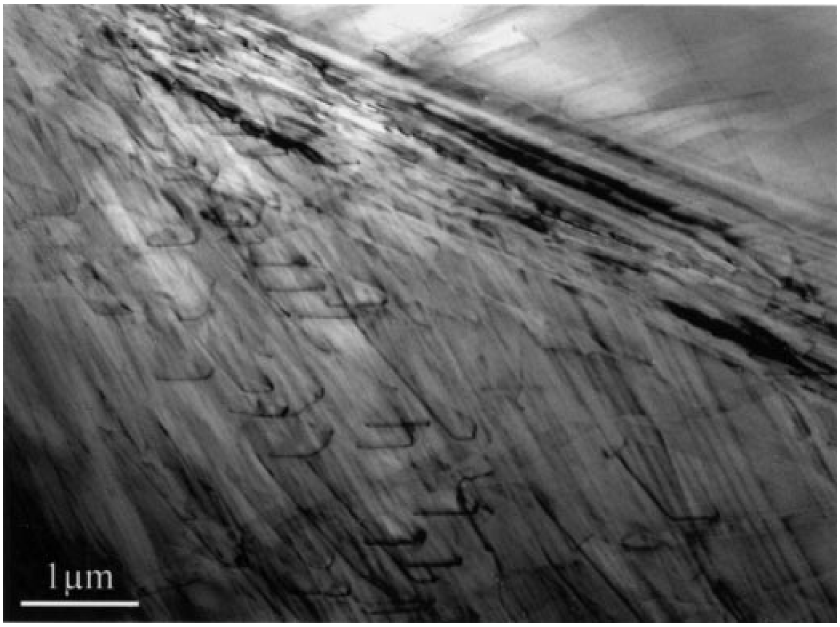

Fig. 3. Moving dislocations and slip traces during the *in situ* deformation of an icosahedral Al-Pd-Mn single quasicrystal at about 750 °C. After Messerschmidt *et al.* (1999c).

(Messerschmidt *et al.*, 2001). During the motion, there will be conservative climb between the partial dislocations. The models are in agreement with the different macroscopic deformation behaviour of the materials, particularly the dependence of the strain rate sensitivity on the stress. In $MoSi_2$ polycrystals, deformation above about 1000 °C is carried by glide and decohesion of a grain boundary phase (Junker *et al.*, 1999). The dislocation processes near cracks in NiAl single crystals are studied by Baither *et al.* (1999).

For the first time, Wollgarten *et al.* (1995) showed directly that plastic deformation in Al-Pd-Mn single quasicrystals is carried by a dislocation mechanism by observing moving dislocations during *in situ* straining experiments at high temperatures. Because single quasicrystals are also very brittle up to about 70% of the melting temperature, these experiments were only possible in a relatively narrow temperature window around 700 °C. Figure 3 shows that many dislocations arrange along crystallographic directions. The dislocations also move in a viscous way. Cross slip was observed frequently but not as often as in most crystalline metals. Cross slip leads to dislocation multiplication (Messerschmidt *et al.*, 2000b). The dislocation velocity was measured from video recordings. It may show a bimodal distribution (Messerschmidt *et al.*, 1998b). Models of the dislocation mobility in quasicrystals are based on the cluster structure of these materials (Feuerbacher *et al.*, 1997; Messerschmidt *et al.*, 1999c; Messerschmidt *et al.*, 2000).

3. Conclusions

- *In situ* straining experiments in a TEM on current high-temperature materials at temperatures above 1000 °C can successfully be performed by heating the specimen grips by electron bombardment and by careful preparation of the frequently brittle microtensile specimens.
- The results show that these experiments yield important information on the processes controlling the dislocation mobility, which cannot be obtained by other methods.
- The coexistence of viscous and unstable dislocation motion in several intermetallic alloys can be explained by diffusion processes in the cores of moving dislocations.

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