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# Dislocation processes during the deformation of NiAl-0.2at.%Ta

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#### Abstract

Macroscopic compression tests and in situ straining experiments in a high-voltage electron microscope were performed on NiAl– 0.2at.%Ta at room temperature and at elevated temperatures. At room temperature in soft orientations, dislocations of  $a\langle 100 \rangle$ Burgers vectors bow out between jogs. In contrast to pure NiAl, the dislocations move in a viscous way between the pinned configurations. At 475°C in a hard orientation, dislocations with  $a\langle 110 \rangle$  Burgers vectors move in a viscous way in configurations strongly depending on the respective slip plane. Preferred orientations of dislocations are of mixed character, most pronounced as very straight dislocations oriented along  $\langle 111 \rangle$  directions on  $\{110\}$  planes. These configurations cannot be explained on the basis of the existing atomistic theories. The flow stress is interpreted in terms of the back stress of the dislocations bowing out between jogs at room temperature, the statistical theory of solid solution hardening, and the formation of atmospheres containing Ta atoms at elevated temperatures. © 1999 Elsevier Science Ltd. All rights reserved.

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# 1. Introduction

Ordered intermetallic alloys may fill the gap between conventional metals and high-temperature ceramics for structural applications at high temperatures, i.e. above about 1000°C. Owing to their ordered crystal structure they show particular mechanical properties which are controlled by various factors including the microscopic structure of the dislocation cores. Materials on the basis of the intermetallic compound NiAl with the B2 crystal structure combine a high melting point (1638°C) with a relatively low density (about 6 g/cm<sup>3</sup>) and a good corrosion resistance. However, NiAl materials suffer from a comparatively low creep strength above 1000°C as well as a poor room temperature ductility. Therefore, considerable efforts are made worldwide to improve these properties.

One possibility to increase the high-temperature creep strength is the incorporation of hard phases via alloying NiAl with Ta. At a sufficiently high concentration, a TaNiAl Laves phase precipitates, preferably at the grain boundaries, to form a hard skeleton, which surrounds the soft NiAl grains and gives rise to strong hardening [1]. As a step towards a deeper understanding of the microprocesses controlling the mechanical behaviour of the composite material, the matrix phase, i.e., the solution-hardened alloy NiAl–0.2at.%Ta is studied in the present paper.

In NiAl, the macroscopic mechanical parameters like strength, hardness and ductility are strongly influenced by the dynamic properties of dislocations. The basic experimental observations and the theoretical interpretation are reviewed in Ref. [2]. The deformation behaviour is characterized by a variety of slip planes and three types of Burgers vectors, i.e., a(100), a(110) and  $a\langle 111\rangle$ . Dislocations with  $a\langle 100\rangle$  Burgers vectors have the highest mobility [3–5]. Single crystals with a tensile or compression axis in a cube orientation, where these dislocations have a zero orientation factor, show a much higher critical shear stress than specimens with loading axes away from  $\langle 100 \rangle$ . The cube orientations are therefore called hard orientations. At room temperature, only dislocations with a(100) Burgers vectors are activated during deformation in soft orientations. In the hard orientations, dislocations with a(111) Burgers vectors move at very high stresses [6,7]. The brittle-toductile transition above about 330°C seems to be connected with a transition from dislocations with a(111)Burgers vectors to such with a(110) ones. Most of these

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results are corroborated by calculations of the Peierls stresses of the different slip systems [8].

The increased ductility at elevated temperatures is certainly also connected with the onset of diffusion [9,10]. Edge dislocations with  $a\langle 110 \rangle$  Burgers vectors decompose under stress into two dislocations with  $a\langle 100 \rangle$  Burgers vectors, separated by distances between about 6 and 500 nm [11]. They do not share a common slip plane so that they cannot glide conservatively together. The propagation of such a decomposed edge segment is described as a viscous lateral motion of macrokinks. It requires the decomposed segment to constrict, which can occur by conservative climb via diffusion between both decomposed edge components [10].

Recently, in situ straining experiments, using a transmission electron microscope, have been performed on polycrystalline NiAl [12] and on single crystals along soft orientations [13,14]. They have shown [14] that at room temperature the dislocations are pinned by high jogs and move in a jerky way by shifting the jogs in the direction of their Burgers vector. This sidewards motion of the jogs may be controlled by the Peierls stress. At elevated temperatures, the jog density is drastically reduced. Long segments move in a viscous way. This has been interpreted by diffusion processes in the dislocation core [14].

It is the aim of the present paper to study the influence of solution hardening by the addition of 0.2 at.% of Ta on the dynamic properties of dislocations in NiAl. To this end, in situ straining experiments have been performed inside a high-voltage electron microscope. The results are compared with previous ones on pure NiAl [14]. Macroscopic tests were carried out under similar conditions as the in situ experiments in order to determine the flow stress and its strain rate sensitivity.

# 2. Experimental

The NiAl-0.2at.%Ta alloy was prepared by ingot metallurgy using basic metals of a purity of 99.95 wt% (Ni), 99.97 wt% (Al), and 99.8 wt% (Ta) [15,1]. It was melted in a vacuum induction furnace and remelted in a Bridgeman furnace. The Ni:Al ratio was chosen as 1:1 corresponding to stoichiometry and the deviations from the nominal alloy composition were less than 0.5 wt% (Ni) and 1.1 wt% (Al).

Macroscopic compression tests were performed at room temperature, 450°C and 650°C/715°C in a singlescrew testing machine on rectangular parallelepipeds of  $2 \times 2 \times 6$  mm<sup>3</sup> in size. The strain rate sensitivity of the flow stress  $\sigma$  was determined by stress relaxation tests. The logarithm of the rate of the stress decrease  $-\dot{\sigma}$  was plotted versus  $\sigma$ . In this diagram, the strain rate sensitivity *r* is given by the inverse slope of the relaxation curves (see the review [16])

$$=\frac{\mathrm{d}\sigma}{\mathrm{d}\ln\dot{\varepsilon}}=\frac{\mathrm{d}\sigma}{\mathrm{d}\ln(-\dot{\sigma})},\tag{1}$$

where  $\dot{\varepsilon}$  is the strain rate.

For the in situ straining experiments, micro-tensile specimens were cut, ground and jet polished in two steps between platinum masks using an electrolyte consisting of methanol and nitric acid  $(-40^{\circ}\text{C}, 11 \text{ V})$ . Usually, the central region of the strip-shaped specimens showed large transparent regions around a single perforation. Because of the relatively large grains, the active gauge length of the in situ specimens resembles a single crystal sample of arbitrary orientation. As it turned out, the specimens investigated at room temperature were of soft orientations while those studied at high temperatures were of a hard orientation.

For in situ straining, the specimens were mounted onto the grips of quantitative straining stages either for room temperature [17] or for grip temperatures up to  $1250^{\circ}$ C [18] at a maximum load of 15 N. The high-voltage electron microscope was operated at 1 MeV. The deformation was carried out by small load increments such that the changes of the microstructure could be recorded under full load either on photographic film or on video tape. In order to minimize unavoidable radiation damage during the in situ experiment, the dislocation structures were usually characterized in more detail in the unloaded state in a wide-angle goniometer ( $\pm 45^{\circ}$ ) at room temperature.

The validity of the results of in situ straining experiments may generally be limited by the low thickness of the specimens even in the high-voltage electron microscope and by the radiation damage inside the latter.

# 3. Results

# 3.1. Macroscopic compression tests

Fig. 1 shows the stress-strain curves of the material at room temperature, 450°C and 650°C/715°C. The critical flow stress decreases only moderately. The stress-strain curves show a pronounced hardening, particularly at small strains at room temperature. Fig. 2 presents some typical stress relaxation curves plotted as  $\ln (-\dot{\sigma})$  versus  $\sigma$  as described in the preceding section. The curves 1 and 3 corresponding to small strains are almost straight. The curves measured at large strains show a slight curvature of the "usual" sign at room temperature (curve 2) and of an "inverse" sign at high temperature (curve 4), i.e., with an increasing strain rate sensitivity at decreasing stress or strain rate. This shape is more pronounced for curve 5 of 715°C.

The results are also summarized in Table 1. For comparison, the data of pure NiAl single crystals [19]. deformed along a [124] compression axis are included.



Fig. 1. Typical stress–strain curves of NiAl–0.2at.%Ta at room temperature,  $450^{\circ}C$  and  $650^{\circ}C/715^{\circ}C$ .

The critical flow stresses of pure NiAl should be reduced by 5–10 MPa to take into account the higher strain rate of these measurements. The strain rate sensitivity r is always determined at the beginning of each relaxation curve, thus corresponding to the macroscopic deformation rate. The strain rate sensitivities are generally very low. The values of NiAl–0.2at.%Ta show a minimum at  $450^{\circ}$ C.

# 3.2. In situ straining experiments in a high-voltage electron microscope

# 3.2.1. Room temperature

In these experiments, the tensile directions were in soft orientations, so that dislocations of  $a\langle 100 \rangle$  Burgers vectors were activated. These dislocations moved mostly on {001} planes, sometimes also on {110} ones. Fig. 3 shows the typical structure of such dislocations, which have moved during room temperature in situ straining.



Fig. 2. Typical stress relaxation curves at room temperature (curve 1 at 0.2%, curve 2 at 2.0% strain), 450°C (curve 3 at 0.8%, curve 4 at 6%) and 715°C (curve 5 at 2.2%).

The active slip plane was identified as (010), the alignment of most of the dislocations parallel to the screw orientation is obvious. As resolved in the video recordings, the dislocations stop in pinned configurations, shown in the micrographs of Figs. 3 and 4. Between the pinned configurations, however, they move in a viscous way, in contrast to pure NiAl, where they move quite jerkily. Between the pinning agents, the dislocations bow out under stress. The bowed segments show an angular shape, in agreement with the theoretical shape illustrated in the inset of Fig. 3 and discussed in Section 4. The density of the pinning agents along the dislocation line varies considerably from dislocation to dislocation, compare, e.g., the dislocation marked A in Fig. 3 with very long segments and that marked B with many short ones. Correspondingly, the distributions of the segment lengths in some specimen areas show a bimodal shape. The histogram of all segments measured does not show the bimodal distribution anymore. The average distances between the obstacles amount to about 150 nm.

The moving dislocations trail a large number of debris, i.e. short dislocation dipoles, as in Fig. 4. The debris may represent effective obstacles to further dislocation motion.

# $3.2.2.\ 475^{\circ}C$

All experiments at elevated temperatures were accidentally performed on grains in hard orientations. Dislocations with  $a\langle 100 \rangle$  and, particularly, with  $a\langle 110 \rangle$  Burgers vectors have been identified. Fig. 5 shows dislocations with  $a\langle 100 \rangle$  Burgers vectors labelled A, which are not expected in the hard cube orientation where they have a low orientation factor. Apparently, internal stresses are responsible for the formation of these dislocations. Similar to room temperature, their bowed-out segments show the characteristic angular shape demonstrated in the inset, but the density of pinning agents is remarkably lower than at room temperature. These dislocations produce debris in the form of lens-shaped loops.

The shape of dislocations with  $a\langle 110 \rangle$  Burgers vectors depends strongly on their glide plane. Fig. 6 shows such dislocations labelled A, moving on {111} planes and trailing slip traces. Segments parallel to the surfaces along  $[0\ \overline{1}\ 1]$  seem to be quite immobile. The moving parts show preferential orientations along  $[1\ 2\ \overline{1}]$  directions. These preferred orientations do not correspond to the edge and screw orientations. The equilibrium shape from line tension calculations is demonstrated in the inset of Fig. 6. The experimental shapes differ from the theoretical equilibrium shape and may therefore be determined by an anisotropic glide resistance. Video records show that these dislocations move in a continuous viscous way. Fig. 6 also exhibits a large number of dislocations of the same type of Burgers vectors

Material	<i>T</i> (°C)	$\dot{\varepsilon}$ (s <sup>-1</sup> )	$\sigma_0$ (MPa)	r (MPa)
NiAl	20	$10^{-4}$	220	3.5–4
single crystal	400	$10^{-4}$	$\approx 50$	2.5–3
	650	$10^{-4}$	47	2
	20	$10^{-5}$	380	6–8
NiAl	450	$10^{-5}$	270	3
0.2 at.%Ta	650	$10^{-5}$	200	4
	715	$10^{-5}$	160	10

Table 1 Results of compression tests

Critical resolved shear stress  $\sigma_0$  and strain rate sensitivity *r* of NiAl single crystals with [124] compression axis and NiAl–0.2%Ta determined for various temperatures *T* and different strain rates  $\dot{\epsilon}$ .

which are not located on {111} slip planes. They have been created during the in situ experiments but have never been seen to move. As observed in stereo pairs, they consist usually of long segments parallel to the foil surfaces. Sometimes, long segments near both surfaces are connected by a segment with another orientation as labelled by B. All segments are arranged along preferred crystallographic orientations. Unfortunately, the crystallographic directions of these dislocation segments and the plane in which they extend could not be determined unequivocally. Most probably, it is not a low-index plane. In some regions of the specimens, these dislocations, which consist of the two segments parallel to the surfaces with a connecting third segment, appear in a high density, as demonstrated in Fig. 7. The trace of their a $\langle 110 \rangle$  Burgers vector is parallel to the [010]  $\vec{g}$ vector. Thus, the orientation factor of these dislocations should be zero. Apparently, they were formed owing to an inhomogeneous stress field in the in situ specimen. The segments connecting parts parallel to the surfaces of different dislocations are arranged in rows oriented along  $\langle 100 \rangle$  directions, most probably owing to mutual



Fig. 3. Dislocations with  $a\langle 100 \rangle$  Burgers vectors moving on {100} planes during in situ deformation in a high-voltage electron microscope at room temperature. (A) Dislocation with long segments bowing out between pinning agents. (B) Dislocation with short segments. Inset in this and the following figures: equilibrium shape of dislocations under stress in the projection of the micrograph calculated by the line tension model using anisotropic elasticity theory, see Section 4. TD: tensile direction.



Fig. 4. Dislocations with  $a\langle 100 \rangle$  Burgers vectors moving during in situ deformation at room temperature and having produced a large number of debris.



Fig. 5. Dislocations of  $a\langle 100\rangle$  Burgers vectors (labelled A) and  $a\langle 110\rangle$  ones (labelled B) during in situ deformation at 475°C.

elastic interaction. Both the origin of these dislocations and their role in the deformation process could not be revealed.

Fig. 8 demonstrates that dislocations with  $a\langle 110 \rangle$ Burgers vectors show a very different morphology when they move on  $\{110\}$  planes. They consist of very straight segments oriented in  $\langle 111 \rangle$  directions, i.e., they are neither of screw nor edge character. The segments are connected by a small arc and move in a viscous way.







Fig. 7. Region of a high density of dislocations with  $a\langle 110 \rangle$  Burgers vectors of similar geometry as the dislocations marked B in Fig. 6, taken during in situ deformation at 475°C.

# 4. Discussion

The in situ experiments at room temperature were performed in soft orientations. They can therefore be compared with similar experiments on pure NiAl [14]. As shown in Table 1, the flow stress in NiAl–0.2at.%Ta is higher than that in pure NiAl in a single slip orientation. The strain rate sensitivity of the flow stress r is slightly higher, too. It corresponds to an activation volume [16]

$$V = kT/(m_{\rm s}r) \tag{2}$$

of 1.75 nm<sup>3</sup> or about 75  $b^3$ . Here, k is Boltzmann's constant, T the absolute temperature, and  $m_s$  the orientation factor. An average value of  $m_s = 0.33$  was chosen. At room temperature, the stress relaxation curves are straight or show the "usual" curvature, i.e., a decreasing strain rate sensitivity with decreasing stress, which indicates that the dislocation mobility is controlled by an obstacle mechanism.

As in pure NiAl, the deformation is carried by dislocations of  $a\langle 100 \rangle$  Burgers vectors, mainly of screw character. Most dislocations moved on  $\{100\}$  or  $\{110\}$ planes.  $\{210\}$  glide planes, which were observed frequently in pure NiAl [14], have not been observed in NiAl–0.2at.%Ta. The screw dislocations are again pinned and bow out between the pinning agents. The shape of the bow-outs is similar to that in pure NiAl and is determined by the line tension. The inset in Fig. 3 shows the equilibrium shape of a dislocation under stress, calculated in the framework of the line tension model using anisotropic elasticity theory. Since the elastic constants of NiAl–0.2at.%Ta are not available, the data were taken for pure NiAl from Fig. 6 of Ref. [2]. As found out previously [20,14], the pure screw dislocation is unstable in NiAl, leading to the characteristic angular shape, which is observed also in this study. The instability of the screw dislocations with a $\langle 100 \rangle$  Burgers vectors occurs on all planes investigated so far, i.e. on the {100}, {110} and {210} planes.

It was concluded from the dynamic behaviour of the dislocations [14] that the pinning agents are jogs in NiAl. The jogs are shifted parallel to the direction of their Burgers vector so that very long segments appear with large bow-outs before these segments become unstable, which is not consistent with a precipitation hardening mechanism. The occurrence of very different segment lengths is also evident in the present material, see dislocations A and B in Fig. 3. These similarities suggest that the jog mechanism is operative in NiAl-0.2at.%Ta, too. In pure NiAl, the flow stress can quantitatively be understood by the back stress of the bowedout segments and a long-range component from the mutual elastic interaction between the dislocations (Taylor hardening [21]). In the present material alloyed with Ta, the obstacle spacing of  $l \approx 150$  nm is larger than in pure NiAl. This may indicate that the jog mechanism is of less importance in NiAl-0.2at.%Ta.





The obstacle distance can be related to the activation volume V, which, for localized obstacles, is given by [16]

$$V = l \, d \, b, \tag{3}$$

where b is the absolute value of the Burgers vector and d the so-called activation distance. The above values of V and l lead to  $d \cong 0.04$  nm. This value is too small for localized pinning due to obstacles with spacings of the jogs. Unfortunately, there does not exist a theory of the activation volume of screw dislocations which are pinned by jogs.

From the in situ experiments, the (shear) back stress of the bowed-out segments can usually be evaluated from the bowing according [22] to

$$\tau_b = E_{\rm e}/(b \, x_{\rm o}) \ln(l/[5 \, b]).$$
 (4)

 $E_{\rm e}$  is the energy factor of the edge dislocation  $(E_e = 5.05 \times 10^{-10} \text{ N}, \text{ using the same elastic constants as})$ above) and  $x_0$  is the half axis parallel to the Burgers vector of the calculated equilibrium loops which fit the experimentally observed dislocation segments. Because of the pronounced angular shape of the unstable screw segments, the  $x_0$  values could not be determined with sufficient accuracy. The segments under stress, e.g., in Figs. 3 and 4, are strongly bowed out. An upper limit of the back stress is then given by considering the bowedout segments to be half-loops, i.e., by taking  $x_0 = l/2$ . This yields  $\tau_b = 110$  MPa. Taking into account the orientation factor, this value is certainly too large when compared with the macroscopic flow stress at room temperature, but it characterizes the pinning by jogs as an essential component of the flow stress.

The pinning of screw dislocations can only be an effective hardening mechanism if the conservative motion of the jogs along the screw orientation is impeded by a lattice friction mechanism. Computer simulation experiments have been carried out to calculate the Peierls stresses of the different slip systems in NiAl [8]. For the  $\langle 100 \rangle \{100\}$  slip system, the Peierls stress should be low, except for the edge dislocation (about 1 GPa). However, the preference of edge dislocations, resulting from a high Peierls stress, has neither been observed in the in situ experiments on pure NiAl [14] nor on the present NiAl–0.2at.%Ta. Nevertheless, the Peierls stress may be effective in stabilizing the pinning jogs, which are segments of edge character.

In Appendix A, the effect of solid solution hardening is estimated which occurs by the addition of Ta. It is based on the size misfit of the Ta atoms owing to their large atomic radius. The maximum interaction force between the dislocations and solutes is estimated [23], which, using the statistical theory of solution hardening [24], gives an estimate of the contribution to the (shear) flow stress at zero temperature as well as of its temperature dependence. In spite of the inaccuracy of the starting data, the following qualitative conclusions can be drawn. The size misfit of  $\delta \cong 0.17$  leads to a large value of the stress contribution at zero temperature of  $\tau_{\rm so} \cong 370$  MPa. The contribution at finite temperature  $\tau_{\rm s}$  decreases very rapidly with increasing temperature. Nevertheless, the difference at room temperature between the critical flow stresses of pure NiAl and NiAl–0.2at.%Ta of about 170 MPa (Table 1) can easily be explained by solution hardening.

If the activation volume, measured macroscopically, is mainly controlled by the solution hardening, the obstacle distance should be given by  $l=b c^{-1/2}$  if the solutes are overcome individually. c is the atomic fraction of the solute. Thus, l=6.4 nm and, using Eq. (3), d = 0.94 nm. The latter value is too large for overcoming the solute atoms individually. However, larger activation volumes can easily be explained on the basis of the statistical theory [24]. The thermally activated overcoming of solute atoms will result in a viscous motion of the dislocations between their pinning configurations due to jogs. This is shown clearly in the in situ experiments, where the viscous motion between the pinned configurations in NiAl-0.2at.%Ta was recorded on video tape, and the dislocations move less jerkily than in pure NiAl.

As a conclusion, the flow stress of NiAl–0.2at.% Ta at room temperature is certainly controlled by pinning due to jogs, solution hardening by the Ta additions and an athermal contribution from long-range dislocation interactions similar to pure NiAl. The high density of debris shown in Fig. 4 is a consequence of the jog mechanism. It is difficult to estimate its effect on the flow stress. It may have little influence on the critical flow stress, but may be an essential contribution to work hardening. At higher temperatures, where debris is only scarcely produced, the work hardening rate is small, as shown in Fig. 1. An influence of the Peierls stress may exist but cannot clearly be proved. The activation volume is too large for the Peierls mechanism, which predicts values of only a few  $b^3$ .

In the following, the behaviour of NiAl–0.2at.%Ta at elevated temperature is discussed. As demonstrated in Table 1 and Fig. 1, the macroscopic tests show only a very moderate reduction of the critical flow stress at high temperature compared to a strong decrease in pure NiAl single crystals. The result is in qualitative agreement with earlier measurements on the same material [1,15]. The smaller values of the flow stresses in the present study are, at least in part, due to the lower strain rate. The difference between NiAl–0.2at.%Ta and pure NiAl is clearly a result of the addition of Ta, either by the solution hardening, discussed above, or by the formation of solute atmospheres around the dislocations at elevated temperatures. As shown in Appendix A, the contribution of solution hardening to the flow stress should strongly decrease between room temperature and 450°C, which is not observed experimentally. Using the strain rate sensitivity of r=3 MPa at 450°C from Table 1 results with Eq. (2) in an activation volume of 10 nm<sup>3</sup> or 420  $b^3$ . This is about 6 times the room temperature value and agrees with the drastically reduced contribution of solid solution hardening at high temperatures.

The addition of Ta may also affect the flow stress at elevated temperatures by forming atmospheres around the dislocations. These atmospheres are connected with strain ageing effects, which have been observed in NiAl. They may be due to interstitial C [25] and are enhanced by additions of Ti, Zr and Hf [26]. As reviewed in Ref. [27], the atmospheres cause a flow stress maximum at increasing dislocation velocity or strain rate  $\dot{\varepsilon}$ . The slope of the stress versus  $\ln \dot{\varepsilon}$  curve is the strain rate sensitivity, which decreases from its value at small strain rates down to zero at the maximum stress. This dependence is opposite to that of obstacles controlling the dislocation mobility like solid solution hardening, where the strain rate sensitivity decreases with decreasing strain rate or stress. The dependence of the strain rate sensitivity on the strain rate or stress is expressed by the shape of the stress relaxation curves. Obstacle mechanisms cause a curve which is bent towards the stress axis as curve 2 in Fig. 2 for room temperature. It was found previously [19] that high-purity NiAl shows such a usual dependence at all temperatures investigated, in contrast to TiAl and MoSi<sub>2</sub>, which show an inverse one, in agreement with the occurrence of a flow stress anomaly in TiAl and MoSi<sub>2</sub> and its missing in NiAl. However, NiAl-0.2at.%Ta exhibits a slightly inverse curvature of the relaxation curve at 450°C, at least after some strain (curve 4 in Fig. 2), and a pronounced one at 715°C (curve 5). The pronounced inverse curvature at the high temperature may additionally be a result of recovery. Nevertheless, the inverse curvatures indicate that the weak temperature dependence of the flow stress may be caused by an additional contribution to the flow stress owing to extrinsic atmospheres of Ta around the dislocations at elevated temperatures. The small yield point effects which are visible in the stress-strain curves of Fig. 1 after the stress relaxation tests and the minimum of the strain rate sensitivity at 450°C may be due to the strain ageing connected with the atmosphere formation.

The in situ experiments at elevated temperatures were all performed in hard orientations. Except for the dislocations with  $a\langle 100 \rangle$  Burgers vectors, which occurred in spite of their orientation factor being zero, a comparison with respective experiments on pure NiAl is not possible. As in pure NiAl [14], the dislocations with  $a\langle 100 \rangle$  Burgers vectors presented in Fig. 5 show the shape controlled by the line tension with the instability of the screw dislocation. The elastic equilibrium shape is inserted in Fig. 5 in the respective projection. Again, the density of pinning jogs is much lower than at room temperature. It may therefore be concluded that the processes controlling the mobility of these dislocations are not very different in pure NiAl and NiAl–0.2at.%Ta also at high temperatures, i.e., that pinning by jogs is not important at high temperatures.

According to the literature [4,11], there is a transition between the dominance of dislocations with a(111)Burgers vectors below about 330°C and such of a(110)Burgers vectors above this temperature during deformation in a hard orientation. This is in accordance with the present experiments where almost exclusively dislocations were observed with a(110) Burgers vectors. It is a new result of the high-temperature in situ straining experiments that the shape of these dislocations under load strongly depends on the respective slip plane. Dislocations moving on {111} planes in Fig. 6 are smoothly bent between segments of preferred orientations. The shapes of these dislocations resemble those resulting from the line tension model as shown in the inset of Fig. 6. In contrast to this, the dislocations in Fig. 8 moving on {110} planes are very straight and are strictly oriented along (111) directions. The preference of these directions is only slightly visible in the shape as calculated by the line tension theory and should therefore be a result mainly of a low mobility of these mixed dislocations. The latter observation is at variance with the results of the literature [10,11], where edge dislocations dominated after deformation at 600°C and were supposed to move by shifting superkinks of non-edge character. High-resolution electron microscopy proved that the edge dislocations with a(110) Burgers vectors decompose into two dislocations with a(100) Burgers vectors, which do not have a common slip plane and can therefore only move at high temperatures by climb inside the dislocation core. The decomposition of a(110)Burgers vectors is also suggested by computer simulation [8]. In the present experiments, the edge components on {110} planes are parallel to the surface and can therefore escape from the specimen. However, their complete missing at 475°C indicates that they should not have a very low mobility as suggested for 600°C [10] [11]. It is open up to now whether the different behaviour of dislocations with a(110) Burgers vectors on {110} planes is due to the different temperatures, the addition of Ta, or the fact that the dislocations were observed in situ in the present experiments but post mortem in the previous ones [10,11]. Fig. 7 demonstrates that in addition to the cases discussed, there exist other preferential orientations on other planes, which have not been identified unequivocally. Further work is necessary to determine these unidentified orientations. The variety of these preference orientations is certainly a consequence of particular core structures. Up to now, there do not exist theoretical calculations of the preferred orientations of dislocations with a(110) Burgers vectors observed in this study, neither for the {111} slip plane nor for the  $\langle 111 \rangle$  orientation on the {110} plane. Besides, high-resolution electron microscopy work is necessary to prove the particular core structures of the mixed dislocations. However, the dislocation properties observed in this paper may occur only at high temperatures under stress so that they can be observed only in respective in situ straining experiments.

#### 5. Conclusions

Addition of 0.2at.%Ta to stoichiometric NiAl results in an increase of the flow stress at room temperature. The flow stress decreases only weakly between room temperature and  $450^{\circ}$ C so that it is drastically higher than that of pure NiAl at this temperature.

In situ straining experiments in a high-voltage electron microscope at room temperature show dislocations of  $a\langle 100 \rangle$  Burgers vectors bowing out between pinning agents as in pure NiAl. It is therefore concluded that the pinning agents are jogs. In contrast to pure NiAl, the dislocations move in a viscous way between the pinned configurations. A consequence of the jog mechanism is the formation of a large number of debris, which may contribute to the work hardening.

The flow stress of NiAl–0.2at.%Ta at room temperature can be explained by the back stress of the bowedout segments between jogs and a contribution of solid solution hardening. An estimation of the latter is given on the basis of the size misfit of the Ta atoms and the statistical theory of solution hardening. The activation volume fits the prediction of the solution hardening mechanism.

The large difference between the flow stresses of NiAl–0.2at.%Ta and pure NiAl at 450°C cannot be explained on the basis of solution hardening alone. The increased activation volume indicates a strong decrease of solid solution hardening with increasing temperature, in accordance with the theory. It is suggested that the added Ta atoms form atmospheres around the dislocations at high temperatures, giving rise to an additional component of the flow stress, to a minimum of the strain rate sensitivity and to strain ageing effects.

In situ straining experiments at 450°C in a hard orientation prove that the configurations of moving dislocations with  $a\langle 110 \rangle$  Burgers vectors depend strongly on the slip plane. On {111} planes, the dislocations show weakly preferred orientations of mixed character, while on {110} planes viscously moving dislocations are very straight and strictly oriented along  $\langle 111 \rangle$  directions. This is at variance with observations in the literature at 600°C, where dislocations with a  $\langle 110 \rangle$ Burgers vectors were arranged along edge orientations. The edge dislocations had a low mobility owing to a climb decomposition. There are no theoretical considerations available explaining the dislocation behaviour observed in this study at 450°C.

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## Appendix A

In the following, the effect of solid solution hardening is estimated which occurs by the addition of Ta. The atomic misfit  $\delta$  of incorporating the Ta atoms of a Goldschmidt radius of  $r_{\text{Ta}} = 0.147$  nm is calculated in the same way as previously [26]. As it is not clear whether the Ta atoms replace the Ni or the Al atoms, an average radius  $r_{\text{NiAI}} = 0.125$  nm based on the lattice constant of 0.288 nm of NiAl is used as a reference value, so that  $\delta = (r_{\text{Ta}} - r_{\text{NiAI}})/r_{\text{NiAI}} \cong 0.17$ . The size misfit results in a parelastic interaction with the dislocations having a maximum interaction force [23]

$$F_{\rm o} \cong \mu b^2 \delta. \tag{A1}$$

 $\mu$  is the shear modulus, which is equal to c<sub>44</sub> for the cube slip system. A value of 112 GPa is taken from Fig. 6 of Ref. [2] for room temperature. With this value, there follows  $F_0 = 1.62 \times 10^{-9}$  N. The contribution of solid solution hardening to the (shear) flow stress at zero temperature is then given by the statistical theory [24] by

$$\tau_{\rm so} = 0.94(1 + 2.5\eta_{\rm o})^{1/3} c^{1/2} F_{\rm o}^{3/2} / (b^2 [2\Gamma]^{1/2}). \tag{A2}$$

Here, c is the atomic fraction of the solute and  $\Gamma$  is the line tension. For a rough estimate,  $\Gamma = 0.5 \ \mu \ b^2$  may be taken.  $\eta_0$  is a normalized obstacle width, which is defined by

$$\eta_{\rm o} = y_{\rm o}/b(2\ c\ \Gamma/F_{\rm o})^{1/2},\tag{A3}$$

where  $y_o$  is the width of the interaction between the solute and the dislocation along the coordinate of forward motion. In Eq. (A.2) and (A.3), the lattice constant was set equal to b for the  $\langle 100 \rangle \{100\}$  slip system. With  $y_o \cong b$  for the individual solutes,  $\eta_o$  equals 0.11. This characterizes the solutes as relatively strong localized obstacles. The contribution of solid solution hardening at zero temperature turns out to be  $\tau_{so} \cong 370$  MPa. In the theory of solution hardening [24], the flow

stress at a finite temperature depends on the total interaction energy  $\Delta G_{\rm o}$  between solutes and dislocations and the normalized obstacle width  $\eta_{\rm o}$ .  $\Delta G_{\rm o}$  determines a maximum temperature of the interaction between individual solutes and dislocations

$$T_{\rm o} = \Delta G_{\rm o} / (k \ln[\dot{\varepsilon}_{\rm o}/\dot{\varepsilon}]), \tag{A4}$$

where  $\dot{\varepsilon}_{o}$  is the preexponential factor in the Arrheniustype relation of the strain rate  $\dot{\varepsilon}$ .

With the values of  $F_{\rm o}$  and  $y_{\rm o}$  taken above,  $\Delta G_{\rm o}$ should be in the order of magnitude of 1.7 eV [23]. This corresponds to To values around 1000 K, if ln  $[\dot{\varepsilon}_{o}/\dot{\varepsilon}] \cong 20$  is taken. Fig. 4 of Ref. [24] describes the temperature dependence of the flow stress. Unfortunately, there is no data for the small value of  $\eta_0 = 0.11$ , estimated above. Nevertheless, it may very roughly be estimated that  $\tau_s/\tau_{so} \cong 0.5$  for room temperature or  $T \cong 0.3 \ T_{\rm o}$  and  $\tau_{\rm s}/\tau_{\rm so} \cong 0.25$  for 450°C or  $T \cong 0.7 \ T_{\rm o}$ . Considering the orientation factor between the engineering stress  $\sigma$  measured in the experiments and the shear stress values  $\tau$  calculated here,  $\sigma = \tau/m_s$ , the contribution of solution hardening to the flow stress should amount to 560 MPa at room temperature and to 280 MPa at 450°C. Comparing the first value with the difference of about 170 MPa between the critical flow stresses of NiAl-0.2at.%Ta and pure NiAl at room temperature, the estimated value is too large by a factor of 3. Certainly, the Ta atoms cause a smaller misfit than that assumed above. This leads to a smaller maximum force  $F_{o}$  and a larger normalized obstacle width  $\eta_{o}$ . However, this does not affect the two qualitative conclusions of the estimation: solution hardening can easily explain the difference between the critical flow stresses of NiAl with and without 0.2at.%Ta at room temperature, and  $\tau_s$  drops down drastically between room temperature and 450°C so that it can only, in part, be responsible for the high flow stress of NiAl-0.2at.%Ta at 450°C.

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