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Alloy softening in Ni_3Al polycrystals

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The alloying effect of boron on localized deformation in Ni_3Al polycrystals containing 24–26 at% Al was studied using microhardness indentation. Alloy softening was observed both along the grain boundaries and in the grain interior. The softening effect decreased as the aluminium concentration increased. For alloys of near-stoichiometric composition, the maximum effect occurred at about 0.23 at% (500 wt p.p.m.) boron. A softening mechanism based on cross slip of screw dislocations was proposed.

1. Introduction

It has been recognized that the addition of solute atoms in the range of a few parts per million in a high purity bcc metal may decrease its flow stress, a phenomenon known as "alloy softening". Both substitutional and interstitial atoms, including self-interstitials, have been found to cause the softening effect, which for bcc metals occurs in the temperature range below 0.15 T_m (melting point temperature). This interesting phenomenon has been reviewed by Christian [1], Ulitchny *et al.* [2] and Pink and Arsenault [3]. The latter two have summarized most of the data available at the time. It appears that alloy softening is a general feature in bcc alloys at low temperatures, e.g. in Fe–Ni/Se [4], Mo–Re [5], Fe–C [6], Fe–N [7] and ordered TiNi [8]. More recently, alloy softening has also been observed in boron-doped Ni_3Al, an ordered intermetallic alloy with $L1_2$ structure [9].

For Ni_3Al alloy, boron was found to be effective in improving its ductility and fabricability [10, 11]. A considerable interest was stimulated in exploiting the possible mechanism for the boron effect. Recent investigations include boron segregation at grain boundaries [12] and at antiphase boundaries [13], and the effect of boron on slip propagation across the grain boundary [14]. It has been postulated [15] that boron exhibits a significant strengthening effect in Ni_3Al due to the large lattice strain produced by the boron atoms. However, on the grain boundaries, addition of boron would cause a softening effect due to the emission of grain boundary dislocations [16]. According to Weihs *et al.* [17], the addition of boron would harden the coarse-grained alloys, but soften the fine-grained alloys ($< 10\ \mu m$). This indicates that boron would soften the boundary but not the grain.

In recent studies on localized deformation using microhardness indentation [9, 18, 19], it was found that the addition of boron (500 wt p.p.m. or 0.23 at %) in a polycrystalline Ni_3Al would soften both grain boundary and grain interior. (In our earlier work [18, 19], the boron concentration was rounded off at 0.2 at %). The purpose of the present investigation is to extend the earlier work and to consider the effects of alloy stoichiometry and boron concentration on boron-induced softening.

2. Experimental procedure

The Ni_3Al intermetallic alloys used in this study were supplied by the Oak Ridge National Laboratory. Their compositions are listed in Table I. The as-cast alloys (Nos. 1–6) were rod shaped with a diameter of ~ 10 mm. A homogenization treatment was carried out in which the samples were vacuum encapsulated ($\sim 10^{-2}$ torr) in fused silica tubes, heated at 1200 $\pm$ 10 °C for a period of 72 h and furnace cooled to room temperature. The homogenized alloys have an average grain size of about 0.9 mm as used in the previous study. Alloys Nos. 7–11 were heat treated at 1050 °C for 2 h in vacuum (10^{-5} torr). The average grain size was about 0.3 mm.

Annealed samples were cut from each alloy (1–6) at three different angles with the longitudinal axis, i.e. at 45°, 90° and 180°. To minimize the error due to surface preparation, a set of six samples from two alloy sets (three boron-free and three boron-doped) was mounted in a bakelite mould. For alloys 7–11, a set of four samples was mounted in a mould. The mounts were mechanically ground to 600 grit emery papers, then polished with 6 μm diamond paste and 0.3 μm alumina powder. In order to obtain reliable microhardness data, the mounts were further polished with 0.05 μm silicon dioxide. Finally, they were chemically etched in a solution containing equal parts of HNO_3, H_2SO_4, and H_3PO_4 by volume for 45 s. Vickers hardness (DPH) was measured on a Tukon microhardness tester equipped with a microprocessor. The dependence of microhardness on indentation load was determined and two levels of loading were used. For grain interior, the load was 500 g and each data point obtained for an individual grain represented the average of five indentation readings. More indentations in a grain would result in interference from deformation in

adjacent indentation sites. The spacing of two adjacent indentations was chosen to be about six units of edge length of the indentation (Fig. 1a). The experimental errors involved in the measurements were within ±3%. For hardness measurements on grain boundaries, a load of 5 g was used, giving an indentation diagonal of 5–7 μm. In this case, ten measurements were taken for each data point with an error of ±7% (Fig. 1b.). A relatively short loading time (30 s) was chosen for both situations to minimize the errors due to the vibration of the hardness tester.

To compare the hardness of a grain boundary with that of the neighbouring interior, a 5 g load was used for both regions. As expected, the phenomenon of grain-boundary hardening exists for all the samples tested. Fig. 2 shows a typical hardness–distance profile near a grain boundary in a stoichiometric Ni_3Al alloy.

TABLE I Alloy compositions

Alloy No.	Composition (at %)		
	Al	B[a]	Ni
1	24	0	Bal.
2	25	0	Bal.
3	26	0	Bal.
4	24	0.23	Bal.
5	25	0.23	Bal.
6	26	0.23	Bal.
7	25.2	0	Bal.
8	25.2	0.09	Bal.
9	25.2	0.23	Bal.
10	25.2	0.47	Bal.
11	25.2	0.93	Bal.

[a] Values are converted from the wt % added in the alloy.

3. Results and discussion

3.1. Experimental results on boron-induced softening

Figs 3–8 illustrate the frequency distributions of 30 averaged Vickers hardness numbers (DPH) in grain

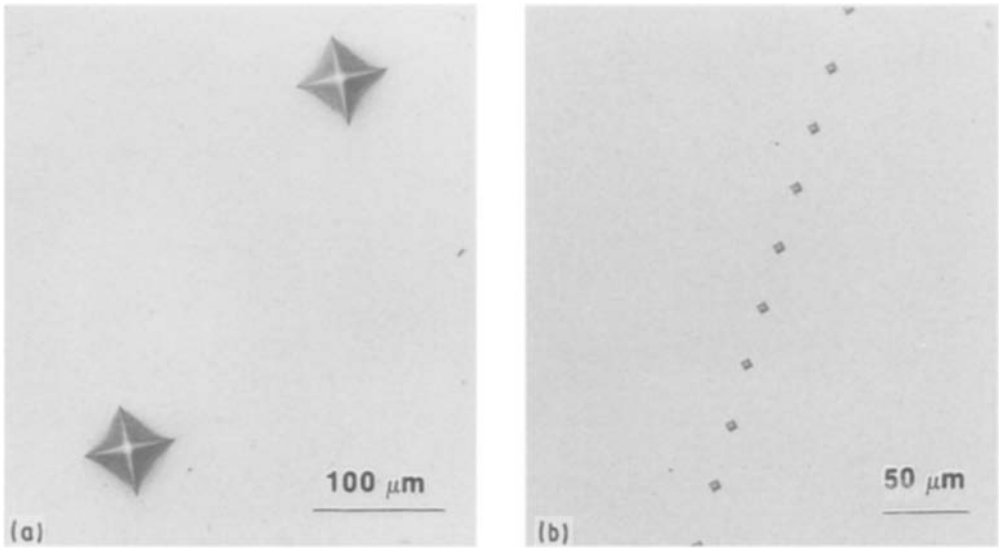

Figure 1 Vickers hardness indentations on stoichiometric Ni_3Al alloy with 0.23 at % boron. (a) In grain interior (500 g load); (b) on grain boundary (5 g load).

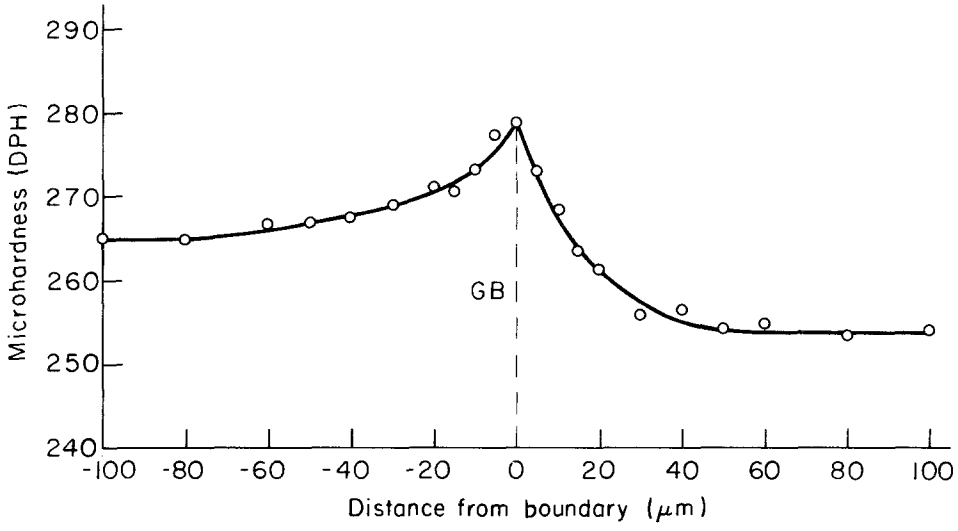

Figure 2 Hardness–distance profile near a grain boundary in a stoichiometric Ni_3Al alloy (5 g load).

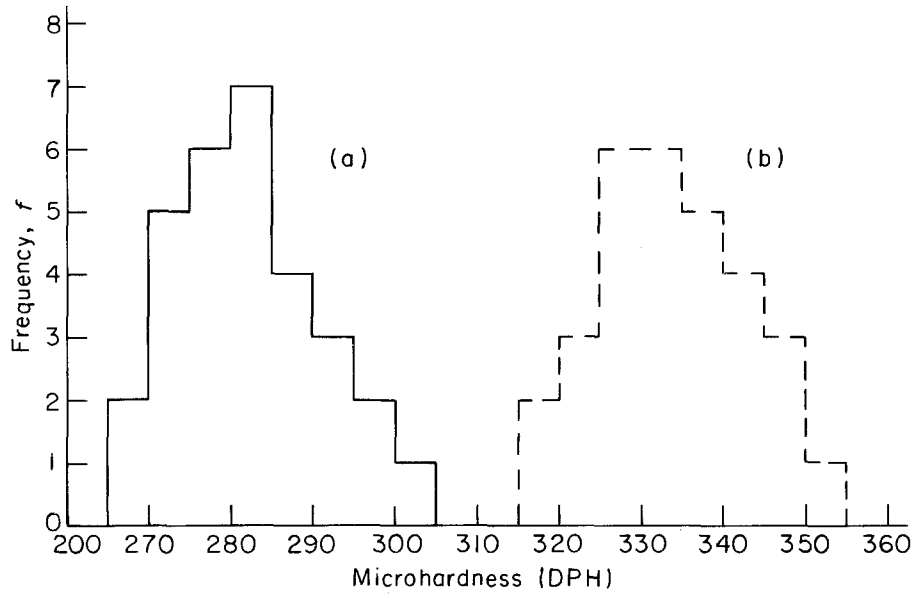

Figure 3 Frequency distributions of 30 averaged Vickers hardness numbers (DPH) on grain boundaries of a hypostoichiometric Ni_3Al alloy at a 5 g load. (a) Alloy doped with 0.23 at % boron (mean = 282.2); (b) boron-free alloy (mean = 333.8).

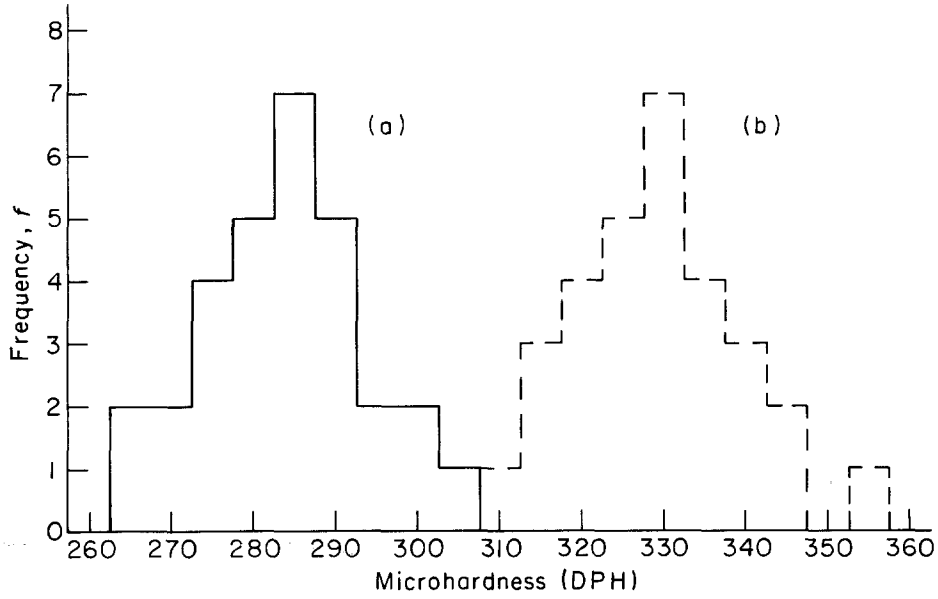

Figure 4 Frequency distributions of 30 averaged Vickers hardness numbers (DPH) in grain boundaries of a stoichiometric Ni_3Al alloy at a 5 g load. (a) Alloy doped with 0.23 at % boron (mean = 283.7); (b) boron-free alloy (mean = 329.2).

interiors (a total of 150 indentations) and on grain boundaries (a total of 300 indentations) for each of the stoichiometric and off-stoichiometric Ni_3Al alloys with and without boron. The 95% confidence intervals [19] for the population means (μ), standard deviations (σ), and the differences between the means of boron-free and boron-doped alloys ($\mu_F - \mu_D$) are shown in Tables II–IV respectively. The statistical data for alloys 1–6 show that the average microhardness of the boron-doped alloy is, in general, lower than that of the boron-free alloy, with the exception that for the hyperstoichiometric alloy (26 at % Al) the trend is reversed.

3.2. Grain-boundary softening and its stoichiometry dependence

The statistical means of microhardness on grain boundaries and in grain interiors with and without boron are plotted in Figs 9 and 10 as a function of aluminium concentration. It is seen that with the addition of 0.23 at % (500 wt p.p.m.) of boron to Ni_3Al, the grain-boundary hardness was consistently decreased. This leads to the hypothesis that boron softens the grain boundaries in Ni_3Al. The experimental results support the argument by Schulson and co-workers [16, 20, 21] that the addition of boron reduces the effectiveness of grain boundary strengthening and increases the ease with which grain boundaries accommodate slip. A second point from the results is that the effect of boron on grain boundary hardness is dependent on alloy stoichiometry, i.e. the extent of grain boundary softening would decrease as the aluminium content varies from hypo- to hyperstoichiometric. This can be partially understood based

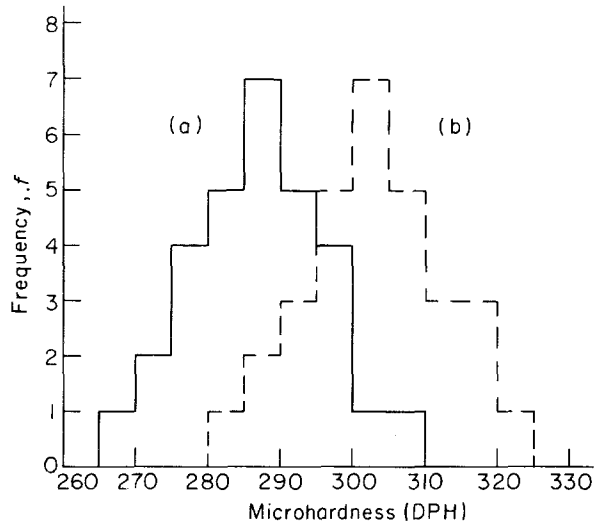

Figure 5 Frequency distributions of 30 averaged Vickers hardness numbers (DPH) on grain boundaries of a hyperstoichiometric Ni_3Al alloy at a 5 g load. (a) Alloy doped with 0.23 at % boron (mean = 287.0); (b) boron-free alloy (mean = 303.0).

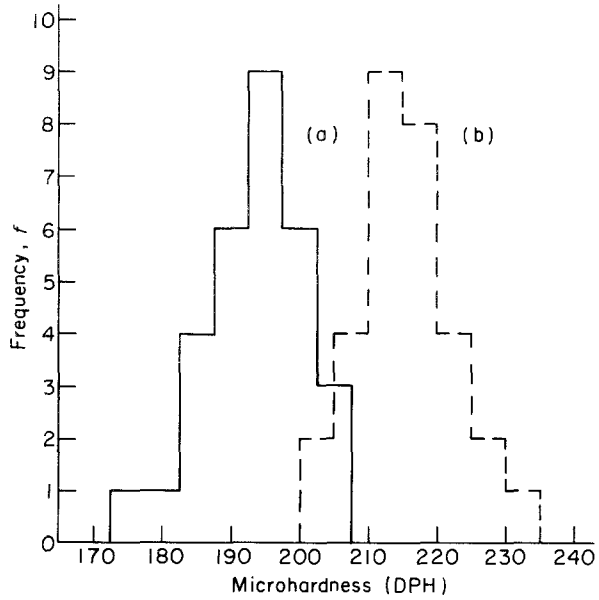

Figure 6 Frequency distributions of 30 averaged Vickers hardness numbers (DPH) in grain interiors of a hypostoichiometric Ni_3Al alloy at a 500 g load. (a) Alloy doped with 0.23 at % boron (mean = 193.5); (b) boron-free alloy (mean = 215.5).

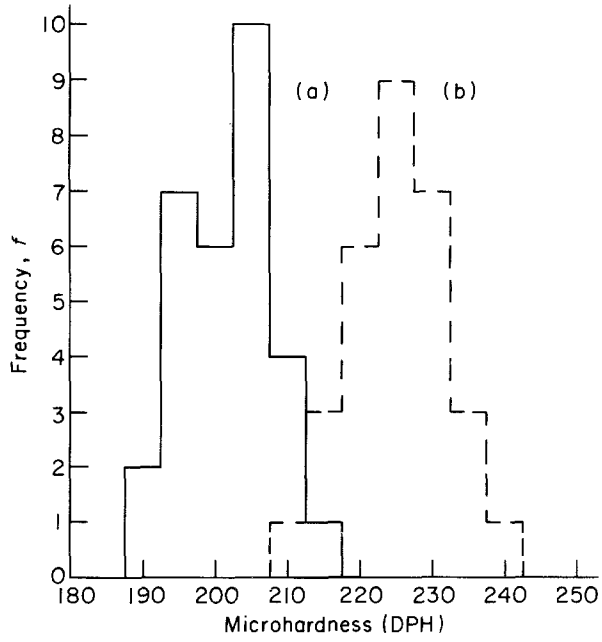

Figure 7 Frequency distributions of 30 averaged Vickers hardness numbers (DPH) on grain-interiors of a stoichiometric Ni_3Al alloy at a 500 g load. (a) Alloy doped with 0.23 at % boron (mean = 201.7); (b) boron-free alloy (mean = 225.2).

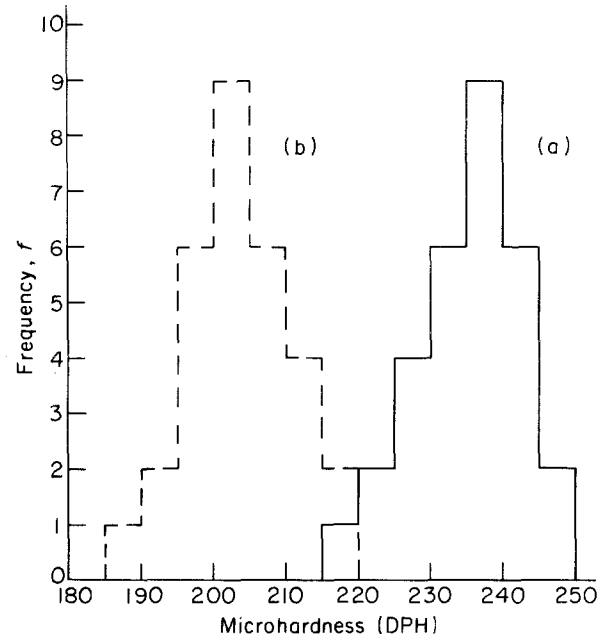

Figure 8 Frequency distributions of 30 averaged Vickers hardness numbers (DPH) in grain interiors of a hyperstoichiometric Ni_3Al alloy at a 500 g load. (a) Alloy doped with 0.23 at % boron (mean = 235.2); (b) boron-free alloy (mean = 203.7).

on the effect of stoichiometry in boron segregation behaviour. The level of boron segregation at the grain boundaries decreases with increasing aluminium concentration in Ni_3Al [12, 22, 23]. That is, the boron becomes less effective in softening Ni_3Al if its content is less than a critical amount ($\sim$ 10 at % B) present at the grain boundaries.

The experimental data also show that in boron-free Ni_3Al alloys, the grain-boundary hardness decreases with increasing aluminium concentration, since aluminium is softer than nickel. In addition, Takasugi *et al.* [24] has concluded that grain boundary hardening with respect to the neighbouring grain interior is dependent on alloy stoichiometry. They postulated that when the excess Ni atoms substituted for Al atoms in the grain boundary region of a hypostoichiometric alloy, the Ni–Ni bonds produced would create more homogeneous charge distribution through the boundary plane, resulting in a higher grain boundary strength. This argument is consistent with our results.

3.3. Grain softening and the proposed mechanism of cross slip

Figs 6–8 demonstrate that small additions of boron can decrease microhardness in grain interiors as well, but only when the intermetallic is stoichiometric or

TABLE II 95% confidence intervals for statistical mean, μ

Micro-alloying	Composition (at %)					
	24Al		25Al		26Al	
	GB[a]	GI[b]	GB	GI	GB	GI
Boron-free	(330.4, 337.2)	(212.8, 218.2)	(325.4, 333.0)	(222.6, 227.8)	(299.3, 306.7)	(200.0, 207.4)
0.23 at % boron	(278.8, 285.6)	(190.8, 196.2)	(280.0, 287.4)	(199.3, 204.1)	(283.6, 290.4)	(232.4, 238.0)

[a] GB, grain boundary.
[b] GI, grain interior.

TABLE III 95% confidence intervals for standard deviation, σ

Micro-alloying	Composition (at %)					
	24Al		25Al		26Al	
	GB[a]	GI[b]	GB	GI	GB	GI
Boron-free	(7.3, 12.6)	(5.6, 9.6)	(8.1, 14.1)	(5.5, 9.3)	(7.7, 13.4)	(5.8, 9.8)
0.23 at % boron	(7.1, 12.3)	(5.8, 9.8)	(7.9, 13.6)	(5.0, 8.5)	(7.3, 12.6)	(5.9, 10.0)

[a] GB, grain boundary.
[b] GI, grain interior.

TABLE IV 95% confidence intervals for the difference between the means of boron-free and boron-doped alloys ($\mu_F - \mu_D$)

	Composition (at %)					
	24Al		25Al		26Al	
	GB[a]	GI[b]	GB	GI	GB	GI
($\mu_F - \mu_D$)	(49.3, 53.9)	(18.3, 25.7)	(20.1, 26.9)	(40.3, 50.7)	(11.1, 20.9)	(−27.7, −35.3)

[a] GB, grain boundary.
[b] GI, grain interior.

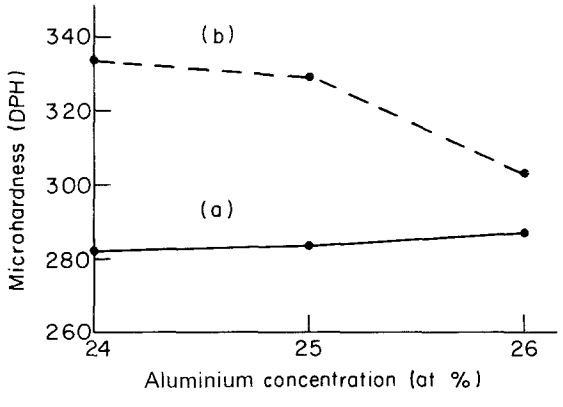

Figure 9 Effect of aluminium concentration on hardness along grain boundaries in Ni_3Al. (a) Alloy doped with 0.23 at % boron; (b) boron-free alloy.

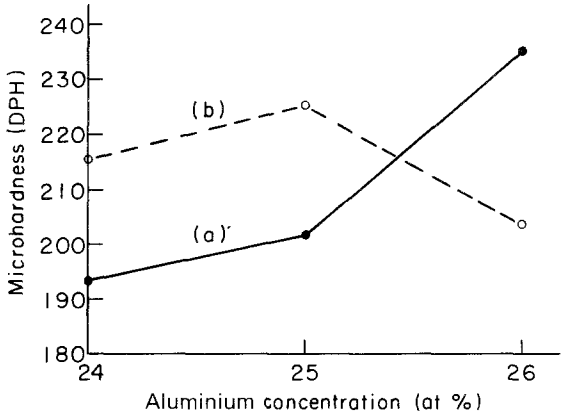

Figure 10 Effect of aluminium concentration on hardness in the grain interior in Ni_3Al. (a) Alloy doped with 0.23 at % boron; (b) boron-free alloy.

aluminium-lean. For hyperstoichiometric Ni_3Al alloy, only the strengthening effect was observed. It is commonly believed that in the Ni_3Al lattice, boron atoms reside interstitially and generate a lattice strain which imparts a marked increase in lattice resistance to slip. The present results show partially the opposite effect.

There are a number of mechanisms proposed for the alloy softening [25]. The two most popular are: (i) the reduction of the Peierls stress by solutes; and (ii) the enhancement of the rate of double-kink formation of screw dislocations as a result of the elastic interaction with solute atoms [26]. These mechanisms are "intrinsic softening" mechanisms and imply that the dislocation mobility is determined by the Peierls lattice potential. Hence they are not applicable to fcc systems in which the dislocation–lattice interaction is expected to be small. For the present case, a different mechanism should be considered.

As mentioned in the review articles [27, 28], the dislocation structure in a deformed Ni_3Al alloy primarily consists of long, straight screw dislocations. The flow stress of Ni_3Al has a very unusual temperature

dependence, i.e. it increases with increasing temperature. This behaviour is the result of thermally-activated cross slip of $a/2\langle 1\,1\,0\rangle$ screw dislocations from the (1 1 1) planes, where they are mobile, to (0 1 0) planes, where they are immobile. At room temperature, the motion of the $a/2$ $[\bar{1}\,0\,1]$ superpartial dislocation on the (1 1 1) plane would result in the production of an APB (anti-phase boundary) on the same plane. This configuration (Fig. 11) is strongly pinned and the flow stress is increased (work hardening). Copley and Kear [29] and Paider *et al.* [30] have shown that under the geometric considerations, cross slip from (1 1 1) to $(1\,\bar{1}\,1)$ planes without changing dislocation mobility is possible only for samples oriented near [0 0 1] at temperatures near the peak in the CRSS (critical resolved shear stress) against temperature plot. Cross slip can be neglected for the samples oriented at other orientations. On the other hand, although it is well known that most alloying additions to Ni_3Al cause strengthening, it is possible that other alloying additions may result in weakening. Pope and Ezz [28] pointed out that the causes of these strengthening or weakening effects appear to be related to the anisotropy of the APB energy, which is the main driving force for cross slip from (1 1 1) to (0 1 0) planes. Suzuki *et al.* [31] have argued that the decrease in the positive temperature dependence of the flow stress is caused by a decrease in the APB energy on (1 1 1) planes as a result of the Fe addition. Wee and coworkers [32] suggested that alloying elements which tend to make a given $L1_2$ alloy unstable with respect to DO_{19} or DO_{22} (or other similar structures) will also decrease the APB energy on the (0 1 0) planes, which leads to an increase in the rate of cross slip from (1 1 1) to (0 1 0). In our case, where a certain content of boron is added to Ni_3Al alloy, it can reasonably be

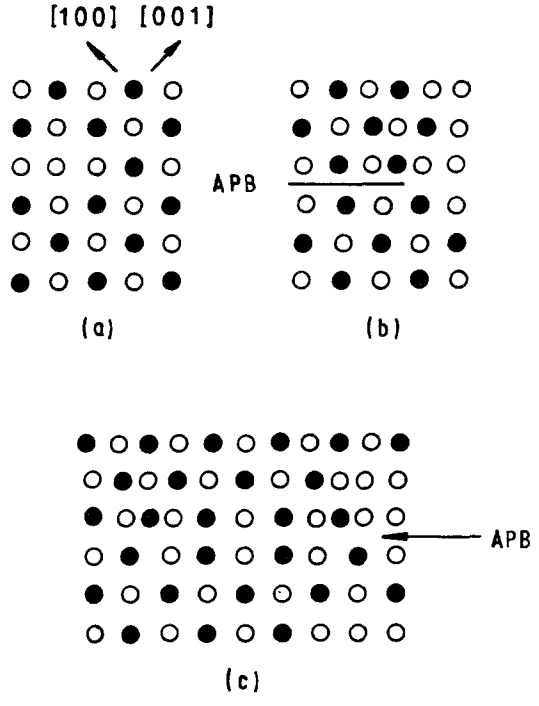

Figure 11 The anti-phase boundaries (APB) in $L1_2$ structure; (a) A (0 1 0) plane of a $L1_2$ structure; (b) as in (a) but with $a/2$ [1 0 1] dislocation on the (1 1 1) plane, producing an APB; (c) two $a/2$ [1 0 1] dislocations, as in (b), connected by an APB [28].

suggested that the energy of APB on the (1 1 1) plane is decreased. In the following analysis, an attempt will be made to show that the boron addition leads to alloy softening.

According to Gilman [33], the stress dependence of the dislocation velocity is given by

$$V = V^* \exp\left(-\frac{Db}{\Sigma f_i}\right), \quad \Sigma f_i \geqslant 0 \tag{1}$$

where V^* is the shear-wave velocity, D is a measure of the friction forces that act on the dislocation (called the characteristic drag-stress coefficient), b is the Burgers vector, f_i is the ith force acting on the dislocation, and Σ stands for summation. Consider a pair of superlattice partial dislocations associated with an APB on the (1 1 1) plane. The velocity of each superpartial is

$$V = V^* \exp\left(-\frac{Db}{\tau b - \gamma + \mu b^2/2\pi\Delta x}\right) \tag{2}$$

where τ is the resolved shear stress, γ is the antiphase boundary energy, $\mu b^2/2\pi\Delta x$ is repulsive force between two partials, and Δx is their equilibrium width. From Equation 2, a reduction of the APB energy on (1 1 1) planes would increase the velocity of the super partials. This means that the mobility of the superlattice screw dislocations would be increased and the rate of work-hardening decreased. Moreover, the frequency for cross slip from the (1 1 1) to the (0 1 0) plane would be decreased due to the decrease in the APB energy on the (1 1 1) plane, and weakening results. In $L1_2$ structure, there is a necessary condition for the cross slip from the (1 1 1) to the (0 1 0) plane [34], given by

$$\left(\frac{3A}{A+2}\right)\frac{\gamma_1}{\gamma_0} > 3^{1/2} \tag{3}$$

where $A = 2c_{44}/(c_{11} - c_{12})$, c_{11}, c_{12} and c_{44} are elastic constants, and γ_1 and γ_0 are the APB energies on $\{1\,1\,1\}$ and $\{1\,0\,0\}$ planes, respectively. Therefore the likelihood for a superpartial dislocation to cross slip from the (1 1 1) to the (0 1 0) plane will be decreased as γ_1 is reduced.

On the other hand, the interaction between an interstitial dipole and superpartial screw dislocation will promote the cross slip from the (1 1 1) to the $(1\,\bar{1}\,1)$ plane and thus relax the flow stress. This mechanism has been used to explain the alloy softening in the Fe–N system near 175 K [35, 36]. Here only an illustrated description is presented. Fig. 12 shows the $(\bar{1}\,0\,1)$ plane containing three $\{1\,1\,1\}$ slip traces and three possible interstitial sites. The circles represent the equipotentials for the interaction when the Burgers vector is normal to the paper and the interstitial is at the type 3 site. The two other types of interstitial positions produce similar equipotential systems and the whole displays trigonal symmetry about the $[\bar{1}\,0\,1]$ direction. While the dislocation is moving along $[\bar{1}\,2\,\bar{1}]$ on the (1 1 1) plane, the total energy increases and a retardation is experienced. Suppose that the dislocation now cross slips onto $(1\,\bar{1}\,1)$ along [1 2 1]. Since this direction is down the gradient of the potential field, the interstitial assists glide on the new slip plane and, hence, promotes cross

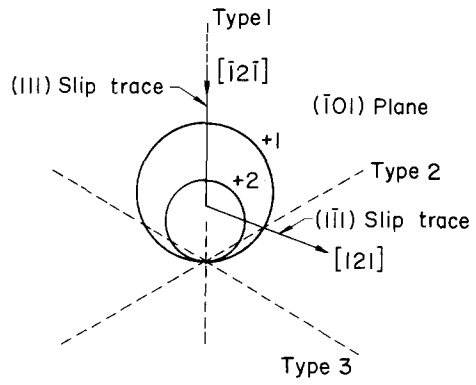

Figure 12 Schematic representation of cross slip from (1 1 1) to (1 $\bar{1}$ 1) plane in Ni_3Al.

slip. The forces depend not only on the type of the site, but also upon whether the interstitial is above or below the slip plane. Although the calculation indicates that only type 2 and 3 interstitial dipoles can produce an effect to promote cross slip of screw dislocations, there always exist circumstances when the properly oriented interstitial dipoles exert forces on the dislocations in a {1 1 1} cross-slip direction.

Assuming that the velocities of superpartial dislocations are represented by Equation 2, it is likely that if the leading dislocation should cross slip from the (1 1 1) into the (1 $\bar{1}$ 1) plane, the trailing dislocation will almost certainly follow the same path into (1 $\bar{1}$ 1). Cross slip may occur when a leading partial dislocation, with Burgers vector $a/6$ [$\bar{2}$ 1 1], dissociates into two partials, one of which, $a/6$ [$\bar{2}$ $\bar{1}$ 1], glides on the cross slip plane and the other, $a/3$ [0 1 0], is sessile at the intersection of the two planes involved. The reaction is

$$a/6[\bar{2}\,1\,1] \rightarrow a/6[\bar{2}\,\bar{1}\,1] + a/3[0\,1\,0] \quad (4)$$

Subsequently, the second of the partials originally on the primary slip plane is attracted to the sessile, and combines with it to give the second $a/6$[$\bar{1}$ 1 2] partial on the cross slip plane, i.e.

$$a/6[\bar{1}\,\bar{1}\,2] + a/3[0\,1\,0] \rightarrow a/6[\bar{1}\,1\,2] \quad (5)$$

An approximate configuration just after Reaction 4 is shown in Fig. 13.

The relaxation of stress can occur by the cross slip mechanism. Once the double cross slip occurs, the Frank–Read source can begin to operate as described by Low and Guard [37], thus increasing the mobile dislocation density. Cross slip occurs where the dislocations are observed to multiply.

3.4. Competition between two deformation processes

Nakada and Keh [7] in their study of the Fe–N system remarked that alloy softening and alloy strengthening are two concurrent but competitive processes in the course of deformation. Under certain circumstances, alloy strengthening is the dominant mechanism, while in other situations alloy softening plays a superior role. The balance of the two processes depends on the interplay of the process parameters, i.e. temperature, alloy concentration, crystallographic orientation, grain size, strain rate, etc. In the present case, for hypostoichiometric Ni_3Al alloys, the softening effect of boron predominates over the strengthening effect. However, it has been suggested [38] that the defect trapping associated with 25 and 26 at % Al causes boron to cluster in the matrix, resulting in an increase in hardness with increasing aluminium concentration. In hyperstoichiometric alloys, the strengthening effect by clustering surpasses the boron softening, leading to apparent hardening (Fig. 8). Lopez and Hancock [39] showed that the excess Al or Ni in the Ni_3Al structure resulted in a higher flow stress and that an excess of Al would have a much more marked effect than an excess of Ni. Rawlings and Stanton-Bevan [40] concluded that the extent of solid-solution strengthening resulting from the alloy addition depends on the atomic-misfit parameter, the stoichiometry of the alloy, and the substitutional nature of the element added. Elements with large misfits that substitute for Al in Al-rich alloys produce the most strengthening effect. Noguchi *et al.* [41] have studied the effects of deviation from stoichiometry on the flow stress of polycrystalline Ni_3Al and Ni_3Ga. They found that an excess of Al produces a greater increase in strength than does an excess of Ni. All these aforementioned experiments and arguments are consistent with our results.

3.5. Effect of boron concentration on alloy softening

The effect of boron concentration on hardness in Ni–25.2 at % Al alloys (samples 7–11) was also examined. As shown in Fig. 14, as the concentration of boron increases from zero to 2000 wt p.p.m., the microhardness first decreases and then increases through a minimum at 500 p.p.m. boron. This can be qualitatively explained based on the ease of cross slip [36]. At a very low concentration of interstitials, the dislocation can sweep forward on the primary slip plane since the interaction between a dislocation and a single interstitial is weak and is not able to promote

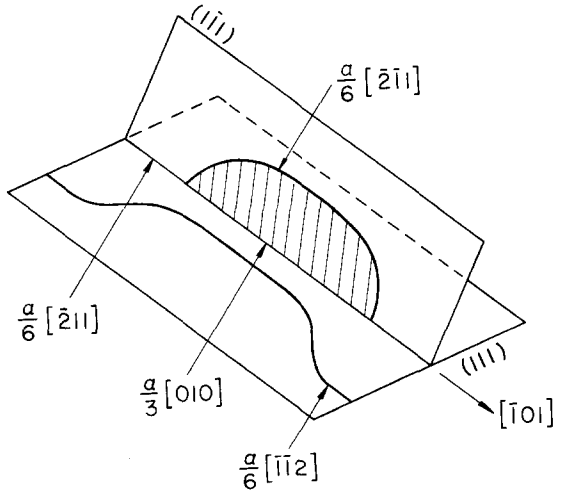

Figure 13 Cross-slip of an extended dislocation in Ni_3Al.

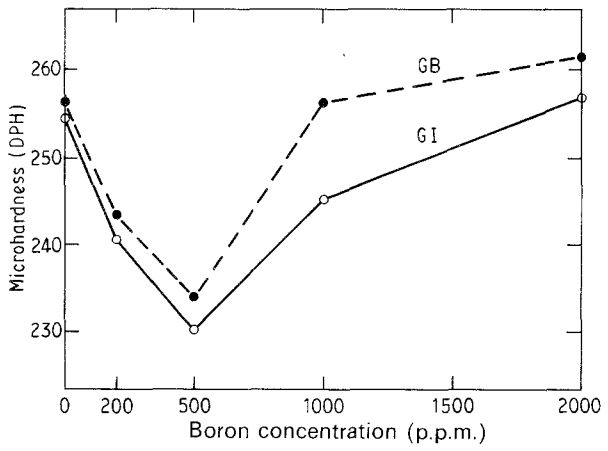

Figure 14 Dependence of boron concentration (wt p.p.m.) on hardness of Ni–25.2 at % Al alloy. (GB), grain boundary; (GI), grain interior.

cross slip. When more interstitials are present and are situated so close together that the distance of forward sweeping is less than one Burgers vector, the dislocation would remain straight and cross slip would then take place by the cooperative action of several interstitials. However when the interstitial concentration is too high, the likelihood that a large number of interstitials are all acting on the dislocation in the same way is negligible. Thus cross slip ought not be significant at this high level of concentration. This would explain why the cross-slip frequency goes through a maximum and the hardness through a minimum as the concentration of boron increases. Chaki [42] has recently proposed a bond-distortion model for boron softening in the Ni_3Al lattice. According to his analysis, an amount of 3900 at p.p.m. ($\sim$830 wt p.p.m.) of boron would be needed to fully effect the softening process. This level of boron content, however, was not studied in the present investigation.

It should be pointed out that the above experimental results do not coincide completely with the compiled data for room temperature tensile yield strengths of Ni_3Al alloys with and without boron [14, 16]. On macrostraining (tensile loading) of Ni_3Al, alloy softening occurs only in the smaller grain size range (<10 μm) and strengthening in a large grain-size range. The salient point of the present investigation is to show that on microstraining (hardness) alloy softening seems to be a general phenomenon and is independent of grain size. It appears that more studies, especially TEM analyses, are needed to clarify the role of boron in the localized deformation of Ni_3Al alloys.

4. Conclusions

1. Alloy softening was observed in Ni_3Al polycrystals due to the addition of boron. The degree of softening decreases with increasing aluminium concentration.

2. Alloy softening in the grain interior is not present in the hyperstoichiometric alloy of 26 at % Al, probably due to the competition between the boron-induced cluster strengthening and the boron-induced cross slip softening. In the hyperstoichiometric alloy, boron clusters were found in the grain and the balance is thus tipped toward hardening.

3. In stoichiometric alloys with different boron concentrations, the maximum softening effect appears near 0.23 at % (500 wt.p.p.m.) boron.

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