Materials Science and Engineering A 387–389 (2004) 109–114

MATERIALS SCIENCE & ENGINEERING A

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Quantitative analysis of dislocation pile-ups in thin foils compared to bulk

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Received 25 August 2003; received in revised form 28 April 2004

Abstract

Pile-ups observed in short-range ordered γ-phase of nickel-base superalloys have been investigated using both post-mortem and in situ transmission electron microscopy experiments. The influence of the foil thickness on the pile-up length, on the distances between the dislocations and on the elastic interaction stresses experienced by them, is analyzed. The results using the approximation of an infinite medium or accurate calculations taking into account the elastic relaxation of the stress by the free surface are compared. The experimental positions of the dislocations are shown to be determined by these latter calculations with a good accuracy so that precise information about short-range order friction forces can be deduced.

Keywords: Thin foil; Dislocation pile-up; Short-range order

1. Introduction

In f.c.c. Ni–Cr concentrated solid solutions, the deformation proceeds by the collective motion of dislocation pile-ups. Both post-mortem and in situ transmission electron microscopy show that, in the γ model phases of nickel-based superalloys noted γ_{MCRe} and γ_{MCRu} (whose chemical compositions are given in [1]), deformed between 25 and 750 °C, the first two dislocations of the pile-ups move closely together, which has been interpreted as the result of short-range order coupling. In previous publications [2–4] the elastic interaction stresses as well as the energies associated with the SRO have been evaluated by measuring the dislocations positions within these pile-ups. However, the elastic interactions were calculated without taking into account the screening effect of the elastic stresses of the dislocations by the free surfaces of the thin foil.

In a recent paper, the distribution of dislocations in a pile-up within a thin foil has been calculated, taking full account of this screening effect, which has allowed the evaluation of the chemical force resulting from SRO in these alloys to be quantified [5].

The aim of this paper is to analyze the effect of the foil thickness on the pile-up properties (length, distribution of the dislocations) using experimental results and to compare the numerical calculations in a thin foil with those obtained under the assumption of an infinite medium. Different samples of γ-phase nickel-base superalloys, with a thickness ranging from 138 to 611 nm were analyzed in a JEOL 200CX electron microscope. A high voltage microscope (1 MV) has been used for samples of the highest thickness. The results are used to determine the friction stress resulting from SRO, and its destruction by the glide of dislocations.

2. Pile-ups in thin foils

2.1. Elastic field in an ideal case

In a solid solution, the structure of a pile-up of screw dislocations either parallel or perpendicular to the surface of a thin foil of thickness h, gliding under the effect of an applied stress τ_a, can be expressed by analytical equations [5], which show that all the meaningful physical quantities depend on the dimensionless parameter $q = \tau_a h/\mu b$, where b is the Burgers vector and μ is the shear modulus. The relevant equations have been solved numerically for various values of q.

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doi:10.1016/j.msea.2004.05.048

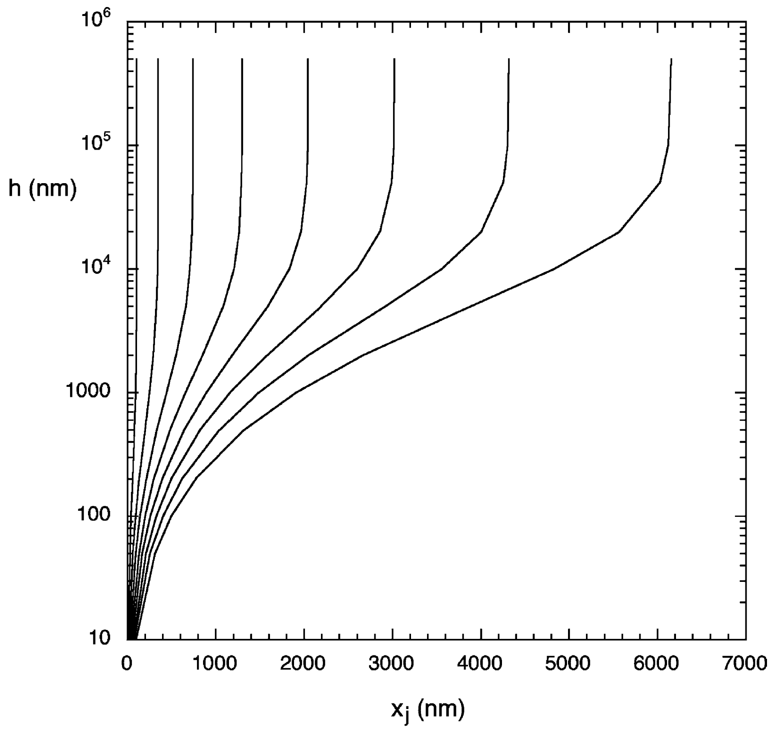

Fig. 1. Pile-up of nine screw dislocations parallel to the surface of a thin foil of thickness h. The positions (x_j) of the dislocations are calculated by taking into account the elastic interactions for h varying from 10 to 5×10^5 nm [5]; the thickness h when 150 nm $< h <$ 300 nm corresponds to typical transmission electron microscopy sample.

The main useful conclusions obtained from this calculation are:

(i) The stress on the leading dislocation of a pile-up of n dislocations is n times the applied stress, as in the bulk.

(ii) The stress resulting from the elastic interaction between dislocations (which we denote interaction stress) on a dislocation located at the distance l from the leading dislocation decreases roughly as exp l/h, which has two important consequences, illustrated in Fig. 1 where we have plotted the positions of the dislocations within a pile-up of nine dislocations calculated for different foil thicknesses, from $h = 10$ nm until it tends to an infinite value:

   (a) The distance d separating the first two dislocations is affected by the foil thickness. The calculation made for a pile-up of nine dislocations shows that for a thickness $h = 100$ nm, $d = 42$ nm, for $h = 500$ nm, $d = 79$ nm and for $h = 5 \times 10^5$ nm, $d = 102$ nm.

   (b) In a thin foil, for example when 150 nm $< h <$ 300 nm which corresponds to typical transmission electron microscopy samples, the interval between adjacent dislocations increases very slowly with the distance to the head of the pile-up. Besides, for a given number of dislocations, the length of the pile-up increases with the thickness of the foil.

(iii) The interesting fact is that parallel and perpendicular screw pile-ups give very similar results [5]. One may therefore postulate that the effect of the stress relaxation at the surface does not depend strongly on the inclination of the dislocations. As pointed out by Eshelby and Stroh [6], the situation is very similar for the case of an edge dislocation perpendicular to the surface. The above results may therefore be used carefully to interpret observations of more general observed pile-ups in thin plates.

2.2. Analysis of the observed pile-ups

An example of a pile-up obtained after a deformation of γ_{MCRu} at 350 °C observed in a foil whose thickness is close to 160 nm is illustrated in Fig. 2. TEM results about the position of each dislocation within a dislocation pile-up are gathered in Fig. 3 which confirms the variation of the pile-up length with the foil thickness. In the investigated foils with a thickness ranging from 150 to 600 nm, the average distance d between the first two dislocations remains constant around 30 ± 5 nm in contradiction with the calculation illustrated in Fig. 1. This pairing is known to be due to short-range order [1]. This effect is taken into account in the calculation illustrated Fig. 4; in this numerical case with SRO, it appears that d remains practically constant ($h = 100$ nm, $d = 19$ nm; $h = 500$ nm, $d = 24$ nm; $h = 5 \times 10^5$ nm, $d = 26$ nm); the effect of short-range order is analyzed in Section 3.1.

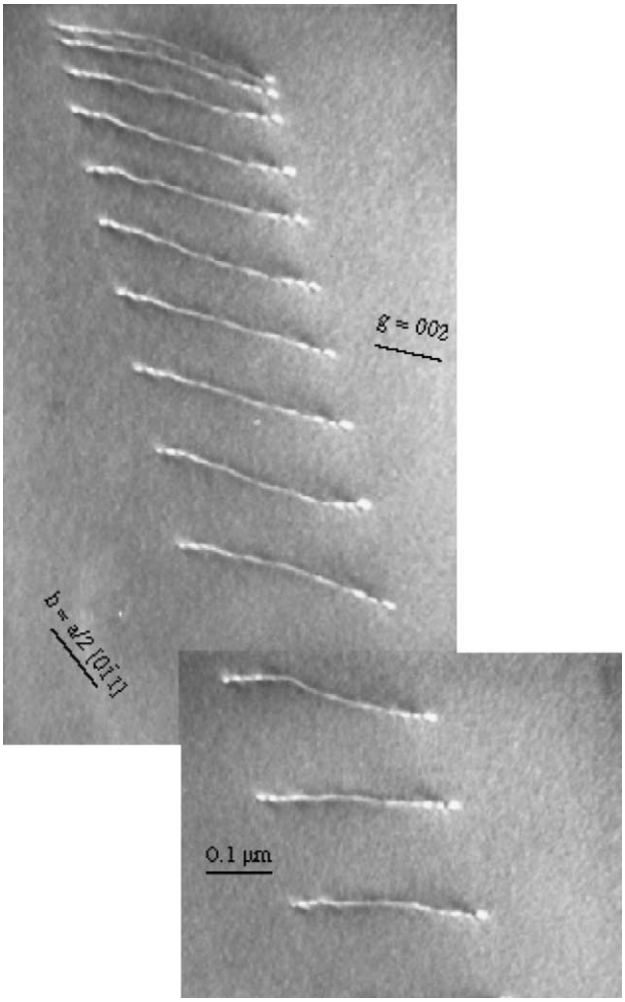

Fig. 2. A typical pile-up illustrated in a post-mortem TEM image of γ_{MCRe} deformed at 350 °C. The spacing between the first two dislocations is 27 nm. The thickness of the foil is 160 nm.

By measuring the positions of the dislocations in pile-ups imaged by TEM, we have evaluated the elastic interaction stresses on the dislocations either by using the bulk approximation [7], or by using the calculations for screw dislocations taking into account the stress relaxation due to the free surface [5], referred to as thin plate approximation. Fig. 5 illustrates a typical example. In this example, the experimental pile-up has a 10° character which is rather close to that of a screw and allows the comparison to be done. Three points are worth mentioning:

(i) Calculations taking into account the effect of the surfaces indicate almost zero elastic stress on the dislocations located at a distance larger than 500 nm, while calculations in the bulk approximation give a stress of about 25 MPa up to the end of the pile-up. This observation indicates that the calculation captures reasonably well the relaxation effect of the surface.
(ii) Both approximations give very close values of the elastic interaction stress on the first dislocation, which is in agreement with the conclusions of Section 2.1.
(iii) The elastic interaction forces on the first dislocations are different from zero, up to about the 4th dislocation, which has been interpreted to be the result of a chemical force acting on the dislocations (see Section 3).

3. Analysis of short-range order in Ni-base alloys

3.1. How SRO affects the position of dislocations within the pile-up?

Since all the dislocations of a pile-up should be at equilibrium, we assume that besides the elastic interaction force, there is a friction force opposing the motion of the dislo-

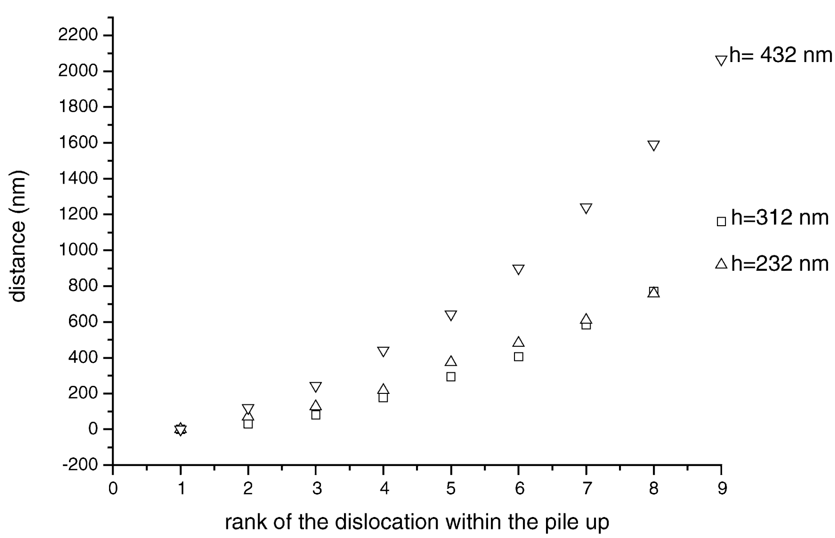

Fig. 3. Measured positions of the dislocations of observed pile-ups in thin foils of different thicknesses $h = 232$, 312 and 432 nm. The lengths of the pile-ups are, respectively, 919, 1161 and 2068 nm. No significant foil thickness effect is observed for the distance between the first two dislocations, while the distance between others dislocations increases very slowly with the distance to the head of the pile-up.

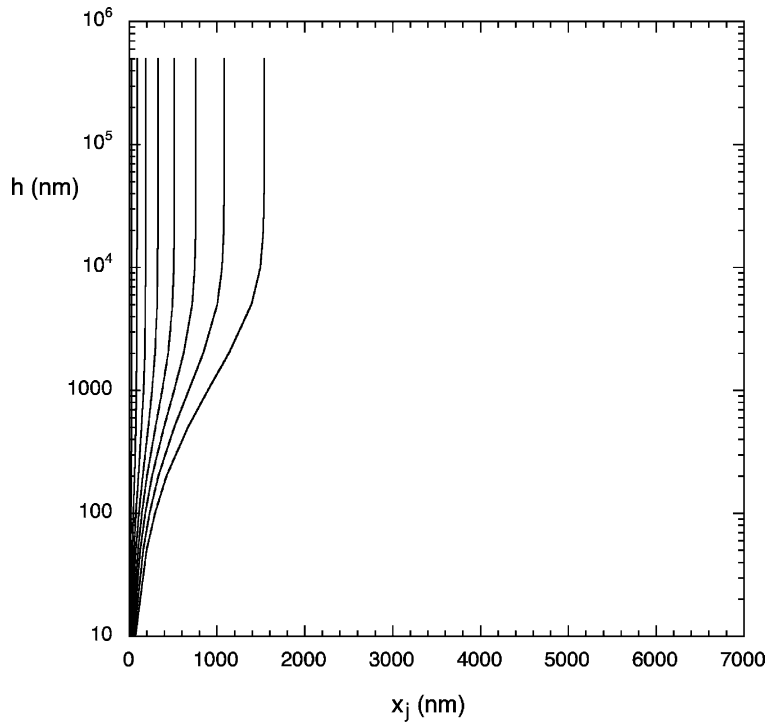

Fig. 4. Pile-up of nine screw dislocations parallel to the surface of a thin foil of thickness h, in a short-range ordered alloy. The positions (x_j) of the dislocations are calculated by taking into account the elastic interactions for h varying from 10 to 5×10^5 nm and short-range order effects (Section 3). The same scale as of Fig. 1 has been chosen for an easier comparison.

cations. This force results from the destruction of SRO by the dislocation glide, i.e. the variation per unit length or equivalently of the energy per unit area of the planar defect formed by the destruction of the order. We assume further that the first dislocation is stopped by a high friction stress.

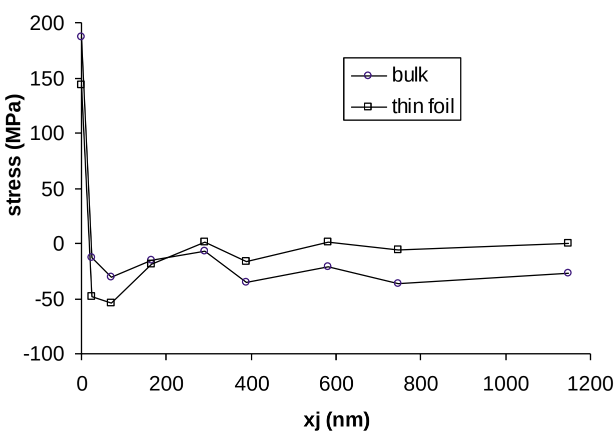

Fig. 5. Elastic interaction stresses obtained using experimental positions of dislocations belonging to a pile-up of 10° character. The calculation has been made for the case of an infinite medium using the expression by Chou et al. [7], and for the case of the real thickness of 208 nm using the expression by Eshelby and Stroh for screw dislocations [6]. The dislocation character has been taken into account in the calculation with the bulk approximation.

In Table 1, the positions of the dislocations have been numerically determined in three different cases and compared with the experimental ones. In the first case, the effect of SRO is not considered, in the 2nd, it is supposed to act only on the first two dislocations, and in the 3rd, it is supposed to be effective up to the 7th dislocation. The best fit corresponds obviously to the last case. This comparison confirms that the positions can be determined using TEM images with a good accuracy (see Fig. 6a; then, it reveals which dislocations experiences SRO. Here, the destruction of SRO needs about seven dislocations.

3.2. Evaluation of the energy due to SRO ("diffuse antiphase boundary energy")

Considering that the glide of a dislocation p in an SRO solid solution introduces some disorder associated with a planar defect whose energy is γ_p, the value of γ_p associated with the glide of each dislocation is determined from the knowledge of their experimental position, using the following equation [5]:

$$\gamma_p = (p+1)b(\tau_{eq} - \tau_f) - \sum_{i=1}^{p} R_i \tag{1}$$

where τ_{eq} is the critical stress necessary to propagate the pile-up, τ_f the friction stress due to the lattice friction, F_{ji} the elastic force exerted by the dislocation j on the dislocation

Table 1
Experimental and calculated positions (in nm) in a pile-up containing nine dislocations in γ_{MCRe} alloy in three different cases: (1) no SRO is considered; (2) SRO is experienced by the 1st and the 2nd dislocations; (3) SRO is experienced by the seven first dislocations

p	Experimental case, position (nm)	Calculated case without SRO, position (nm)	Calculated case with SRO behind the first two dislocations		Calculated case with SRO behind the first seven dislocations	
			Position (nm)	γ_p (mJ/m^2)	Position (nm)	γ_p (mJ/m^2)
1	0	0	0	27.7	0	27.7
2	31	86.1	27.2	22.4	31	22.4
3	81	197.1	72.3		81	10.9
4	177	317.7	143.8		176.8	6.7
5	294	447.8	329.2		293.8	5.7
6	405	589.8	496.6		404.6	2.3
7	582	748.1	681.5		581.8	1.6
8	770	931.3	893.4		769.4	
9	1161	1161.6	1156.7		1160	

γ_p is the energy of the fault behind dislocation p calculated using Eq. (1). The applied stress is different in the three cases and has been chosen to fit the total length of the observed pile-up.

i, and $R_i = \sum_{j\neq i} F_{ji}$ the total elastic force exerted on the dislocation i by the other dislocations j. Fig. 6b shows the result of the determination of γ_p obtained from the analysis of Fig. 6a. The following results are obtained:

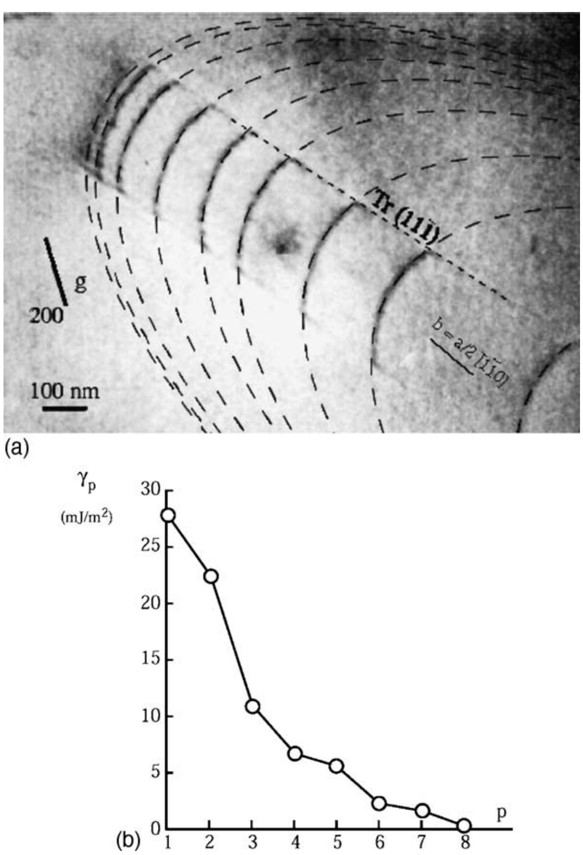

Fig. 6. (a) Example of a pile-up found during in situ deformation of γ_{MCRe} at 25 °C. The dislocations are located in the $(1\,1\,\bar{1})$ plane. The positions of the dislocations have been calculated assuming fault energies plotted in (b). The calculated positions are superimposed (dot lines), the equilibrium shape corresponds to a dislocation pinned at the surface submitted to an effective stress of 11 MPa. (b) Energies γ_p of the defects between dislocation p and $p + 1$ resulting from short-range order.

- The energies of the faults are found to decrease down to zero only after the 6th dislocation. Thus, the passage of seven dislocations appears necessary to destroy totally the SRO.
- As a consequence, as the last dislocations are at equilibrium, the effective stress $\tau_{eq} - \tau_f$ acting on the observed pile-up is very small, of the order or less than 1 MPa.
- The friction force γ_0, or equivalently the stress τ_0 ($=\gamma_0/b$) on the 1st dislocation, is obtained from Fig 6b. Observations show that it decreases with temperature from about 30 mJ/m^2 ($\tau_0 \approx 120$ MPa) at 25 °C down to 10 mJ/m^2 ($\tau_0 \approx 39$ MPa) at 750 °C, in agreement with the results obtained by Schwander et al. in Ni–21% Cr [8]. Furthermore, the force due to SRO opposed to the movement of the dislocations is so high (from 40 to 120 MPa compared to the applied stress in the order of 10 MPa) that the propagation of dislocations is only possible by the formation of pile-ups, even at this temperature.

4. Conclusions

Transmission electron microscopy observations of pile-ups in alloys presenting short-range order have been interpreted with the help of calculations taking into account the stress relaxation at the surface of a thin foil.

Experiments show that the length of the pile-ups and the general distribution of the dislocations within it are influenced by the foil thickness but that the spacing between the first two dislocations is not sensitive to the thickness of the foil. This has been attributed to the existence of short-range order in the alloys. The evaluation of the friction force due to SRO, as well as the associated energy values, were determined by comparison of measured dislocations positions and numerical calculations taking into account the thin foil effect. The results attest that the thin foil influence must be taken into account for a better accuracy of the results.

References

[1] F. Pettinari, M. Prem, G. Krexner, P. Caron, A. Coujou, H.O.K. Kirchner, N. Clément, Acta Mater. 49 (2001) 2549–2556.
[2] M. Jouiad, F. Pettinari, N. Clément, A. Coujou, Phil. Mag. A 79 (1999) 2591–2602.
[3] M. Jouiad, N. Clément, A. Coujou, Phil. Mag. A 77 (1998) 689–699.
[4] F. Pettinari, Doctorate Thesis, Toulouse University, February 1999.
[5] G. Saada, J. Douin, F. Pettinari-Sturmel, A. Coujou, N. Clément, Phil. Mag. 84 (2004) 807–824.
[6] J.D. Eshelby, A.N. Stroh, Phil. Mag. 42 (1951) 1401–1405.
[7] Y.T. Chou, F. Garofalo, R.W. Whitmore, Acta Metall. 8 (1960) 480–488.
[8] P. Schwander, B. Schönfeld, G. Kostorz, Phys. Stat. Sol. (b) 172 (1992) 73–85.