

Some Deformation Micromechanisms in Ni-based Superalloys Evidenced Using TEM In Situ Experiments

Florence PETTINARI-STURMEL^{1,2,a}, Mustafa BENYUCEF^{3,b},
Joël DOUIN^{1,2,c}, Pierre CARON^{4,d}, Didier LOCQ^{4,e}, Nicole CLEMENT^{1,f}
and Armand COUJOU^{1,2,h}

¹ CEMES-CNRS, 29 rue Jeanne Marvig, BP 94347 31055 Toulouse, France

² Université Paul Sabatier, 118 route de Narbonne 31062 Toulouse, France

³ Université Cadi Ayyad, Marrakech, Maroc

⁴ ONERA, 29 avenue de la Division Leclerc, BP 72, 92322 Châtillon, France

^aflorence.pettinari@cemes.fr, ^bbenyucef_mustafa@yahoo.fr, ^cdouin@cemes.fr, ^dlocq@onera.fr,
^ecaron@onera.fr, ^fm_nicole@orange.fr, ^hcoujou@cemes.fr

Key words: TEM in situ, deformation micromechanisms, moving dislocation.

Abstract. *In situ* straining of microsamples have been carried out at different temperatures in a transmission electron microscope (TEM) to provide information about the elementary micromechanisms, which control the deformation in Ni-based superalloys at the nano and microscopic scales. The obstacles to the propagation of the dislocations have been identified and quantified when possible. The strengthening effect in the γ -matrix channels and the shearing of γ' precipitates have been chosen to illustrate some results deduced from moving dislocation analysis. A quantitative evaluation of the stresses acting on dislocations has been performed. The shearing process of the γ' precipitates at 850°C has been precisely analysed.

Introduction

The aim of fundamental studies in plasticity is to correlate the macroscopic mechanical behavior to the elementary deformation processes controlling the dislocation motion. TEM *in situ* experiments offer the unique advantage to allow the direct observation of the dislocation multiplication and propagation during the deformation and to access to the dynamics of the events. Developed for few decades, this technique has demonstrated its capability when applied to a great variety of metallic alloys [1 - 4]. The deformation mechanisms occurring in Ni-based superalloys have been widely investigated using TEM by many authors. Depending on the temperature, on the strain rate and on the applied stress, shearing or by-passing of γ' precipitates may occur through the movement of paired perfect dislocations ($b = a/2\langle 110 \rangle$) surrounding an antiphase boundary (A.P.B) [5 - 9] or through the movement of super-Shockley partials ($b = a/3\langle 112 \rangle$) trailing a superlattice intrinsic or extrinsic stacking fault (S.I.S.F or S.E.S.F) or else through the movement of Shockley partials ($b = a/6\langle 112 \rangle$) trailing a stacking fault [10 - 18]. Whatever the temperature, when deforming this two-phase material, in addition to the obvious role of the γ/γ' interface, two other processes influence the deformation rate: the propagation of moving dislocations in the γ -channels and the shearing of γ' -precipitates. The roles of these two “obstacles” at the origin of the exceptional strength of the Ni-based superalloys can be summarized as follows:

- the γ matrix channels which force the dislocations to be strongly curved and the short range order present in this phase which impedes the dislocation movement,
- the γ' -precipitates whose interfaces with the γ -phase consist in structural and chemical discontinuities and whose ordered structure is at the origin of a three dimensional core structure for the non glissile screw dislocations and hence induces particular shearing.

The aim of this paper is not to give an exhaustive description of all the processes involved during the deformation of the Ni-based superalloys but although to highlight some specific results deduced from *in situ* TEM experiments.

Experimental details

The *in situ* straining experiments have been carried out using a JEOL 200 CX or a JEOL 2010 transmission electron microscope operating at 200 kV, and at 25°C, 350°C and 850°C. The TEM *in situ* tensile tests have been performed on two Ni-based superalloys: the MC2 and the NR3 both provided by ONERA [16]. In order to analyze precisely the deformation mechanisms associated with the γ -phase, single crystals of γ -phase corresponding to the γ -phase of the MC2 superalloy have been especially elaborated at ONERA and investigated [19].

Strengthening effect in the γ -matrix

The most commonly reported mechanism operating at low temperature and high stress level is the deformation by $a/2 \langle 110 \rangle$ slip on the $\{111\}$ octahedral planes in the γ -matrix and the shearing of the γ' -precipitates by paired $a/2 \langle 110 \rangle$ dislocations. We focused here on the glide of perfect dislocations in the γ -phase. One of the contributions to the strength of the alloy is the Short Range Order (SRO) present in the γ -phase. It is now admitted for many years that the presence of SRO induces a local glide resistance higher than the critical resolved shear stress, so that individual movements are impossible and collective motions of dislocation pile-ups are observed in the γ -phase [20 – 23]. In order to evaluate quantitatively the strengthening effect due to SRO, we have developed and improved in the last few years a methodology based on the analysis of dynamic dislocation pile-ups observed in the γ -single-phase during *in situ* tensile tests performed from 25°C to 350°C [19, 24]. The experimental determination of the dislocation positions within the pile-up allows to determine the elastic interaction forces between dislocations and then to access to the stresses associated with SRO. An example of dislocation pile-up analysis is illustrated Fig. 1. It has to be pointed out that: i) the SRO friction effect acts mainly for the leading dislocation, ii) the calculated value of the SRO-stress encountered by the first dislocation is effectively higher than the critical resolved shear stress (close to 100 MPa for the γ -phase). This last result justifies the formation of pile-ups which induces a stress multiplication at the head of the pile-up and thus helps to overcome the SRO friction stress.

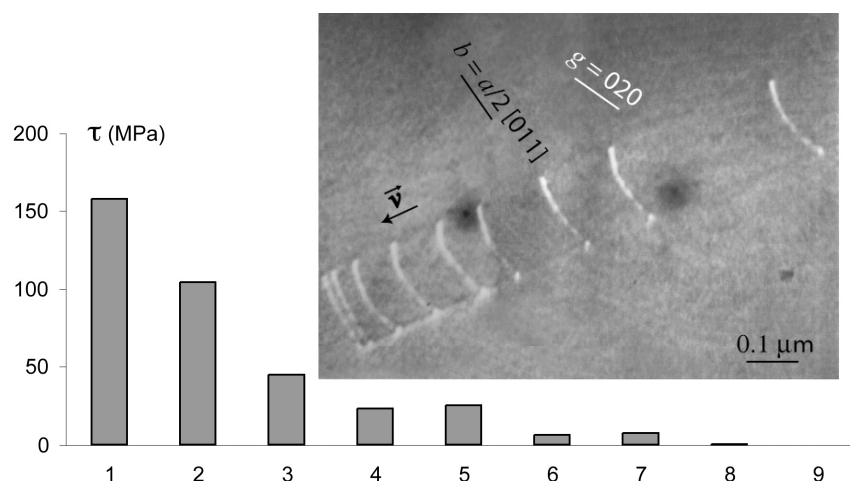


Figure 1. Experimental dynamic dislocation pile-up observed in the γ -single-phase and the corresponding friction stress due to SRO experienced by each dislocation (the value for the 9th is so close to zero that it is not visible). The value for the 1st dislocation has to be compared with the 100MPa of the critical resolved shear stress.

Note that the configuration of the dislocations is different in the γ -single-phase than in the two-phase superalloy: small pile-ups containing about 4 dislocations are observed in the two-phase material [25]. As a justification, the critical resolved shear stress is about four times higher in the two-phase material than in the single phase, and the microstructure is completely different. Indeed,

the presence of ordered γ' -particles generates narrow channels and interfaces, so that SRO becomes one contribution amongst the other ones. However, the obtained values for the SRO friction stresses are useful when calculating the total effective local stresses acting on the dislocations and there is no physical reason for the SRO friction stress to be different in the γ -phase compared with the two-phase alloy. It is worth emphasizing that the SRO friction stress decreases with temperature [19] and is close to zero at 900°C, so that no contribution of the SRO to the strengthening can be considered at high temperature.

To enter the γ -channels, a dislocation has to be curved. The corresponding effective stress required for this mechanism to occur has to be at least equal to the well-known Orowan stress τ_{OR} (the total effective stress combines the applied stress, the friction stress, and the misfit stress. The Orowan stress is proportional to the line tension of the dislocation over the channel width [26]). As the line tension decreases with temperature, whereas the shearing of γ' -precipitates is the prevalent mechanism at low temperature, the bypassing by perfect dislocation is favored at high temperature. When the local effective stress is too low to propagate perfect dislocations, other more subtle mechanisms occur involving partial dislocations. This has been observed both in single crystal and in polycrystalline superalloys during tensile or creep tests. An example of propagation of partial dislocation is illustrated Fig. 2. It concerns the NR3 superalloy deformed at room temperature. The description of this particular mechanism and its occurrence is discussed in detail in another paper [27].

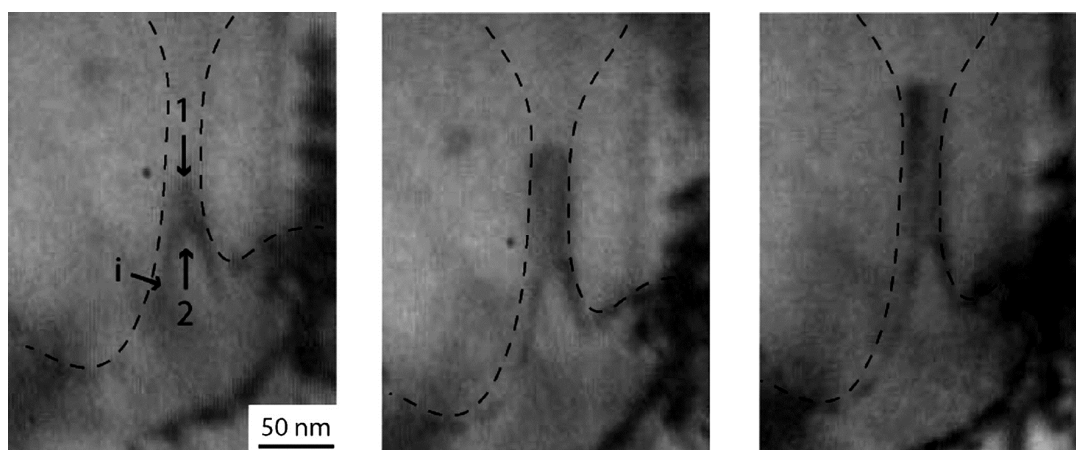


Figure 2. Propagation of a partial dislocation trailing a stacking fault in a narrow γ -channel. The moving leading dislocation is noted 1, and the trailing dislocation stopped at the entrance of the channel is noted 2.

In the intermediate temperature regime (i.e. 650°C – 850°C), the dislocation behavior is more complex because of a variety of micromechanisms involving partial dislocations as fault formation either only in the precipitates, either continuously in the matrix and the precipitates, or else microtwinning [10 -18]. One example of shearing by partial dislocation is described in details in the following.

Shearing of γ' -precipitates at 850°C

The shearing mechanism involving super-Shockley partials has been frequently observed during primary creep of nickel-based superalloys at intermediate temperature [28 - 31]. Two different models have been proposed, based on distinct hypotheses:

- Kear *et al.* [30] assume that $a/3 \langle 112 \rangle$ are created by the dissociation of dislocations $a/2 \langle 112 \rangle$.
- Condat *et al.* [17] developed the ideas previously exposed by Kear *et al.* [29] that $a/2 \langle 110 \rangle$ matrix dislocation, entering the γ' particle acts as a superpartial and transforming its APB, creates a superlattice stacking fault by nucleation of a super-Shockley partial. This mechanism is favourable because the original antiphase boundary gives rise to a superlattice stacking fault of less energy. Our aim was to analyse this shearing mechanism.

A representative dynamic process of the shearing at 850°C observed in the MC2 superalloy is illustrated Fig. 3. At the beginning of the sequence, a matrix dislocation noted *i* which is at the origin of the shearing, is stopped at the γ/γ' interface as it is out of contrast, its Burgers vector is $\pm DA = \pm a/2[101]$ (Fig. 3a). Then, while gliding in $(\bar{1}\bar{1}1)$ plane, it appears with a very faint contrast trailing a S.I.S.F (Fig. 3b). The crossing begins at the corner of the precipitate B (Fig. 3b). From this corner the S.I.S.F expands continuously while the super-Shockley is observed to remain parallel to the $[011]$ direction (Fig. 3c). When the shearing is completed, the fault covers the whole precipitate (Fig. 3d). A dislocation noted *j* remains at the interface, surrounding the precipitate. In this sequence, the shearing by super-Shockley dislocation originates from one perfect matrix dislocation.

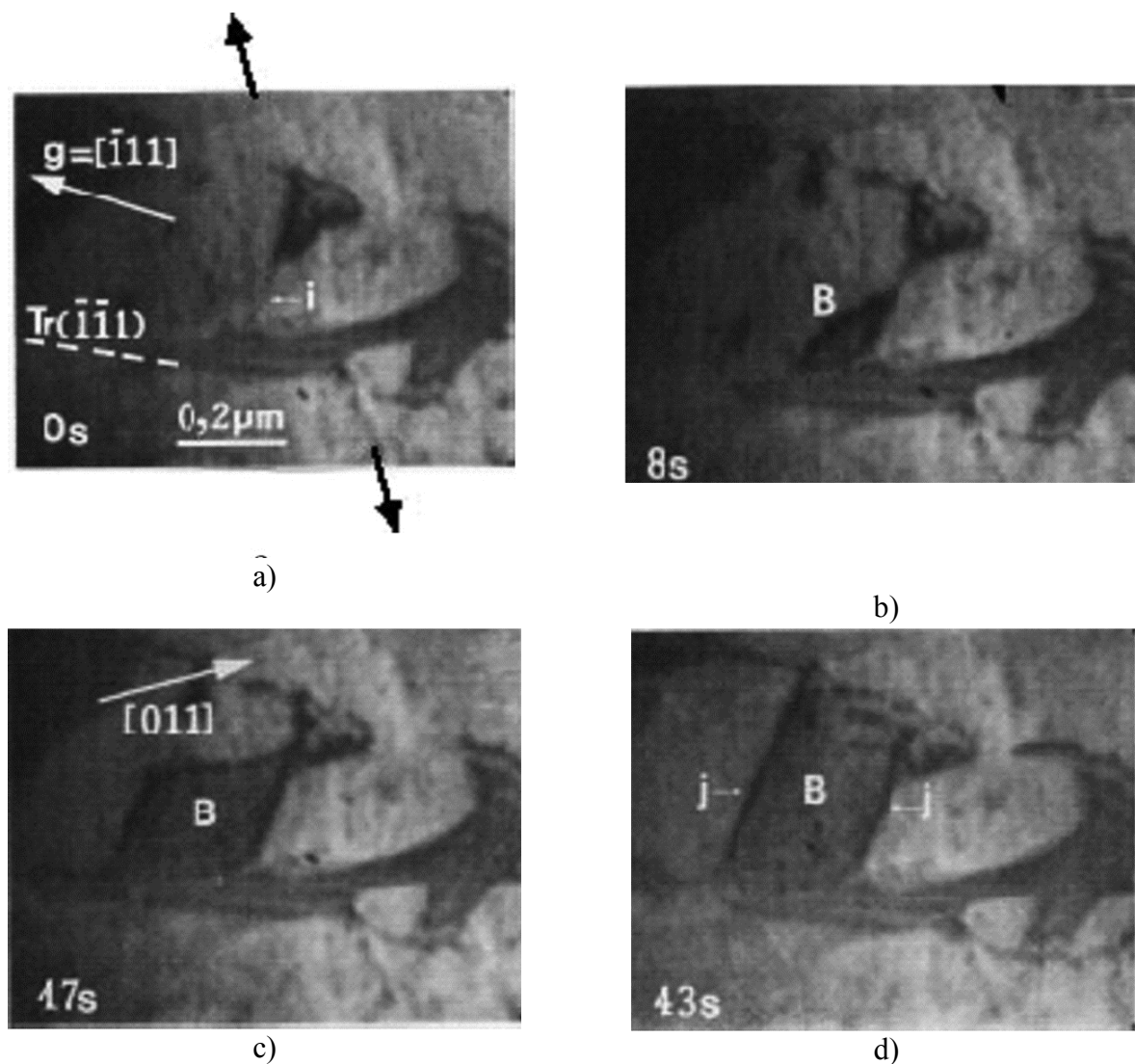


Figure 3. *In situ* observation of γ' -precipitates shearing by super-Shockley partials. a) initial state, b) the shearing begins at the corner of precipitate B, a S.I.S.F is left behind the moving super-Shockley, c) the super-Shockley appears to be aligned along $[011]$, d) complete shearing of the γ' precipitate.

Within the γ' phase the propagation rate of the super-Shockley partial has been measured. It varies, as a function of its position: a strong decrease of the velocity is observed when the super-Shockley direction becomes nearly parallel to $[011]$ direction. The velocity varies from 45 nm/s to 5 nm/s. The *in situ* experiments have shown that such a shearing can exist repeatedly in a shear band owing to the stress multiplication effect when several coplanar dislocations are piling-up against the interface.

Then, the equilibrium of a matrix dislocation stopped at the apex of a γ' precipitate has been quantitatively analysed using the experimental data. The dislocation segment $d\ell$ undergoes four forces (Fig. 4.):

- the force due to the applied stress $\tau_a b d\ell$,
- the forces due to the projection of the line tensions ξ_1 and ξ_2 associated with the straight segments of the dislocation not in contact with the precipitate,
- the force F exerted by the precipitate and opposing to the motion of the dislocation.

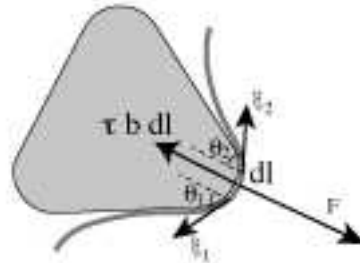


Figure 4. Schematic representation of a dislocation entering a γ' -precipitate by its apex.

For a rough estimate, in the frame of isotropic elasticity and using the De Witt and Koehler formulation [32], the line tension contribution is given by $\xi_1 \cos \theta_1 + \xi_2 \cos \theta_2 = 1.75$ nN (with $b = 0.245$ nm, $\mu = 58.6$ GPa and $\nu = 1/3$). This value has to be compared with the resolved applied force which is $\tau_a b d\ell = 1.6$ nN.

To conclude, these dynamic experiments are consistent with those mentioned in literature in the case of relaxed situations: the fact that the shearing process begins at the corner of the γ' particles [9] and that the dissociation of one perfect matrix dislocation leaving on the interface a Shockley partial have already been reported [17, 31]. Else, the decrease of the velocity of the moving super-Shockley when aligned along a $\langle 110 \rangle$ direction is directly connected with a core dissociation described in [28]. Nevertheless our *in situ* experiment emphasized the fact that:

- the shearing process is initiated from the apex of a γ' particle and is assisted by the line tension, which has been evaluated and is in the same order of magnitude as the applied force,
- the velocity of the moving super-Shockley has been measured to decrease when aligned along a $\langle 110 \rangle$ direction in relation with a core dissociation.

Summary

In situ straining experiments have been performed on Ni-based superalloys in order to precise some deformation characteristics. Two controlling deformation parameters have been investigated: the strengthening effect of the γ -phase and the shearing of the γ' -precipitates by partial dislocations. As the $a/2\langle 110 \rangle$ perfect dislocation propagates through short range ordered γ -channels, the friction stress associated with the SRO was estimated at about 150 MPa. The shearing of the γ' -precipitates at 850°C has been analysed: it occurs through the viscous movement of a super-Shockley partial trailing a super intrinsic stacking fault resulting from a perfect dislocation.

References

- [1] A. Coujou: Acta Metall. 31 (1983), p.1505.
- [2] A. Coujou, M. Benyoucef, M. Legros and N. Clément: Sol. Stat. Phen. 59-60 (1988), p. 185.
- [3] D. Caillard, N. Clément, A. Couret, P. Lours and A. Coujou: Phil. Mag. Lett. 58 (1988), p. 263.
- [4] N. Clément, A. Couret and D. Caillard: Phil. Mag. A64 (1991), p. 669.
- [5] D.P. Pope and S.S. Ezz: Int. Metals Rev. 29 (1984), p. 136.
- [6] E. Nembach and G. Neite: Prog. Mater. Sci. 29 (1985), p. 177.
- [7] G.F. Eggeler and A. Dlouhy: Acta Metall. Mater. 45 (1997) p. 4251.
- [8] T.S. Rong, I.P. Jones and R.E. Sallman: Acta Metall. Mater. 53 (2005), p. 3041.
- [9] P. Caron, T. Khan and P. Veyssière: Phil. Mag. A 6 (1988), p. 859.
- [10] M.G. Ardakani, M. McLean and B.A. Shollock: Acta Mater. 47 (1999), p. 2593.
- [11] D.M. Knowles and Q.Z. Chen: Mater. Sci. Eng. A340 (2003), p. 88.
- [12] G.B. Viswanathan, P.M. Sarosi and M.F. Henry: Acta Metall. Mater. 53 (2005), p. 3041.
- [13] G.B. Viswanathan, S. Karthiekeyan, P.M. Sarosi, R.R. Unocic and M.J. Mills: Phil. Mag. 29-31 (2006), p. 4823.
- [14] M. Kolbe: Mater. Sci. Eng. A 319/321 (2001), p. 383.
- [15] B. Décamps, S. Raujol, A. Coujou, F. Pettinari-Sturmél, N. Clément, D. Locq and P. Caron: Phil. Mag., 84, 1 (2004), p. 91.
- [16] S. Raujol, M. Benyoucef, D. Locq, P. Caron, F. Pettinari, N. Clément and A. Coujou: Phil. Mag. 86, 9 (2006), p. 1189.
- [17] M. Condat and B. Décamps: Scripta Metall., 21, 5 (1987), p. 607.
- [18] B. Décamps, J.M. Péniisson, M. Condat, L. Guétaz and A.J. Morton: Scripta Metall., 30, 11 (1994), p. 1425.
- [19] F. Pettinari-Sturmél, J. Douin, A. Coujou and N. Clément : Z. Metallkd. 97 (2006) p. 200.
- [20] J. Olfe and H. Neuhäuser: Phys. Stat. Sol. (a) 109 (1988) p. 149.
- [21] V. Gerold and H-P. Karnthaler: Acta Metall. 37 (1989) p. 2177.
- [22] J. Plessing, Ch. Achmus, H. Neuhäuser, B. Schönfeld and G. Kostorz: Z. Metallkd. 88 (1997), p. 630.
- [23] N. Clément, in: *L'ordre et le désordre dans les matériaux*, edited by F. Reynaud, N. Clément, J.J. Couderc (Les Editions de Physique, Aussois, Paris) (1984).
- [24] G. Saada, J. Douin, F. Pettinari-Sturmél, A. Coujou and N. Clément : Phil. Mag. A 84 (2004) p. 807.
- [25] M. Benyoucef, N. Clément and A. Coujou: Phil. Mag. A71-72 (1995), p. 1043.
- [26] E. Orowan, in: *Proceedings of the Symposium on Internal Stresses in Metals and Alloys*, (London: Institute of Metals) (1948), p. 451.
- [27] F. Pettinari-Sturmél, J. Douin, , D. Locq , P. Caron and A. Coujou, in: *Eurosuperalloys 2010*, Trans Tech. Publications (2010) to be published.
- [28] A.F. Giamei, J. Oblack and B.H. Kear, in: *Proceedings of the 29th Annual EMSA Meeting* (Baton Rouge, Louisiana : Claitor's), II2 (1971).
- [29] B.H. Kear., A.F. Giamei, J.M. Silcock and R.K. Ham: Scripta Metall. 2 (1968) p. 281.
- [30] B.H. Kear., A.F. Giamei, G.R. Leverant and J.M. Oblak: Scripta Metall 3 (1969), p. 455.
- [31] A.J. Huis in't Veld, G. Boom, P.M. Bronsveld and J.T. De Hosson: Scripta Metall. 19 (1985) p. 1123.
- [32] G. De Witt, J.S. Koehler: Phys. Rev. 116 (1959) p.1113