LOCAL ORDER AND MECHANICAL PROPERTIES OF THE γ MATRIX OF NICKEL-BASE SUPERALLOYS

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Abstract

For the first time γ single crystals, closely matching with the chemical composition of the matrices of the industrial single crystal superalloys AM3 and MC2 have been made. Macroscopic tensile tests as well as "in situ" deformation tests show that in both alloys a strong localization is the main characteristic of the deformation at different scales of observation. Collective movements of dense planar arrays of dislocations are observed related to the presence of order which appears to be different depending on the alloy. These ordering phenomena introduce high friction forces larger than the applied stress ($\tau_f \approx 2.4 \tau_{el}$) opposing dislocation movement.

The MC2 matrix has a tensile strength 15 % higher than that of the AM3 matrix. This improvement in strength is partially due to the higher content in W. From deformation and neutron diffraction experiments, it appears that the hardening mechanism is not only a solid solution one, but also associated with a local order hardening which depends on the alloy. The superior mechanical properties of the MC2 γ phase are attributed presumably to the presence of a DO₂₂ short range order due to W.

Introduction

In nickel base superalloys, most of the studies reported in the literature have, up to now, been devoted to the mechanical properties of the two phase γ/γ' material or the strengthening γ' phase. Even though the creation and propagation of dislocations in those materials, especially at high temperature, occurs in the γ matrix, the hardening contribution of the γ matrix has been neglected or underestimated. With the exception of the paper by Beardmore et al. [1] very little information is available on the intrinsic characteristics of this f.c.c. phase, often considered to be disordered. Two main reasons can explain this :

- Direct microscopic observations within the very narrow γ channels are not easy,
- The precise composition of the γ phase is difficult to determine and due to solidification difficulties no matrix single crystals were available until now.

In order to improve the mechanical properties of superalloys, it is necessary to understand the hardening mechanisms operating in the γ phase itself, which, of course, are related to its structure and composition.

For the first time, we succeeded in the fabrication of γ single crystals, almost exactly of the chemical composition of the matrices of AM3 and MC2 industrial nickel-base superalloys [2]. These alloys were recently developed at ONERA for single crystal turbine blade applications. AM3 is a low density ($d = 8.25 \text{ g.cm}^{-3}$) superalloy which exhibits a mechanical strength comparable to that of the first generation single crystal superalloys with densities close

to 8.6 g.cm^{-3} . MC2 ($d = 8.62 \text{ g.cm}^{-3}$) is a very high strength single crystal superalloy showing a 50°C operating temperature advantage over AM3. With a chemical composition quite similar to those of many conventional superalloys, its creep behaviour is comparable to that of some recent superalloys which contain rhenium.

The aim of this study is to elucidate the reasons for such a difference considering the microstructural aspects and the microscopic deformation processes occurring in the γ phase. Here we have to deal with the question of order vs. disorder complicated by the fact that in the matrix of complex industrial alloys, not only short range order, but also long range order (for example, DO₂₂ as well as L1₂ type) may be present. For Cr (20 - 30 at %) rich Ni-Cr alloys, neutron scattering, deformation experiments and in situ electron microscopy indicate the presence of short range order, of the type Ni₂Cr [4, 6] or Ni₃Cr [3, 5, 7, 8, 9]. Short range order of the Ni-Cr pairs has also been found by X-ray scattering in Ni₂CoCr, and the same was confirmed to exist in the MC2 matrix single crystals used in the present study [3, 10].

Experimental procedure

The microchemistries of the γ MC2 and γ AM3 phases in the two phase materials were determined by atom probe nanoanalysis [11]. It must, however, be noted that the compositions chosen and prepared correspond to the equilibrium composition at 850°C (the temperature of the final ageing treatment of the alloys). The composition of the corresponding γ single crystals were checked to be very similar to those of the matrices of the two-phase materials. They are given in Table I. The W content is significantly higher in the MC2 matrix, while the concentrations of Al, Ta and Ti are higher in the AM3 matrix. As Al, Ti and Ta are considered to favour precipitation of the γ' phase and in order to check their influence on ordering, MC2 type matrix crystals without these γ' formers were also grown. All these specimens were homogenized 3h at 1300°C and air cooled.

Table I : Composition (at.%) of the three different matrices investigated, as checked by chemical analysis.

	Ni	Cr	Co	Al	Ta	W	Ti	Mo
γ AM3 matrix	52.19	27.1	11.81	3.69	0.42	1.53	0.36	2.9
γ MC2 matrix	54.93	25.58	9.21	2.84	0.29	4.23	0.25	2.67
γ MC2 matrix without γ' formers	57.42	26.5	9.16	-----	-----	4.19	-----	2.72

In order to determine unambiguously the controlling mechanisms in such complex industrial alloys, experiments were conducted at different scales :

- Macroscopic tensile tests,
- S.E.M. observations of the surfaces,
- "In situ" deformation tests in a T.E.M.,
- "Post mortem" T.E.M. observations of macroscopically deformed samples,
- Neutron diffuse scattering.

Experimental Results

Tensile tests

Figure 1 : True stress-strain curves of three different matrices. [001] tensile axis, room temperature.

Figure 2 : True stress-strain curves of three different matrices. [001] tensile axis, $T = 750^{\circ}\text{C}$.

Figure 3 : True stress-strain curves of three different matrices. Tension [001] tensile axis, $T = 900^{\circ}\text{C}$.

Figure 4 : Yield stress as a function of temperature for γMC2 , γAM3 and γMC2 without γ' formers. [001] tensile axes.

Tensile tests were conducted on single crystals with [001] orientation. The test temperature was varied from -196°C to 900°C , and the crosshead speed corresponded to a strain rate of 10^{-4} s^{-1} . The deformation was interrupted at around 2.5 % maximum plastic strain. Samples were then cut from the tensile specimens for observation in a JEOL 200CX transmission electron microscope.

The "true" curves corresponding to instantaneous values of section and length during the test are shown in Figures 1, 2 and 3. Plastic instabilities corresponding to load drops exist on these curves. The onset of the serrations indicated by arrows in Figures 1, 2 and 3, is seen to appear at decreasing strains, while their amplitude increases. The temperature dependence of the yield stress is shown in Figure 4.

From these stress-strain curves it appears that :

- The strength of the γ phase is higher than expected [12], i.e. 243 MPa at 20°C and 121 MPa at 900°C (Figures 1 and 3).

- The strength of γMC2 is about 15% higher than that of γAM3 , from room temperature to 500°C (Figures 1 and 4).

- A stress peak exists at 760°C in γ alloys containing γ' elements while it disappears in the alloy without these γ' formers (Figure 4) where a continuous decrease of the flow stress is observed as expected for thermally activated phenomena.

The comparison of the data obtained on the two MC2 matrices with and without γ' forming elements shows that Al, Ti and Ta do not have a significant influence on the yield strength below the stress peak.

Scanning Electron Microscopy

Strong localization is the main characteristic of the deformation observed at different scales :

At a mesoscopic scale (Cambridge S.E.M., 20 kV), Figure 5 shows that, independent of the temperature, for a low deformation of about 2 %, slip bands of bundles of {111} slip lines are created at the sample surface. They are very heterogeneous: slip zones 2.5 to 10 μm large separated by 50 to 100 μm wide zones, free of any traces, are observed.

Figure 5 : Observation of slip traces at the surface of deformed samples (S.E.M.) : a) $T = -196^{\circ}\text{C}$; b) $T = 25^{\circ}\text{C}$; c) $T = 600^{\circ}$; d) $T = 900^{\circ}\text{C}$.

"In situ" Deformation Tests in a T.E.M.

"In situ" deformation tests were performed at various temperatures in a JEOL 200 CX T.E.M., at 200 kV. The tensile axis of the microsamples was $\langle 001 \rangle$ (Schmid factor = 0.43 for $\{111\}$ planes).

As in the macroscopic observations at low and medium temperatures, the deformation appears to be very localized. The microstructure developed during these deformation tests consists of dense planar arrays of mobile dislocations. In Figure 6, such a moving group is observed with three pairs clearly visible at the head of the group. Here the average velocity is 85 nm.s^{-1} and the movement is observed to be correlated for dislocations belonging to the same pair. This microstructure is very different from a F.C.C. disordered phase microstructure where individual dislocation movements create an homogeneously distributed deformation. Several physical mechanisms have been suggested to explain this behaviour observed in concentrated solid solutions [13, 14, 15]. Here the presence of tiny ordered coherent precipitates, or the presence of short range order is emphasized and corroborated by the neutron diffraction experiments.

"Post mortem" T.E.M. Observations of Macroscopically Deformed Samples

Samples cut parallel to $\{111\}$ planes in deformed tensile specimens allow large dislocation arrays to be imaged in their glide planes. Up to 900°C , as in the "in situ" experiments, the deformation is very heterogeneous; above this temperature, on the contrary, it is homogeneous (Figure 7).

In Figure 12a such a planar group in γ AM3 is shown : a source located at S emitted 16 dislocations on the $\{111\}$ plane, the sense of the Burgers vectors $b = 1/2[-1 \ 0 \ 1]$, being different on each side of the source.

Figures 8, 11a, 12a, 13a and 14a correspond to static and relaxed configurations : unlike a standard pile-up, the spacing between dislocations inside the first group does not follow the expected scheme. Instead of a regular increase of the spacing from the front to the back of the pile-up, pairs exist at the head of the pile-up. Measurements of the distance between two successive dislocations, $d_{ij} = f(i)$, allow their identification when a minimum appears on the distribution (in γ MC2, Figures 11c, 13b and 14c, in γ AM3 Figure 14c).

Figure 6 : $T = 350^{\circ}\text{C}$. "In situ" dynamic sequence in γ MC2. Y is a fixed point.

Figure 7 : $T = 900^{\circ}\text{C}$; "Post mortem" observations in γ MC2 macroscopically deformed samples. Homogeneous distribution of dislocations. Presence of loops.

When the γ MC2 and γ AM3 samples are deformed at increasing temperatures, the number of pairs increases (Figure 8) : 2 pairs at 25°C , 3 pairs at 350°C , 5-6 pairs at 600°C . On the contrary, in γ MC2 samples devoid of γ' formers, only one pair is observed at the head of the moving pile-ups independent of the temperature between 25°C and 600°C . (Figure 14a, $T = 500^{\circ}\text{C}$).

From these observations one can deduce that some tiny ordered particles exist in the γ MC2 and γ AM3 phases even at room temperature. With this hypothesis the reason for pairing is clear : if during the deformation a dislocation glides through a long range ordered particle, its order is destroyed. Dislocations therefore move in pairs, the leading one creates an antiphase boundary and the trailing one restores order. The complete shearing of a microprecipitate, whose radius is r , is obtained after the crossing of r/b dislocations, ($b = 0.25$ nm is the Burgers vector of the moving dislocations). Measuring the pair number (from 2 to 6 depending on the temperature) allows the magnitude of the ordered zone to be determined (here d varies from 0.5 to 3.0 nm). This value is in good agreement with the dimensions of ordered zones estimated by atom-probe techniques which were found to be around 4 to $5 \times 2d_{002} = 1.5$ to 2 nm [3, 11]. Since they are so tiny, they do not give any superstructure spots in the electron diagrams at room temperature.

Neutron Diffuse Scattering

As these pairings indicate the presence of order, different types of ordering seem to exist in different matrices. In order to evaluate and compare them, all these experiments were completed with neutron diffraction on γ AM3 and γ MC2 single crystals. They prove that, contrary to the widely accepted idea, these solid solutions are not homogeneously disordered [5].

In agreement with T.E.M. experiments, $L1_2$ long range order appears to exist in both samples : the special points for this type of ordering are $(1\ 0\ 0)$ and $(1\ 1\ 0)$. In both the MC2 (Figure 9a) and AM3 specimens (Figure 9c) well defined (100) peaks are present. This is in good agreement with the microscopic observations (Figure 10) where beyond 700°C superstructure $\langle 100 \rangle$ and $\langle 110 \rangle$ diffraction spots are clearly visible on electron diffraction patterns (Figures 10b and 10d) of both samples annealed in the microscope. When they are selected they image small precipitates appearing in dark field contrast conditions. The existence of γ' $L1_2$ microprecipitates growing when the temperature is raised is therefore corroborated, their dimension being $d = 15$ nm at 760°C (Figure 10c).

Short range order seems to exist too : taken in $[4\ 2\ 0]$ direction, $(1\ 1/2\ 0)$ diffuse maxima are observed in both samples (Figures 9b and 9d). They can only come from DO_{22} ordering.

As in the γ MC2 alloy (Figures 9a and 9b), the three characteristic reflections $(1\ 0\ 0)$, $(1\ 1/2\ 0)$ and $(2\ 1\ 0)$ are present, while $(1\ 1/2\ 0)$ and $(2\ 1\ 0)$ only are visible in γ AM3, (Figures 9c and 9d), it seems that γ MC2 has a greater propensity for DO_{22} ordering than γ AM3.

If one considers that Ni-Cr pairs only are responsible of such an order, it must be recalled that in binary Ni 75 at.% - Cr 25 at.% the order parameters at 530°C agree with a Ni_3Cr [16] type order with a critical transition temperature of less than 150°C . Due to the chemical composition of the alloy it is also possible in terms of off-stoichiometric compounds based on Ni-Cr, Ni-W ($T_c = 820^{\circ}\text{C}$) [17] or Ni-Mo ($T_c = 700^{\circ}\text{C}$) pairs participating in DO_{22} short range ordering type [8].

In any case, these ordering phenomena introduce friction forces opposing dislocation movement and thus increase the flow stress. Close inspection of the arrangement of moving dislocations in the pile-ups allows to evaluate them.

Figure 8 : Planar groups of dislocations as a function of temperature, in the deformed γ matrix of MC2 superalloy. "Post mortem" observations. An increase of the number of pairs at the head of the pile-up is observed : a) one at $T = -196^{\circ}\text{C}$; b) one at $T = 25^{\circ}\text{C}$; c) three at $T = 350^{\circ}\text{C}$; d) four at $T = 500^{\circ}\text{C}$; e) five at $T = 600^{\circ}\text{C}$.

Figure 9 : Diffuse neutron scattering
In $[1\ 0\ 0]$ direction : (a) γ MC2 ; (b) γ AM3.
In $[4\ 2\ 0]$ direction : (c) γ MC2 ; (d) γ AM3.

Figure 10 : Annealing of the MC2 matrix in the T.E.M. :
 $T = 700^{\circ}\text{C}$: a) diffraction pattern ; b) corresponding dark field image with a (110) superstructure spot.
 $T = 760^{\circ}\text{C}$: c) diffraction pattern ; d) dark field image with a (110) superstructure spot.

Figure 11 : γ MC2 ; $T = 25^\circ\text{C}$

a) Experimental pile-up.

b) Determination of the friction stress, from the experimental distribution of dislocations within the pile-up.

c) Determination of the number of pairs

$$\tau_{f/i} \text{ max} = 249 \text{ MPa} \quad \tau_{ss} = 27 \text{ MPa} \quad \tau_{f/i} = 2.4 \tau_{cl}$$

Figure 12 : γ AM3 ; $T = 25^\circ\text{C}$

a) Experimental pile-up

b) Determination of the friction stress, from the experimental distribution of dislocations within the pile-up.

c) Determination of the number of pairs

$$\tau_{f/i} \text{ max} = 221 \text{ MPa} \quad \tau_{ss} = 15 \text{ MPa} \quad \tau_{f/i} = 2.4 \tau_{cl}$$

Evaluation of Friction Stresses

The friction stress τ_f opposing the moving dislocations can be deduced by measuring the equilibrium position of each dislocation in the pile-ups [18]. Here we use an improved method compared with the estimation made in the case of Ni-Cr binary alloys [4].

As the observed pile-ups are relaxed, the applied stress has disappeared, $\tau_a = 0$. Since they are observed in a thin foil, the long range stress due to other dislocations inside the samples can be neglected. The position of each dislocation i in a pile-up is the result of two opposing forces which are in equilibrium :

$$b\tau_{fi} = -b\tau_{int/i}$$

- $b\tau_{int/i}$ is the force due to the elastic interaction of all the dislocations of the pile-up on dislocation i with :

$$\tau_{int/i} = \sum_{j \neq i} \frac{A}{x_i - x_j}$$

and

$$A = \frac{\mu b}{2\pi} \left[\cos^2 \phi + \frac{\sin^2 \phi}{1 - \nu} \right]$$

μ : Shear modulus

ν : Poisson's ratio

- $b\tau_{fi}$ is the friction force opposing the movement.

From the experimental positions x_i and x_j , measured in the observed pile-ups, $\tau_{int/i}$ can be calculated (Figures 11b, 12b and 13c) and the corresponding friction stress τ_{fi} deduced. The results are given in Table II.

Table II : Friction stresses τ_f and solid solution stresses τ_{ss} compared with the elastic limit τ_{el} . $T = 25^\circ\text{C}$

	γ AM3	γ MC2	γ MC2 without γ' formers
τ_f (MPa)	220	249	240
τ_{ss} (MPa)	10 to 20	20 to 30	10 to 20
τ_{el} (MPa)	91	104	54

$T = 25^\circ\text{C}$

On the average the friction stresses were found to be larger at the head of the pile-up than the yield stress τ_{el} , with $\tau_f = 2.4 \tau_{el}$ for the three matrices at 25°C , while they are smaller, $\tau_f = 0.2 \tau_{el}$ at the tail. This latter value corresponds to τ_{ss} , the solid solution stress, which is constant all along the pile-up.

This is in agreement with the observed mechanical behaviour. At the elastic limit, a single dislocation cannot move under the applied stress alone and pile-ups are necessary to enhance the stress and allow the deformation to proceed. The planar glide observed in the groups is associated with a softening mechanism. The first dislocations destroy the ordered arrangement existing in the glide plane, the following ones face a lower resistance to their movement. As a consequence they remain in the same glide plane.

At room temperature both short range order and long range order coexist in γ AM3 and γ MC2 samples as shown by neutron diffraction, while only S.R.O. can exist in γ MC2 without γ' formers. But it is not easy to separate their contribution to the measured friction stresses since only one pair of dislocations exists independent of the state of order.

$T = 500^\circ\text{C}$

At this temperature a difference appears depending on the nature of the matrix. Several pairs of dislocations are observed in γ MC2 and γ AM3 pile-ups, serrations corresponding to these pairs exist in $\tau_{int/i}$ curves (Figure 13c) which are a consequence of the shearing of L.R.O. small particles. On the contrary, in γ MC2 without γ' formers only one pair is visible at this temperature (Figure 14).

Figure 13 : γ MC2 ; $T = 500^\circ\text{C}$: a) Experimental pile-up; b) Determination of the number of pairs; c) Determination of the friction stress from the experimental distribution of dislocations within the pile-up.

$$\tau_{fi} \text{ max} = 72 \text{ MPa}$$

$$\tau_{ss} = 10 \text{ MPa}$$

$$\tau_{fi} \approx 1.3 \tau_{el}$$

Figure 14 : T = 500°C : a) "Post mortem" observation in γ MC2 without γ' formers. Macroscopically deformed samples.

One pair is observed at the head of the pile-up.
b) Determination of the friction stress, from the experimental distribution of dislocations within the pile-up.

c) Determination of the number of pairs
 $\tau_{f/i} \text{ max} = 135 \text{ MPa}$ $\tau_{ss} = 10 \text{ MPa}$ $\tau_{f/i} = 2.5 \tau_{el}$

As proposed by Schwander *et al.* [8] for binary Ni-Cr alloys, we use the concept of diffuse antiphase boundary energy, γ_{DAPB} , left between two successive dislocations moving in a S.R.O. structure. We denote by Γ , the total A.P.B. energy existing in a sample when both L.R.O. and S.R.O. coexist :

$$\Gamma = \gamma_{S.R.O.} + f \gamma_{L.R.O.}$$

f is the surface fraction of L.R.O. precipitates encountered by the dislocation during shearing. Two successive dislocations within a pair are then linked by a force $b \gamma_{DAPB}$, or $b \Gamma$ and these different antiphase energies can be separated [18] assuming that S.R.O. only acts on the first pair while L.R.O. exerts its influence on the four following ones. In this case :

$$b \tau_f = b \tau_{int} = b (\tau_{ss} + \Gamma / b)$$

Figures 13c and 14b which compare γ MC2 and γ MC2 without γ' formers are analyzed with this hypothesis. The results of this analysis are given in Table III.

Table III : Friction stresses and solid solution stresses compared with the elastic limits at 500°C for the MC2 matrices.

	γ MC2	γ MC2 without γ' formers
τ_f (MPa)	$i=1$ 72 $i=2$ 20 $i=3$ 18 $i \geq 4$ 20 $\tau_{f \text{ total}} =$ 130	$i=1$ 135 $i \geq 2$ 0 $\tau_{f \text{ total}} =$ 135
τ_{el} (MPa)	55	55
τ_{ss} (MPa)	≥ 10	≥ 10

Finally, through the variation of $\tau_{int}(i)$ calculated from the observed relaxed pile-ups ($\tau_a = 0$), we are able to identify and evaluate three different contributions to the friction stress in the matrices investigated :

$$\tau_f = \tau_{S.R.O.} + \tau_{L.R.O.} + \tau_{ss}$$

At 500°C comparing γ MC2 and γ MC2 without γ' formers it appears that :

1 - the contribution of the solid solution is the same ($\tau_{ss} \geq 10 \text{ MPa}$).

2 - $\tau_{S.R.O.}$ in γ MC2 without γ' formers is $\geq 135 \text{ MPa}$, it acts only on the first pair.

3 - summarizing the resulting stresses due to L.R.O. which exist on the fourth first pairs in γ MC2 a similar value is obtained ($\tau_{L.R.O.} \geq 130 \text{ MPa}$).

Points 2 and 3 are in good agreement with the observed mechanical properties of these two alloys, where similar elastic limits are observed in both samples at this temperature (Figure 4 : $\tau_{el} = 55 \text{ MPa}$). S.R.O. effect in γ MC2 without γ' formers appears to balance the effect of L.R.O. found in γ MC2.

Conclusion

According to our knowledge, for the first time, the strengthening mechanism of different γ matrices of industrial superalloys has been identified and compared on several levels, by various means of investigations (chemical analysis, tensile tests, T.E.M. "in situ" deformation tests at various temperatures, "post-mortem" observations of macroscopically deformed samples, neutron diffraction).

Regardless of the scale of observation and temperature, in the range 25°C - 600°C, the deformation in these single crystals appears to be very localized. The deformation proceeds through the collective movements of planar groups of dislocations. At the head of these groups dislocations clearly move in pairs which indicates the presence of local order (S.R.O. and L.R.O.). By a close inspection of these planar arrays the friction forces τ_f involved in each material have been calculated. They appear to be very high at the head of the moving groups, $\tau_f = 2.4 \tau_{el}$ and much lower at the tail, $\tau_f = 0.2 \tau_{el}$, in agreement with a softening mechanism postulated if the local order is destroyed by the moving groups.

In γ MC2 and γ AM3 alloys an hyperfine precipitation can occur when the sample is annealed. This is due to a supersaturation of the γ' forming elements (Al, Ti, Ta) at room temperature allowing a demixing to appear when the temperature is raised. Such a phenomenon has probably to be also considered when a γ/γ' superalloy is cooled from high temperature.

From tensile tests on γ single crystals, it appears that the γ phase of the MC2 and AM3 superalloys have a higher strength than generally expected. Below 500°C, in γ MC2, the presence or absence of γ' formers does not contribute significantly to the yield stress. Its high strength therefore appears to be due to the presence of elements such as Cr, Co, W and Mo. The strengthening of these matrices is not only due to solid solution hardening, but also to a local order hardening which depends on the alloy. The better mechanical properties of the MC2 γ phase compared to AM3 γ phase can be partially attributed to the occurrence of a DO₂₂ short range type order due to the presence of W.

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