The mobile dislocation pile-ups observed during in situ straining experiments have been used as probes to describe the short-range order (SRO) present in the $\gamma$-phase of nickel-base superalloys. An experimental approach based on the measurement of the dislocation positions to characterise the SRO has been developed. This paper focuses on the main results we have obtained on this topic. A quantitative analysis allows to get the chemical forces associated with SRO and also the SRO degree. For the first time the energies associated with SRO is evaluated along a dislocation pile-up as a function of temperature. More generally, a description of the SRO is proposed: SRO is seen as a heterogeneous distribution at the micrometer scale of nanometric areas. Another characteristic has been evidenced: the reversible character of the SRO creation.

**Keywords:** SRO; Dislocation pile-ups; SRO-energy

1. Introduction

The fcc concentrated Ni–Cr solid solutions, commonly found as the $\gamma$-phase of nickel-base superalloys, are of special industrial interest because of their high mechanical strength. The resistance to the dislocation motion is higher than the macroscopic elastic limit so that the formation of dislocation pile-ups is necessary. These pile-ups induce high internal stresses which contribute predominantly to the plastic deformation. Such a collective behaviour of the dislocations has been observed in different concentrated solid solutions by many authors [1–4]. Our analysis is based on TEM observations and lid solutions by many authors [1–4]. Our analysis is based on TEM observations and lid solutions by many authors [1–4].

The determination of the different $F_{ip}$ is based on the experimental measurements of the positions of the

2. Methodology

2.1. Experimental details

The investigated $\gamma$-phases ($\text{Ni}_{65.6}\text{Cr}_{26.2}\text{Mo}_{2.0}\text{W}_{1.9}\text{Re}_{4.2}$) and ($\text{Ni}_{65.9}\text{Cr}_{26.1}\text{Mo}_{2.0}\text{W}_{1.9}\text{Ru}_{4.0}$) are single crystals cast at the “Office National d’Etudes et de Recherches Aéronautiques” (ONERA) with the withdrawal process, using <001> seeds. For solid solution homogenisation, the samples were heat-treated using the “standard” heat treatment of the ONERA as follows: 1300°C during 3 h, subsequently air-cooled, heat treated again at 1100°C during 4 h, air-cooled, then heated at 850°C during 24 h and air-cooled. As the experimental results analysed in this paper are almost identical for Re- and Ru-containing alloys, no distinction between these two alloys will be made in the following.

For TEM in situ tensile tests, microsamples were spark machined in order to obtain a tensile axis parallel to a $<100>$ direction. They were electropolished and observed in a JEOL 200 CX. Dynamic sequences were recorded in real time, digitized with the MASSRAM software.

2.2. Quantitative approach

We consider a pile-up under stress with $n$ parallel dislocations in a short-range ordered phase. Each dislocation $p$ experiences the applied force $b\tau_{s}$, where $b$ is the Burgers vector of the dislocation and $\tau_{s}$ the applied stress, the uniform alloy friction force $b\tau_{ss}$ and the total elastic interaction force $R_{p}$ are exerted on the dislocation $p$ due to the other dislocations defined by:

$$R_{p} = \sum_{i\neq p} F_{ip}$$

where $F_{ip}$ is the elastic force exerted by dislocation $i$ on dislocation $p$. The determination of the different $F_{ip}$ is based on the experimental measurements of the positions of the
dislocations observed in a thin foil. The calculation is done taking into account the effect of a free surface as detailed by Saada et al. [5].

As the glide of each successive dislocation $p$ modifies SRO, a planar defect is created behind it, as illustrated in Fig. 1. An energy $c_p$ is then associated with this defect. Such a concept has been introduced for the first time by Schwander et al. [4] with the “diffuse antiphase boundary energy” (DAB). If the energy $c_p$ differs from the DAB energy, we will use the term “$p$-SRO-energy”, or more generally the form “SRO-energy”.

Finally the equilibrium of the dislocation $p$ in a short-range ordered solid solution is obtained when the total force acting on it is zero:

$$F_p = b \tau_a - b \tau_{SS} + R_p - (\gamma_p - \gamma_{p-1}) = 0, \quad 2 = p = n$$  \hspace{1cm} (2)

Our experimental approach has allowed us to evaluate the quantity $(b \tau_a - b \tau_{SS})$, which is close to zero [5]. Thus, the $p$-SRO-energies can be determined using:

$$\gamma_p = \sum_{i=1}^{p} R_i \quad \text{with} \quad 1 \leq p \leq n$$  \hspace{1cm} (3)

These SRO-energies are of great interest because they are directly connected with the “strength of the SRO”. They give a quantitative description of the “obstacle” at the origin of the strength of the alloy. In the following, we will also focus on their determination (i) as a function of the dislocation position within the pile-up, (ii) as a function of the position of the pile-up within the thin foil, (iii) as a function of the temperature.

<table>
<thead>
<tr>
<th>$\gamma_1$ (mJ/m²)</th>
<th>$\gamma_2$ (mJ/m²)</th>
<th>$\gamma_3$ (mJ/m²)</th>
<th>$\gamma_4$ (mJ/m²)</th>
<th>$\gamma_5$ (mJ/m²)</th>
<th>$\gamma_6$ (mJ/m²)</th>
<th>$\gamma_7$ (mJ/m²)</th>
<th>$\gamma_8$ (mJ/m²)</th>
</tr>
</thead>
<tbody>
<tr>
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<td>5.6</td>
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<td>1.5</td>
<td>0.1</td>
<td>0.3</td>
<td>0.0</td>
</tr>
<tr>
<td>11.3</td>
<td>4.5</td>
<td>2.5</td>
<td>1.7</td>
<td>0.4</td>
<td>0.2</td>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td>27.4</td>
<td>8.7</td>
<td>4.0</td>
<td>2.9</td>
<td>0.5</td>
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<td>0.3</td>
<td>0.0</td>
</tr>
<tr>
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<tr>
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<td>3.0</td>
<td>1.5</td>
<td>0.1</td>
<td>0.3</td>
<td>0.0</td>
</tr>
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</table>

Table 1. Determination of the $p$-SRO energies when the pile-up imaged in Fig. 2 is propagating in a 2 μm zone. The different positions of the pile-up correspond to the 5 positions imaged respectively in Figs. 2a, b, c, d and e; $x_1$ refers to the position of the first dislocation.
3. Results

The propagation of dislocation pile-ups has been recorded during TEM in situ straining tests at 25 °C. Some images taken from a sequence are shown Fig. 2. The first images (Fig. 2a, b) were taken when the pile-up is propagating with a velocity around 25 nm/s. Then, the dislocations accelerate so that their velocity attains around 180 nm/s (Fig. 2c, d). Finally, the movement is so fast (Fig. 2e) that it becomes difficult to image the dislocations correctly. Such an experiment clearly shows two points: (i) the paired dislocations at the head of the pile-up indicates the presence of a defect between the two dislocations, that is attributed to the presence of SRO as analysed already [4, 6, 7], (ii) the non-uniform propagation of the dislocations indicates an heterogenous resistance of the solid solution.

Using the analysis of the pile-up detailed in the previous section, the SRO-energies have been determined for each dislocation and for the 15 positions (imaged in Fig. 2) of the pile-up during its propagation within the sample. In the results reported in Table 1, some fluctuations of the SRO-energies are evident. The magnitude of these fluctuations is more pronounced for the leading dislocation. The values of $\gamma_1$ and $\gamma_2$ are correlated and vary, increasing or decreasing, rather in the same way, while this effect is not significant for the other SRO-energies (see $\gamma_3$, for example). Then, from the third dislocation, the $\gamma_p$ values are in the range of 0–5 mJ/m$^2$, which indicates a very low SRO-degree. As the effect of SRO is most pronounced on the first dislocation, the fluctuations of $\gamma_1$ can be considered as the pertinent parameter of the SRO degree. The experimental case analysed in Fig. 2 can be described as a 2 μm zone of the sample where two hard areas (the first one corresponding to the position in Figs. 2a, b and c, the second one to the position in Fig. 2e) are separated by a soft area (position in Fig. 2d). Our previous results [8] have shown the same type of SRO variations for the other SRO-energies (see $\gamma_3$ for example). Then, from the third dislocation, the $\gamma_p$ values are in the range of 0–5 mJ/m$^2$, which indicates a very low SRO-degree. As the effect of SRO is most pronounced on the first dislocation, the fluctuations of $\gamma_1$ can be considered as the pertinent parameter of the SRO degree. The experimental case analysed in Fig. 2 can be described as a 2 μm zone of the sample where two hard areas (the first one corresponding to the position in Figs. 2a, b and c, the second one to the position in Fig. 2e) are separated by a soft area (position in Fig. 2d). Our previous results [8] have shown the same type of fluctuations for $\gamma_1$, with a clear periodicity of hard and soft areas extended over a few micrometers. The wavelength characterising the distribution of hard zones can be also estimated while dislocations are propagating over several micrometers during an in-situ test: here it is in the range of a micrometer.

The analysis of dislocation pile-ups has been carried out as a function of temperature. TEM in situ experiments have been carried out at 25 °C and 350 °C and TEM post mortem observations have been realised on samples deformed at 600 °C and 750 °C. For comparison, pile-ups with the same number of dislocations have been chosen. The different $p$-SRO-energies have been evaluated for pile-ups containing 2 dislocations (Fig. 3). Several points are noticeable:

(i) SRO-energies vary very similarly at 25 °C and 350 °C. This is characterised by high values for the two first SRO-energies ($\gamma_3$ around 30–35 mJ/m$^2$), then a decrease in the following SRO-energies which becomes close to zero after the passage of about 7 dislocations;

(ii) a clear decrease is observed for the first SRO-energy at 600 °C in comparison with those at 25 °C and 350 °C. At high temperature, $\gamma_1$ is of the same order of magnitude as other $\gamma_p$ values;

(iii) all $p$-SRO energies are low at 750 °C, suggesting a vanishing degree of SRO.

The dislocation distribution can be summarise as follows (see [7] for more details):

- At low temperatures (25 °C and 350 °C), dislocation pile-ups and paired leading dislocations have been observed.
- At 600 °C, the pile-ups are still present but the pairing disappears.
- At 750 °C, some pile-ups are still present and coexist with isolated dislocations.

Finally, a noticeable result is illustrated with the in situ sequence imaged on Fig. 4. Here a pile-up was nucleated during the loading (this step was not recorded). A to relaxing the stress, a reverse movement of the dislocation has been observed. Important is that the two trailing dislocations (marked by 1 and 2 in Fig. 4) propagate as a pair. Such a phenomenon of paired dislocations at the tail of the pile-up indicates a reverse movement of the pile-up. It has been observed several times and has been analysed in detail in [9]. It is worth noting that the velocity of the dislocations strongly increases at the end of the sequence (Fig. 4e, f), which is in good agreement with a mechanism of SRO reconstruction induced by the dislocations movement.

4. Discussion

The results presented above, allow us to propose a description of SRO. First, a qualitative description can be made based on the simple observation of the dislocation distribution. Because of the presence of the pile-up, we may attribute the high resistance at the origin of the piling-up to SRO. The existence of SRO has been confirmed by diffuse neutron scattering experiments [7]. As the first dislocation plays a major role in SRO destruction, this high strength must be correlated with a high value of $\gamma_1$. Then, as paired dislocations have been seen at the head of the pile-up, one may conclude roughly that only two dislocations are necessary to destroy most of SRO. In a first approximation the SRO could be then described, by an ordered zone extending over 2$b$ (b being the magnitude of the Burgers vector of the perfect $d/2<110>$ dislocation) which corresponds to approximately 0.5 nm. However, there are defects with significantly higher energies linking the first dislocations. These SRO-energies are associated with the disorder left by the propagation of the successive dislocations. A value of $\gamma_p$ close to zero indicates that SRO is no more present after the glide of $p$ dislocations. As a consequence, SRO can be described as a diffuse obstacle whose influence ranges over a distance $d$ given by

$$d = bp$$

where $b$ is the magnitude of the Burgers vector of a perfect dislocation and $p$ is the number of dislocations required to destroy SRO. Experimentally, we find that $\gamma_p$ is close to zero when $p$ is about 6–7. This means that SRO can be seen as a diffuse obstacle extended over 1.5 to 2 nm.

In addition, some noticeable fluctuations of the SRO-energies are observed, especially for the first two dislocations. This suggests the existence of alternative soft and hard areas. The quantitative analysis of several dislocation pile-ups reveals a mean distance between hard zones (or between soft zones) in the range of one micrometer. This has been confirmed by in situ atomic force microscopy (AFM) deformation tests on the investigated alloy, where the deformation was observed in (soft) areas distant from one (or some) to a few micrometers [8]. All these results allow a description of the SRO in concentrated solid solution, as a het-
Fig. 3. Determination of the $p$-SRO-energies for a pile-up of 9-dislocations for different temperatures: (a) 25 °C; (b) 350 °C; (c) 600 °C and (d) 750 °C.
Finally, the creation and destruction of SRO appear to be a completely reversible dislocation-assisted phenomenon. It is clear from the experiments that SRO is destroyed by moving dislocations but also that it is fully reconstructed when the dislocations are exactly moving back. Thus, the dislocations assist the destruction of SRO, and reciprocally, SRO favours the propagation of the dislocations when they are moving back.

5. Conclusion

Based on experimental results and a precise investigation of the dislocations observed by TEM, a description of SRO in concentrated solid solution is proposed. Because of the dimension of our "probe" (the mobile dislocations) the nanometer scale can be achieved. The SRO influence is extended over nanometric areas which are heterogeneously distributed at a micrometer scale. The dislocations assist the destruction of SRO, and SRO favours the dislocation propagation which induces its reconstruction. The analysis of the position of the dislocations allows to measure the variation of the SRO-energies within a pile-up. These values are of special interest in understanding the distribution of the dislocations (their pairing, the pile-up formation or their individual movement) and also to determine the possible strengthening effect due to SRO.

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References


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Correspondence address
Dr. Pettinari-Sturmel
CEMES/CNRS 29 rue Jeanne Marvig BP 94347
31055 Toulouse cedex 4 France
Tel.: +33 5 62 25 78 73
Fax: +33 5 62 25 79 99
E-mail: florence.pettinari@cemes.fr