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# Annealing behavior of the ODS nickel-based superalloy PM 1000

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## Abstract

PM 1000 is a powder-metallurgy (P/M) nickel-based superalloy containing about 1% (volume) of fine and uniformly dispersed incoherent particles in an austenitic matrix. In the present paper, we have investigated the annealing behavior of rods deformed by cold swaging to reductions of 24 and 44% followed by annealing in temperatures varying from 800 to 1350 °C. The microstructural changes were followed by channeling contrast in the scanning electron microscopy (SEM), electron backscattering diffraction (EBSD), and transmission electron microscopy (TEM). Results show that discontinuous recrystallization and extended recovery are responsible for the softening of this alloy. A few grains found preferentially at grain boundary regions and within deformation heterogeneities like shear bands are able to grow corresponding to a recrystallized volume fraction lower than 10%. These new grains are arranged in colonies having a significant amount of annealing twins with  $\Sigma$ 3-symmetry boundaries. The pinning effect on boundaries exerted by hard non-deformable particles (Zener drag) tends to suppress growth of most recrystallized grains. In the less deformed regions of the microstructure, a particle-stabilized subgrain structure is present and further softening is not significant even when longer annealing is performed.

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# 1. Introduction

Oxide dispersion strengthened (ODS) alloys are commonly used in high-temperature applications including the manufacture of devices for aerospace and glass processing industries, just to mention a few examples [1]. ODS alloys like PM 1000,<sup>1</sup> a nickel-based cold-workable superalloy, have high tensile and creep rupture strengths even at temperatures close to their melting point [2]. These ODS alloys are usually manufactured by mechanical alloying (a powder metallurgy route). Processing of these alloys involves high energy milling of elementary or pre-alloyed powders with stable oxides ( $Y_2O_3$ , for instance), followed by pressure-assisted consolidation at high temperatures [3]. The microstructure of the PM 1000 alloy consists of coarse elongated grains parallel to the longitudinal direction. A strong texture resulting from secondary recrystallization pro-

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cess is also present in this alloy [4]. This coarse-elongated microstructure is more creep-resistant than alloys with an equiaxed grain structure [5]. In this regard, recrystallization is not intended in ODS alloys and should be virtually suppressed to maximize creep resistance. The presence of fine and well-distributed incoherent particles in the nm-range exerts an appreciable pinning force on free dislocations and boundaries, especially those with low-angle character.

Most of the literature concerning the recrystallization in P/M ODS alloys refers to aspects of the primary recrystallization from the deformed structure (as-milled and as-extruded powders upon hot consolidation) and further secondary recrystallization to obtain a coarse microstructure [3,6,7]. Secondary recrystallization is usually carried out under a high temperature gradient (directional recrystallization) to promote the formation of elongated grains with a high grain aspect ratio (GAR) [8,9].

In the present paper we have investigated the annealing behavior of the PM 1000 alloy samples deformed by cold swaging. The combination of a coarse-grained structure (oligocrystalline material), a strong initial texture, and the presence of fine particles make this alloy a very interesting

<sup>&</sup>lt;sup>1</sup> PM 1000 is a trademark of Plansee AG, Reutte, Austria.

material for recrystallization studies. The microstructure of this alloy was characterized by scanning (SEM) and transmission electron microscopy (TEM) in the as-received condition, in the cold-worked state and after annealing at high temperatures. The microtexture of partially recrystallized samples was also investigated using the electron backscattering diffraction (EBSD) technique. The main results of this characterization will be presented and discussed.

# 2. Experimental

The nominal chemical composition of the nickel-based superalloy PM 1000 used in the present investigation was Ni–20Cr–3Fe–0.5Ti–0.3Al–0.6Y<sub>2</sub>O<sub>3</sub> (wt.%) [2]. The austenitic matrix consists of a Ni–Cr–Al–Fe–Ti solid solution. Cylindrical rods with 5.5 mm in diameter were cold swaged to 24 and 44% reductions (in area), respectively. These rods were cut from a plate using spark erosion in such a way that the longitudinal direction of rods was parallel with the rolling direction. After cold swaging the specimens were sealed in quartz glass and annealed in temperatures ranging from 800 to 1300 °C from 1 to 120 min. A few specimens were annealed for longer times and at higher temperatures (1350 °C).

The longitudinal sections were ground and polished using conventional techniques. Chemical etching of the polished sections was performed using an aqueous  $HNO_3$ –HF solution at room temperature. A chemical method has been chosen for the bulk extraction of the precipitates in the PM-1000 superalloy. The metallic matrix was dissolved in a Berzelius-type solution at room temperature. The extraction residue was cleaned in a 0.25 M hydrochloric acid wash.

The structural analysis of the residue was performed by X-ray diffraction using a Cu Ka radiation. Microhardness testing was performed on the transversal section of polished samples. The microstructures of as-received, deformed, and annealed specimens were observed on a VP-1450 model LEO scanning electron microscope (SEM) operating at 10 kV using backscattered electrons to reveal orientation contrast. Transmission electron microscopy was performed on a Philips CM 120 operating at 120 kV. Thinning down to electron transparency was carried out in 3 mm disks using a solution consisting of 15 parts of perchloric acid (HClO<sub>4</sub>) and 85 parts of anhydrous ethanol at 40 V and cooled to 5 °C. Orientation image microscopy (OIM) results and microtexture evaluation were determined by means of automatic indexing of Kikuchi patterns after suitable image processing in a TSL 3.3 system interfaced to a Philips XL-30 SEM operating at 30 kV with a conventional W-filament. EBSD sampling points were acquired automatically at steps ranging from 2 to 10 µm (step size), depending on the magnification and intended resolution level.

## 3. Results and discussion

#### 3.1. Starting material

The microstructure of the as-received rods is shown in Fig. 1a. The length of these elongated grains determined by the linear intercept method is about 2 mm (longitudinal direction). The grain aspect ratio is about 10. Grain boundaries display a serrated morphology as a result of particle-boundary interaction during the manufacturing process (secondary recrystallization). There is also a low



Fig. 1. Longitudinal section of PM 1000 alloy in the as-received condition: (a) light optical micrograph showing elongated grains; (b) pole figure from EBSD mapping. LD is the longitudinal direction in the pole figure. Arrow in the left lower part of this figure marks the longitudinal direction.





Fig. 2. Microstructure of PM 1000 alloy in the as-received condition: (a) TEM micrograph showing a large number of particles (bright field); (b) Size distribution based on 2300 particles.



Fig. 3. X-ray diffraction pattern of the isolate (precipitate extraction) showing the presence of  $\alpha$ -alumina and Y-Al-O compounds.

fraction of coarse pores at grain boundaries. These pores likely result from the manufacturing process of this alloy. Fig. 1b shows the pole figure for (001) reflections obtained from EBSD mapping. This material displays a clear (001)-fiber texture parallel to the rod longitudinal direction (LD). Many low-angle boundaries aligned parallel to the LD are found in the corresponding orientation image maps (not shown in this paper). The presence of a large amount of low-angle boundaries is expected in materials with pronounced texture.

TEM investigation of as-received samples shows that the mean particle size is about 16 nm. Fig. 2 shows a TEM micrograph where a large population of particles is present. In our investigation, particle size varies from 5 to 250 nm. The spherical morphology is predominant for the smaller particles; however, cuboidal particles (usually coarser) are also found. These coarse particles with cuboidal morphology were identified as  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> by EDS analyses in TEM. Similar results were reported elsewhere [10]. The result of the chemical analysis of the extracted precipitates reveals that the residue consists of a mixture of stable Y-Al-O compounds (Fig. 3). Peaks corresponding to individual  $Y_2O_3$  particles were not identified. In this figure one can note the presence of peaks corresponding to  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>, YAM  $(Al_2Y_4O_9)$ , YAG  $(Al_5Y_3O_{12})$ , and YA  $(AlYO_3)$ compounds. These compounds belong to the  $Al_2O_3-Y_2O_3$ system and were reported in other ODS superalloys [11]. The presence of yttrium aluminates instead of pure  $Y_2O_3$ was already reported for MA 754 alloy, an alloy with very similar microstructure as well as chemical composition [12]. These findings are also in accord with those reported for precipitates extracted from the ODS Fe-base MA 956 alloy [13]. These evidences suggest that most of the  $Y_2O_3$ particles react with aluminum available in solid solution during the manufacture of this alloy [7]. The possibility of partial dissolution of Y2O3 particles during chemical extraction of precipitates has to be also taken into account.

## 3.2. Deformed state

The presence of a fine dispersion of particles tends to homogenize the dislocation structure in cold worked materials [14]. In addition, alloys containing such a fine dispersion display a pronounced work hardening, even at low strains. The work hardening behavior of the PM 1000 alloy during cold swaging is shown in Fig. 4. In the present work strain was limited to 44%. Larger reductions led to cracking and fracture of the rods.

The cold-worked structure of PM 1000 is not completely homogeneous. Banded structures are found in the microstructure of the deformed samples for both reductions. The extent of banding appears to vary from grain to grain. Because of their high dislocation densities it was not possible to get sound TEM micrographs in cold swaged samples. The SEM micrograph shown in Fig. 5 corresponds to a region of sample deformed to 24% and annealed at 1000 °C



Fig. 4. Work hardening behavior of the PM 1000 alloy during cold swaging.



Fig. 5. SEM micrograph showing a banded structure in grain A between two non-banded grains B and C in a sample deformed to 24% followed by annealing at 1000  $^{\circ}$ C for 1 min.

for 1 min. Recrystallization did not occur for this annealing condition. Grain A is fully banded whereas the adjacent ones (grains B and C) are not. It is worth mentioning that it is not possible determining the rotations across these banded structures using EBSD data in the as-deformed state. Due to the large amount of stored dislocations, Kikuchi patterns are too diffuse and cannot be properly indexed. In a general manner, only recrystallized grains or strongly recovered regions can be mapped with an acceptable quality index. Fig. 6 shows a similar feature. Bands are present throughout the microstructure. In some cases, groups of intersecting bands are easily seen within grains. Shear bands crossing many grains are also found in the microstructure. They probably result from the inhomogeneous deformation imposed during cold swaging, a high-strain-rate process. The amount of deformation heterogeneities (banded structures) increases with strain. Shear bands are noticeable in many regions of deformed specimens (both reductions) and are favorable sites for recrystallization upon annealing. Evidences for such a feature will be presented in the following item.

## 3.3. Annealed state

## 3.3.1. Softening kinetics

Fig. 7 shows the softening kinetics curves for PM 1000 deformed at 24 and 44% and annealed in the range 800–1300 °C. Metallographic examination of annealed



Fig. 6. SEM micrograph showing grains with banded structures in a sample deformed to 24% followed by annealing at 1100 °C for 30 min.

samples reveals that the recrystallized fraction is lower than 10%. This fraction corresponds to grains large enough to be distinguished by LOM. The softening behavior expressed by the hardness versus time isothermal plots displays a classical behavior. It is noticeable that the material softens in a pronounced manner in the first 15 min; however, after 20 min hardness is leveled out remaining nearly unchanged for longer annealing times. For both reductions, the values of hardness in the plateau regime are higher than in the as-received condition. The amount of softening depends on the annealing temperature; however, this effect is more pronounced for the more deformed samples. The relative standard deviation (R.S.D.) of the hardness tests was about 5%.

A simple model based on the comparison between the driving pressure (elastic energy from stored dislocations) and the drag force associated to finely dispersed particles (Zener pinning) was used to validate the experimental results found in this investigation. It should be noted that this simple model assumes that the stored energy is homogeneously distributed in the material. It also assumes the presence of a narrow distribution of particles also equally spaced. Upon annealing, recovery reactions tend to lower the driving pressure for recrystallization. When a certain value is reached, Zener pinning prevails and recrystallization tends to be suppressed. The increase in dislocation density ( $\rho$ ) can be estimated from microhardness testing results using Eq. (1) [15]

$$\rho = \left(\frac{1}{3(AGb)}(H - H_{\rm i})\right)^2\tag{1}$$

where A is a constant ( $\approx 0.4$ ), G the shear modulus, b the Burgers vector length, H the hardness at a given annealing condition, and  $H_i$  is the hardness of the material in the as-received condition. We have adopted G = 150 GPa [2] for the  $\langle 0.01 \rangle$ -textured PM 1000 alloy, and  $b = 2.49 \times 10^{-10}$  m. The hardness of this alloy in the as-received condition was about 294  $\pm$  6 VHN. The changes on the driving pressure during isothermal annealing (*E*) can be determined at any



Fig. 7. Softening kinetics for ODS-PM-1000 deformed by cold swaging to the following reductions: (a) 24%; (b) 44%.



Fig. 8. Stored energy as a function of the annealing time for ODS-PM-1000 deformed by cold swaging to the following reductions: (a) 24%; (b) 44%. The dashed line marks the Zener pressure due to particle pinning.



Fig. 9. Details of recrystallized grains and annealing twins in several samples deformed to 44% reduction and annealed at: (a)  $1200 \degree C$  for 2 h; (b)  $1300 \degree C$  for 2 h; (c)  $1300 \degree C$  for only 1 min (SEM, BSE).

time by using the following equation [15]

$$E = \frac{Gb^2}{2}\rho \tag{2}$$

The drag force associated to Zener pinning  $(E_Z)$  is determined by using the well-known equation [16]:

$$E_Z = \frac{3f\gamma}{d} \tag{3}$$

where *f* is the volume fraction of particles,  $\gamma$  the grain boundary energy, and *d* is the particle diameter. In our calculations,  $\gamma = 866 \text{ mJ/m}^2$  [17] corresponding to pure nickel,  $f \approx 0.01$  [18], and  $d \approx 16 \text{ nm}$ . The occurrence of particle coarsening was not evidenced in this investigation. It should be noted that the size distribution of samples annealed at 1300 °C for 8 h did not differ too much compared to the as-received condition (17 and 16 nm, respectively). For practical purposes, we assume that particle size remains nearly unaffected during annealing, at least for the experimental conditions (time and temperature) employed during this investigation.

The dislocation density in the as-deformed samples was calculated for 24 and 44% reductions, being equal to  $4.9 \times 10^{14}$  and  $7.2 \times 10^{14}$  m<sup>-2</sup>, respectively. From these values,  $E_0$  is equal to 2.3 and 3.3 MJ/m<sup>3</sup>, respectively. During annealing the initial driving pressure is lowered to values close to  $E_{\infty}$  (residual stored energy of the fully recovered material). When the driving pressure for recrystallization *E* equals  $E_Z$ , recrystallization stops and Zener pinning prevails resulting in a partially recrystallized structure. The driving force for recrystallization is plotted as a function of time for several annealing temperatures (Fig. 8). It can be seen from it that Zener pinning overcomes the driving pressures for recrystallized structures for recrystallized structures.

tallization at very short times. For instance, for 24% deformed samples only 2 min are necessary to lower E and stop recrystallization. For 44% deformed samples, this time increases to about 8 min, depending on the annealing temperature. These findings are in agreement with our quantitative metallographic data. The recrystallized volume fractions in samples annealed for 5 min do not differ too much to those annealed for longer times.

#### 3.3.2. Microstructural characterization

The large recrystallized grains are found mainly at some grain boundary regions and also at deformation heterogeneities. Fig. 9 depicts examples of preferential recrystallization along grain boundaries. The large curvature associated to these regions and the higher amount of stored dislocations might explain why recrystallization is favored at grain boundaries and virtually suppressed within grains. Annealing twins are found within most of the recrystallized grains. The black spots observed within these new grains result from the enlargement of pores due to chemical etching. Fig. 10 shows a SEM micrograph where recrystallization occurs preferentially within shear bands. The new grains are not elongated as commonly reported for other MA–ODS superalloys [19].

Channelling contrast allows the identification of details of the microstructure. It consists of subgrains with irregular morphology. A close inspection shows that there are clear changes in orientation from one region to another. The shape of the subgrains depends on the extent of pinning exerted by particles, as clearly shown in Fig. 11. In this TEM micrograph, pinning of low-angle boundaries by arrays of particles is evident. The dislocation density inside these subgrains is



Fig. 10. Recrystallized grains within a set of parallel shear bands in a sample deformed to 44% and annealed at 1200 °C for 10 min: (a) general view of the banded structure crossing many grains (LOM, Nomarski contrast); (b) mirrored image of the central region showing details of the surrounding substructure (SEM, BSE).



Fig. 11. TEM micrographs (bright field) showing details of the particle-boundary interaction in a sample deformed to 44% and annealed at 1300 °C for 8 h: (a) general view of the substructure; (b) heterogeneity in terms of morphology and size of subgrains in another region of the same sample.

reduced as a result of recovery. It was not possible to resolve details of the dislocation structure in the subgrain boundaries. At a first sight, they appear to be less organized than the boundary structures commonly found in pure high-SFE metals.

Despite of its low volume fraction, a few recrystallized grains could be found in the microstructure, as shown in



(a)



Fig. 12. TEM micrographs (bright field) showing recrystallized grains and surrounding structures in a sample deformed to 44% and annealed at 1350 °C for 4 h.

Fig. 12. We did not succeed on finding any large recrystallized grains during the TEM investigation. A good example showing the early stages of primary recrystallization in the PM 1000 alloy is given in Fig. 12a. Dislocation-free volumes limited by high-angle boundaries are present in this micrograph. Within this new grain, a coarse particle surrounded by smaller ones is also noticeable. Due to the small size of the individual particles, particle-stimulated nucleation (PSN), promoted by individual particles, even by the coarser ones (the larger particles found had about 200 nm diameter), seems to be unlikely to occur in this alloy. We did not perform any extensive TEM investigation to evaluate the distribution of particles across the sample; however, particle clustering is evident in many regions. In some circumstances, particle clusters may act as potential sites for PSN depending on their sizes. This might be an interesting point to be investigated in a future work.



Fig. 13. EBSD mapping of a sample deformed to 44% and annealed at 1300 °C for 30 min: (a) OIM showing recrystallized grains and deformation heterogeneities in neighboring grains; (b) inverse pole figure corresponding to the recrystallized grains (lower part of the figure). Longitudinal direction is parallel to the scale bar.



Fig. 14. EBSD mapping of a sample deformed to 44% and annealed at 1300 °C for 1 h: (a) general view of the microstructure (SEM-BSE); (b) OIM showing a strip of recrystallized grains; (c) inverse pole figure corresponding to the recrystallized grains. Longitudinal direction is perpendicular to the scale bar.

In this regard, why do some large grains evolve in the microstructure giving rise to a partially recrystallized structure? The driving pressure at deformation heterogeneities and grain boundary regions is much higher than the one found in the homogeneously strained material. Large curvatures are also associated with these regions, in particular at grain boundaries. Moreover, the extent of pinning also depends on the homogeneity of particle distribution. If particles are not randomly distributed in the material (local inhomogeneities are feasible), pinning pressure varies along the boundary [16]. Hence, some subgrains might gain the necessary mobility to migrate, easing effective nucleation. In the present work there are good evidences that only a few subgrains have enough mobility to grow towards the substructure as annealing proceeds. In this case, particle pinning is ineffective to avoid the boundary breakaway of some of these new grains. This is in agreement with the findings reported in this paper regarding the preferential recrystallization in regions where large curvatures and larger stored energies are present.

The behavior exhibited by PM 1000 during annealing is a good example of the competition between recovery and recrystallization. Recovery and recrystallization are concurrent events both driven by stored energy in the deformed state. During the first 15 min softening is very pronounced. This result can be interpreted in terms of the occurrence of recrystallization in areas with higher stored energies like grain boundary regions and deformation heterogeneities. This is a typical example of recrystallization with site-saturated nucleation. Further growth of most of these new grains is impeded by particle drag. The growth rate of these new grains tends to zero with time since the driving force for recrystallization is continuously reduced by recovery.

In less deformed regions, discontinuous recrystallization is virtually absent and a particle-stabilized subgrain structure is predominant in the microstructure even for longer annealing times. This is a characteristic feature of extended recovery.

## 3.3.3. EBSD mappings

The microstructural characterization of annealed samples using EBSD revealed many interesting aspects concerning the recrystallization of this alloy. Contrasting with the results provided by TEM investigation, large areas containing recrystallized grains could be mapped by EBSD. There are two important remarks concerning recrystallization in



Fig. 15. EBSD mapping of a sample deformed to 44% and annealed at 1300 °C for 8 h: (a) general view of the microstructure (SEM-BSE); (b) OIM showing a strip of recrystallized grains; (c) inverse pole figure corresponding to the recrystallized grains. Longitudinal direction is perpendicular to the scale bar.

this PM 1000 superalloy. As a general trend, a strong local texture is found in most of the recrystallized areas. The recrystallization microtexture associated to these regions is far from the possibility of being considered random.

Moreover, it must be highlighted that not all grain boundaries are preferential sites for recrystallization. This likely has to do with the nature of the boundary distribution (mesotexture) found in the initial condition. Further work is necessary to clarify this point. Fig. 13 brings an EBSD mapping in a sample deformed to 44% and annealed at 1300 °C for 30 min. The lower part of this OIM image shows recrystallized grains with a strong [411] fiber texture. They were nucleated in a former [001]-oriented grain. Two coarse recovered grains (upper part) could be also mapped. A family of parallel bands is visible within both grains indicating the presence of deformation heterogeneities. For this particular annealing condition, they did not influence recrystallization since new grains were not nucleated at these structures. The misorientation associated to these bands was found to be lower than 10°. Non-indexed points were purposely left in the OIM image. The majority of the mapped points could be indexed because of the positive effect of recovery on sharpening of Kikuchi patterns.

In another OIM, a narrow strip of recrystallized grains was identified along a grain boundary (see Fig. 14). The surrounding substructure was subtracted in this OIM image. These grains alter [2 2 7] and [0 1 1] orientations. Two other grains, present in the left lower part of the OIM image, but not belonging to the strip, are [5 3 6]- and [2 1 4]-oriented. This morphology is representative of partially recrystallized samples.

Groups of annealing twins were successfully mapped in a sample deformed to 44% and annealed at 1300 °C for 8 h, as shown in Fig. 15. Most of the twins are bounded by low-energy coherent  $\Sigma$ -3 boundaries. Annealing twins are commonly found during the recrystallization of face-centered cubic metals of intermediate-to-low stacking fault energy (SFE). Some of these twins appear to be originated during primary recrystallization; however, some of them could have their origin during grain growth. Further experimental evidence (in this work) is needed to make this point clear.

## 4. Conclusions

Based on the microstructural evidences presented in this paper, the following conclusions are drawn:

- (a) The original  $Y_2O_3$  particles react during high-temperature processing of the PM 1000 alloy. Results of chemical extraction of precipitates followed by X-ray diffraction confirm the presence of yttrium aluminates and  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> instead of pure  $Y_2O_3$  in the residue.
- (b) Results of TEM investigation confirm that particles are very effective to prevent growth of subgrains and recrys-

tallized grains during annealing of this alloy. Growth of new grains is also hindered by concurrent recovery. Particle coarsening was not evidenced, at least in the annealing conditions used in this investigation.

(c) The annealing behavior of the PM 1000 superalloy is very complex. Discontinuous recrystallization and extended recovery are responsible for the softening of this alloy. Only a few grains overcome pinning effects and are able grow in the microstructure. They represent about  $X_{RX} \approx 10\%$  in volume and are found mostly at grain boundary regions and within deformation heterogeneities like shear bands. This behavior is explained mainly to the presence of larger curvatures as well as larger stored energies found at these regions. Nevertheless, in the less deformed regions of the microstructure, a particle-stabilized subgrain structure (extended recovery) is also evidenced.

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