INFLUENCE OF DEFORMATION ON RECRYSTALLISATION OF A YTTRIA OXIDE
DISPERSION-STRENGTHENED IRON ALLOY (PM2000)

C. Capdevila^{*} and H. K. D. H. Bhadeshia

University of Cambridge

Department of Materials Science and Metallurgy, Pembroke Street, Cambridge CB2 3QZ, UK,

www.msm.cam.ac.uk/phase-trans

ABSTRACT

Mechanically alloyed and compacted dispersion-strengthened iron-base alloys are materials which have been severely deformed, though uniformly deformed. However, these materials show a strange behaviour, that the recrystallisation temperature drops when the compacted sample is cold deformed. Controlled experiments have been conducted on PM2000, which is an yttria oxide dispersion-strengthened iron alloy. This involved the study of grain structure evolution in samples which were systematically bent and then heat-treated. A bend test introduces a strain gradient and compressive or tensile deformation about the neutral axis. It has therefore been possible to characterise the recrystallisation behaviour in both tension and compression in a single test, with varying levels of strain. The results imply anything that introduces heterogeneity into the microstructure stimulates recrystallisation with large effects on the evolution of the grain microstructure.

^{*} C. Capdevila is currently at the National Centre for Metallurgical Research (CENIM-CSIC), Department of Physical Metallurgy, Avda. Gregorio del Amo, 8, Madrid, E-28040, Spain (E-mail: ccm@cenim.csic.es).

INTRODUCTION

An alloy can be created without melting, by violently deforming mixtures of different powders [1]. Inert oxides can, using this technique, be introduced uniformly into the microstructure. The dispersion-strengthened alloyed powders are then consolidated using hot-isostatic pressing and extrusion, to produce a solid with a very fine grain structure. Heat treatment then induces recrystallisation, either into a coarse columnar grain structure or into a fine, equiaxed set of grains. Columnar grains occur for two reasons: the oxide particles tend to become aligned along the extrusion direction, making that a favoured growth direction. Alternatively, and in the absence of particle alignment, columnar growth can be stimulated by recrystallising in a temperature gradient; the latter may be a stationary gradient or one which moves along the sample, as in zone annealing. The columnar microstructure is desirable in applications where the resistance to creep deformation is paramount. [2]

The ferritic oxide dispersion-strengthened alloy PM2000 is manufactured using the mechanical alloying process [3-5]. The development of a coarse grained microstructure during the recrystallisation of PM2000, has been noted and discussed by a number of authors, [6-8] but the mechanism of grain control remains uncertain. Recent work on MA957, another mechanically alloyed ODS ferritic steel, has emphasised the large influence of non-uniformities on the development of the recrystallised microstructure [9-11]. Thus, it is argued that anything which introduces a heterogeneity into the microstructure stimulates recrystallisation. This has the effect of greatly reducing the recrystallisation temperatures and of enhancing the nucleation of recrystallisation thereby giving an undesirable, fine-grained microstructure with poor creep properties. The heterogeneity can be introduced by having a non-uniform starting microstructure or by introducing a non-uniform plastic strain in the sample.

The purpose of the present work was to study the latter effect. PM2000 is sometimes used in tube form; the process by which the tubes are manufactured can introduce non-uniform plastic strains,

which may radically alter the recrystallised microstructure. It is therefore important to understand the influence of strain heterogeneities in determining both the scale and anisotropy of the recrystallised microstructure.

EXPERIMENTAL PROCEDURE

The nominal composition of the alloy PM2000 used in this work is shown in Table 1. The alloy was supplied by PLANSEE GmbH. The essential feature of PM2000 is that it contains 5.5 wt % of Al and 0.5 wt % of Y_2O_3 . The aluminium enhances corrosion and oxidation resistance and it is claimed that PM2000 is better than other ODS in gaseous environments containing SO_2 0.24%, CO_2 15%, O_2 4%, N_2 to balance [12,13]. The creep performance has been found to be optimum with a Y_2O_3 content of 0.5 wt %.

Bend tests were employed to evaluate the role of deformation on subsequent recrystallisation of as-received PM2000 in the form of extruded tubes with 4.7 mm wall thickness. The bend samples were machined into bars, each of 100 mm length, parallel to the extrusion direction, and with a rectangular in cross-section of 7 mm width (w) and 4 mm thickness (h). Guided-bend tests were carried out using a MAYES-100 kN bend machine. The axis of the bend was oriented at 90° to the direction of extrusion. The specimen was placed over two rounded supports separated by a clearance (L) equal to 40 mm. The specimen was bent by applying a force through a plunger in contact with the specimen at the mid-length between supports.

Optical microscopy was used to observe the microstructures after etching using a mixture of 2 g $CuCl_2$, 40 ml HCl , and 40-80 ml ethanol. Metallographic examination in longitudinal and cross sections taken along the centre line of the specimens were carried out. A Jeol JSM6500 Field Emission Gun Scanning Electron Microscope (FEG-SEM) has been used to reveal the fine grain structures.

RESULTS AND DISCUSSION

The work presented here is inspired by Regle and Alamo [11], who studied the recrystallisation behaviour of MA956 and MA957, for samples which were cold deformed after extrusion. Two deformation processes were used, swaging and drawing, with reductions ranging from 10-60%. Swaging and drawing led to quite different changes in the recrystallisation behaviour. In all cases, deformation led to a reduction in the recrystallisation temperature, the change being largest for the cold-drawn samples.

In the present work, PM2000 samples that were cold-deformed by bending after extrusion. It is important to point out that the material is already in a severely deformed state, though uniformly deformed, before the bending. Bend test samples are ideal for simulating deformation gradients. Table 2 and Fig. 1 summarise the bend-test parameters for the two different levels of deformation implemented (B1 and B2).

The following analysis [14] deals with the problem of bending strains in a bar assuming plane-strain conditions, which probably is a good approximation for the central portion of the sample, where there are no large changes in width. The radial strains are given by

$$\varepsilon_r = \frac{R}{4} \left(1 - \frac{R^2 - (h/2)^2}{r^2} \right) \quad (1)$$

where R is the internal radius of curvature and h is the thickness of the sample. The radial strains are plotted in Fig. 2 using the input data listed in Table 2. For bending the strain passes through zero halfway through the thickness of the sample at the neutral axis, which is consistent with the plot presented in Fig. 2. Likewise, the strain *gradient* in B2 sample is clearly much higher than that of B1.

Recrystallisation in iron-base ODS alloys occurs at exceptionally high temperatures, of the order of 0.9 of the melting temperature (in PM2000 the melting temperature T_m is 1756 K). Recrystallisation in such alloys nucleates by the bowing of grain boundaries. With the sub-micrometer grain size of mechanically alloyed metals, the grain junctions themselves act as severe pinning lines for grain boundary bowing [15]. The activation energy for the nucleation of recrystallisation is then very large so the few successful nuclei then give the very coarse-grained recrystallised microstructure. Deformation gradients assist nucleation by favouring some grains over others; the resulting increase in the nucleation rate leads to a finer recrystallised grain size together with a reduction in the recrystallisation temperature, as shown in Figs. 3 to 5. Whereas recrystallisation does not occur after heat treatment at 1295 °C for 1 h in the unbent sample (Fig. 3), B1 and B2 clearly show recrystallisation in tensile and compression areas of the samples (Fig. 4).

Figure 5 shows the cross section of the B1 and B2 samples. It is clear that the centre of the sample remains unrecrystallised since it corresponds to the neutral axis. Therefore, it is concluded from Figs. 3 to 5 that deformation clearly decreases the minimum temperature at which recrystallisation begins. Moreover, it is clear from the comparison between Figs. 4(a) and 5(a), and Figs. 4(b) and 5(b) the unrecrystallised area of the B2 sample is closer to the compression region than that of the B1 sample. This could indicate that the neutral axis approximate to the inner surface of the sample as bending becomes more severe. This is consistent with the bending theory where it is indicated that beyond the elastic limit the neutral axis moves closer to the inside surface of the bend as the bending proceeds.

The grain structures in the tensile and compression areas of the bend samples are presented in detail in Figs. 6 and 7. The larger strain gradient of B2 leads to more a equiaxed and smaller recrystallised grain structure. The grains in the compressed inner regions (Fig.6) are more equiaxed and refined than those in the tensile regions (Fig. 7), which is consistent with strain gradient analysis shown in Fig. 2. Likewise, the recrystallised grains in the compression and the tension areas of B2 (Fig.6(b) and Fig. 7(b)) are finer than those in B1 (Fig.6(a) and Fig. 7(a)). These results are consistent with

the idea that anything which introduces heterogeneity into the microstructure stimulates recrystallisation [9, 10].

Since the grains in B2 sample becomes very fine and they can not be properly discriminate with optical micrographs techniques, Fig. 8 shows FEG-SEM micrographs of the compressed inner, centre and tensile outer parts of the B2 sample. The comparison between Figs. 4 to 8 evidence the extreme variation on grain size due to the strain gradients introduced in the sample by bending. Likewise, Fig. 8(b) shows the grain morphology in the neutral-axis region of the sample. It is clear that although not recrystallisation has been produced, the grains are deformed resulting in an elongated grain structure as compared with the microstructure showed in Fig. 3(b).

From these results it could be concluded that non-uniform deformation promotes the nucleation of recrystallisation, and consequently leads to a finer grain microstructure in PM2000. However, PM2000 is designed for high temperature applications. High-temperature strength is enhanced by the development of a coarse-grain microstructure with a high aspect ratio. Therefore, more homogeneous deformation has to be produced in order to reduce nucleation, and then promote coarse grain structure.

The tensile properties of PM2000 have been analysed using the neural network model created by Badmos et al., [16] which has as inputs virtually all the parameters that are known to affect the strength. This includes the detailed chemical composition, any recrystallisation or ageing heat-treatment, the extent of cold work, the test temperature, and the strain rate. Figure 9 shows the evolution of the yield strength with temperature for as-flow formed material. The solid line indicates the evolution of yield strength with temperature, meanwhile the upper and lower dashed lines indicates the range of uncertainty (error bars) of the predicted yield strength. There is a significant decrease in the yield strength at and beyond about 500 °C. Therefore, bending at 500 °C might induce more homogeneous deformation instead of that induced at room temperature where the yield strength is much higher. Figure 10 shows the microstructure obtained after recrystallisation heat treatment at 1295 °C for 1 h in the compressed inner and tensile outer regions

of the B2 sample but bent at 500 °C instead of at room temperature. A notable difference in the grain structure as compare with that for room temperature is observed (Figs. 6(b) and 7(b)). A more homogeneous and coarse grain structure is obtained.

CONCLUSIONS

Bearing in mind that PM2000 has been severely deformed, though uniformly deformed, during manufacturing processes, the effect of cold deformation on the recrystallisation of PM2000 samples is two fold. Firstly, the recrystallisation temperature decreases, consistent with the hypothesis that anything that makes the original microstructure heterogeneous will encourage recrystallisation. This is because the microstructure prior to recrystallisation is relatively uniform with grains that are so fine their junctions are powerful pinning points. The second effect is that the increase in the number and density of recrystallisation nuclei, due to (non-uniform) cold deformation, leads to fine grain structures, which are also more isotropic in three dimensions.

If coarse, columnar grain structures are desirable in the context of creep strength, then the present work indicates that the processing of PM2000, or similar materials in order to produce tubes, should avoid plastic strain gradients. Indeed, it is predicted that coarse, directional grain structures are only expected when the deformation following consolidation is either zero or very large, since both cases lead to a uniform distribution of plastic strain.

ACKNOWLEDGEMENTS

The authors are grateful to Professor A. H. Windle for the provision of laboratory facilities at the University of Cambridge and the European Commission for funding the work via a BRITE-EURAM III project. It is a pleasure to acknowledge our project partners: Plansee GmbH, Metall-

Spezialrohr GmbH (MSR), Sydkraft Konsult, Risø, Mitsui Babcock Technology Centre and Liverpool University.

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TABLE 1
Chemical composition (wt %).

Cr	Ti	Y ₂ O ₃	Al	Fe
20	0.5	0.5	5.5	Bal.

TABLE 2
Bend test parameters.

	Displacement (m)	Maximum Load (N)	R* (m)
B1	10.9×10^{-3}	4.03×10^3	11.5×10^{-3}
B2	18.1×10^{-3}	4.33×10^3	6.0×10^{-3}

*R is the radius of curvature at the internal surface

Figure 1. Bend test samples with different levels of deformation.

Figure 2. Radial strains into the bend sample at room temperature

Figure 3. Longitudinal section of unbent sample annealed at 1295 °C for 1h (a) Optical micrograph, and (b) TEM micrograph.

Figure 4. Longitudinal section of (a) B1 and (b) B2 bend samples recrystallised at 1295 °C for 1 h. The layer of light-etching material along the centreline is unrecrystallised.

Figure 5. Cross section of (a) B1 and (b) B2 bend samples recrystallised at 1295 °C for 1 h. The layer of light-etching material along the centreline is unrecrystallised.

Figure 6. Details of compression regions of a longitudinal section of (a) B1 and (b) B2 bend samples recrystallised at 1295 °C for 1 h. The area inside the square is enlarged on the right.

Figure 7. Details of tensile regions of a longitudinal section of (a) B1 and (b) B2 bend samples recrystallised at 1295 °C for 1 h. The area inside the square is enlarged on the right.

Figure 8. Details of the secondary electron images (SEI) obtained from the (a) compression, (b) centre, and (c) tensile regions of the B2 sample.

Figure 9. Effect of the temperature on the yield strength (YS) of PM2000 calculated using the method of Badmos et al. [16]

Figure 10. Microstructure of the (a) compression, and (b) tensile region in a longitudinal section of a bend sample deformed at 500 °C.

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