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Strain heterogeneity and the production of coarse grains in mechanically alloyed iron-based PM2000 alloy

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Abstract

Mechanically alloyed iron-based ODS alloys have the potential for application in heat exchangers for biomass processing, with gas operating temperatures and pressures of approximately 1100°C and 15–30 bar. The yttria dispersion in such alloys improves the high-temperature creep and stress rupture life. The elevated temperature strength is enhanced by the development of a coarse-grained microstructure during recrystallisation. Factors controlling the evolution of this desirable microstructure are explored in this work, focusing specifically on PM2000, which is a yttria dispersion strengthened, mechanically alloyed material. The microstructure following mechanical alloying and consolidation is fine and uniform, making it difficult to nucleate recrystallisation. Therefore, the introduction of a strain heterogeneity in the microstructure is found to promote the nucleation of recrystallisation. In contrast, uniform deformation reduces the chances of nucleation and hence leads to the development of a coarser microstrutructure.

Keywords: Cold deformation; Yttria particles; Superalloys; Directional recrystallisation

1. Introduction

Coarse grained, Fe-based mechanical alloys which are oxide dispersion strengthened (MA-ODS) offer good combinations of creep and oxidation resistance in the context of tubes used to construct heat exchangers in equipment exploiting biomass for power generation.

PM2000 is an iron-based superalloy designed for power plant applications, strengthened with yttria particles [1]. It is manufactured using mechanical alloying which is a process in which mixtures of elemental or master-alloy powders are subjected to high-energy milling. This causes the deformation, fragmention and rewelding of the powders until they mix intimately to form true solid solutions. Inert oxides such as yttria may be added during milling to form a stable dispersion which helps resist deformation during service [2]. The alloyed powder is then canned and hot-extruded; the microstructure at this stage consists of fine ($\cong 0.5$ μm) equiaxed grains of ferrite [3]. Subsequent annealing at exceptionally high temperatures, of the order of 0.9 of the absolute melting temperature, causes recrystallisation into grains which are columnar and aligned along the extrusion direction, giving excellent creep strength along the tube axis [4,5]. The columnar growth is primarily a consequence of the alignment of oxide particles along the extrusion direction.

Recrystallisation in commercial MA-ODS alloys occurs at very high temperatures, close to melting, in spite of their large stored energies [6–8]. This is unlike conventional metallic alloys which recrystallise at much lower homologous temperatures [9]. It could be argued that recrystallisation is inhibited by the fine yttria dispersoids. However, this is inconsistent with experimental observations which indicate an insensitivity of the recrystallisation temperature to variations in the overall pinning force [10]. Indeed, the limiting grain size in the presence of particles is far greater than the starting grain size.

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The anomalous recrystallisation behaviour of MA-ODS metallic alloys have been explained in terms of the nucleation stage. Recrystallisation in these alloys nucleates by the classic mechanism involving the bowing of an existing grain boundary. This process is straightforward in normal deformed metals because the size of the boundary perturbation is small when compared with the grain size (Fig. 1). However, for fine grained microstructures such as those found in the consolidated MA-ODS alloys, the boundary junctions themselves act as severe pinning lines restricting the bowing process; small grains can no longer be regarded as being topologically independent. This leads to an enormous activation energy for nucleation which is much larger than the activation energy for self-diffusion [11]. It is for this reason that the MA-ODS alloys recrystallise at exceptionally high temperatures [18,19]. Naturally, any non-uniformity in the microstructure, such as the occasional grain which is larger than the critical boundary perturbation, will make for easier nucleation. Regle and Alamo have shown that heterogeneous deformation of the consolidated product can have the same effect, leading to dramatic reductions in the recrystallisation temperature [12]. This is presumably because of the introduction of strain gradients which create non-uniform microstructures. The purpose of the present study was to analyse the effect of a particular kind of technologically important torsional cold-deformation on the nucleation and growth of recrystallised grains in a commercial MA-ODS iron alloy designated PM2000. The nature of the deformation is determined by a need to control the growth orientation of the recrystallised grains.

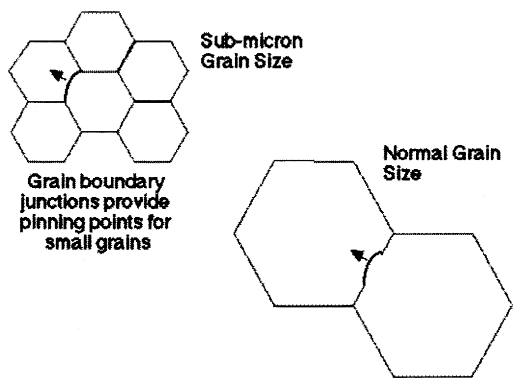

Fig. 1. The nucleation of recrystallisation occurs by the formation of a grain boundary bulge. This can occur with less constraint when the grain junctions are spaced at distances greater than the critical bulge size. With the ultra-fine grains of mechanically alloyed metals, the grain junctions are themselves pinning points, making it very difficult to form large enough bulges.

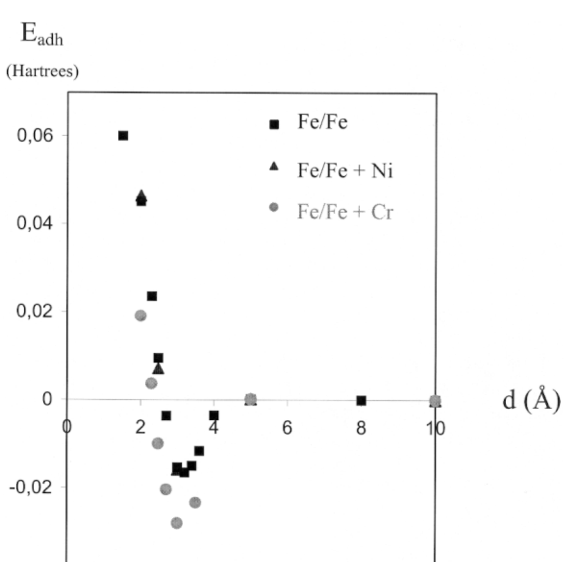

Fig. 2. Diagram illustrating the sections on which metallographic examination was carried out.

2. Experimental procedure

Tubes of PM2000 (Fe–20Cr–5.5Al–0.5Y_2O_3–0.5 Ti wt.%) were 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 and chromium improve the corrosion and oxidation resistance, making PM2000 better than other ODS alloys in gaseous environments containing SO_2 [13,14]. The creep performance has been found to be optimum with a Y_2O_3 content of 0.5 wt.%.

PM 2000 tubing is usually processed by unidirectional extrusion followed by heat treatment. This results in tubes with anisotropic, very coarse grained, axially aligned microstructures which exhibit excellent axial creep properties. However, when pressurised, the maximum principal stress occurs in the hoop direction of the tube; axially aligned columnar grains do not therefore provide adequate creep strength in the hoop direction [15]. To overcome this problem, a novel processing route has been developed at MSR Metall-Spezialrohr GmbH. In this, the extruded tubes are flow formed in a process of torsional extrusion, which leads to a reduction in wall thickness from 4.5 mm to 2.3 mm. Flow forming is a process in which the tube is subjected to compressive stress between a mandrel located inside the tube, and three work-rollers located symmetrically around its circumference. The design of the rollers is such that the tube not only extends but also twists since the total deformation is a combination of torsion and extrusion.

It is found the yttria particles tend to align along the extrusion direction [16,17]; torsional deformation causes this alignment to become helical in three dimensions. Such a materials should therefore recrystallised into columnar grains which twist along the extrusion

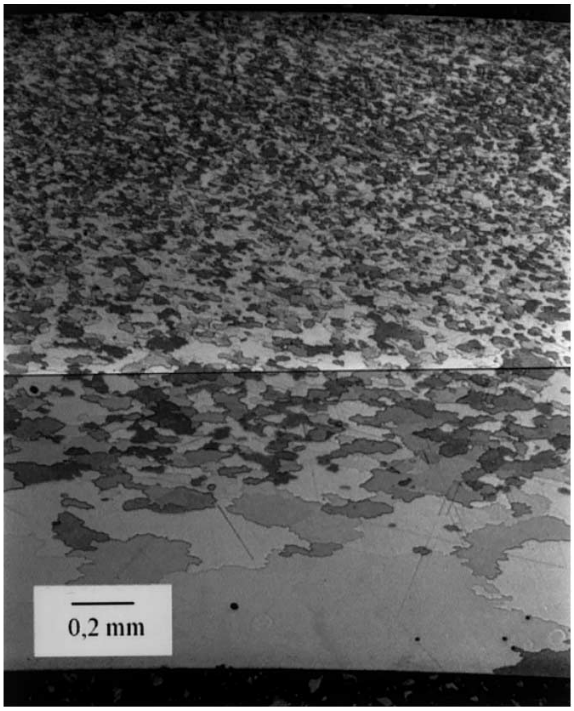

Fig. 3. Cross-section of tube R after subsequent recrystallisation at 1380°C for 1 h.

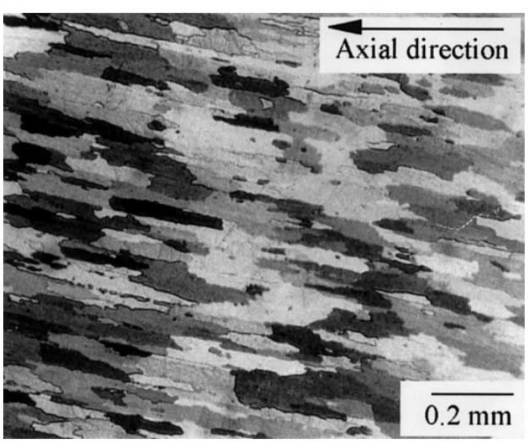

Fig. 4. Flat section of tube R after subsequent recrystallisation at 1380°C for 1 h.

axis and hence lead to better creep-resistance along the hoop direction.

Two types of samples were produced, one in which the flow forming was conducted at room temperature (tube R) and the other in which the flow-forming was carried out at approximately 600°C (tube W). The reasons for these two treatments will be explained later in the text. Samples for metallographic examination were cut from the tubes as it is schematically shown in Fig. 2. Optical microscopy was used to observe the microstructures of heat treated specimens. The etchant used was 2 g $CuCl_2$, 40 ml HCl, and 40–80 ml ethanol.

3. Results and discussion

A variety of isothermal heat treatment experiments were conducted to study the recrystallisation behaviour of the flow-formed tubes. The minimum recrystallisation temperature (T_R) is defined as the minimum temperature at which optical microscopy indicates some signs of recrystallisation following 1 h of heat treatment. Values of these temperatures, for the inner and outer surfaces, were found to be 975 and 750°C, respectively, for tube R in the flow-formed condition.

Fig. 3 shows the microstructure of tube R after a recrystallisation heat treatment at 1380°C for 1 h. The outer microstructure consists of sheets of grains which have some tendency to helical alignment (Fig. 4), with the thin dimension along the radius of the tube. The inner microstructure comprises essentially coarse grains which from a creep point of view represent a better microstructure than that of the outer surface.

As stated in Section 1, the grain junctions in the fine-grained mechanically alloyed and consolidated material act as severe pinning lines hindering grain boundary bowing [18]. The activation energy for the nucleation of recrystallisation thus becomes very large, giving the high recrystallisation temperatures characteristic of this material. However, recrystallisation can be stimulated to occur at much lower temperatures if a few grains happen to be slightly larger, i.e. if the grains are not uniform in size [19,20], or if there are significant local strain heterogeneities which assist nucleation.

During flow-forming, the surface of the tube is in contact with the rollers whereas the interior is in contact with a stationary mandrel. The torsional influence of the rollers does not therefore penetrate effectively through the thickness of the tube when reduction in area is small. Tube R was flow-formed at ambient temperature, leaving the tube surface more intensely deformed than the tube interior. The outer region should therefore find it easier to recrystallise. This is confirmed by Fig. 3 which shows that the outer part of the tube has fine grains with small grain aspect ratios (GAR), whereas the inner part of the tube is coarse-grained.

Since coarse grains are desired for elevated temperature applications, it was argued that a flow-forming operation should be designed which leads to a more uniform distribution of strain through the cross-section of the tube. To prove this, the strength of PM2000 was estimated using the model created by Badmos et al. [21], as a function of the chemical composition, heat-treatment, the extent of cold work, temperature, and the strain rate. Fig. 5 shows how the yield strength changes with temperature for alloys prior to the recrystallisation heat treatment. There is a significant drop in the yield strength for temperatures in excess of 600°C. It is expected therefore that flow forming at 600°C should lead to more a more uniform distribution of deformation when compared with room temperature where the material is so much stronger. This was the basis for the manufacture of tube W.

Fig. 6 shows the microstructure obtained in the tube W after recrystallisation heat treatment at 1380°C for 1 h. There is a clear difference in the grain structure as compared with that of tube R (Figs. 3 and 4). The grains are coarser and as for tube R, there is a tendency for the desired helical alignment of grains.

4. Conclusions

The influence of deformation on the recrystallisation of mechanically alloyed PM2000 has been studied. Two different tubes manufactured at room temperature and 600°C following a flow forming tube production route to introduce torsional flow to the material have been studied. Subsequent recrystallisation will develop coarse, torsionally wound grain structures. The lower yield strength of PM2000 at 600°C predicted using a published strength model, permitted the warm flow-forming which led to a more homogeneous deformation through the wall-thickness of the manufactured tube. Therefore, a coarser grain structure was obtained after recrystallisation. In future work, we hope to investigate the long-term creep and oxidation properties of suitably flow-formed tubes, both in laboratory tests and by installation in a pilot plant.

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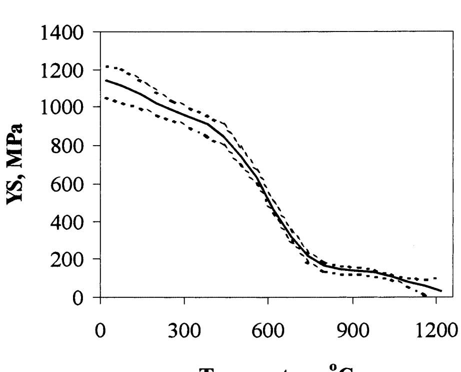

Fig. 5. Effect of the temperature on yield strength (YS) of PM2000 calculated using the method of Badmos et al. [21].

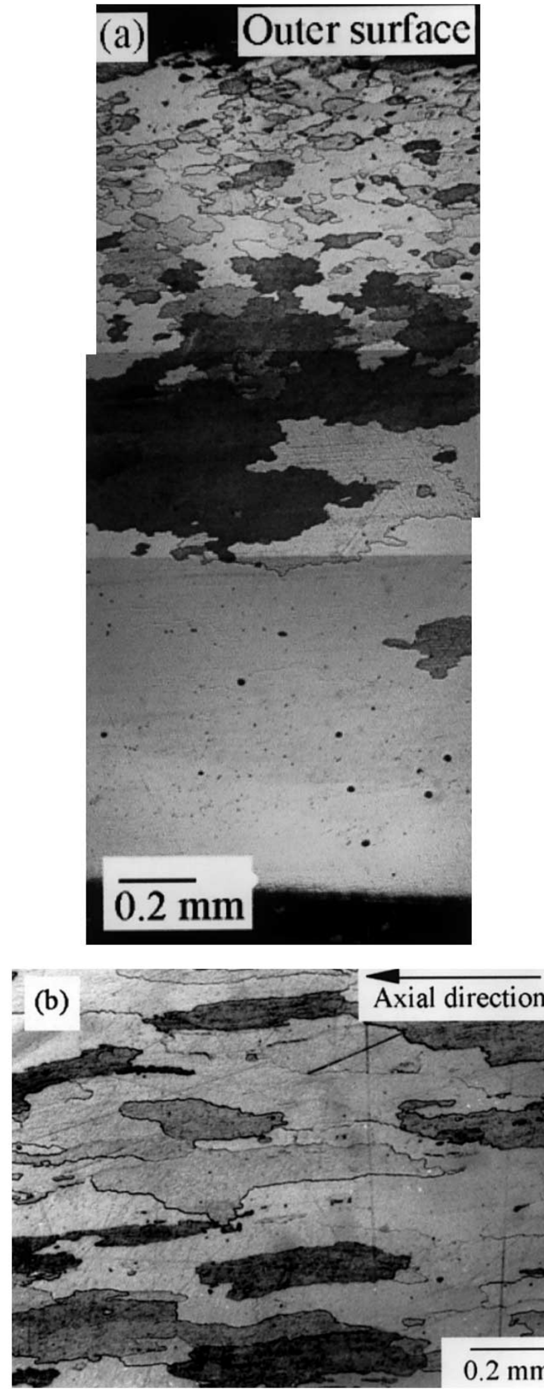

Fig. 6. Microstructures obtained after recrystallisation heat treatment at 1380°C for 1 h in tube W: (a) cross-section and (b) flat section.

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