EFFECT OF STRAIN HETEROGENEITY ON RECRYSTALLISATION OF PM2000

C. Capdevila*, Y. L. Chen**, A. R. Jones** and H. K. D. H. Bhadeshia*

*Department of Materials Science and Metallurgy
University of Cambridge, Pembroke Street, Cambridge CB2 3QZ
United Kingdom, www.msm.cam.ac.uk/phase-trans
**Materials Science and Engineering
Department of Engineering, University of Liverpool, L69 3GH
United Kingdom, www.liv.ac.uk/mateng/home.html

ABSTRACT

The recrystallisation behaviour of PM2000 oxide dispersion strengthened ferritic alloy has been investigated for samples which were cold deformed after extrusion. The evolution of the recrystallisation temperature T_R, defined as the minimum temperature at which the sample begins to recrystallise, has been studied in detail as a function of the level of deformation. The microstructure was characterised using transmission electron and optical microscopy, together with microhardness measurements. The results can be interpreted if it is assumed that anything which makes the microstructure heterogeneous, stimulates recrystallisation. In this sense, larger strain gradients lead to more refined and more isotropic grain structures. The way in which these results can be exploited for commercial applications are discussed.

1. INTRODUCTION

There is commitment in Europe to renewable energy; biomass is likely to make the largest single contribution to this type of power generation. The use of biomass for power generation does not require radically new technologies when compared with alternative sources of renewable energy. However, the thermodynamic efficiency of the process is dependent on the maximum temperature which can be attained in the operating cycle. To reach high efficiencies, it will be necessary to develop a heat exchanger capable of gas operating temperatures and pressures of around 1100 °C and 15-30 bar, respectively; the heat exchanger is needed for the fluid that enters the gas turbine. This demands metal operating temperatures up to 1150 °C for heat exchanger tubing.

The oxidation resistance and good creep performance of mechanically alloyed Fe-based oxide-dispersion strengthened (ODS) alloys such as PM2000 and MA956, makes them prime candidates for the heat exchangers (HE) (Sporer and Lempenauer 1993; Starr *et al.* 1994). Creep

and stress rupture properties are strongly influenced by grain size and shape (Wilson *et al.* 1978). Only coarse recrystallised grains have adequate high temperature creep strength. This coarse grained condition is produced by subjecting the alloy to a controlled degree of working and a finally, high temperature heat treatment.

The consolidated mechanically alloyed materials have a cold deformed microstructure with grains which tends to be submicrometre in width (Jaeger and Jones 1994; Baloch 1989; Chou *et al.* 1993). The material therefore contains an enormous amount of stored energy in this state (Chou and Bhadeshia 1993a and 1993b). The steels usually tend to recrystallise directionally into coarse columnar grains with an overall appearance which resembles that of a directionally solidified microstructure. There is considerable interest in controlling the microstructure. Many of the methods which seek to alter the microstructure rely on some type of deformation before recrystallisation at temperatures close to the melting temperature. Regle and Alamo (1993) studied the influence of cold deformation on the recrystallisation and obtained fascinating results in which recrystallisation temperature and grain structure was radically altered by the degree of deformation. The purpose of this study was to analyse the role of a *non-uniform* deformation on recrystallisation behaviour in PM2000 and a method to produce coarse grain structures was proposed.

2. EXPERIMENTAL PROCEDURES

The nominal composition of the alloy PM2000 used in this work is shown in Table 1. The alloy was supplied by PLANSEE GmbH / MSR Metall-Spezialrohr 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 alloys in gaseous environments containing SO_2 0.24%, CO_2 15%, O_2 4%, N_2 balance (Hedrich 1986; DeGaudenzi 1994). The creep performance has been found to be optimum with a Y_2O_3 content of 0.5 wt %.

Table 1. Chemical composition (wt %).

Cr	Ti	Y_2O_3	Al	Fe
20	0.5	0.5	5.5	Bal.

PM2000 is produced by mechanical alloying of the components in a high-energy mill to produce a solid solution which contains a uniform dispersion of yttria, in the form of coarse powder. The powder is then consolidated using hot isostatic pressing and these tube preforms are then extruded at 1050 °C. The extruded tubes are then flow formed, which is a process of *torsional extrusion* at room temperature to achieve reductions in area of 72% (T1) and 90% (T2).

Samples for metallographic examination were cut from the transverse direction of the tube. Optical microscopy was used to observe the microstructures both of as-flow formed and heat treated specimens. The etchant used was 2 g $CuCl_2$, 40 ml HCl, and 40-80 ml ethanol. Transmission electron microscopy was carried out using a JEOL 2000FX microscope operated at 200 kV to evaluate the deformed microstructure in flow formed tubes. In order to minimise the defects introduced by sample preparation foils were extracted by spark erosion. Samples were ground and polished to ~ 30 - 50 μm in thickness, then thinned to perforation by ion milling. Microhardness measurements were carried out in as flow formed samples by means of Mitutoyo Mvk-H2 hardness tester machine with a load of 200 g.

3. RESULTS AND DISCUSSION

The aim of this work was to analyse the effect of deformation induced by the flow forming processes on the recrystallisation of tubes for heat exchanger application. Tubes with two different levels of reduction in area have been studied.

The temperature T_R is defined as the minimum temperature at which recrystallisation takes place during one hour of heat treatment. Measured values of T_R for the inner and outer surfaces of the tubes are listed in Table 2. Tube T2 shows a uniform behaviour whereas T_R of the outer surface in T1 is much smaller than that of the inner surface. Furthermore, the recrystallisation behaviours of T1 and T2 are found to be quite different. In tube T1, recrystallisation begins close to the outer surface (Fig. 1a), whereas it initiates in the centre of the sample in the case of T2 (Fig. 1b). Moreover, the shapes of the recrystallised grains are very different in the two cases. More refined and equiaxed grains are obtained after recrystallisation in T1 when compared against T2, as is shown in Fig. 2.

Table 2. Measured values of T_R temperatures.

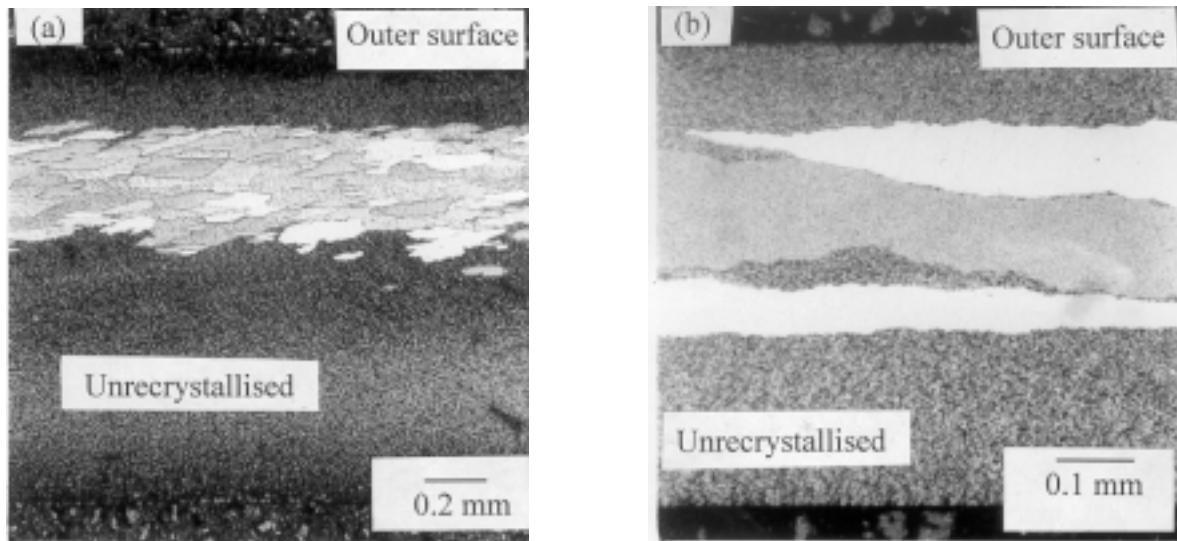|	Reduction in area / %	Outer surface	Inner surface
T1	72	835 °C	1190 °C
T2	90	1175 °C	1200 °C

Fig. 1. Transverse sections of (a) T1 and (b) T2 recrystallised at 870 °C for 30 min and 1180 °C for 1h, respectively.

Fig. 3a, which is a transmission electron microscope (TEM) image from T1, shows the inhomogeneous nature of the deformation, with some regions where sub-grains have a clearly elongated structure. In contrast, other regions of the same sample have more equiaxed grain structures. This should be contrasted with Fig. 3b, which is of tube T2 and shows a uniform elongated grain structure. These observations might correlate against recrystallised grain structures obtained following heat treatment.
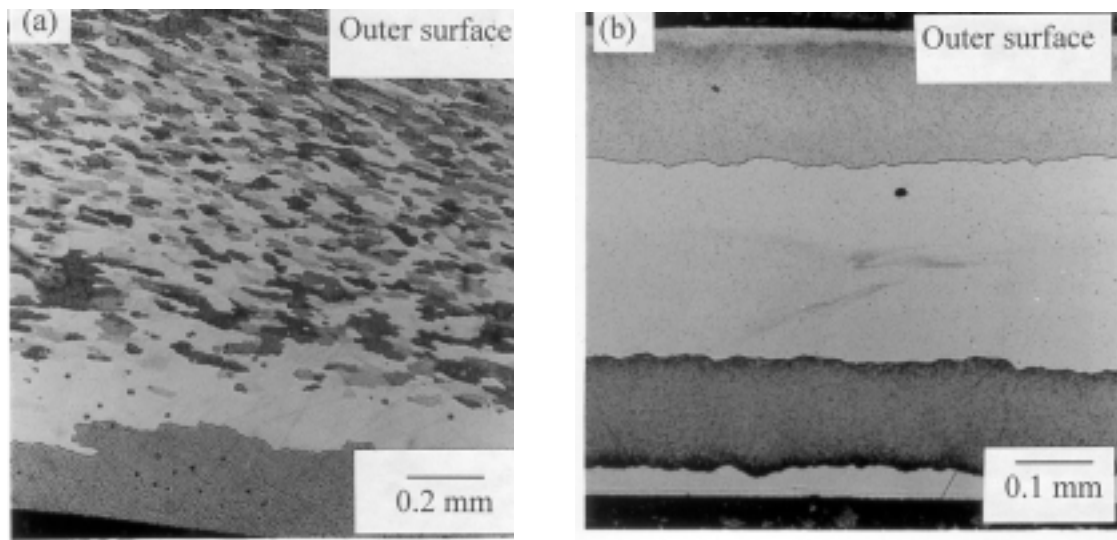
Fig. 2. Transverse sections of (a) T1 and (b) T2 recrystallised both at 1380 °C for 1h.

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 (Bhadeshia 1997). The activation energy for the nucleation of recrystallisation should be very large in order to achieve high recrystallisation temperatures characteristic of this material. However, this energy drops if a few grains happen to be slightly larger, *i.e.* if the grains are not uniform in size.
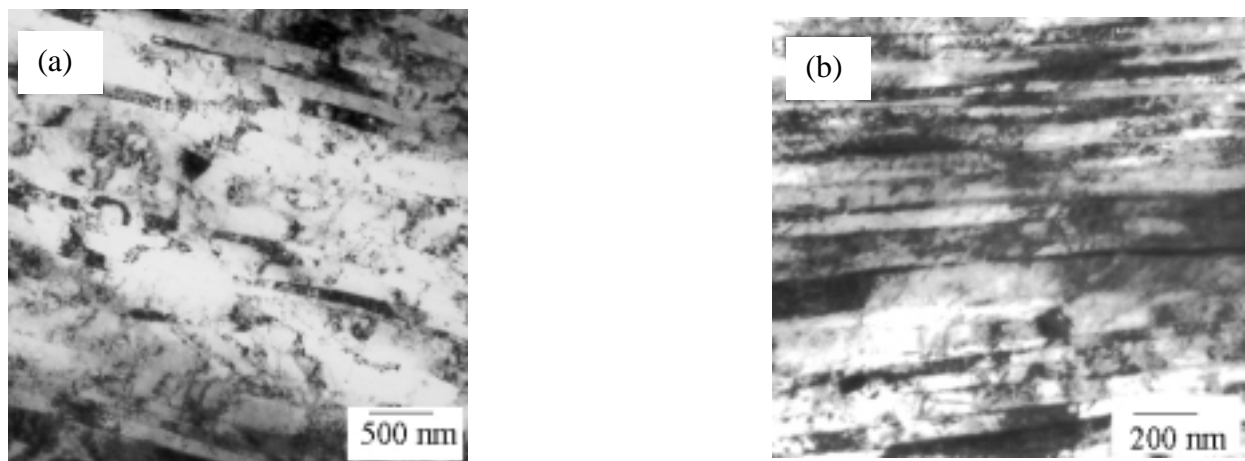

Fig. 3. Transverse sections of (a) T1 and (b) T2 in the as-flow formed condition.

Assuming that recrystallisation takes place by means of a bowing mechanism, a non-uniform microstructure corresponding to inhomogeneous deformation assists nucleation by making available some grains which are sufficiently large to permit easy bowing. This would lead to a greater nucleation rate and hence a finer recrystallised grain size together with a smaller value of T_R, as is observed for tube T1. By contrast, a uniform fine grain structure would find it more difficult to recrystallise and hence the few successful nuclei would develop into a coarse grained microstructure and T_R would be abnormally high; this is exactly the case for T2.

The fact that the overall deformation is less homogeneous in T1 than in T2 is confirmed by the data presented in Figure 4, which shows the evolution of the hardness across the wall thickness of the two tubes. The strain distribution in T2 is more homogeneous than that in T1. These results are consistent with the fact that nucleation of recrystallisation in T1 is promoted. Therefore, coarse grain structures should be obtained after uniform distribution of strain across the whole tube.
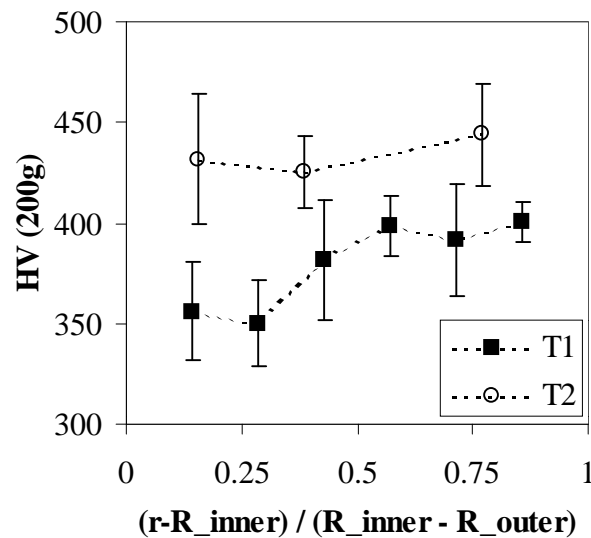

Fig. 4. Comparison of strain gradient across the wall-thickness of the tube between T1 and T2.

4. SUMMARY

The influence of deformation on the recrystallisation of mechanically alloyed PM2000 has been studied. Tubes with two different levels of deformation after flow-forming have been studied. As the reduction in area increases, a more homogeneous sub micron microstructure and strain gradient across the wall thickness of the tube is observed. Likewise, the differences between the minimum temperature at which recrystallisation in the outer and inner surface of the tube takes place is reduce as deformation increase.

These results are consistent with the broad idea that anything, which introduces heterogeneity into the microstructure, stimulates nucleation of recrystallisation. An inhomogeneous distribution of strain produce an inhomogeneous distortion of the sub-micron cold worked structure, increasing the differences in size and shape between the unrecrystallised grains themselves. Therefore, an easier nucleation process for recrystallisation can be expected and the grains can grow at lower temperatures resulting in a more equiaxed microstructure.

ACKNOWLEDGEMENT

C. Capdevila, Y. L. Chen, A. R. Jones and H. K. D. H. Bhadeshia

partners: Plansee GmbH, Metall-Spezialrohr GmbH (MSR), Sydkraft, Risø and Mitsui Babcock Technology Centre.

REFERENCES

Baloch, M.M. (1989). PhD Thesis. University of Cambridge.

Bhadeshia, H.K.D.H. (1997) Recrystallisation of practical mechanically alloyed iron-base and nickel-base superalloys. Mat. Sci. Engineering. A223, 64-77.

Chou, T.S. and Bhadeshia, H.K.D.H. (1993a). Crystallographic texture in mechanically alloyed oxide dispersion-strengthened MA957 and MA957 steels. Metall. Trans. 24A, 773-779.

Chou, T.S. and Bhadeshia, H.K.D.H (1993b). Grain control in mechanically alloyed oxide dispersion strengthened MA957 steel. Mater. Sci. Technol. 9, 890-897.

Chou, T.S., Bhadeshia, H.K.D.H., McColvin, G. and Elliott, I.C. (1993). Atomic Structure of mechanically alloyed steels. In: Mechanical Alloying for Structural Applications. Proceedings of the 2nd International Conference on Structural Applications of Mechanical Alloying. Vancouver, British Columbia, Canada, Ed: ASM (Materials Park, Ohio, USA) 77-82.

DeGaudenzi,G.P., Uberti,F., Bregani,F. and Toledo,G. P. (1994). ODS Alloys for High Temperature Heat Exchangers- Evaluation of Corrosion Resistance of MA956 and PM2000. In: Proceedings of High Temperature Materials for Power Engineering. Part II. Ed: D. Coutsouradis *et al.* (Kluwer Academic Publishers, The Netherlands) 1563-1572.

Hedrich, H.D. (1986). Properties and applications of ODS alloys. In: New materials by mechanical alloying techniques. Ed: E.D. Artz and L. Schultz. (Informationsgesellschaft Verlag, Berlin, Germany) 217-230.

Jaeger, D.M and Jones, A.R. (1994). The development of grain shape in Iron Base ODS Alloys. In: Proceedings of Materials Advanced Power Engineering. Ed: D. Coutsouradis *et al.* (Kluwer Academic Publishers, The Netherlands) 1507-1514.

Regle, H. and Alamo, A. (1993) J. Phys. IV 3, C7, 727-730.

Sporer, D. and Lempenauer, K. (1993). The structural stability of work hardened ODS superalloys. In: Proceedings of 13th International Plansee Seminar. Ed. Bildstein-Eck. (PLANSEE GmbH, Lechbruck, Germany) 796-810

Starr, F., White, A.R. and Kazimierza, B. (1994). Preassurised heat exchangers for 1100 °C operation using ODS. In: Proceedings of Materials Advanced Power Engineering. Ed: D. Coutsouradis *et al.* (Kluwer Academic Publishers, The Netherlands) 1393-1411

Wilson, F.G., Knott, B.R., and Desforges, C.D. (1978). Preparation and properties of some ODS Fe-Cr-Al-Alloys. Metall. Trans., 9A, 275-287.