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INFLUENCE OF Y_2O_3 PARTICLES AND DEFORMATION ON RECRYSTALLISATION OF ODS STEELS MA956 AND MA957

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Abstract

Commercialised mechanically alloyed yttria dispersion strengthened alloys exhibit unusual recrystallisation behaviour i.e. instead of recrystallising in an equiaxed grain structure they recrystallise in a manner which closely resembles that obtained by directional solidification, with the elongated columnar grains aligned along a particular sample axis. In spite of their large stored energy content, they tend to recrystallise at temperatures close to melting. The recrystallised microstructure is often very coarse and highly anisotropic, characterised by columnar grains. In the present paper influence of dispersed particles and deformation on recrystallisation behaviour of oxide dispersion strengthened MA956 and MA957 are investigated. It was found that degree of deformation prior to recrystallisation and manufacturing process, which tends to align the dispersoid particles along the principal fabrication direction, encourages recrystallisation to be directional.

Keywords: Dispersion strengthened, recrystallisation, deformation, columnar grains, mechanical alloying.

1. Introduction

Some of the most successful mechanically alloyed materials include the oxide-dispersion-strengthened (ODS) alloys are based on iron or nickel Capdevilas, *et al.,* (2003) the mechanical alloying process has been described in detail else where, Benjamin, Gessinger (1984) and relatively recently by Bhadeshia (1997) Briefly, mechanical alloying involves the severe deformation of mixtures of powders until they form the most intimate of atomic solutions. Inert oxides can also be introduced to form a uniform dispersion of fine particles which strengthened the consolidated product Bhadeshia (1997).

After mechanical alloying and processing into bulk form, the ODS ferritic stainless steels posses an ultra fine microstructure containing sub-micrometer sized grains of ferrite and a dislocation density of about $10^{15}m^{-2}$ Capdevila, (2001) and Bhadeshia. (2000) Chao, (2004) and Gonzalez the hardness in this condition is unacceptably high Chou (1993) and Bhadeshhia (2000) and contains very high levels of stored energy. Therefore, it has to be recrystallised into a coarse columnar grained microstructure before use Capdevila, and Bhadeshia. However, the recrystallisation behaviour of iron based ODS alloys is peculiar in that they recrystallise into a grain structure which resembles that obtained by directional solidification, with coarse, columnar grains having their longest axis along the extrusion direction Baloch, (1991) and Bhadeshia (2000). and Furthermore, recrystallisation usually does not occur until temperatures close to melting are reached Baloch, Elongated grains are desirable where the creep resistance at high temperatures is of paramount importance and iron based oxide-dispersion-strengthened alloys exhibit a high creep strength at temperatures from 900 to 1100°C Miller *etal.,* and Wasilkowaska *et al.,* (2003).

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Oxide dispersion strengthened Fe-based alloys are the only materials presenting jointly two important properties: (a) a high dimensional stability under irradiation, i.e. high resistance to swelling and irradiation creep, due to their bcc structure, and (b) a potential high temperature strength resulting from the homogeneous dispersion of nanometric oxide particles Almo *et al.*(2004), and Haken *et al.*, (2007) in the case of oxide dispersion strengthened ferritic alloys, the main difficulty could be the anisotropy in mechanical properties resulting from the anisotropy of microstructure developed during fabrication.

In the present work, the effect of yittrium oxide particles and deformation is examined in as received/deformed condition and after recrystallisation at various temperatures. The microstructural investigations were performed using transmission electron microscope and light optical microscope.

2. Material

Oxide dispersion strengthened ferritic stainless steels Incoloy MA956 and MA957 were supplied by INCOMAP (Wiggin Alloys Ltd. United Kingdom) in "hot-rolled" condition, their chemical composition is given in Table 1. The alloys were fabricated by charging three primary powders (elemental iron, prealloyed metallic alloys, and yittria) into a water cooled vertical attritor for the mechanical alloying. The consolidation of the resultant powder was achieved by extrusion at 1000°C while packed in a mild steel can. This was followed by a rolling at 1000°C, with a reduction in diameter from 54mm diameter to 25mm diameter (an 54% reduction in area) being achieved in one pass and from 54mm diameter to 9.5mm diameter (an 82% reduction in area) being achieved in one pass for oxide-dispersion-strengthened Incoloy MA956 and MA957 respectively. The extrusion and rolling are not to be thought of as "hot-working" processes, since the microstructure in as received condition is in the cold deformed state with a grain size of 0.45μm and 0.4μm for MA956 and MA957 respectively. For further work specimens for alloy MA956 were cut in the form of square rods of dimension 5×5×20mm and for alloy MA957 specimen were cut in the rectangular shape with the final dimensions of 2.5×4×20mm, with the length direction parallel to the rolling direction and others were prepared with the long dimension perpendicular to the rolling direction.

Alloy	C	Cr	Al	Ti	O	Y_2O_3	Balance
MA956	0.014	19.88	4.52	-	-	0.52	Fe
MA957	0.01	14.29	1.01	0.04	0.21	0.27	Fe

Table 1. Chemical composition (wt%) of Inconel Alloys MA956 and MA957.

3. Experimental techniques

3.1 Zone annealing

Zone annealing experiments were carried using a crystal grower as the way of achieving directional recrystallisation. The specimens were placed in a silica tube, mounted on a carriage driven by a variable speed electric motor. A Pt/Pt-13wt%Rh thermocouple was used to measure the temperature during operation, with one end of thermocouple in the middle of the specimen and the other end attached to a Comark (a device which converts electrical potential to temperature) for measuring the temperature during the movement of the specimen through the coil. The zone annealing experiments were performed in air. A schematic diagram of the crystal grower adapted and used is shown in (**Fig. 1**).

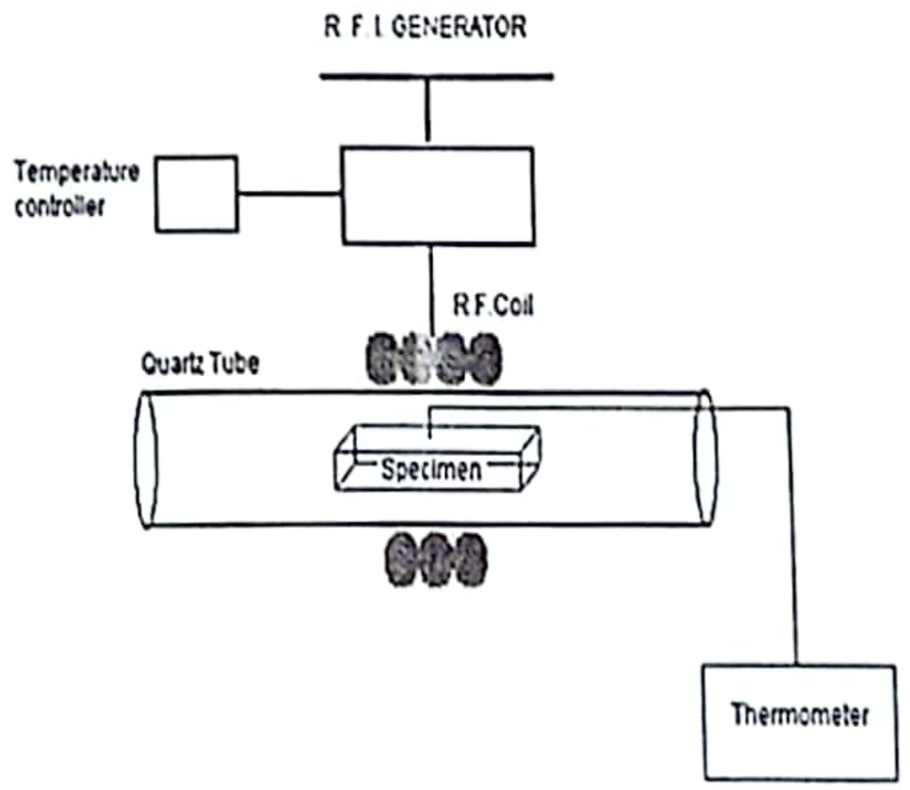


Fig. 1. Schematic diagram of crystal grower, used for zone annealing experiments.

3.2 Microscopy

Mechanically polished samples were etched for optical microscopy using a mixture of 2g $CuCl_2$ in 40ml HCl and 40-80ml ethanol. Thin foils for transmission electron microscopy (TEM) were prepared from 0.25mm thick discs machined from the sample concerned. After reducing the thickness to 0.05mm by abrasion on SiC paper, the samples were electropolished using a Fishione twin jet unit. The polishing solution consisted of 20 vol-% perchloric acid in methnol and was used at ambient temperature with the polishing potential set to 25-30 V. Unless otherwise stated, all the microscopy reported on the zone annealed samples was conducted on the longitudinal section, where the anisotropy of grain structure is most obvious. The hardness tests were performed using Vicker's hardness testing machine. A load of 10kg applied for each indentation.

4. Results

The manufacturing process of oxide dispersion strengthened alloys MA956 and MA957 as described in section 2 involves rolling at a temperature of 1000°C, with a reduction in diameter from 54 mm to 25 mm (an 54% reduction in area) and from 54 mm to 9.5 mm diameter (an 82% reduction in area respectively, being achieved in one pass. On examining the thin foils, a true cold deformed structure was observed with a very high dislocation density in the as received condition, as shown in (Fig. 2). From (Fig. 2a), which was recorded during the examination of the thin foils prepared from the transverse section of the bar of alloy MA956, an equiaxed grain structure (consistent with the circular section of the extruded bar) can be seen, by contrast, (Fig. 2b) longitudinal section revealed an elongated grain structure, with tangled dislocation net works. Where as thin foils examined for alloy MA957 from transverse and longitudinal sections exhibited slightly anisotropic grain structure in the transverse section of the bar (Fig.2c) but, highly elongated grains for the longitudinal section (Fig.2d). The microstructure was found to be consistent with a cold-deformed state, with an obviously high density of dislocation.

The hardness measured in the as received condition was also found to be very high i.e. 350 and 410 HVN (10kg) for alloys MA956 and MA957 respectively, again consistent with a cold deformed microstructure; fully annealed iron can have a hardness of less than 200 HVN (10kg).

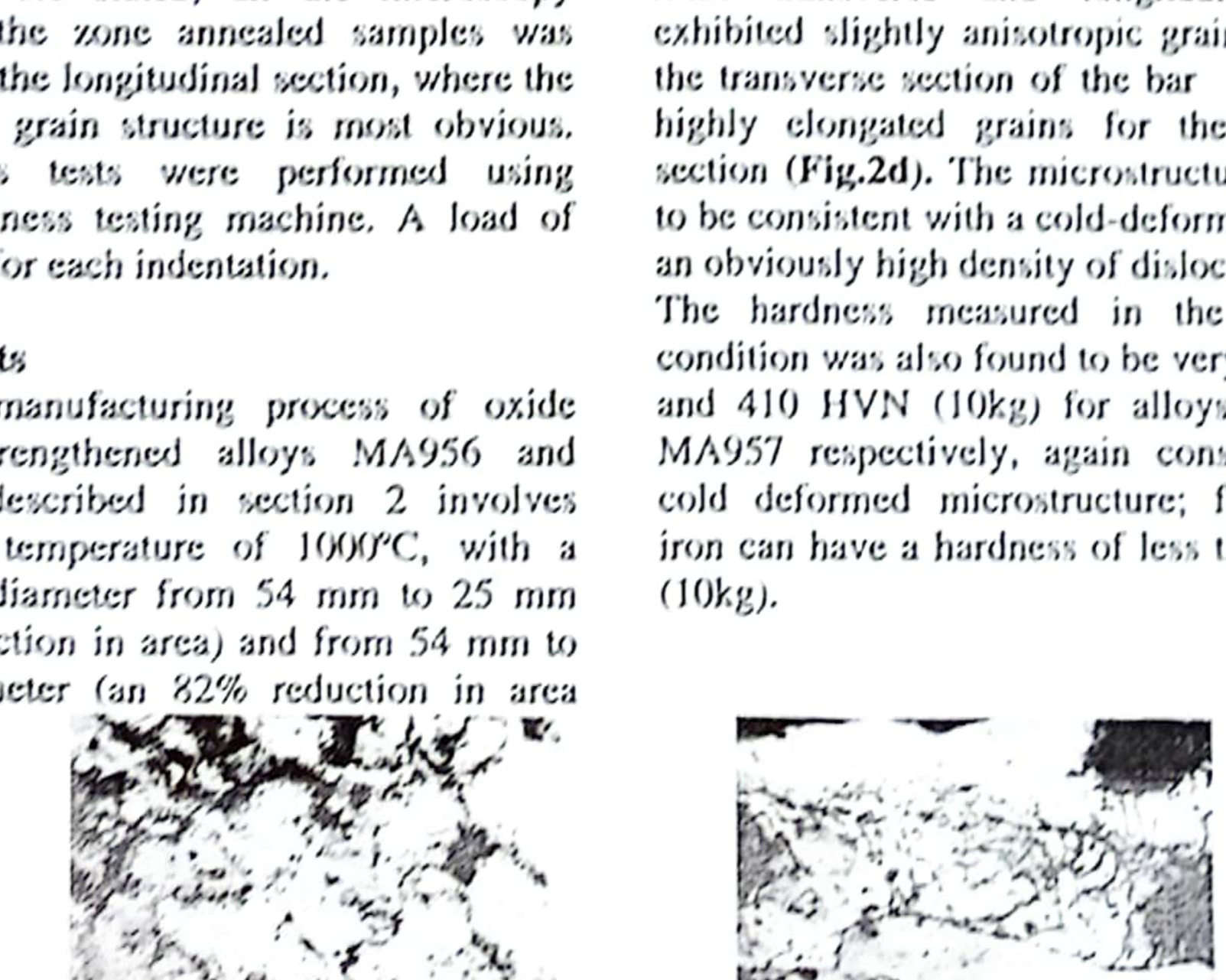


Fig. 2.Transmission electron micrographs reveals the microstructure of alloys MA956 and MA957 in the as-received condition. a and b are from transverse and longitudinal sections of MA956 and c and d are from transverse and longitudinal sections of MA957.

Experiments were performed to directionally recrystallise the samples of MA956 at various peak temperatures (T_P) ranging from 1150, 1180 to 1380°C in a temperature gradient using crystal grower as a means to achieve columnar grain structure with various specimen travel speeds (0.2, 0.4, 0.8, 1.4, 3.2, 5.0, 7.7 and 10.0 mm/min).

Zone annealing at $T_P \approx 1150$°C failed to produce any substantial change in the optical microstructure (Figure 3) but only a very small amount of directional recrystallisation could be seen at even the lowest travel speed (0.2mm/min) utilized, and the overall hardness showed a little change relative to the hardness before annealing (322 HVN (10kg). This observation suggested that either a further lowering of specimen travel speed (i.e., less than 0.2mm/min, which was not possible with our equipment) or an increase in temperature can successfully produce columnar grains in ODS MA956.

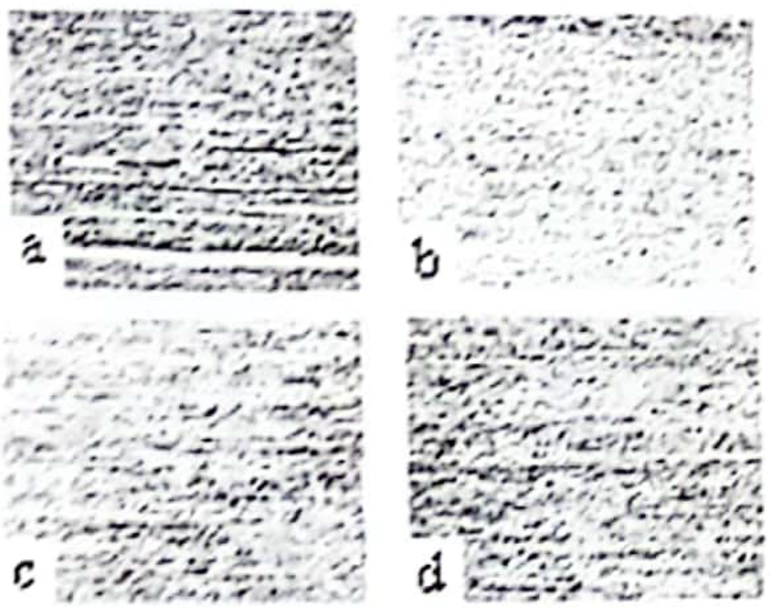


Fig. 3. Optical micrographs recorded after zone annealing specimens at $T_P \approx 1150$°C. The micrographs are from the regions where T_P was measured.
a. Specimen travel speed 0.2mm min^{-1}. b. Specimen travel speed 5.0mm min^{-1} (Alloy MA956) c. Specimen travel speed 0.8mm min^{-1}. d. Specimen travel speed 5.0mm min^{-1} (Alloy MA957)

In order to resolve the ambiguities further zone annealing experiments were performed at higher peak temperatures i.e. $T_P \approx$ 1280 and 1380°C. The optical microstructure recorded after these experiments are shown in figures 4 and 5. From Figures 4 and 5, it can be clearly seen that the specimens of alloy MA956 are fully directionally recrystallised at minimum specimen travel speeds used (0.4 and 0.8 mm/min at 1280 and 1380°C respectively) and the hardness values measured dropped significantly i.e. 288 and 248 HVN(10kg) at 1280 and 1380°C respectively and the columnar grains were found to be straight along the length of specimen and as long as the sample size.

Zone annealing experiments were also performed on an identical ODS ferritic stainless steel MA957 at peak temperatures (T_P) ranging from 1150 to1350°C using various specimen travel speeds (0.2, 0.4, 0.8, 1.4, 3.2, 5.0, 7.7 and 10.0 mm/min).



Fig. 4. Optical micrographs recorded after zone annealing MA956 at 1280°C.
a. 0.8mm min^{-1} b. 1.4mm min^{-1} c. 3.2mm min^{-1} d. 5.0mm min^{-1} e. 7.7mm min^{-1} f. 10.0mm min^{-1}

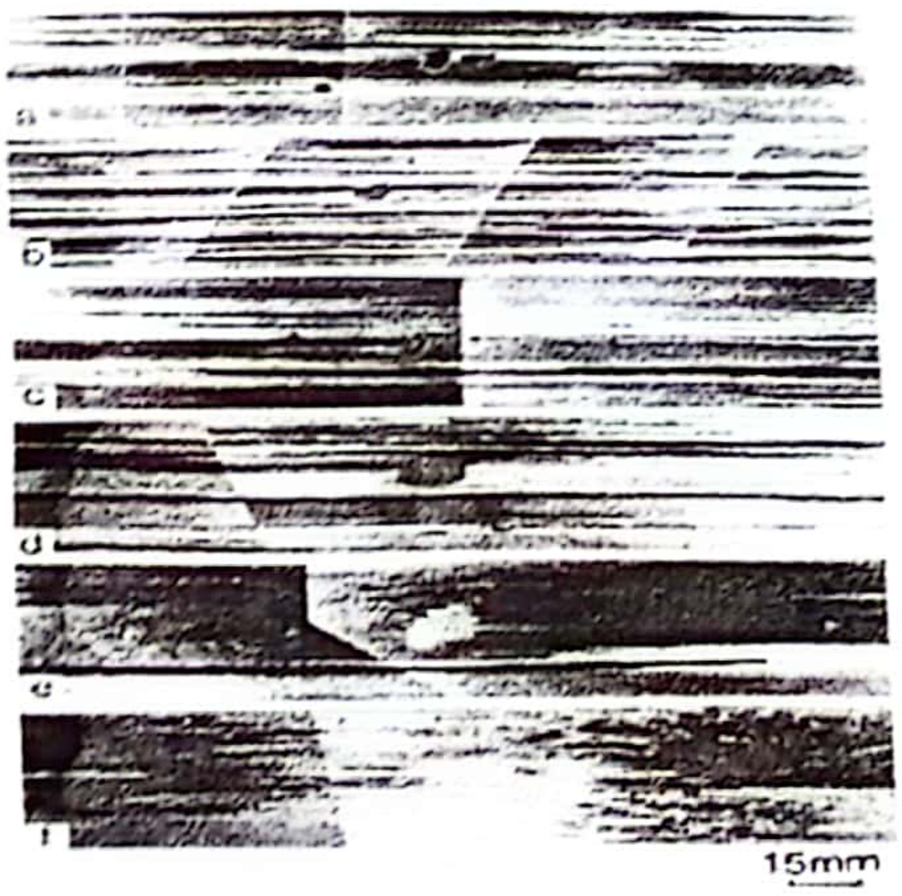


Fig. 5. Optical micrographs recorded after zone annealing MA956 at 1380°C.

a. 0.8mm min^{-1} b. 1.4mm min^{-1} c. 3.2mm min^{-1} d. 5.0mm min^{-1} e. 7.7mm min^{-1} f. 10.0mm min^{-1}

The minimum peak temperature applied to directionally recrystallise the samples in a temperature gradient (i.e. $T_P \approx 1150$°C) does not

seems to alter the microstructure as can be seen from Figure 3B, and the hardness measured turn out to be 374HVN (10kg). Since the optical microstructure at $T_P \approx 1150°C$ resembles the microstructure obtained in the as-received / deformed condition but slightly lower hardness value as mentioned above suggest some kind of recovery in the material at this temperature.

As the zone annealing experiments performed at relatively higher temperatures ($T_P \approx 1250$ and 1350°C) and the micrographs recorded show complete directional recrystallisation with a specimen travel speed of 0.2 mm min^{-1} at 1250°C, with a substantial drop in measured hardness value (i.e. 178 HVN 10kg) but at relatively faster specimen travel speeds (0.4 and 3.2 mm min^{-1}) partial directional recrystallisation was observed (Figure 6A). As the temperature raised to maximum peak temperature ($T_P \approx 1350°C$), used to zone anneal the specimens of ODS alloy MA957 found to directionally recrystallised at even faster specimen travel speeds (**Fig. 6B**) and hardness value measured were decreased further as161, 164, 167, 167, 201 and 293 HVN (10kg) for specimen travel speeds of 0.8, 1.4, 3.2, 5.0, 7.7 and 10.0 mm min^{-1} respectively.

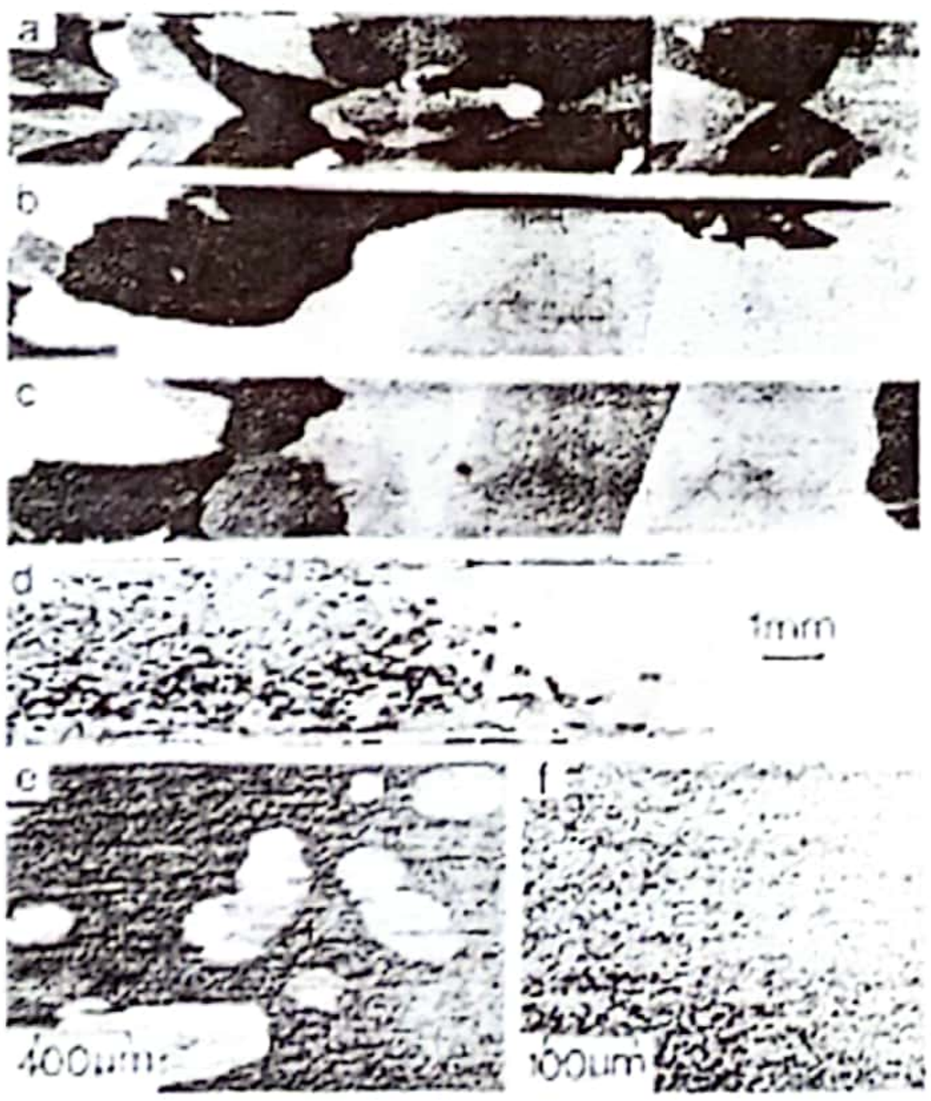


Fig. 6A. Optical micrographs recorded after zone annealing MA957 at 1250°C.
a. 0.2mm min^{-1} b. 0.4mm min^{-1} c. 0.8mm min^{-1}
d. 1.4mm min^{-1} e. 3.2mm min^{-1} f. 5.0mm min^{-1}

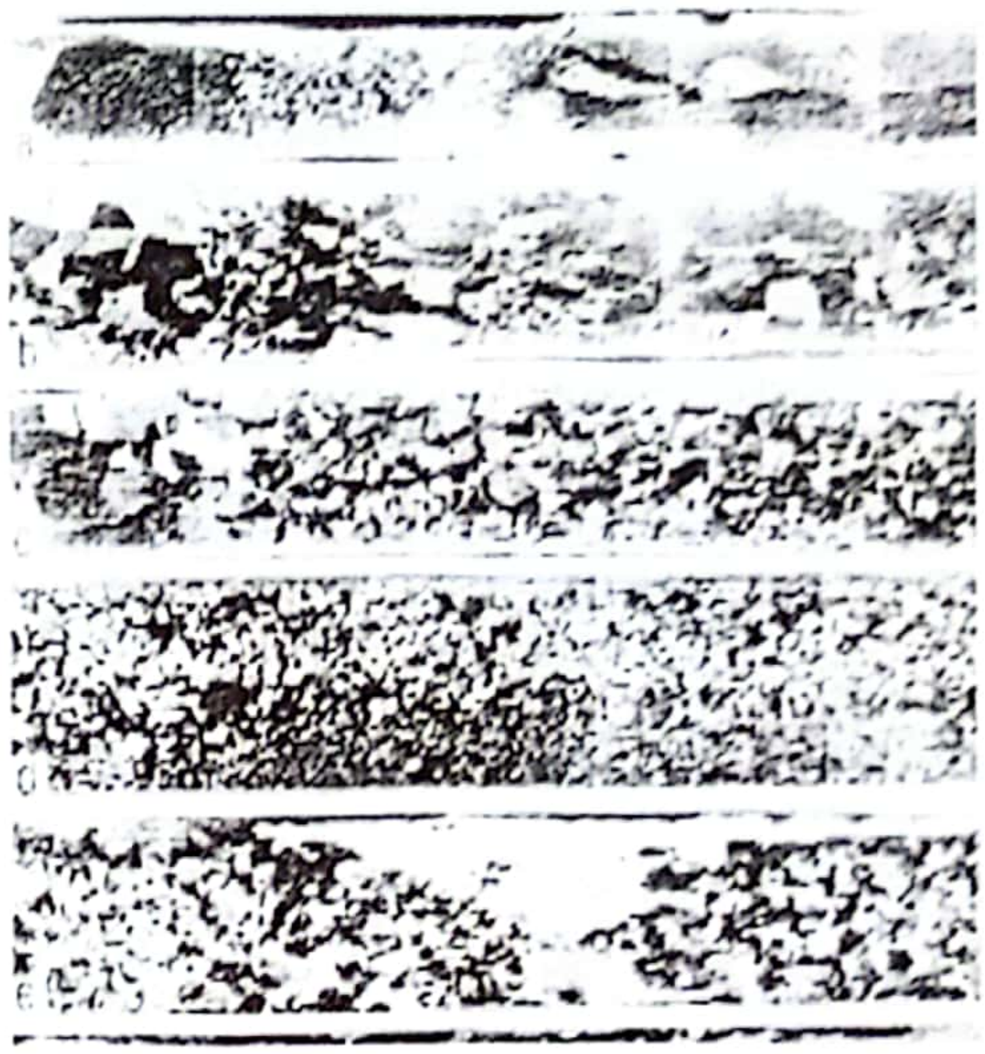


Fig. 6B. Optical micrographs recorded after zone annealing MA957 at 1350°C.
a. 0.8mm min^{-1} b. 1.4mm min^{-1} c. 3.2mm min^{-1}
d. 5.0mm min^{-1} e. 7.7mm min^{-1} f. 10.0mm min^{-1}

The optical microstructure recorded from the samples zone annealed at various peak temperatures exhibit columnar grains with an appearance of cast ingot type grain structure, an effect which was thought to be due to dominance of grains growing from the surface of the sample during zone annealing.

It may be argued, that the unidirectional grain growth takes place during directional recrystallisation in oxide dispersion strengthened ferritic steels MA956 and MA957 is a consequence of the fabrication process.

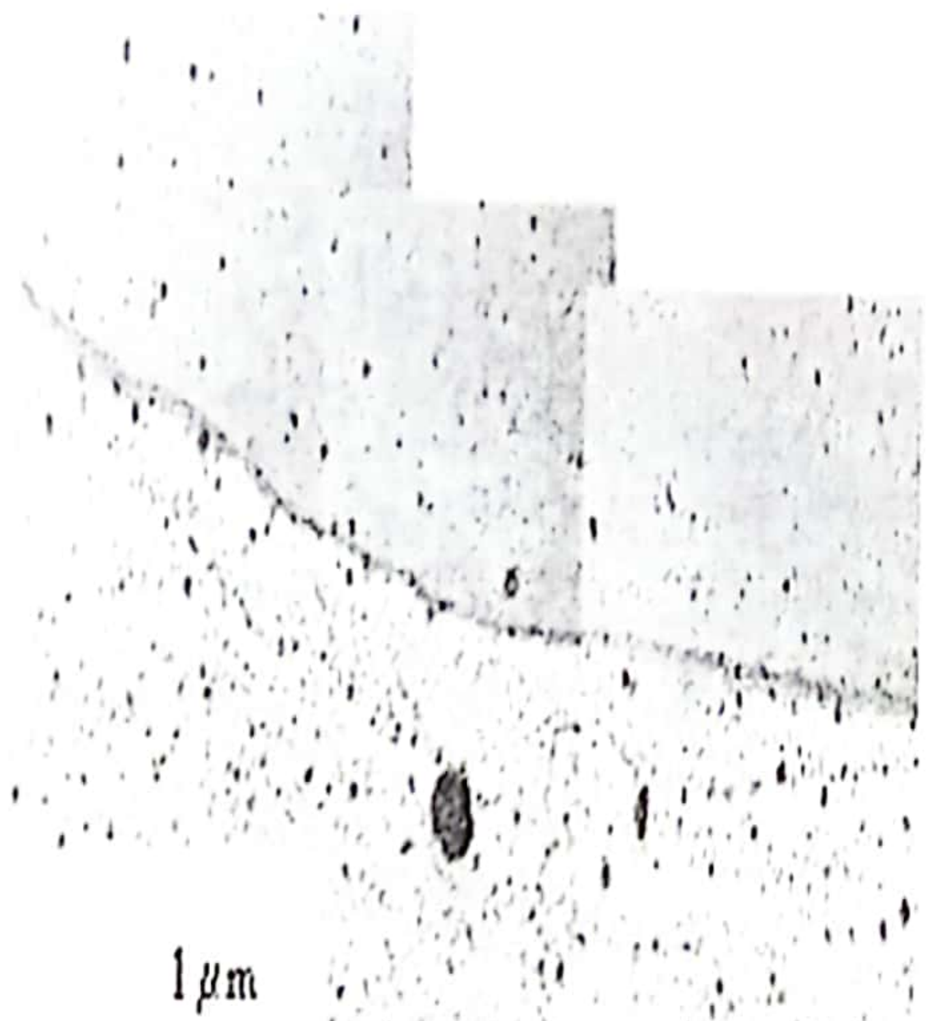
An attempt was made to analyse the effect of yittrium oxide (Y_2O_3) particle alignment on the recrystallisation of MA956. Alignment that can be seen in (**Fig. 7**), which was taken from an unetched specimen zone annealed at $T_P \approx 1280°C$ and 1.4 mm min^{-1}. It is apparent that some of the particles are aligned along the deformation direction, the spacing between adjacent rows of aligned particles being approximately equal to the maximum lateral spacing observed between two adjacent grain boundaries in directionally recrystallised specimens.The transmission electron micrograph shown in (**Fig. 8A**), further confirms the effect

of particles on the columnar grain growth process for MA956. The micrograph further reveals that very fine and relatively coarser Y_2O_3 particles are aligned along the grain boundary, which was found after considerable effort because of large grain size obtained after zone annealing.



Fig. 7A. Showing alignment of particles along the rolling direction, recorded from an unetched sample of MA956.

An examination of thin foils prepared from the longitudinal section of directionally recrystallised sample of ODS MA957, revealed tendency for particle alignment along the working direction (Figure 8B), although the tendency for alignment appeared less than in alloy MA956. This could be due to the relatively small additions of Y_2O_3 particles (0.27 wt%) (Table 1), compared with MA956 which contains 0.52 wt% Y_2O_3 particles. There is an obvious tendency for grain boundary to be pinned by the particles, although the pinning is not adequate to completely stop recrystallisation. However, the effect of the Y_2O_3 particles was evident in the observation of small regions of unrecrystallised material pinned and left behind as the remainder of the recrystallisation front advanced relatively rapidly (**Fig. 9**).



A.



B.

Fig. 8. (A) Transmission electron micrographs of the MA956 samples zone annealed at 1280°C at a specimen travel speed of 3.2mm min^{-1}. (B) Transmission electron micrographs of the MA957 samples zone annealed at 1250°C at a specimen travel speed of 0.2mm min^{-1}. Note the degree of particle alignment along the rolling direction in both MA956 and MA957.

As completely different grain structure i.e. cast ingot type and straight and long grains extending to the length of specimen, was observed for two identically processed ferritic steels MA957 and MA956 respectively, and heat treated under almost similar conditions, it was thought necessary to measure the activation energy, stored in the material in the form of defects (i.e. dislocations).

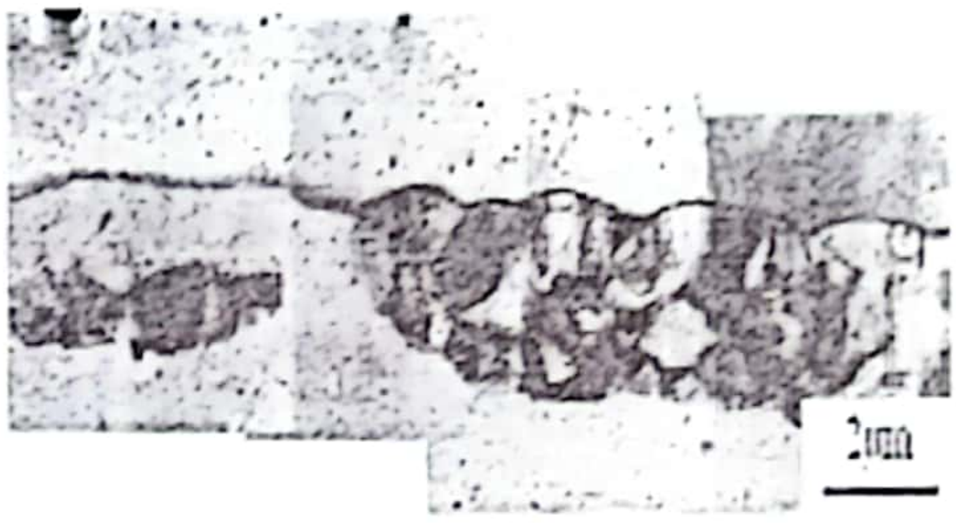


Fig. 9. Transmission electron micrographs of the MA956 samples zone annealed at 1280°C at a specimen travel speed of 3.2mm min^{-1}. Streams of unrecrystallised grains are seen to be trapped between two fully recrystallised, "dislocation free" grains.

Using experimental data obtained after zone annealing experiments at two different peak temperatures and a range of specimen travel speed for both the materials individually, the activation energy Q required to directionally recrystallise was measured. The measured value of activation energy for alloy MA957 was found to be 654257 J mol^{-1} which is much larger than that of alloy MA956 (402791 J mol^{-1}). This significant difference in the measured activation energies of two identically processed alloys (MA957 and MA956) may be due to the amount of deformation applied to each alloy 82% and 54% reduction respectively. Although MA957 has a lower oxide content, the tendency for particle alignment, as discussed above, is smaller, so that grain boundary mobility along the rolling direction may be more effectively impeded.

5. Conclusions

The influence of dispersed yittrium oxide particles and deformation on directional recrystallisation behaviour of two oxide dispersion strengthened ferritic steels MA956 and MA957 processed in identical conditions, were investigated and following conclusions were made:

1. The recrystallisation behaviour of alloys MA956 and MA957 is highly influenced by the fabrication process.

2. Strong alignment of Y_2O_3 particles along the rolling direction for MA956, was found to boost the grains to grow unidirectionally, whereas, smaller particle alignment tendency was found to appear in MA957.

3. The activation energy measured is MA957 (654257 J mol^{-1}) was much larger than that of alloy MA956 (402791 J mol^{-1}) which may be due to the amount of deformation applied to each alloy 82% and 54% reduction respectively.

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