Dynamic recrystallisation in hot deformed oxide dispersion strengthened MA956 and MA957 steels

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The hot deformation behaviour of the oxide dispersion strengthened stainless steels MA956 and MA957 has been studied. The alloys are made by a mechanical alloying process which leaves them in a very fine grained, cold deformed state immediately after consolidation. It is found that deformation is accompanied by dynamic recrystallisation, even when the deformation temperatures are far less than the ordinary recrystallisation temperatures of the two alloys. These and other results on the strength and anisotropy of the alloys are interpreted in terms of their microstructures. MST/3043

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Introduction

Mechanical alloying is a process in which powders of different compositions are deformed together to such an extent that a solid solution is formed.[1] Stable oxides can also be incorporated into the alloy during this process.[2,3] Many mechanically alloyed steels are now commercially available. Among these, MA956 is a chromium rich ferritic stainless steel containing ~8 at.-%Al, together with a dispersion of yttrium oxide particles for creep resistance.[4,5] An alternative ferritic stainless steel variant (MA957) contains titanium rather than aluminium and is designed for applications in the nuclear industry.[6]

The mechanically alloyed powders are consolidated by extrusion and hot rolling. The consolidated alloys have a cold deformed microstructure with grains which tend to be submicrometre in width.[7,8] The materials therefore contain an enormous amount of stored energy in this state.[9,10] 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 heat treatment at temperatures close to the melting temperatures of the alloys. Regle and Alamo[11] have recently made a comprehensive study of the influence of cold deformation on the subsequent recrystallisation behaviour of MA956 and MA957 steels. The purpose of the present work was to extend these studies to deformation carried out at temperatures which are relatively high, but still well below the recrystallisation temperature of the undeformed steels.

Experimental procedure

The alloys, the chemical compositions of which are given in Table 1, were supplied by Inco Alloys (Hereford, UK) in an unrecrystallised state. They were fabricated by charging three primary powders (elemental iron, prealloyed metallic alloys, and yttria) 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 rolling at 1000°C, with a reduction in diameter from 54 to 25 mm for both MA956 and MA957 steels.

Compression tests were employed to evaluate the hot deformation behaviour of as received MA956 and MA957 steels, both of which were obtained as bars 25 mm in diameter. The samples for the compression experiments were cut from the longitudinal and transverse directions and machined into cylinders 8 mm in diameter and 12 mm in length. The experiments were carried out in an adapted Thermecmaster Z thermomechanical simulator. The diametral strain was monitored using a laser device with an absolute accuracy of ±1 μm, which over an 8 mm specimen diameter gave an accuracy in the strain measurement of about ±1%. The specimens were heated by induction, with a measured temperature variation along the length direction of only ±5 K. The uniaxial compressive load was applied via quartz discs. Test temperatures were selected at 950, 1000, and 1050°C, with a 50% total reduction strain and 0·01 s^{-1} constant strain rate control.

Both optical microscopy and transmission electron microscopy were used to observe the deformed microstructures. The hardness was measured after hot deformation.

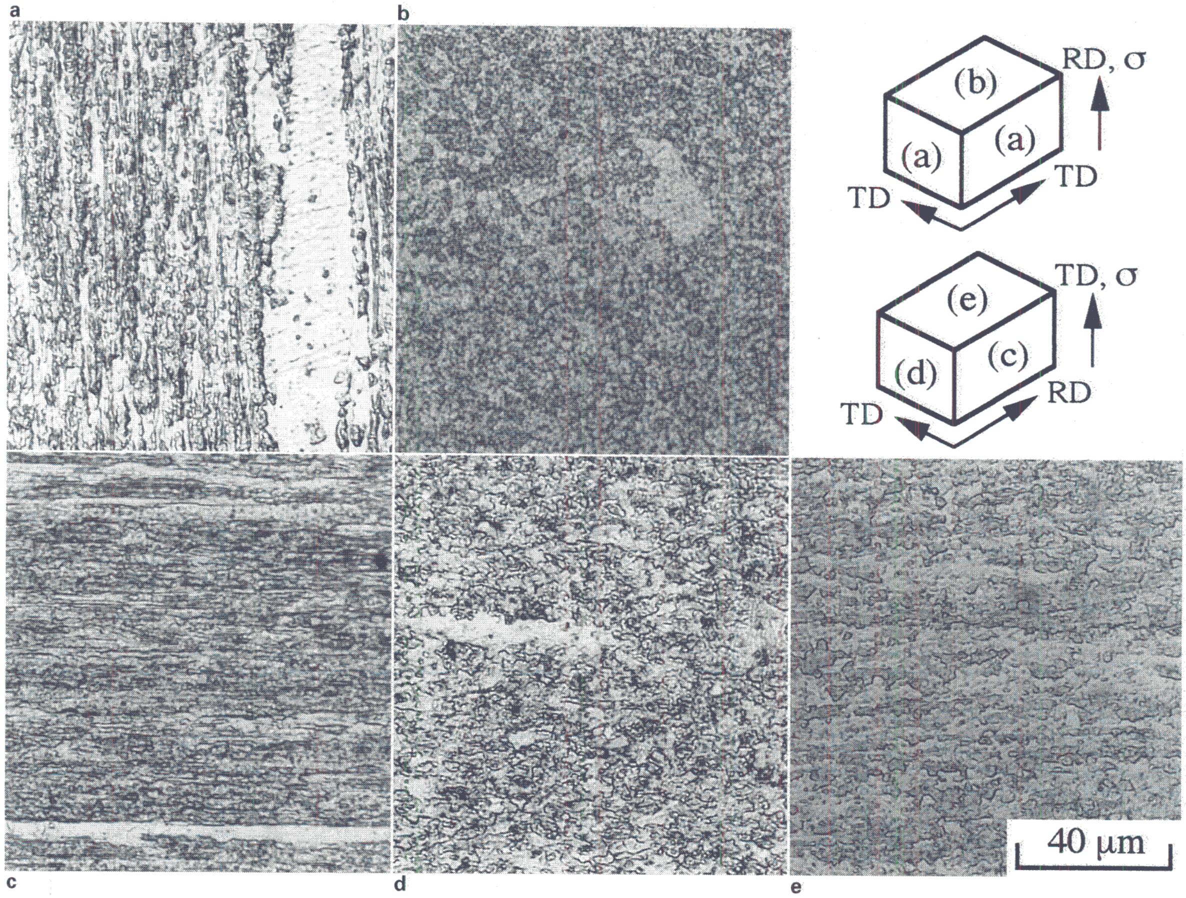
Results and discussion

HOT DEFORMED MICROSTRUCTURES

Figures 1–3 show the optical micrographs of hot deformed MA956 steel, with the compression axis parallel or normal to the rolling direction for temperatures ranging from 950 to 1050°C. In spite of these relatively low temperatures, elongated grain structures can be seen in all the samples. Furthermore, the elongated grains always occur with their long axes along the rolling direction, irrespective of the orientation of the compression direction. It also appears that the fraction of recrystallised microstructure increases with increasing hot deformation temperature, and the hardness data presented later confirm this more quantitatively. The samples deformed along the rolling direction recrystallise more readily. It should be noted that the micrographs shown in Figs. 1–3 are from barrelled hot deformed samples; the recrystallised grains are rodlike in three dimensions but are curved by the barrelling. The meridional sections (Figs. 1*c*, 2*c*, and 3*c*) therefore illustrate sections approximately along the long axes of the recrystallised grain. Figures 1*e*, 2*e*, and 3*e* also contain the rolling direction but the grain anisotropy is less pronounced

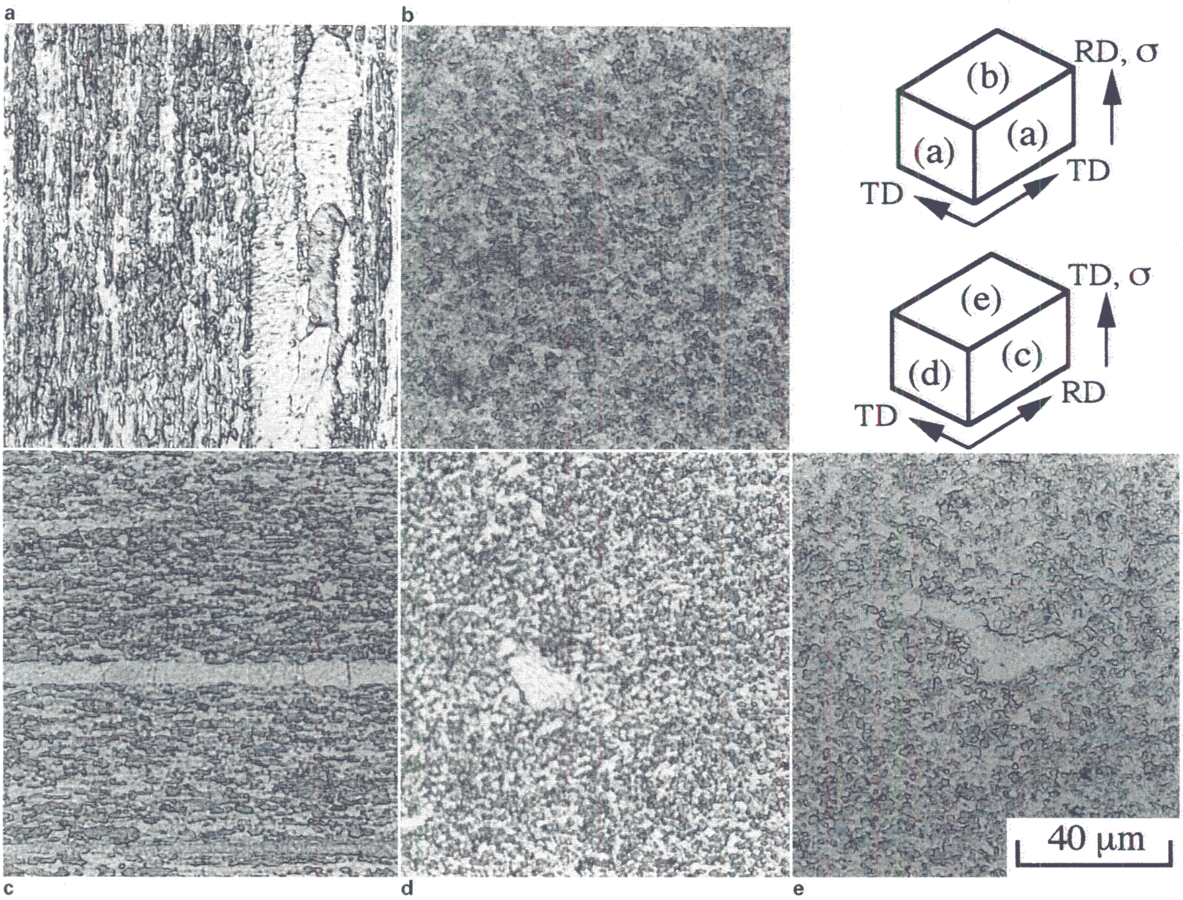
Table 1 Chemical composition of mechanically alloyed steels MA956 and MA957, wt-%

Alloy	C	Cr	Mo	Al	Ti	Y_2O_3	Fe
MA956	0·01	20·0	...	4·5	0·5	0·5	Bal.
MA957	0·01	14·0	0·3	...	1·0	0·27	Bal.



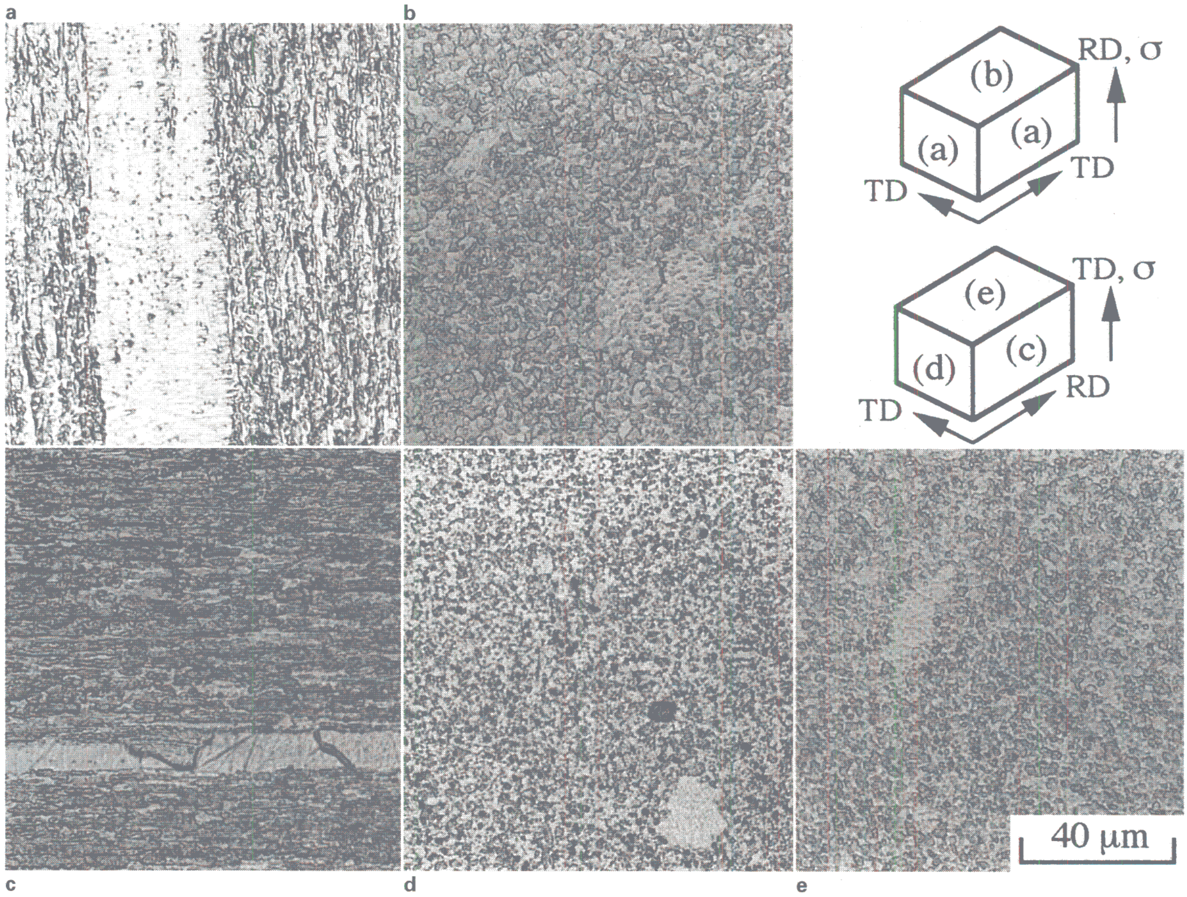
a, b compressed along rolling direction; *c–e* compressed normal to rolling direction

1 Micrographs illustrating hot deformed microstructures of MA956 steel compressed at 950°C



a, b compressed along rolling direction; *c–e* compressed normal to rolling direction

2 Micrographs illustrating hot deformed microstructures of MA956 steel compressed at 1000°C



a, *b* compressed along rolling direction; *c–e* compressed normal to rolling direction

3 Micrographs illustrating hot deformed microstructures of MA956 steel compressed at 1050°C

because the observations are close to the sample surface and hence cut the curved rods in a glancing manner.

Figures 4–6 illustrate similar observations for hot deformed MA957 steel. The recrystallised grains tend to be coarser in MA957 compared with MA956, consistent with the usual recrystallisation microstructures[7–11] of these alloys. Steel MA956 was found to recrystallise more easily than MA957 during hot deformation; this may be expected since MA956 steel has a lower recrystallisation temperature in normal circumstances.

HOT DEFORMATION BEHAVIOUR

Figure 7 shows the stress–strain curves for MA956 samples compressed along the rolling direction and normal to the rolling direction for a variety of temperatures. The true stress does not increase monotonically with strain, so that the work hardening characteristics usually observed are not found for the as received alloys. Instead, softening occurs during hot deformation. This is due to dynamic recrystallisation during deformation, as is evident from the metallographic observations (Figs. 1–6). The grain structure in the as received material is incredibly fine (submicrometre),[7–11] so that the optically visible grains shown in Figs. 1–6 are unambiguously a result of dynamic recrystallisation. It is interesting to note that the type of stress–strain curve reported in the present paper is similar to those found during the hot rolling of steel sheet where dynamic recrystallisation is common.[12]

To further analyse the data, the critical strain ε_c for the start of recrystallisation, and the strain ε_x equivalent to that required to recrystallise a significant fraction of the material for each test temperature (compressed along the rolling direction) are given in Table 2.

According to Rossard,[13] ε_c should be slightly less than the peak strain ε_p because, while the first nuclei are softening the material locally, the remaining material continues to become stronger by work hardening. The parameter ε_c is given by the approximate relation[12]

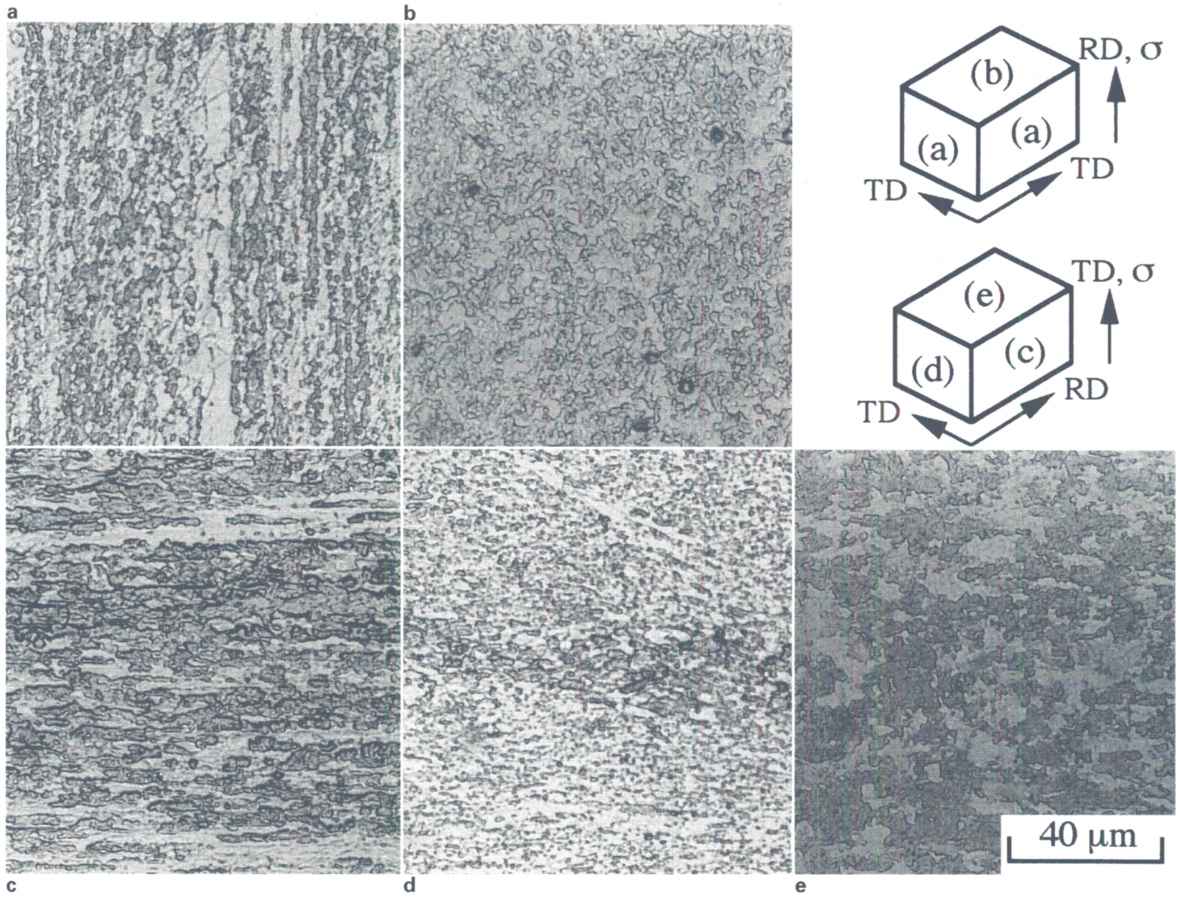
$$\varepsilon_c \approx 5\varepsilon_p/6 \approx 0{\cdot}83\varepsilon_p$$

However, the critical strain for dynamic recrystallisation may not in fact be attained for a variety of reasons. For example, the material could continue to strain harden, developing a higher and higher flow stress, until failure occurs. The definition of ε_x is therefore useful, as the strain from the peak stress to the minimum value when dynamic recrystallisation occurs (e.g. Fig. 7*b*), or from the peak to the steady state when there is continuous softening.[14,15]

In general, the critical strain for recrystallisation ε_c increases with the strain rate and with decreasing deformation temperature.[16] Consistent with this, the results given

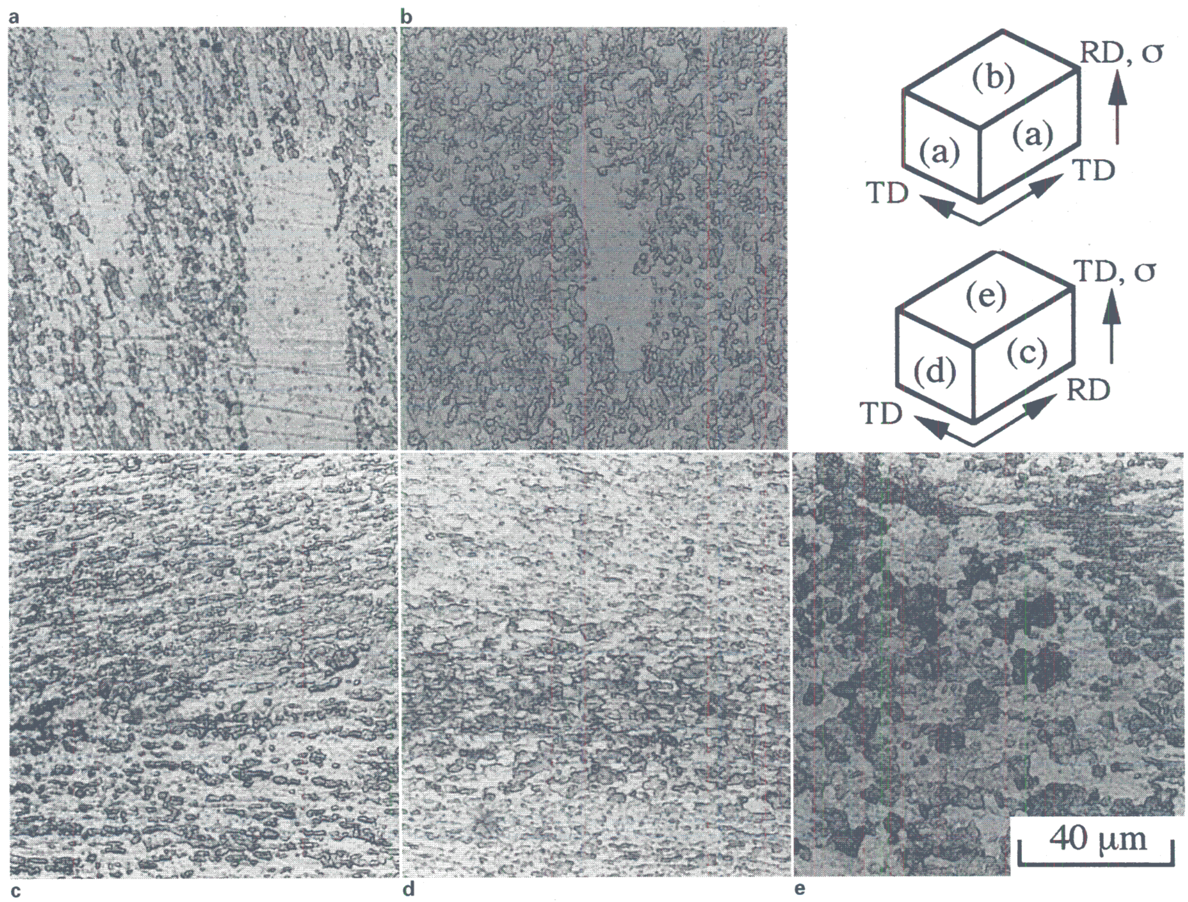
Table 2 Critical strain ε_c, peak strain ε_p, and equivalent strain ε_x required to recrystallise significant fraction of material for dynamic recrystallisation for MA956 steel compressed along and normal to rolling direction

Deformation temp., °C	ε_p	ε_c	ε_x
Along rolling direction			
900	0·12	0·10	0·28
1000	0·10	0·08	0·20
1050	0·04	0·03	0·16
Normal to rolling direction			
900	0·024	0·020	0·046
1000	0·240	0·200	0·130
1050	0·220	0·183	0·045



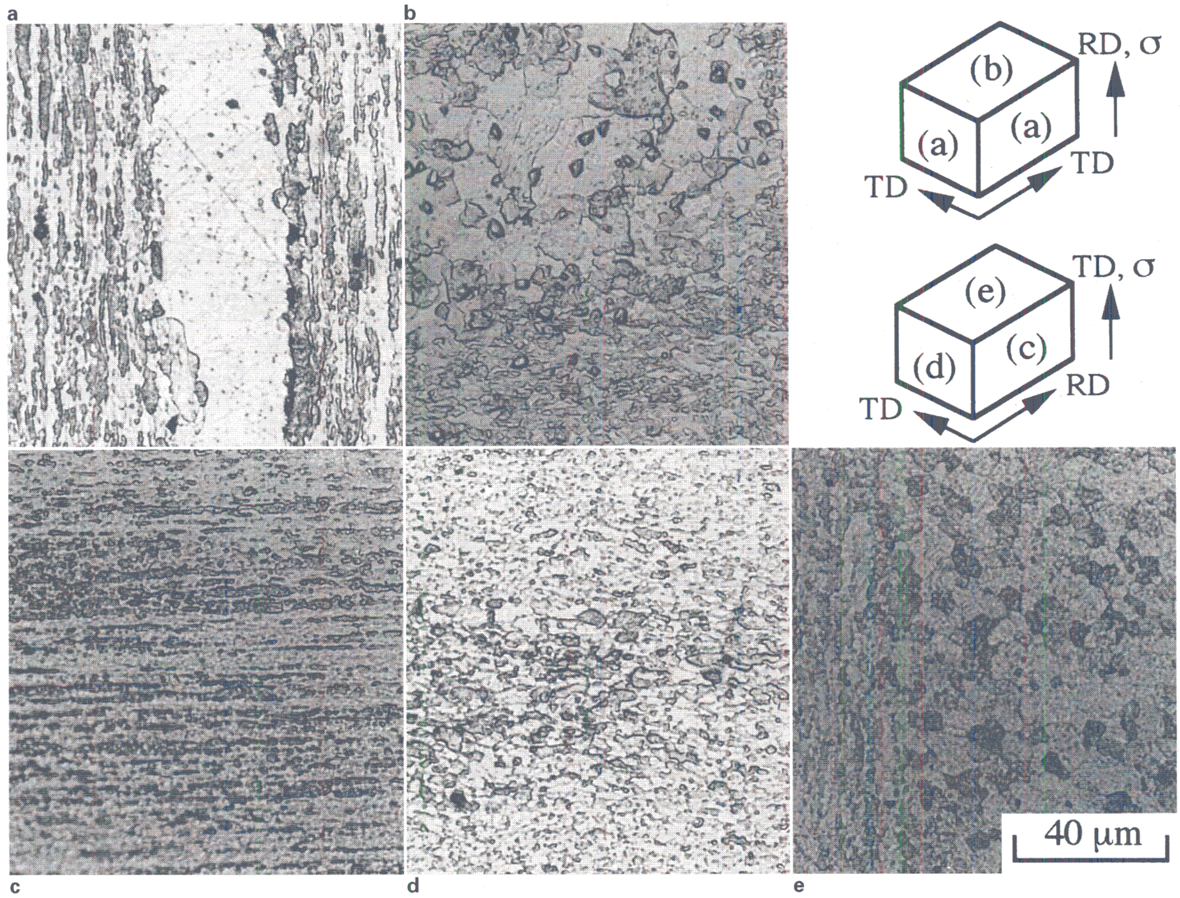
a, b compressed along rolling direction; *c–e* compressed normal to rolling direction

4 Micrographs illustrating hot deformed microstructures of MA957 steel compressed at 950°C



a, b compressed along rolling direction; *c–e* compressed normal to rolling direction

5 Micrographs illustrating hot deformed microstructures of MA957 steel compressed at 1000°C



a, b compressed along rolling direction; *c–e* compressed normal to rolling direction

6 Micrographs illustrating hot deformed microstructures of MA957 steel compressed at 1050°C

in Table 2 show that ε_c and ε_x both decrease with increasing hot deformation temperature. Recrystallisation at lower temperatures requires a longer time to be completed, so that the observed trend in ε_x is also reasonable.

Similar changes in ε_c and ε_x with the deformation temperature were found for deformation normal to the rolling direction, except the sample deformed at 950°C (Table 2). The reason for this discrepancy is unclear but Fig. 7*b* shows an unusual deformation curve with a large 'yield drop'.

From Fig. 7, it seems that there are two stages of dynamic recrystallisation for MA956, hot compressed both along the rolling direction and normal to the rolling direction. It is suspected that this non-uniformity of the recrystallisation behaviour is related to corresponding heterogeneities in the dispersoid distributions.

The investigation of hot deformed MA957 steel was carried out under the same conditions as MA956. However, owing to the poor workability of this steel, serious cracking was generated during hot deformation. Hence, the curves for MA957 shown in Fig. 8 should be treated with caution. These are from the samples compressed along the rolling direction, and normal to the rolling direction, for a variety of temperatures. There is an obvious decrease in stress with increasing compression strain due to the dynamic recrystallisation, as shown in Fig. 8*c* and *e*. In addition, the strength decreases with increasing temperature. The monotonic decrease in stress as a function of strain is due to the cracking generated during hot deformation.

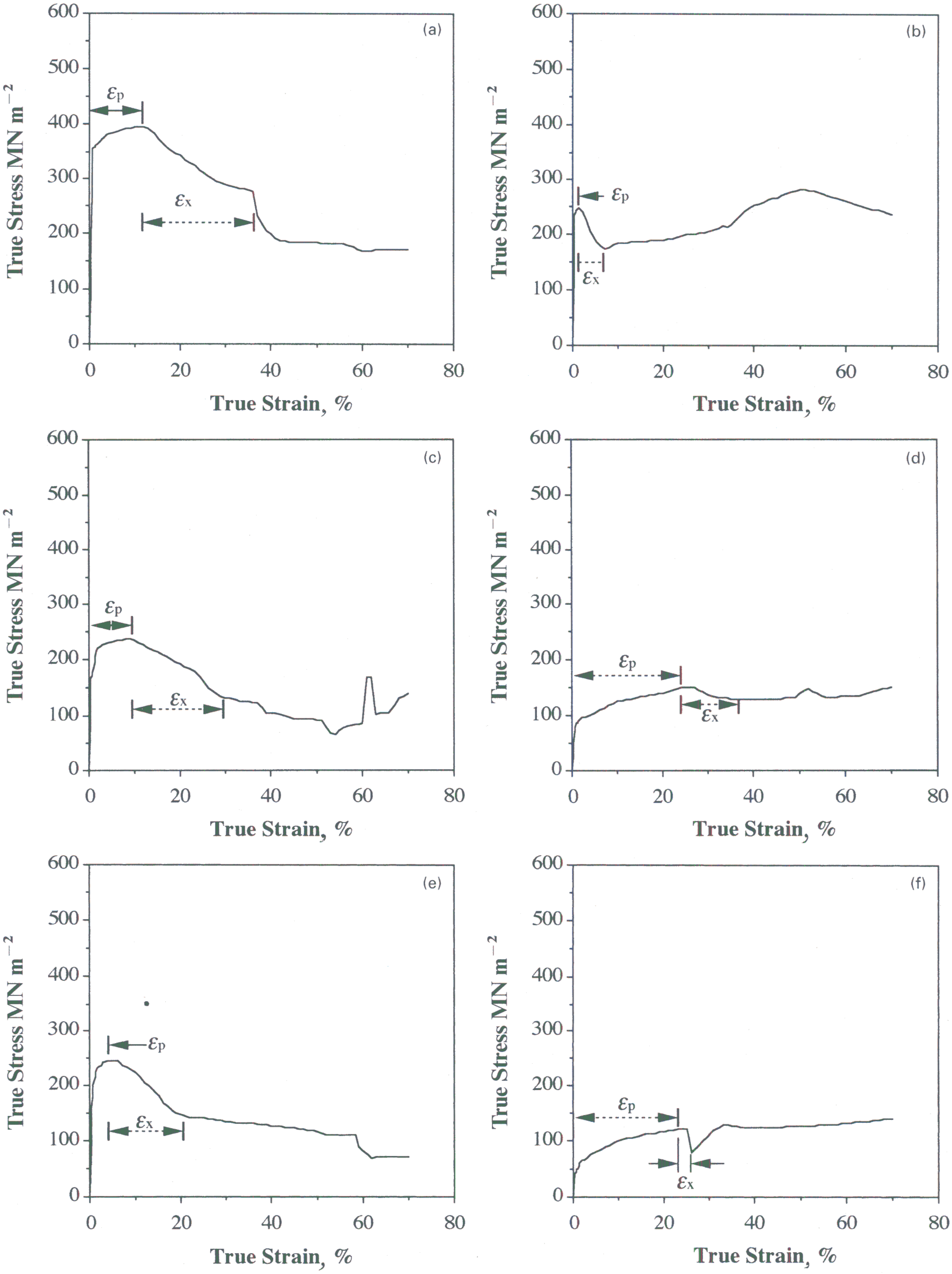
Table 3 Proof strength (0·2% of offset) of hot compressed MA956 and MA957 steels at various deformation temperatures

	Proof strength, MN m^{-2}		
Steel	950°C	1000°C	1050°C
MA956 RD	355	208	178
MA956 TD	225	80	48
MA957 RD	>530	430	380
MA957 TD	273	165	70

RD compressed along rolling direction; TD compressed normal to rolling direction.

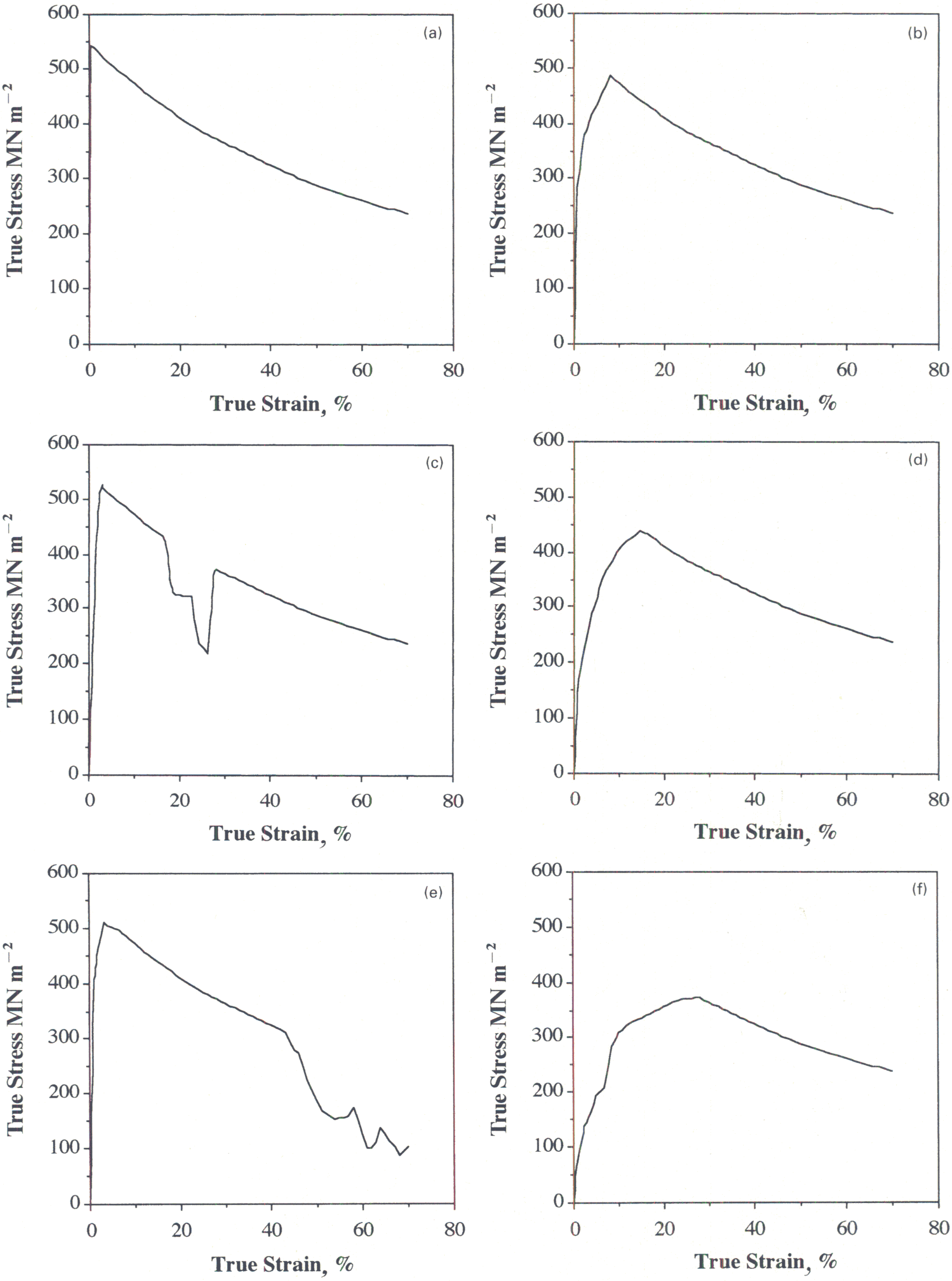
Whittenberger investigated the hot working behaviour of MA956 and reported that it has poor ductility in the temperature range 1144–1477 K (Ref. 17). It is said that MA956 deforms via a crack nucleation and growth mechanism within this temperature range, eventually leading to sudden fracture. It is demonstrated in the present work that MA957 has poor hot workability compared with MA956. Consistent with the present work, Alamo *et al.* reported a higher ductility in MA956 than MA957 (Ref. 18). In their work, the peak value of elongation was found to be at a test temperature of ~650°C.

The yield strength of MA957 is always found to be higher than that of MA956 (*see* Table 3 and Fig. 9). Consistent with this, Alamo *et al.* also reported that MA957 always has a higher hardness and strength than MA956 (Ref. 18). The anomalously large strength of MA957 is not expected at first. Strengthening factors include grain size effects, second phase particles, dislocations, and solid solution composition.[19] Both MA956 and MA957 have submicrometre grains (~0·3 μm) and high dislocation densities in the as received state.[7,8] However, MA956 contains a larger yttria and solute concentration and should therefore be stronger than MA957. Nevertheless, the hardness and yield strength of MA957 are larger, indicating a higher degree of deformation in the microstructure of the



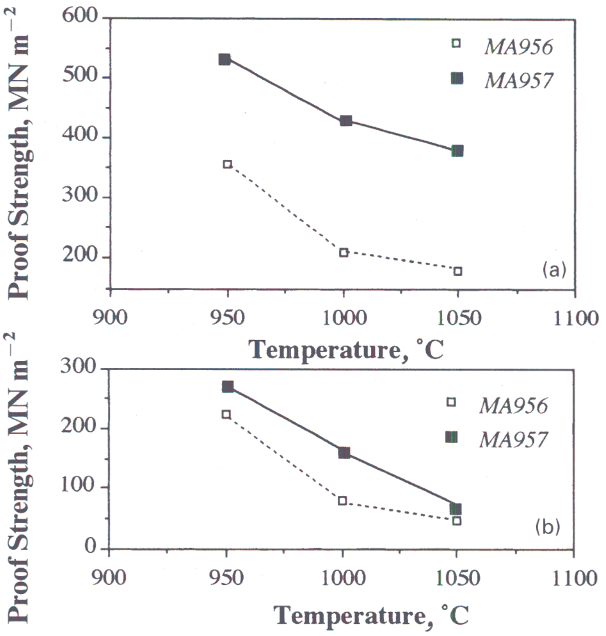
a, *c*, *e* compressed along rolling direction; *b*, *d*, *f* compressed normal to rolling direction

7 Stress–strain curves for MA956 as deformed at *a*, *b* 950, *c*, *d* 1000, and *e*, *f* 1050°C



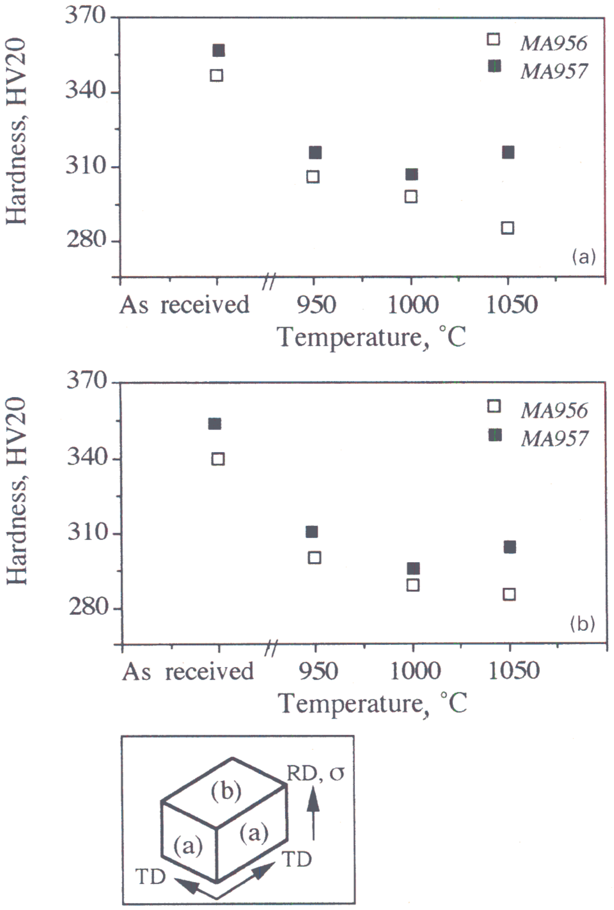
a, c, e compressed along rolling direction; *b, d, f* compressed normal to rolling direction

8 Stress–strain curves for MA957 as deformed at *a, b* 950, *c, d* 1000, and *e, f* 1050°C



a compressed along rolling direction; *b* compressed normal to rolling direction

9 Comparison of proof strength (0·2%) of MA956 and MA957 at both ambient temperature and high temperature



a section containing stress axis and rolling direction; *b* section normal to stress axis and rolling direction

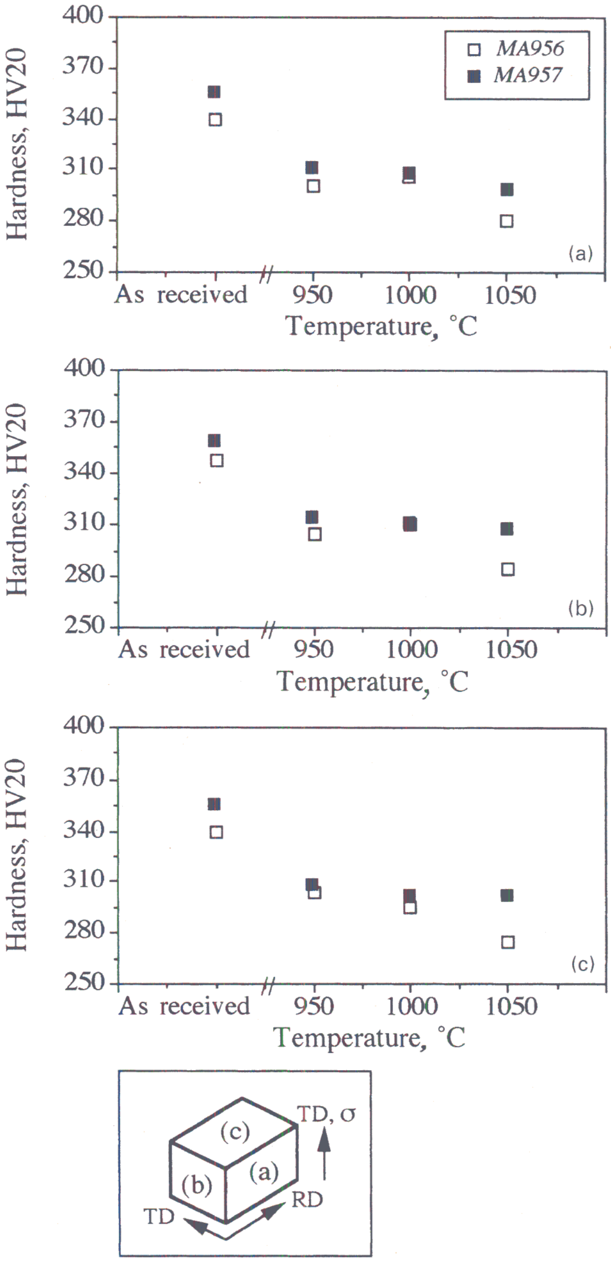
10 Recorded hardness changes in hot deformed MA956 and MA957 steels as compressed along rolling direction for variety of deformation temperatures

as received state. This is consistent with the greater stored energy.[8–10] Thus, MA957 probably lacks ductility relative to MA956 because of its higher starting strength.

MECHANISM FOR DYNAMIC RECRYSTALLISATION

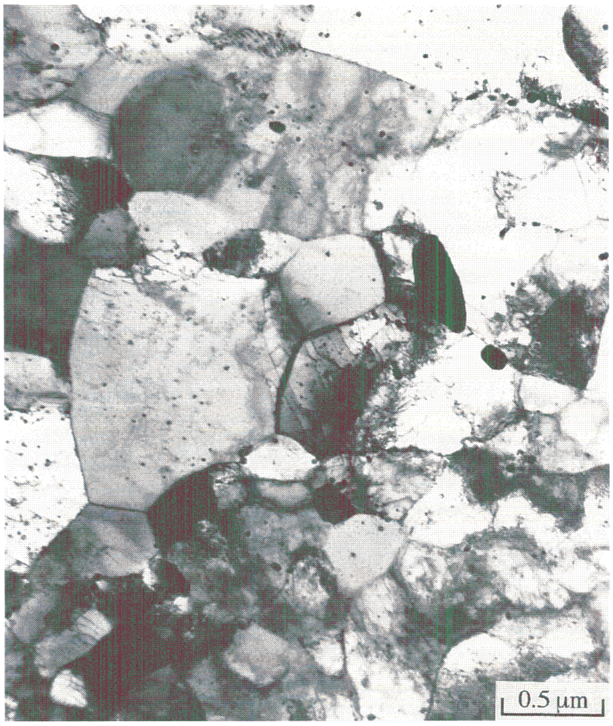
Static recovery and recrystallisation occur in the absence of applied stress. The adjective 'dynamic' on the other hand implies that recovery and recrystallisation processes occur during deformation.

Whether only dynamic recovery, or dynamic recovery and recrystallisation, are observed during hot working depends on the stresses and the strain rates involved. Larger strains result in a greater tendency for recrystallisation. Materials that do not dynamically recrystallise exhibit rapid recovery rates, and thus a dislocation density

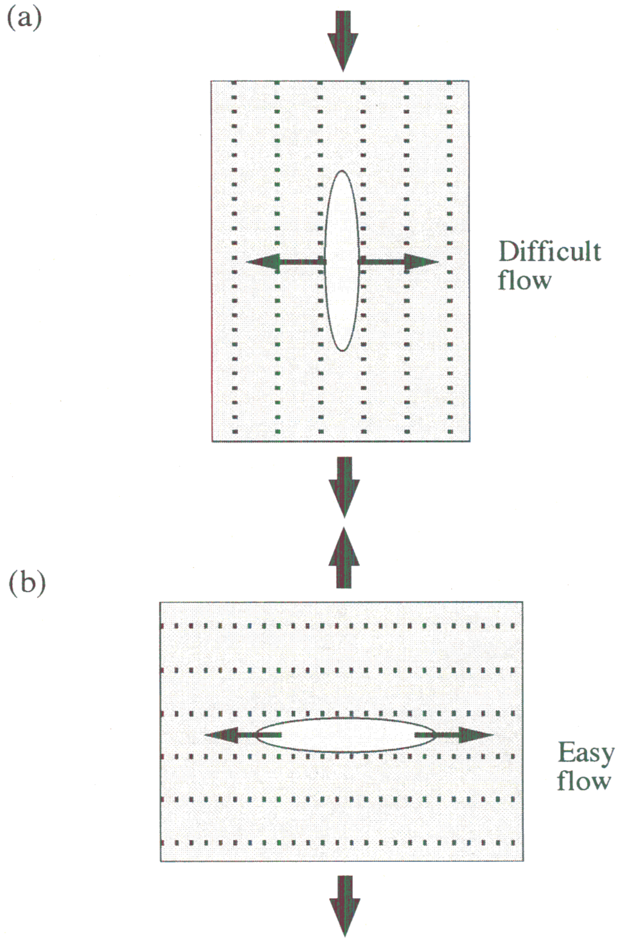


a section containing stress axis and rolling direction; *b* section containing stress axis but normal to rolling direction; *c* section containing rolling direction but normal to stress axis

11 Recorded hardness changes in hot deformed MA956 and MA957 steels as compressed along rolling direction for variety of deformation temperatures



12 Micrograph obtained using transmission electron microscopy (TEM) showing partially recrystallised microstructure in MA956 steel as hot deformed at 1000°C (sample compressed along rolling direction)



a compressed along rolling direction; *b* compressed normal to rolling direction

13 Schematic diagrams illustrating effect of dispersoid alignment on material flow with change of deformation configuration

and configuration sufficient to nucleate and drive the recrystallisation process is not developed. This includes materials with a high stacking fault energy such as aluminium, ferritic iron, and zirconium. By contrast, those materials with a low stacking fault energy tend to dynamically recrystallise more readily. Thus, nickel and its alloys, austenitic iron, copper, and brass recrystallise dynamically.[15]

The present results indicate that the ferritic steels undergo dynamic recrystallisation and this may be considered inconsistent with the general trends discussed above. However, mechanically alloyed steels contain an incredible amount of stored energy, the key parameter which determines whether or not dynamic recrystallisation should occur. The softening evident in the as deformed hardness data is also consistent with dynamic recrystallisation (Figs. 10 and 11) and indeed with an increasing fraction of dynamic recrystallisation as the deformation temperature increases. It is noted that MA957 always has a higher hardness than MA956 in both the as received and deformed states. However, there is no significant difference in the hardnesss data for the two test orientations (*compare* Figs. 10 and 11), because a hardness indent involves a three-dimensional state of stress. Moreover, transmission electron metallographic observations which revealed the partial recrystallised microstructure (Fig. 12) confirmed the optical observations.

There are two features which need explanation: (i) the fact that recrystallisation occurs more readily during deformation when compared with static heat treatment, and (ii) the differences in recrystallisation behaviour as a function of the orientation of the deformation axes with respect to the rolling direction. The first observation probably cannot be attributed to any increase in stored energy as a consequence of deformation because the materials already contain an unusually large amount of excess energy. It is likely instead that the stress assists the migration of the glissile components of subgrain or grain boundaries, thereby permitting easier recrystallisation. This would be true irrespective of the orientation of the strain axis, since it is shear stresses which are important in the movement of dislocations.

From Table 3, the yield stress is smaller when the compression axis is normal to the rolling direction. This is a very useful result because in compression, a net transport of material in a direction normal to the compression axis may be expected. When this latter direction is also parallel to the rolling direction, the net flow should be easier since the particles are aligned parallel to the flow direction (Fig. 13).

For the same reason, the stress needed to overcome the barriers should be less sensitive to temperature when the compression axis is normal to the rolling direction, since the boundaries are essentially migrating in particle free regions. It should be noted that work hardening is also more rapid for this deformation configuration. This is also expected since when the particles are aligned to the compression axis, it is predominantly the need to overcome the particles that determine yield.

Conclusions

The hot deformation behaviour and its related effect on deformed microstructures in MA956 and MA957 steels has been investigated. It is found that dynamic recrystallisation occurs during hot deformation, causing the softening. The occurrence of dynamic recrystallisation in mechanically alloyed steels is attributed to the high stored energy in the as received state. Stress appears to assist the migration of the glissile components of subgrain or grain boundaries, thereby permitting easier recrystallisation. In addition, the dynamically recrystallised microstructure shows an anisotropic character.

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