

DIRECTIONAL RECRYSTALLISATION & ITS EXPLOITATION
IN MECHANICALLY ALLOYED METALS

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ABSTRACT

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 strengthen the consolidated product. Significant quantities of iron and nickel-base alloys with unusual properties are produced commercially using this process.

The total true strain during mechanical alloying can be as large as 9; there is proof that this leads to mixing on an atomic scale and to the development of a uniform grain structure which is submicron in size. The large stored energy, approaching 1 J g^{-1} , ought to make it easy to induce recrystallisation, but in practice the alloys fail to recrystallise except at very high temperatures close to melting. On the other hand, the recrystallisation temperature can be dramatically reduced by a small additional deformation prior to heat treatment. When recrystallisation does occur, the grains that evolve can be enormous and anisotropic in shape. In some cases, the principal growth direction can be controlled by applying thermal gradients; in other cases that direction obstinately remains parallel to the extrusion direction, irrespective of the nature of the heat treatment. These and other anomalous aspects of the recrystallisation behaviour of these remarkable materials are assessed and discussed in the context of applications.

1. INTRODUCTION

It is a privilege to be able to present at this conference, which marks the formal retirement of Neils Hansen. I first met him in 1986 in Cambridge when he was working with Brian Ralph on an *Acta Metallurgica* paper on the recrystallisation of copper. My next contact was many years later, when I wrote asking for samples of Al/SiC composites, which he so generously delivered without any hesitation. But more than anything else, I have benefited from his science and continue to do so; we are currently working together on some intriguing microstructures which form the subject of this paper.

An alloy can be created without melting, by violently deforming mixtures of different powders, Fig. 1 (Benjamin, 1970; Gessinger, 1984; Hack, 1984). 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

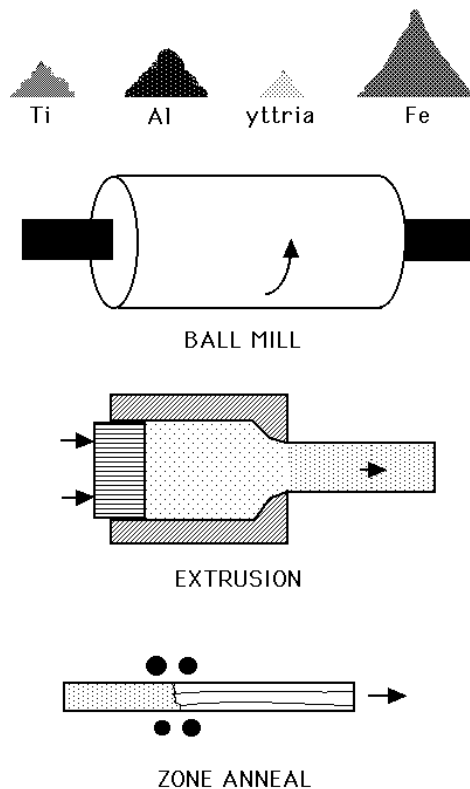


Fig. 1: The manufacture of mechanically alloyed metals for engineering applications. The elemental powders/master-alloys/oxides are milled together to produce solid solutions with uniform dispersions of oxide particles. This powder is consolidated and the resulting material heat-treated to achieve a coarse, directional grain structure.

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.

The chemical compositions of some of the commercial alloys produced using this method are listed in Table 1. They all contain chromium and/or aluminium for corrosion and oxidation resistance, and yttrium or titanium oxides for creep strength. Yttrium oxide cannot be introduced into either iron or nickel by any method other than mechanical alloying; indeed, this was the motivation for the original work by Benjamin (1970).

The alloys have wide-ranging applications, mostly in circumstances where creep strength and oxidation resistance are the paramount design criteria. There are also potential applications of the iron-base alloys in the nuclear-power generation industry. The ferritic state makes them less susceptible to radiation induced swelling. MA957 and DT2203Y05 are therefore designed for use in a liquid sodium environment at temperatures of the order of 700 °C. Both have a high void swelling resistance, and a low carbon concentration in order to avoid the formation of titanium carbides. The titanium is meant to combine with chromium, molybdenum and iron to form a stable body-centred cubic FeCrTiMo intermetallic χ -phase during ageing at around 800 °C, which can further boost the creep strength. The rupture strength of ODM751 is larger than that of MA956 (Jaeger and Jones, 1991). ODM751 has an additional 1.5 wt.% Mo which, either via the χ -phase or through solid solution strengthening, presumably adds to the creep strength of ODM751. However, the results are confusing because PM2000, which does not contain molybdenum, virtually matches the rupture strength of ODM751.

Typical applications of PM2000 include use in furnace construction as shields or carrier systems;

Table 1: Compositions (wt%) of some typical alloys. MA758 and PM1000 are nickel base mechanical alloys without γ' strengthening. The compositions of ODM061, DT and DY are from Regle (1994), as are the nitrogen data for MA956 and MA957. The compositions of PM2000 and PM2010 are from Krautwasser *et al.* (1994).

Fe-base	C	Cr	Al	Mo	Ti	N		Ti ₂ O ₃	Y ₂ O ₃	Fe
MA957	0.01	14.0	–	0.3	1.0	0.012		–	0.27	Balance
DT2203Y05		13.0	–	1.5	2.2			–	0.5	Balance
ODM 331		13.0	3.0	1.5	0.6			–	0.5	Balance
ODM 751		16.5	4.5	1.5	0.6			–	0.5	Balance
ODM 061		20.0	6.0	1.5	0.6			–	0.5	Balance
MA956	0.01	20.0	4.5	–	0.5	0.045		–	0.50	Balance
PM2000	< 0.04	20.0	5.5		0.5			–	0.5	Balance
PM2010	< 0.04	20.0	5.5		0.5			–	1.0	Balance
DT		13.0	–	1.5	2.9			1.8	–	Balance
DY		13.0	–	1.5	2.2			0.9	0.5	Balance
Ni-Base	C	Cr	Al	Ti	W	Fe	N	Total O	Y ₂ O ₃	Ni
MA6000	0.06	15.0	4.5	2.3	3.9	1.5	0.2	0.57	1.1	Balance
MA760	0.06	19.5	6.0	–	3.4	1.2	0.3	0.6	1.0	Balance
MA758	0.05	30.0	0.3	–	0.5	–	–	0.37	0.6	Balance
PM1000		20.0	0.3	0.5		3.0			0.6	Balance

in the glass industry as stirrers or plungers in molten glass; in the combustion of waste materials; as thermocouple protection tubes; in high temperature testing-equipment; as burner tubes; a variety of applications in automotive diesel engines. Similarly, PM1000 is used in the manufacture of rotating discs for glass fibre production; for high temperature screws and fasteners; as face sheets in thermal protection panels; for space and aerospace engineering in general (Miller, 2000). MA956 sheet has been used in combustion chambers for turbines, for burner hardware in coal and oil burning power stations; MA974 for brazed nozzle guides in aeroengines (Hack, 1984).

2. INITIAL ATOMIC STRUCTURE

The intense deformation associated with mechanical alloying can force atoms into phases where they may not prefer to be at equilibrium. This is known as “solute-trapping” and can influence the stability of the alloy. The atomic structure of solid solutions in commercially important metals formed by the mechanical alloying process has been studied using field ion microscopy and the atom-probe (Chou *et al.*, 1993a).

A solution which is homogeneous will nevertheless exhibit concentration differences of increasing magnitude as the size of the region which is chemically analysed decreases (Landau and Lifshitz, 1958; Russell, 1971). These are random fluctuations which obey the laws of stochastic processes, and represent the real distribution of atoms in the solution. These equilibrium variations cannot usually be observed directly because of the lack of spatial resolution and noise in the usual microanalytical techniques. The fluctuations only become apparent when the resolution of chemical analysis falls to less than about a thousand atoms block. The atom probe technique collects the experimental data on an atom by atom basis. The atom by atom data can be presented at any

block size.

Fig. 2 illustrates the variation in the iron and chromium concentrations in fifty atom blocks, of the ferrite in *MA956*. There are real fluctuations but further analysis is needed to show whether they are beyond what is expected in homogeneous solutions

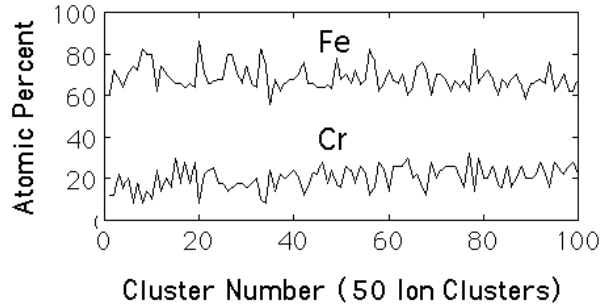


Fig. 2: The variation in the iron and chromium concentrations of 50 atom samples of MA956 (Chou *et al.*, 1993a).

For a random solution, the distribution of concentrations should be binomial since the fluctuations are random; any significant deviations from the binomial distribution would indicate either the clustering of like-atoms or the ordering of unlike pairs.

The frequency distribution is obtained by plotting the total number of composition blocks with a given number of atoms of a specified element against the concentration. Fig. 3 shows that the experimental distributions are essentially identical to the calculated binomial distributions, indicating that the solutions are random.

The atom probe data can be analysed further if it is assumed, fairly reasonably, that the successive atoms picked up by the mass spectrometer were near neighbour atoms in the sample. Successive atoms which are identical then represent bonds between like atoms *etc.* so that pair probabilities used in solid solution theory can be measured experimentally. These data can be compared against calculated pair probabilities. Thus, in a random $A-B$ solution, the probability p_{AB} of finding $A-B$ or $B-A$ bonds is given by $p_{AB} = 2x_Ax_B$ where x_i is the atom fraction of element i . Similarly, $p_{AA} = x_A^2$ and $p_{BB} = x_B^2$. Table 2 shows the excellent agreement between the experimentally measured pair probabilities and those calculated assuming a random distribution of atoms.

Table 2: Pair probability analysis. B is the solute element (such as Cr or Al) whereas A represents the remainder of atoms. N represents the total number of atoms included in the analysis. The calculations assume a random solution.

Alloy	Element	p_{AA}	p_{AB}	p_{BB}	N
<i>MA956</i>	Cr (Measured)	0.638	0.324	0.038	12168
<i>MA956</i>	Cr (Calculated)	0.637	0.322	0.041	
<i>MA956</i>	Al (Measured)	0.808	0.184	0.008	12168
<i>MA956</i>	Al (Calculated)	0.833	0.160	0.008	

This does not mean that the solutions are thermodynamically ideal, but rather that the alloy preparation method which involves intense deformation which forces a random dispersal of atoms.

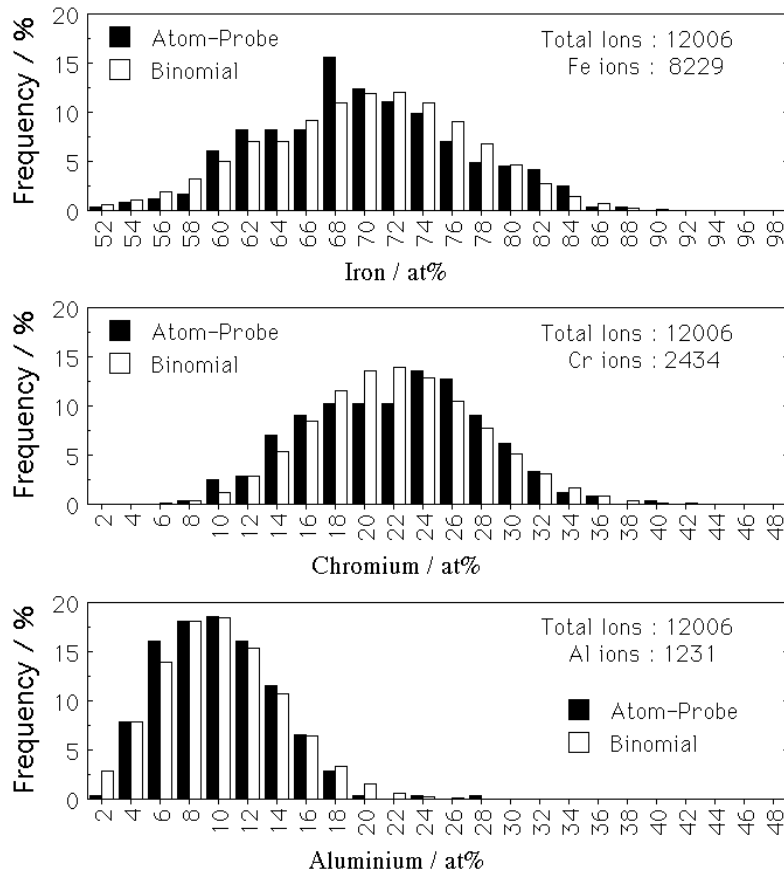


Fig. 3: Frequency distribution curves for iron, chromium and aluminium in mechanically alloyed MA956, (Chou *et al.*, 1993a).

Indeed, Fe–Cr solutions are known to deviate significantly from ideality, with a tendency for like atoms to cluster (Uemori, 1988; Miller, 1988). Thus, it can be concluded that the alloy is in a mechanically homogenised nonequilibrium state, and that prolonged annealing at low temperatures should lead to, for example, the clustering of chromium atoms. In recent atom–probe work, Read and Hono (1996) have shown this to happen in MA956, during prolonged ageing at 475 °C.

3. INITIAL MICROSTRUCTURE

Immediately after the mechanical alloying process, the powders have a grain size which can be as fine as 1–2 nm locally (Jaeger & Jones, 1991–1994). This is hardly surprising given the extent of the deformation during mechanical alloying, with true strains of the order of 9, equivalent to stretching a unit length by a factor of 8000. The consolidation process involves hot extrusion and rolling at temperatures of about 1000 °C, which causes recrystallisation into a sub–micron grain size. It is known that during the course of consolidation, the material may dynamically recrystallise several times (Jaeger & Jones, 1991–1994). It should be emphasised that the sub–micron grains are not low–misorientation cell structures, but true grains with large relative misorientations (Bhadeshia, 1997). Subsequent heat–treatment leads to primary recrystallisation into a very coarse grained microstructure whose dimensions may be comparable to those of the sample.

The extrusion and hot–rolling process leaves the microstructure of iron–base alloys in a cold–deformed state with elongated grains and a dislocation density of about 10^{15} m^{-2} (Little *et al.*, 1991). By contrast, the nickel–base alloys have fine, clean, equiaxed grains in a primary recrystallised state. The deformation also causes an alignment of dispersoids along the working direction,

particularly in the iron–base alloys (Baloch, 1989; Bhadeshia, 1997). This alignment reflects inhomogeneities in the fabrication process arising at the single particle level (Jaeger & Jones, 1992) and below.

The reason why the yttrium oxide particles are aligned along the extrusion direction in iron–base alloys but not in the nickel variants has yet to be solved. We shall see that there are consequences on the recrystallisation behaviour.

4. RECRYSTALLISATION

4.1 Why Recrystallise? The alloys are very hard in the consolidated state (Table 3) and the fine grain structure is not appropriate for elevated temperature applications. They are therefore given a recrystallisation heat treatment which leads to an enormous coarsening of the microstructure and a reduction in the amount of grain surface per unit volume by 2–3 orders of magnitude.

Table 3: Vicker’s Hardness data for commercial alloys, before and after recrystallisation.

Alloy	HV, before recrystallisation	HV, after recrystallisation
PM2000	400	290
MA957	400–410	230–240
MA956	350–390	225–245
MA956 Sheet	410	250
PM1000	550	250
MA6000	645	500–520
MA760	720–790	500–515
MA758	405	214

4.2 Columnar Grain Growth. One of the most remarkable features of the alloys listed in Table 1 is that they can recrystallise into a columnar grain microstructure; the columnar grains can be as long as the sample size and some hundreds of microns in width (*e.g.* Hack, 1984). The grains grow anisotropically because of the particle alignment discussed in the previous section. Hence, iron–base alloys, which have strong particle alignment, develop columnar grains parallel to the extrusion direction irrespective of whether they are isothermally annealed, zone–annealed or cross annealed (*i.e.* zone annealed along a direction normal to the extrusion direction). The distribution of particles is more isotropic in the nickel alloys; consequently, the direction of columnar grain growth can be controlled by the orientation of the temperature gradient during zone annealing. Indeed, equiaxed coarse grained secondary recrystallised microstructures can be readily generated either by isothermal annealing or by zone annealing at high speeds (Baloch & Bhadeshia, 1991).

One feature not explained by particle alignment is the coarse scale of the columnar microstructure, which indicates some difficulty in nucleating recrystallisation.

Notice that the limiting grain size, obtained by balancing the particle pinning against the force driving grain boundary motion, is very large. Although particle pinning has a role in the development of anisotropic recrystallisation microstructures, it does not control the scale of the grains that grow. The scale seems to be determined by the nucleation process.

4.3 Recrystallisation Temperature. Another strong indication of difficulties in nucleation is that recrystallisation occurs at exceptionally high homologous temperatures, of the order of 0.9 of the melting temperature (T_M). This contrasts with 0.6 T_M in ordinary variants of similar metallic alloys. Strangely enough, the mechanically alloyed metals contain a more stored energy than conventional materials which recrystallise at lower temperatures (Table 4). Notice that the whereas the stored energy is large in the context of deformed metals, it is quite small when compared with other metastable materials such as amorphous metals or highly supersaturated solutions where solutes are trapped.

Table 4: Enthalpy of Recrystallisation (Chou & Bhadeshia, 1993b, 1994; Murakami *et al.*, 1993; Sha & Bhadeshia, 1994). The last two columns place the excess energy in context, with some data are from Turnbull (1981). T_M is the melting temperature and R the gas constant. For details see Bhadeshia (1997).

Alloy	Stored Energy / J g^{-1}	Materials Example	Stored Energy / RT_M
MA957	1.0	Supersaturated Solution	< 1
MA956	0.4	Intermetallic Compounds	< 0.5
MA956 sheet	$\simeq 0.4$	Amorphous Solids	< 0.5
MA6000	0.6	Compositionally Modulated Films	< 0.1
MA760	1.0	Interphase Dispersions	< 0.1
MA758	0.3	Commercial Mechanical Alloys	< 0.005

The following mechanisms have in the past been proposed to explain the high recrystallisation temperatures (T_R) in the range 1300–1450 °C (see Bhadeshia, 1997, for original references):

- (a) In the nickel alloys, γ' precipitates were considered to inhibit recrystallisation. However, T_R is sometimes less than the temperature at which γ' dissolves and the iron–base alloys do not contain γ' and yet show exceptionally large values of T_R .
- (b) Solute–drag limits grain boundary mobility until a temperature is reached where the solute atmosphere evaporates. There is no direct evidence to support this, and the idea is inconsistent with the observations that the recrystallisation temperature can be greatly reduced by a low–temperature heat treatment in the recovery range (Chou and Bhadeshia, 1993b), or by other processing (Alamo *et al.*, 1992). In any event, solute–drag is a feature of all commercial alloys which are impure so it is hard to see why the MA–ODS alloys should have especially high recrystallisation temperatures. There is a claim that molybdenum segregates to grain boundaries in MA957 (Evens *et al.*, 1992), and that the low diffusivity of molybdenum inhibits grain boundary mobility. In fact, molybdenum diffuses faster in iron than iron itself (Fridberg *et al.*, 1969). Miodownik *et al.* (1993) found excess titanium at a grain boundary in an extruded and heat treated bar of MA957 but the experiment should have been carried out on an unrecrystallised sample since it is segregation at the recrystallisation front which matters.
- (c) It has been argued that the particles of yttrium oxide or various yttria/alumina compounds (spinel) must coarsen before recrystallisation can occur. However, as pointed out earlier, the limiting grain size is in fact very large. Furthermore, T_R is found to be insensitive to changes in the particle pinning force (Murakami, 1993).
- (d) It has been suggested that a critical value of kinetic strength is required before the onset of recrystallisation (Baloch and Bhadeshia, 1991). Kinetic strength is the product

$t \exp\{-Q/RT\}$ for an isothermal heat treatment, where t is the heat treatment time and Q is an empirical activation energy. The concept is readily generalised for anisothermal processes by integrating over time and temperature. But there is no explanation in it for the particular kinetic strength needed, nor for the magnitude of Q which turns out to be some ten times greater than that for the self diffusion of the base element. More fundamental approaches using conventional nucleation and growth theory in combination with the Johnson–Mehl–Avrami method for overall transformation kinetics, also reveal a very large activation energy (Murakami, 1993; Sha & Bhadeshia, 1997).

The difficulties are resolved as follows. Nucleation in the present context begins by the bowing of a grain boundary. This normally is straightforward because the size of the boundary perturbation is small when compared with the grain size. However, with very small grains, the boundary junctions themselves act as severe pinning lines restricting bowing (Fig. 4). This leads to an enormous activation energy Q for the nucleation of recrystallisation, ten times larger than Q_D which is the activation energy for self diffusion (Murakami, 1993; Sha & Bhadeshia, 1997). Q is reduced if a few grains happen to be larger; indeed, any nonuniformity introduced into the microstructure, for example by inhomogeneous deformation, will lead to a large decrease in T_R (see Capdevila and co-workers, these proceedings).

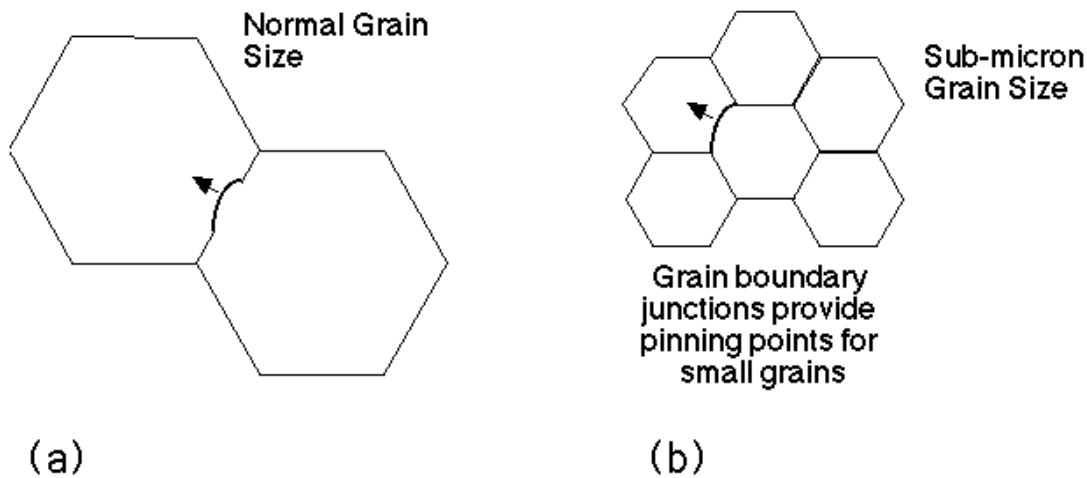


Fig. 4: (a) Grain boundary bulging leading to the nucleation of recrystallisation can occur readily when the grain junctions are widely spaced, at distances greater than the critical bulge size. (b) With sufficiently fine grains in the unrecrystallised microstructure, the grain junctions themselves are pinning points, making it very difficult to form recrystallisation nuclei.

This model suggests that the individual grains cannot be considered to be topologically independent when the grain size becomes small. It explains the observed high values of T_R which are independent of alloy type; the common feature of the iron and nickel base alloys is the fine grain structure following the mechanical alloying process. It is predicted correctly that the recrystallisation temperature should decrease if the the stored energy is reduced by a low-temperature heat treatment which leads to uniform grain coarsening. Since recrystallisation should eventually become more difficult as the stored energy is reduced, the curve of recrystallisation temperature versus grain size (or stored energy) should show a minimum. This too has been verified experimentally (Sha & Bhadeshia, 1997).

5. CRYSTALLOGRAPHIC TEXTURE

The major components of crystallographic texture in the mechanically alloyed steels are $\{001\}$

$\langle 110 \rangle$, $\{111\} \langle \bar{1}10 \rangle$, which belong to the so-called α and γ fibres in crystal orientation distribution space (Alamo *et al.*, 1992; Chou & Bhadeshia, 1993c). This applies both before and after recrystallisation; the latter simply changes the strength of the texture components relative to a random distribution of orientations. For reasons which are not obvious, recrystallisation seems easiest whenever the $\{111\} \langle \bar{1}10 \rangle$ is prominent (Table 5). Thus, MA956 prior to recrystallisation is rich in this particular component whereas MA957 is not, probably due to the presence of austenite in MA957 at the fabrication temperature (Chou & Bhadeshia, 1993b). The former has a lower T_R compared with the latter, even though MA957 has a higher stored energy and lower yttria content than MA956. However, if MA957 is pre-annealed (*i.e.* heat-treated without recrystallisation) in order to make its texture comparable to that of MA956, then its recrystallisation temperature drops in spite of the reduction in stored energy due to the preannealing heat-treatment.

Table 5: Crystallographic texture and recrystallisation (Chou & Bhadeshia, 1994).

	As-Received MA956	As-Received MA957	Preannealed MA957
Stored Energy J g^{-1}	0.4	1.00	0.70
Recrystallisation Start	1273 °C	1429 °C	1362 °C
Recrystallisation Finish	1334 °C	1447 °C	1382 °C
Texture Summary	$\{111\} \langle \bar{1}10 \rangle$ $\{001\} \langle 110 \rangle$	Random Random	$\{111\} \langle \bar{1}10 \rangle$ $\{001\} \langle 110 \rangle$
Recrystallised Grains	Highly anisotropic	anisotropic	Equiaxed fine

Evens *et al.* (1992) have also conducted microtextural measurements and interpreted their recrystallisation results in terms of the mobilities of grain boundaries with different orientations.

6. GRAIN BOUNDARY TOPOLOGY

Surface tension favours smooth boundaries, but the presence of pinning particles in MA-ODS alloys gives the grain boundaries a roughness related to the particle spacing (Murakami *et al.*, 1992). The phenomenon depends also on the stored energy. Grain boundaries become more ragged as the stored energy is reduced; this is because the force driving the recrystallisation front is reduced relative to the pinning force due to oxide particles. Consequently, smoother boundaries are obtained for large values of stored energy.

7. GRAIN SHAPE

We have seen that anisotropic grain structures result either from a zone annealing heat treatment which favours growth along the moving temperature gradient, or due to the aligned dispersoids in the microstructure.

The nickel alloys have a more uniform distribution of dispersoids. Thus, MA6000, when isothermally annealed, often recrystallises into an equiaxed grain structure. The same alloy will recrystallise directionally when zone annealed. The orientation of the columnar grains relative to the extrusion direction can be altered by changing the zone annealing direction (cross annealing). Zone annealing at high speeds can even cause a transition from a columnar to an equiaxed grain microstructure (Baloch & Bhadeshia, 1991).

The nickel-base alloy MA760 has is similar in its recrystallisation behaviour to MA6000, but with a stronger alignment of particles along the extrusion direction. Thus, isothermal annealing does not

lead to equiaxed grains, but cross annealing can alter the direction of the columnar recrystallised grains.

By contrast, the yttria particles in the iron alloys are strongly aligned along the extrusion direction. Simple, isothermal annealing therefore causes the development of columnar grains parallel to the extrusion direction. MA957 with its low yttria content recrystallises into stubby columnar grains following isothermal heat treatment. No amount of cross annealing or any other heat treatment has succeeded in causing a change in the direction of columnar grain growth in iron alloys. The development of columnar grains during secondary recrystallisation due to a nonuniform dispersion of HfC or TiB₂ has also been demonstrated in a NiAl intermetallic matrix (Lee *et al.*, 1994).

8. PREANNEALING EFFECTS

Preannealing describes heat treatment at a temperature which is below T_R , but high enough cause recovery with an accompanying reduction in stored energy.

Mild preannealing ($\simeq 1150^\circ\text{C}$) has little or no effect on subsequent recrystallisation in MA957 (Chou & Bhadeshia, 1993b). An increase in the preannealing time causes a transition from a coarse columnar grain structure to one which is fine and equiaxed. The reduction in stored energy also reduces the grain boundary velocity, permitting nucleation in many locations thereby giving an equiaxed grain structure.

Continued preannealing causes the development of a bimodal equiaxed grain structure. This is because there is an inhomogeneous distribution of pinning particles in the alloy. The now substantial reduction in stored energy retards recrystallisation more in some regions compared with others which are less strongly pinned. For MA957 the preannealing time at 1150°C is in excess of 160 hours for this condition to be reached.

Further preannealing leads to such a large reduction in the stored energy that subsequent recrystallisation is completely suppressed.

It is much more difficult to similarly control the grain structure of MA956 using preannealing heat treatments. Grain refinement certainly occurs, as in MA957, but the fine grains tend not to be equiaxed. This may be because MA956 contains a larger concentration of yttria. The anisotropic pinning due to the inhomogeneous distribution of the oxide particles is more difficult to overcome if the fraction of particles is large. It would be very interesting to test this with MA956 containing a smaller quantity of yttria dispersoids.

9. RECRYSTALLISATION DURING DIFFUSION BONDING

Fusion and friction welding processes have not been successful in achieving joint integrity in the MA-ODS alloys. Transient liquid phase (TLP) diffusion bonding using a Fe-13B-9Si wt% interlayer, generally causes the agglomeration of oxide particles during the transient melting part of the bond cycle. The bond-line then tends to be a region of weakness. A further difficulty is that the microstructures from either side of the bond meet and form a discontinuity at the bond interface, which also contributes to a weak bond. TLP bonding is therefore usually carried out with the substrate in the unrecrystallised state, in the hope that a subsequent recrystallisation heat treatment causes the grains to cross the bond interface and hence remove it as a discontinuity.

The agglomeration problem can be mitigated by making the interlayer sufficiently thin, for example by sputtering it on to the bond surfaces (Khan and Wallach, 1996a,b). Recrystallised grains which nucleate at the bond interface, succeed in growing across the TLP bond in MA956, but not in MA957 or PM2000. In MA957 the formation of a premature recrystallisation zone with fine grains at the bond line acts as a barrier to further grain growth across the bond line on subsequent zone annealing. In PM2000, coarse recrystallised grains grow in both directions from the bond interface, but do not cross that interface. The reasons for the different observations in similar alloys are not clear; in MA957, the premature recrystallisation at the bond-line has been attributed to boron

entering the base metal (Khan and Wallach, 1996b) but it is not obvious why this effect is limited to MA957.

10. NOVEL PROCESSING

Mechanical alloying is a difficult process. There are inevitable composition differences between powder particles/aggregates. Jaeger and Jones (1994a,b) demonstrated that these differences develop into bands in the consolidated product which has been extruded. Their study is particularly useful in that the materials were examined both before and after consolidation. When a powder which is inhomogeneous in its dispersoid distribution is fabricated into a tube form, the particles align in concentric cylinders parallel to the tube axis. This in turn leads to an onion-peel type of recrystallised grain structure, which may be of some benefit from the point of view of mechanical properties. Indeed, this work has led to processing technology which aims to produce, in tubes, helical grain structures which optimise both the longitudinal and transverse mechanical properties. More can be found about this and other interesting recrystallisation phenomena in work by Capdevila, Chen and Jones, described in papers published in these proceedings. The effect of cold deformation on the development of microstructure is also discussed, building on some classical work by Regle and Alamo.

11. SUMMARY

Commercial mechanically alloyed, oxide dispersion-strengthened materials have a fascination both in industry and in science. In industry they offer the possibility of metals which can serve under conditions normally reserved for the less reliable structural ceramics. In academia, they have revealed new phenomena about the recrystallisation process, apart from providing some of the most beautiful grain structures visible to the naked eye. To paraphrase, a uniformly fine grain structure finds it incredibly difficult to recrystallise because the individual grains can no longer be considered to be topologically independent. Anything which introduces a heterogeneity in such a microstructure will lead to a reduction in the recrystallisation temperature and a refinement of the grain structure due to increased nucleation.

There are a number of unanswered questions. An explanation is needed for the observed differences in the alignment of particles between iron and nickel alloys; measurements of the velocity of the recrystallisation front during isothermal heat treatment might reveal more about the mechanism by which coarse, columnar grain structures develop; what is the consequence of solute trapping, and the associated stored energy, on the recrystallisation behaviour and long term stability of the iron-base alloys?

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