Directional recrystallisation in Inconel MA 6000 nickel base oxide dispersion strengthened superalloy

The directional recrystallisation process which leads to the growth of highly anisotropic 'new' grains in an oxide dispersion strengthened nickel base superalloy is investigated to elucidate the mechanism of recrystallisation and the process variables important in controlling microstructural evolution. It is found that after extrusion and hot deformation, the alloy consists of an ultrafine equiaxed grain microstructure characteristic of a material which has undergone dynamic primary recrystallisation. Thus, the directional recrystallisation which follows when the hot rolled sample is annealed in a moving temperature gradient (zone annealed) is really a secondary recrystallisation phenomenon. By comparing grain boundary mobility with the rate of zone annealing, it was theoretically predicted and experimentally observed that directional recrystallisation should give way to equiaxial recrystallisation when the interface velocity is less than the speed of zone annealing. The variables involved in the anisothermal zone annealing process appear to rationalise when the effects of temperature and time are consolidated into a single parameter, the kinetic strength of the heat treatment. Directional recrystallisation is found to occur only when a critical value of the kinetic strength is exceeded, irrespective of the peak temperature or traverse speed during zone annealing. *MST/1268*

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Introduction

Recrystallisation occurs when cold worked metals are heated to temperatures high enough to permit the movement of high energy grain boundaries, which sweep through the metal and replace the cold worked grains by a new set of more nearly perfect crystals. In general, recrystallised metals have an equiaxed grain microstructure. However, there are now many cases reported in the literature in which unconventional alloying or processing leads to the development of a highly anisotropic grain structure as a consequence of recrystallisation.[1–10] This directionally recrystallised microstructure closely resembles that obtained by directional solidification, with the elongated columnar grains aligned along a particular sample axis.

The majority of reported examples of directional recrystallisation are found in oxide dispersion strengthened (ODS) alloys, designed primarily for their exceptional creep resistance and high temperature stability.[1,3,11–13] These ODS materials are usually produced by mechanical alloying, in which mixtures of metal powders (or master alloy powders) and fine refractory oxide powders are milled together, extruded, and subsequently fabricated according to the intended application. In some cases, directional recrystallisation is found to occur during isothermal annealing, but in other cases only on annealing in a temperature gradient. The latter process is most effective when the annealing does not involve a static temperature gradient – when, instead, a hot zone is traversed across the specimen. The recrystallisation front then follows the hot zone, giving extremely anisotropic grain structures, with the new grains sometimes extending over large distances (~1 m).[1,3,5,6] The alloys are in practice used in the directionally recrystallised condition, where the highly anisotropic grains contribute to creep strength in the direction parallel to their longest axes.

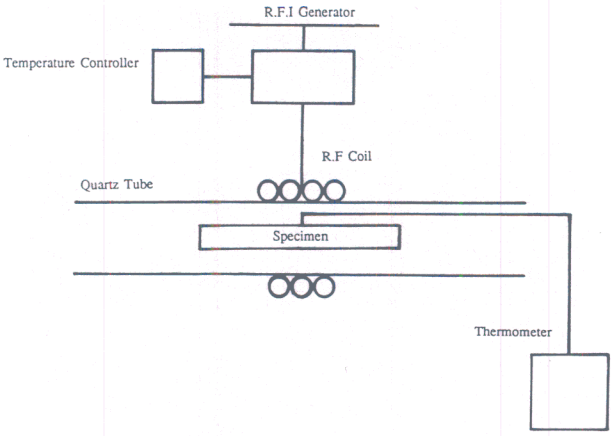
The purpose of the present work was to study the directional recrystallisation process in a commercial alloy, Inconel MA 6000, which is essentially a nickel base superalloy designed for aerospace applications, strengthened with both γ'-precipitates and oxide particles. The microstructure of this particular alloy, after fabrication but before any heat treatment, is believed to be the result of dynamic recrystallisation during the hot rolling process. Thus, growth of a directional microstructure during subsequent heat treatment (at temperatures above the γ'-solvus of about 1160°C) is thought to be a consequence of secondary recrystallisation,[1] driven essentially by reduction in total surface energy. Furthermore, the alloy does not undergo directional recrystallisation during isothermal annealing, only when it is heat treated in a static or moving temperature gradient, the latter being the more effective in producing the anisotropic microstructure.[1] The alloy is therefore suitable for the study of the microstructural changes that take place during directional recrystallisation, and also in establishing the factors controlling the zone annealing process. It has been the practice for the zone annealing conditions to be established empirically, and it was a major aim of the present work to obtain a theoretical framework for the process of anisothermal annealing.

Experimental techniques

The alloy studied was an ODS nickel base superalloy, with the commercial designation Inconel MA 6000; its chemical composition is given in Table 1. It was prepared using a mechanical alloying technique by INCOMAP. This involves the ball milling of a mixture of nickel powder (particle size 4–7 μm) and powdered chromium, molybdenum, tungsten, tantalum, and a master nickel base alloy powder containing

Table 1 Chemical composition of nickel base superalloy MA 6000, wt-%

C	Cr	Al	Ti	Ta	Mo	W	Zr	Si	Mn	P	S	Co	Cu	Fe	N	Nb	V	Y_2O_3	Ni
0·058	14·96	4·44	2·28	1·97	1·96	3·91	0·13	0·08	0·01	0·006	0·001	0·22	0·01	1·49	0·20	0·05	0·01	1·08	Bal.



1 Schematic diagram of crystal growth equipment adapted for zone annealing experiments; heating system is based on rf induction coil, and zone annealing is achieved as specimen traverses through coil

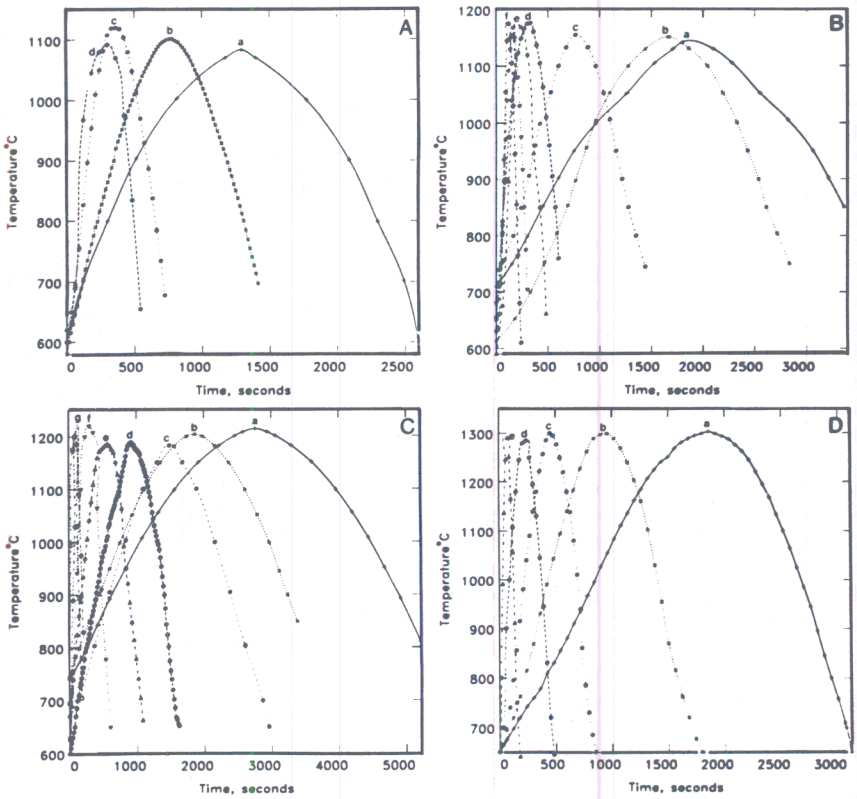
the reactive elements aluminium, titanium, boron, and zirconium (particle size $\sim 150\,\mu m$). The yttrium oxide is introduced into the mixture in the form of 1 μm aggregates each consisting of numerous individual particles of 20–40 nm dia.

The mechanically alloyed powder is then extruded using a mild steel canning material, into a 54 mm dia. rod before hot rolling at 1040°C into a 20 mm dia. bar of 800 mm length. The normal commercial practice at this stage is to anneal to induce recrystallisation into an anisotropic grain microstructure, but for the present purposes, the alloy was supplied without having been given any heat treatment after rolling.

ZONE ANNEALING

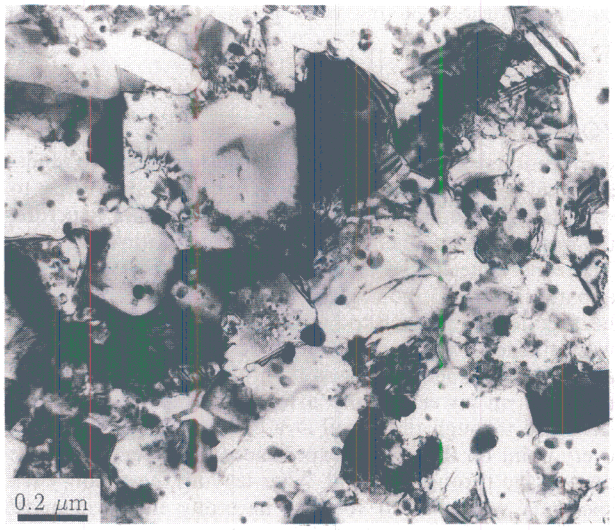
Conventional isothermal recrystallisation processes generally lead to the formation of equiaxed grain structures; to stimulate recrystallisation into an elongated grain structure, the samples were zone annealed in a temperature gradient. The zone annealing was carried out using crystal growth apparatus (Fig. 1), consisting of a narrow rf coil as the heating device. To each rod shaped sample ($4 \times 4 \times 20$ mm) was attached a Pt/Pt–13Rh thermocouple, welded halfway along the rod's length; it was then placed in a quartz tube mounted on an electrically driven carriage. The rod was traversed through the rf coil at a controlled speed, so that every point on the sample experienced a temperature pulse on passage through the coil. After leaving the rf coil, each specimen was allowed to cool naturally in air. Typical cooling curves are discussed later in the text.

To obtain directional recrystallisation, $4 \times 4 \times 20$ mm rectangular rods were cut from the hot rolled MA 6000 alloy, with their major axes parallel to the rolling direction. The samples were then zone annealed, the thermal cycle experienced at each point being characterised by the peak temperature (T_p) and the specimen travel speed through the rf coil. The thermal cycles, as measured by the Pt/Pt–Rh thermocouples, are presented in Fig. 2. Note that the actual peak temperatures are slightly different from their nominal values as quoted throughout this paper. 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.



a $T_p \approx 1100$°C: curves a, b, c, and d represent specimen travel speeds of 0·8, 1·4, 3·2, and 5·0 mm min^{-1}, respectively; *b* $T_p \approx 1160$°C: curves a, b, c, d, e, and f represent specimen travel speeds of 0·8, 1·4, 3·2, 5·0, 7·7, and 10·0 mm min^{-1}, respectively; *c* $T_p \approx 1200$°C: curves a, b, c, d, e, f, and g represent specimen travel speeds of 0·4, 0·8, 1·4, 3·2, 5·0, 7·7, and 10·0 mm min^{-1}, respectively; *d* $T_p \approx 1300$°C: curves a, b, c, d, e, and f represent specimen travel speeds of 0·8, 1·4, 3·2, 5·0, 7·7, and 10·0 mm min^{-1}, respectively;

2 Measured thermal cycles during zone annealing experiments



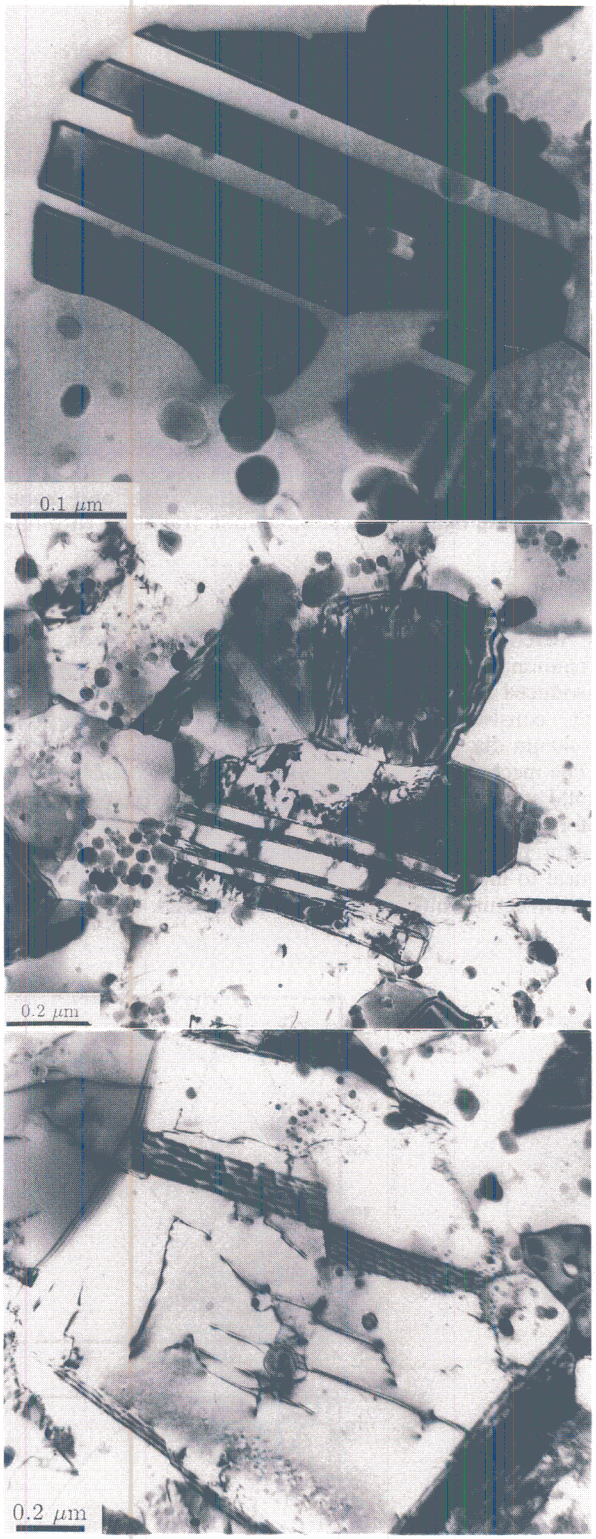
3 General microstructure observed in longitudinal section of hot rolled sample, showing equiaxed grains

MICROSCOPY

Mechanically polished samples were etched for optical microscopy using a mixture of 2 g $CuCl_2$ in 40 ml HCl and 80 ml ethanol. Thin foils for transmission electron microscopy (TEM) were prepared from 0·25 mm thick discs machined from the sample concerned. After reducing the thickness to 0·05 mm by abrasion on SiC paper, the samples were electropolished using a Fischione twin jet unit. The electrolyte consisted of 20 vol.-% perchloric acid in methanol and was used at ambient temperature with the polishing potential set to 25–30 V.

As-received microstructure

The final process in the manufacture of MA 6000 as an alloy for component manufacture involves rolling at a temperature of 1040°C. This is a relatively high temperature compared with the melting temperature of the alloy, and it is not obvious whether the conditions during the final processing lead to dynamic recovery and recrystallisation, or whether a cold deformation type microstructure is expected when fabrication is completed. A true cold deformed microstructure is unlikely, since the grain structure has been reported to be equiaxed and extremely fine.[1] It is therefore believed that the microstructure of MA 6000 before zone annealing is a consequence of primary recrystallisation, and that it is secondary recrystallisation that leads to the directional microstructure during zone annealing.[1] However, with aluminium alloys it is quite common to observe an apparently ultrafine grained, equiaxed microstructure which evolves during hot deformation by a process of dynamic recovery sometimes called 'continuous recrystallisation'.[14] Because the grain boundaries in the continuously recrystallised microstructure form by extensive polygonisation of dislocations, the misorientations between adjacent grains tend to be rather small. Hence, an examination of the orientations between adjacent grains should enable a distinction to be made between what could be essentially a recovered microstructure with low misorientation subgrain boundaries, and one which represents a recrystallised microstructure with large misorientations between adjacent grains.

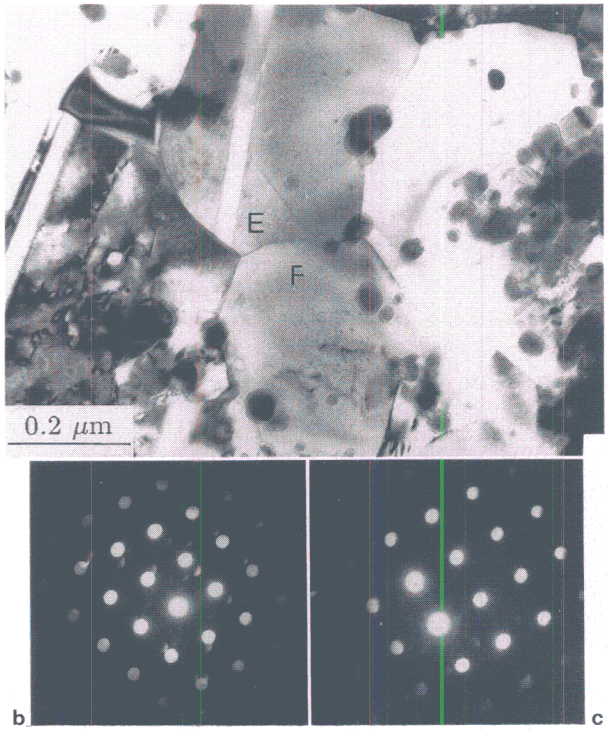


a undistorted annealing twins, longitudinal section; *b* undistorted annealing twins, transverse section; *c* 'clean' subgrain boundary

4 High magnification images showing evidence of recrystallised microstructure

GENERAL OBSERVATIONS

Thin foil samples prepared from the hot rolled MA 6000 (hardness 645 HV(10)) revealed an equiaxed grain structure (Fig. 3) irrespective of whether longitudinal or transverse sections were examined. (The hardness data presented in this study are intended to give a coarse and macroscopic



a bright field image; *b* electron diffraction pattern for grain E; *c* electron diffraction pattern for grain F

5 Example of orientation relationship between adjacent grains, axis–angle pair representation for this particular case being ⟨0·501 0·499 0·707⟩ 180°

measure of the extent of recrystallisation. The hardness in fact depends on factors such as the grain size and substructure, and on levels of oxide particles and γ'-precipitation, although only the grain size varies significantly between the samples studied here.) The mean linear intercept as measured on transmission electron micrographs was found to be 0·17 μm, representing an unusually small grain size. The dislocation density within the grains appeared to be rather low, with numerous annealing twins and subgrain boundaries (Fig. 4). The traces of the annealing twin boundaries were always straight, showing no signs of deformation, and the subgrain boundaries consisted of neat arrays of dislocations rather than the tangles of dislocations that characterise dislocation cells typical of cold deformed microstructures. All these observations are consistent with the hypothesis that the microstructure has undergone primary recrystallisation during hot rolling.

Table 2 Orientation relationships between adjacent grains of MA 6000 in as-deformed condition; orientation relations are expressed as axis–angle pairs, first three columns representing components of unit axis of rotation, followed by right handed angle of rotation (degrees) and approximate Σ value

Axis of rotation			Angle	Σ
0·000	0·608	0·793	180	1
0·372	0·656	0·656	180	9
0·501	0·499	0·707	180	17b
0·553	0·622	0·553	180	3
0·650	0·322	0·687	154	9
0·777	0·629	0·000	180	1
0·743	0·669	0·000	180	1
0·687	0·650	0·322	154	9
0·435	0·676	0·593	173	9
0·566	0·799	0·200	179	13b
0·268	0·632	0·726	169	7
0·332	0·609	0·719	174	9
0·792	0·297	0·532	180	7
0·302	0·624	0·720	156	9
0·596	0·743	0·301	176	7
0·928	0·263	0·263	180	9
0·204	0·453	0·867	174	21b
0·333	0·666	0·666	180	9
0·243	0·402	0·882	173	21b
0·167	0·576	0·799	177	21
0·889	0·337	0·316	177	11
0·291	0·698	0·653	164	9
0·669	0·246	0·700	160	19a
0·323	0·669	0·669	180	9
0·000	0·707	0·707	179	1
0·591	0·716	0·255	180	7

ORIENTATION RELATIONSHIPS

As discussed earlier, a measurement of the orientation relationships between adjacent grains can help distinguish between dynamic recovery effects and primary recrystallisation.

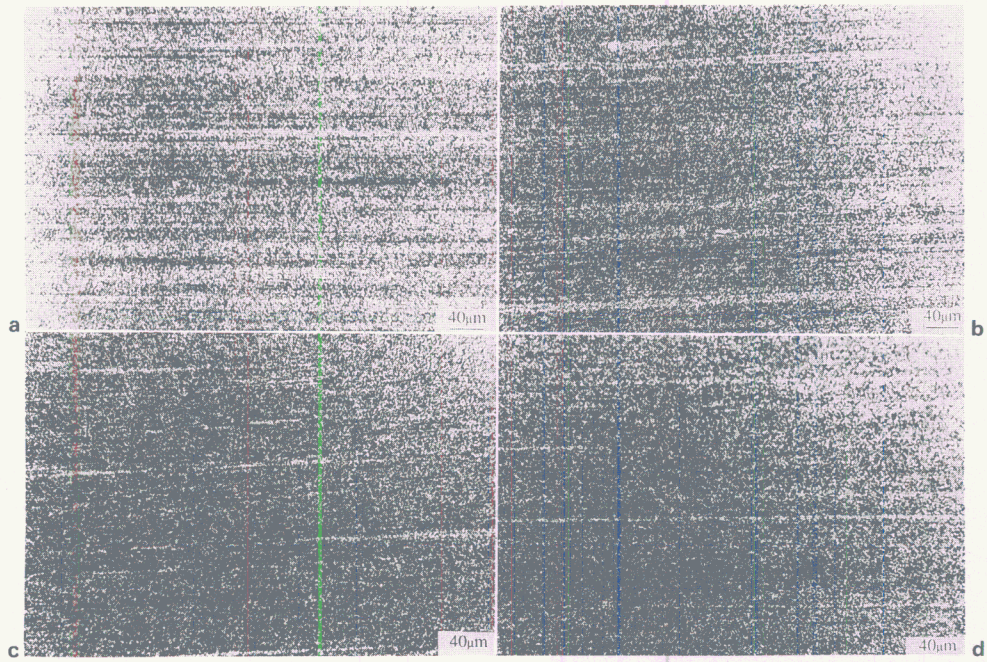
The orientation relationship between a pair of like crystals whose crystallographic bases are defined from a common origin can be described using an axis–angle pair. This means that if one of the crystals is rotated rigidly about the specified axis which passes through the origin, through a right handed angle of rotation θ, its orientation coincides with that of the other.

Accurate measurements of orientation relationships are best carried out on Kikuchi lines, but the distinction between a large and a small misorientation does not require extreme precision; the experimental data presented here are based on an analysis of the recriprocal lattice vectors observed in conventional selected area electron diffraction patterns (Fig. 5). The zone axis of each such pattern can then deviate typically by 5° from the optical axis of the microscope, and this can be used as an estimate of error. In fact, the error should be somewhat lower since the orientations were deduced not from the zone axes, but by examining the relationship between pairs of recriprocal lattice vectors (in the same diffraction pattern) from each of the two crystals concerned. The four recriprocal lattice vectors were used to deduce the rotation matrix, and hence the axis–angle pair, in the manner described in Ref. 15.*

Because the γ-matrix phase in the nickel base superalloy has an fcc crystal structure, there are in general 24 crystallographically equivalent descriptions (in terms of axis–angle pairs or rotation matrices) of any orientation relationship. The equivalent operations can be generated by multiplying the rotation matrix by the symmetry operations of the fcc structure. Hence, for each experimentally determined orientation relation, all 24 possibilities were calculated; following convention, the one with the highest angle of rotation was chosen for presentation in Table 2.

Also given in Table 2 are the approximate values of Σ (the recriprocal density of coincidence points), included simply as a rough indicator of the level of misorientation; subgrain boundaries of the type associated with dynamic recovery effects would not be expected to be far from a $\Sigma = 1$ orientation. The results provide strong evidence that the ultrafine grained microstructure obtained after hot rolling MA 6000 is a result of primary recrystallisation, and not the so-called continuous recrystallisation

*The procedure for doing such calculations is illustrated with worked examples in Ref. 15; examples 4–6 are particularly relevant. A computer program capable of carrying out these calculations, on any IBM PC compatible computer using an MS DOS operating system, is available free from the authors. All requests must be accompanied by a securely packed, double sided, double density 5·25 in. formatted floppy diskette together with an addressed envelope.



a 0·8 mm min^{-1}, 615 HV(10); *b* 1·4 mm min^{-1}, 624 HV(10); *c* 3·2 mm min^{-1}, 637 HV(10); *d* 5·0 mm min^{-1}, 639 HV(10)

6 Microstructures and hardness obtained after zone annealing at $T_p \approx 1000°C$

observed in other alloy systems. Consistent with previous suggestions,[8] the driving force for secondary recrystallisation during zone annealing is therefore the large amount of energy stored in the material in the form of high misorientation grain boundaries.

Zone annealing experiments

GENERAL OBSERVATIONS

Zone annealing at $T_p \approx 1100°C$ failed to produce any discernible change in the optical microstructure (Fig. 6), and the overall hardness showed little change relative to the hardness before annealing (Table 3). On the other hand, the microstructure changed quite spectacularly during zone annealing at $T_p \approx 1160°C$, as illustrated in Fig. 7; this shows clear evidence for directional grain growth, at least at the lower travel speeds. The microstructures obtained for $T_p \approx 1200°C$ are presented in Fig. 8, and for $T_p \approx 1300°C$ in Fig. 9.

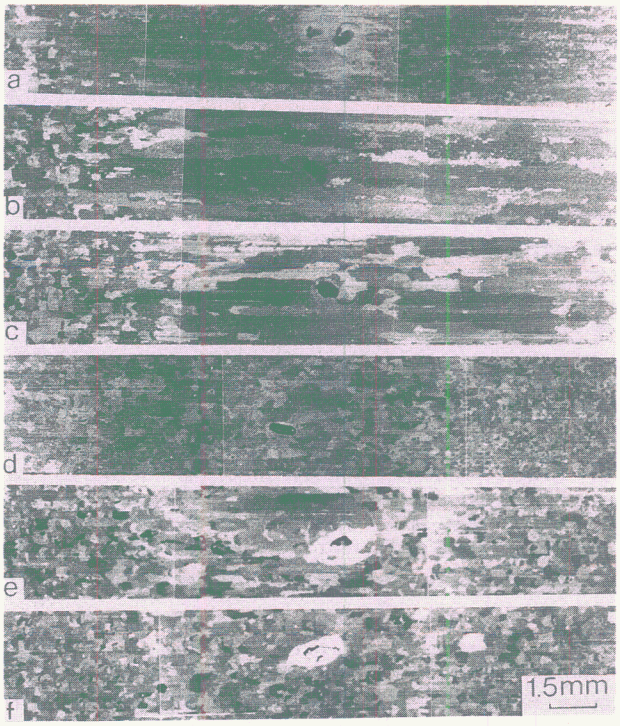
TRANSITION FROM DIRECTIONAL TO EQUIAXED MICROSTRUCTURE

For all the experiments, it is clear that at faster travel speeds the microstructure tends to change from one containing predominantly anisotropic grains to a more or less equiaxed grain structure of a slightly higher hardness. The change to the equiaxed structure at the higher speeds is, as discussed later, attributable to incomplete recrystallisation during the stage when the temperature is rising, with recrystallisation continuing in the now hot region of the sample which proceeds out of the rf coil (Fig. 10). In effect, the sample moves at a rate which is faster than can be tolerated by any recrystallisation front that forms. As the peak temperature rises, so does the mobility of the recrystallisation front, so that the speed at which the microstructure changes from directionally recrystallised (DX) to equiaxial recrystallisation (X) should increase. This is exactly as observed in the present work (Table 4).

The hypothesis describing the DX→X transition can be expressed quantitatively in terms of the maximum velocity v_1^1 at which the grain boundaries constituting the recrystallisation front can move at a temperature T_1. The transition

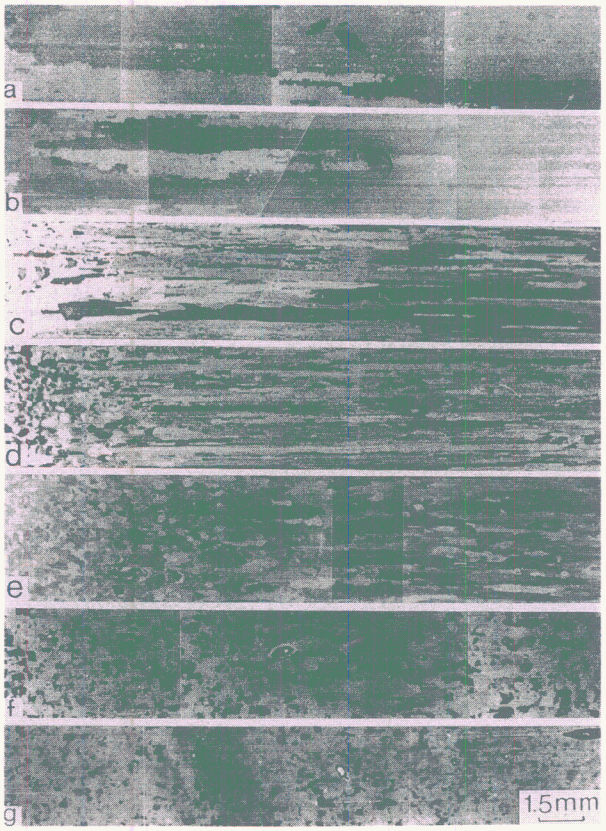
Table 3 Mean hardness (averaged over five results) of samples after zone annealing. As received, hot rolled MA 6000 alloy has mean hardness of 645 HV(10). Speed is that at which sample traverses through rf coil during zone annealing. PL indicates zone annealing direction parallel to hot rolling direction; PR indicates zone annealing direction normal to rolling direction

Zone annealing direction	Speed, mm min^{-1}	Mean Vickers hardness with 10 kg load, HV(10)			
		$T_p \approx 1100°C$	$T_p \approx 1160°C$	$T_p \approx 1200°C$	$T_p \approx 1300°C$
PL	0·4	...	...	476	...
PL	0·8	615	496	488	420
PR	0·8	617	...	...	...
PL	1·4	624	499	482	489
PR	1·4	631	...	508	482
PL	3·2	637	517	490	469
PR	3·2	640	...	523	503
PL	5·0	639	525	486	489
PR	5·0	643	...	533	513
PL	7·7	...	527	524	499
PL	10·0	...	534	528	512



a 0·8 mm min^{-1}, 496 HV(10); *b* 1·4 mm min^{-1}, 499 HV(10); *c* 3·2 mm min^{-1}, 517 HV(10); *d* 5·0 mm min^{-1}, 520 HV(10); *e* 7·7 mm min^{-1}, 527 HV(10); *f* 10·0 mm min^{-1}, 534 HV(10)

7 Microstructures and hardnesses obtained after zone annealing at $T_p \approx 1160°C$



a 0·4 mm min^{-1}, 476 HV(10); *b* 0·8 mm min^{-1}, 488 HV(10); *c* 1·4 mm min^{-1}, 482 HV(10); *d* 3·2 mm min^{-1}, 490 HV(10); *e* 5·0 mm min^{-1}, 486 HV(10); *f* 7·7 mm min^{-1}, 524 HV(10); *g* 10·0 mm min^{-1}, 528 HV(10)

8 Microstructures and hardnesses obtained after zone annealing at $T_p \approx 1200°C$

is predicted to occur if the actual velocity of the sample exceeds v^t. Since the driving force for secondary recrystallisation is independent of temperature (it depends only on the energy stored in the form of defects such as grain boundaries), the velocity v_2^t expected at a different temperature T_2 is given by

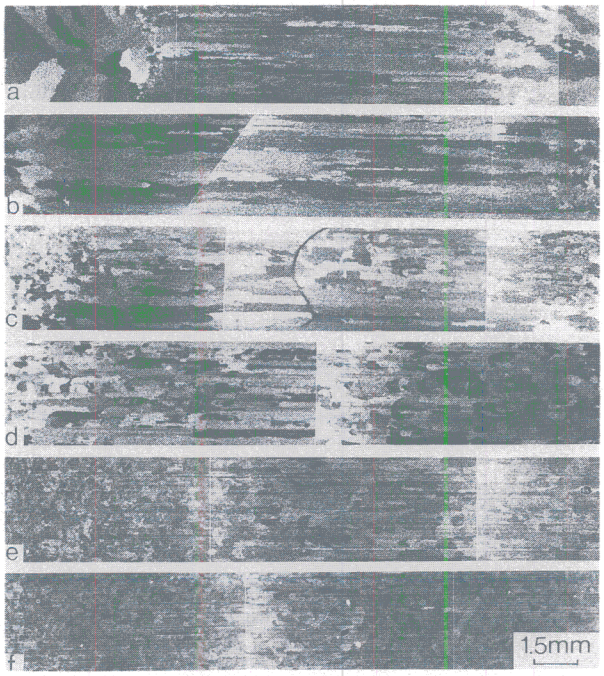
$$\ln\left(\frac{v_1^t}{v_2^t}\right) = \frac{Q}{R}\left(\frac{1}{T_2} - \frac{1}{T_1}\right)$$

where Q is the activation energy for the transfer of atoms across a grain boundary during recrystallisation, R is the universal gas constant, and T_1 and T_2 are absolute temperatures. If it is assumed that $Q = 284\,716$ J mol^{-1}, the activation energy for self-diffusion in nickel,[16] and that most of the boundary motion occurs at the peak temperature, it should be possible to estimate v^t as a function of T_p. Thus, taking $v_1^t = 4{\cdot}1$ mm min^{-1} (i.e. between 3·2 and 5 mm min^{-1} from the data for the DX→X transition in Table 4) for $T_p \approx 1160°C$, it can be shown that for $T_p \approx 1200$ and 1300°C the transition velocities should be 7·8 and 34 mm min^{-1}, respectively. This is in reasonable agreement with the data presented in Table 4, especially given that, at $T_p \approx 1300°C$, even the specimen treated at the highest speed showed some directional grain structure.

The ends of all the zone annealed specimens revealed an equiaxed grain structure, presumably since rf induction heating is more effective there because of the extra free surface associated with the ends. Consequently, temperature gradients should tend to be gentler at the sample ends.

Table 4 Microstructural condition of samples after zone annealing: D, deformed (unrecrystallised); DX, directionally recrystallised; PDX, partially directionally recrystallised; X, recrystallised with equiaxed grain structure; PX, partially recrystallised with equiaxed structure (zone annealing direction indicated as in Table 3)

Zone annealing direction	Speed, mm min^{-1}	Microstructure			
		$T_p \approx 1100°C$	$T_p \approx 1160°C$	$T_p \approx 1200°C$	$T_p \approx 1300°C$
PL	0·4	...	...	DX	...
PL	0·8	D	DX	DX	DX
PR	0·8	D	...	...	...
PL	1·4	D	DX	DX	DX
PR	1·4	D	...	DX	DX
PL	3·2	D	DX	DX	DX
PR	3·2	D	...	PDX	PDX
PL	5·0	D	X	DX	DX
PR	5·0	D	...	X	X
PL	7·7	...	X	PDX	PDX
PL	10·0	...	X	PX	'X'

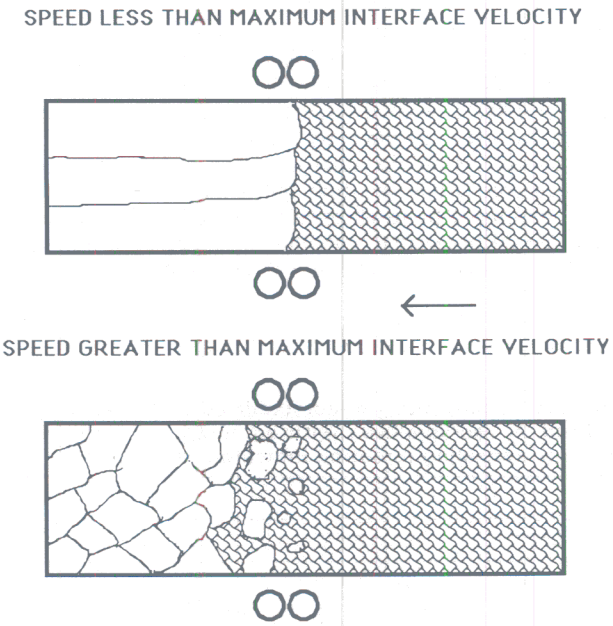


a 0·8 mm min^{-1}, 420 HV(10); *b* 1·4 mm min^{-1}, 489 HV(10); *c* 3·2 mm min^{-1}, 469 HV(10); *d* 5·0 mm min^{-1}, 489 HV(10); *e* 7·7 mm min^{-1}, 499 HV(10); *f* 10·0 mm min^{-1}, 512 HV(10)

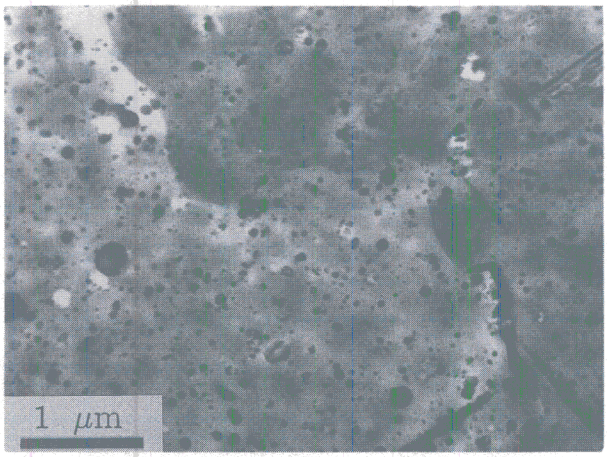
9 Microstructures and hardnesses obtained after zone annealing at $T_p \approx 1300°C$

PARTICLE ALIGNMENT

It might be argued that the anisotropic grain growth that takes place during directional recrystallisation in MA 6000 is a consequence of the non-uniform distribution of oxide particles, which may be aligned along the rolling direction.

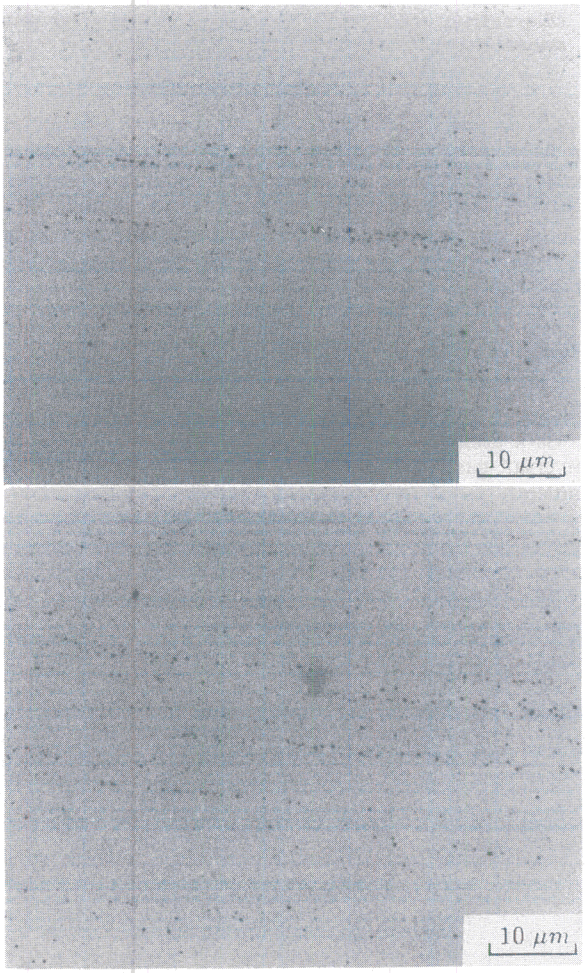


10 Schematic illustration of transition from directionally recrystallised microstructure to one in which recrystallised grains are equiaxed; in latter case, recrystallisation is not complete when specimen leaves rf coil because maximum rate at which interfaces can move is less than sample speed (arrow indicates direction of motion through rf coil)



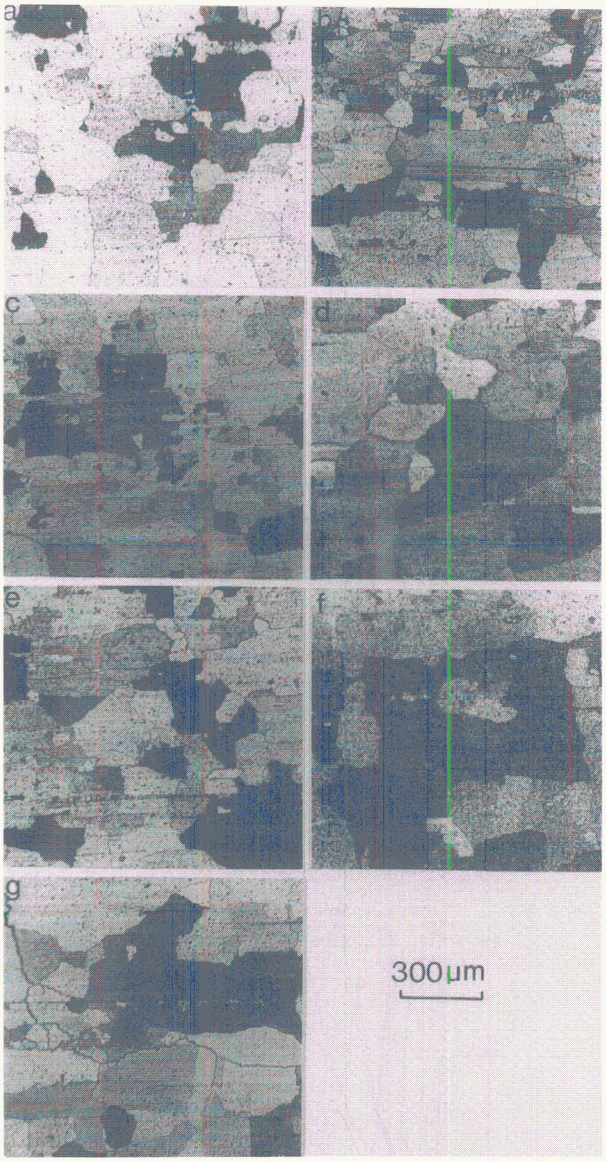
11 Bright field transmission electron micrograph of longitudinal section of directionally recrystallised ($T_p \approx 1160°C$, 5 mm min^{-1}) MA 6000 sample; there is no obvious alignment of oxide particles (rolling direction is approximately horizontal)

This cannot easily be resolved by TEM for the hot rolled samples, since contrast effects resulting from the ultrafine grain size distort the images and prevent the particle distribution from being examined over a large enough



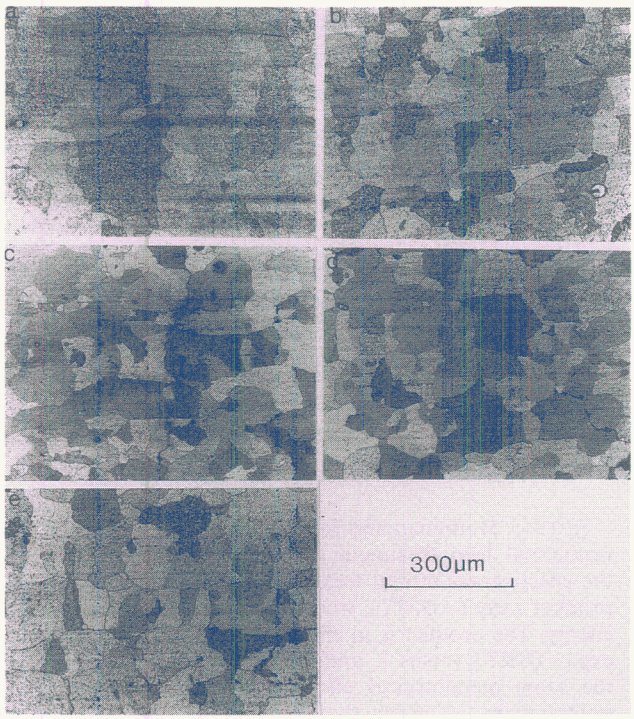
a specimen prepared by grinding and polishing in direction parallel to rolling direction; *b* specimen prepared by grinding and polishing in direction normal to rolling direction

12 Optical micrographs illustrating degree of alignment of yttrium oxide particles along hot rolling direction, which is approximately horizontal



a 15 min, 511 HV(10); *b* 30 min, 507 HV(10); *c* 1 h, 501 HV(10); *d* 2 h, 501 HV(10); *e* 4 h, 502 HV(10); *f* 8 h, 501 HV(10); *g* 16 h, 500 HV(10)

13 Microstructures and hardnesses of hot rolled MA 6000 specimens after isothermal annealing at $T_p \approx 1200°C$ for various times. Hardness values are means of five tests on each sample



a 15 min, 520 HV(10); *b* 30 min, 516 HV(10); *c* 1 h, 511 HV(10); *d* 2 h, 506 HV(10); *e* 4 h, 504 HV(10)

14 Microstructures and hardnesses of hot rolled MA 6000 specimens after isothermal annealing at $T_p \approx 1300°C$ for various times. Hardness values are means of five tests on each sample

region (Fig. 3). However, the distribution cannot alter during recrystallisation, and TEM examination of recrystallised samples failed to reveal any particle alignment (Fig. 11). Optical microscopy of unetched specimens revealed however, a small degree of particle alignment of relatively coarse particles along the hot rolling direction (Fig. 12). To check that these observations were not artefacts of preparation, two samples were examined, with the grinding direction maintained either parallel or perpendicular to the rolling direction (Fig. 12).

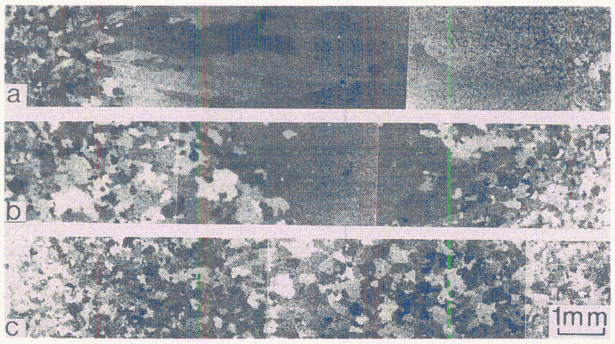
Although there appears to be some oxide particle alignment along the hot rolling direction, this cannot have much of an influence on grain morphology, since equiaxed microstructures could be obtained by zone annealing at high speeds. Thus, any directional grain growth cannot be attributed to anisotropic pinning caused by a non-uniform distribution of particles.

Two further sets of experiments were done to confirm this conclusion. Hot rolled specimens of MA 6000 were isothermally annealed at 1200 or 1300°C, and it was found that equiaxed grain microstructures developed during secondary recrystallisation (Figs. 13 and 14). This confirms that the variations in the intensity of grain boundary pinning along different directions are not responsible for the development of an elongated grain microstructure.

In the second set of experiments, rods of hot rolled MA 6000 specimens were machined with their length directions perpendicular to the hot rolling direction. Thus, during zone annealing, the rolling direction was normal to the specimen's direction of travel (i.e. cross-annealing). Directional recrystallisation could then be observed normal to the rolling direction (Figs. 15 and 16). However, comparison with experiments in which the rolling direction was parallel to the zone annealing direction indicates that the DX→X transition occurs at a lower speed for the cross-annealed samples (Table 4), indicating that the maximum rate at which grain boundaries can move in a direction normal to the rolling direction is smaller than the mobility parallel to the rolling direction. However, the effect is marginal since equiaxed grain microstructures can easily be obtained.

KINETIC STRENGTH ANALYSIS

Although the transition between a directionally recrystallised and an equiaxially recrystallised microstructure has been rationalised by comparing grain boundary mobility with the sample travel speed during zone annealing, the conditions (peak temperature, travel speed, and so on) necessary to ensure that any kind of recrystallisation actually happens have not been established. To do this, it is necessary to estimate the effectiveness of the continuous heating and cooling thermal cycle to achieve the defined purpose of recrystallisation.



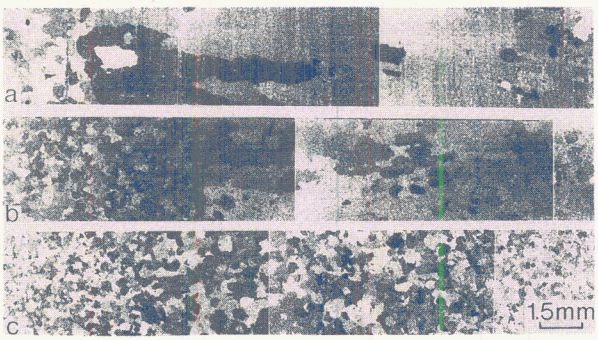
a 1·4 mm min^{-1}, 508 HV(10); *b* 3·2 mm min^{-1}, 523 HV(10); *c* 5·0 mm min^{-1}, 533 HV(10)

15 Microstructures and hardnesses obtained after cross-annealing at $T_p \approx 1200$°C

In 1945, Holloman and Jaffe[17] proposed that for a given isothermal heat treatment (at temperature T for time t), the effectiveness of that treatment should be related to the product $t \exp(-Q/RT)$, where Q is an effective activation energy. The product is in effect the integral of the curve of $\exp(-Q/RT)$ versus t, and the time required to achieve the same metallurgical effect at another temperature is estimated by assuming that the product, once evaluated, is constant for any other heat treatment. The product is therefore the kinetic strength of the heat treatment, and can easily be used to estimate the effectiveness of an anisothermal heat treatment. Alberry and co-workers[18–20] have used a similar procedure in the prediction of microstructure in welds, as have Ashby, Ion, and Easterling.[21–23]

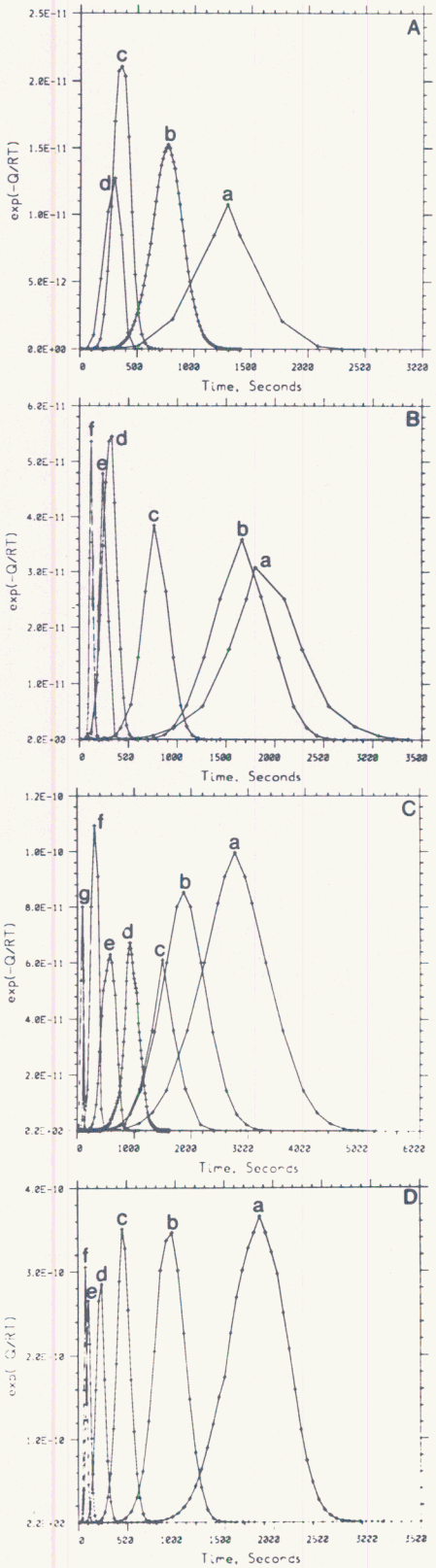
The concept is particularly applicable in the present work because the driving force for recrystallisation is independent of temperature, so that grain boundary mobility (a factor of vital importance in any recrystallisation process) depends largely on the thermally activated transfer of atoms across the boundary. Assuming, as before, an activation energy Q equal to the self-diffusivity of nickel, curves of $\exp(-Q/RT)$ were calculated for each of the thermal cycles presented in Fig. 2, as illustrated in Fig. 17. The area under each curve represents the kinetic strength of that heat treatment. It is evident from Fig. 18 that the kinetic strength concept is very useful in that it enables the variation in hardness caused by zone annealing to be rationalised for a wide variety of conditions. Thus, to achieve a given hardness would require a specific value of kinetic strength, which could in practice be achieved using a variety of anisothermal heat treatments.

A more significant application of the concept is illustrated in Fig. 19, where it is seen that a minimum value of kinetic



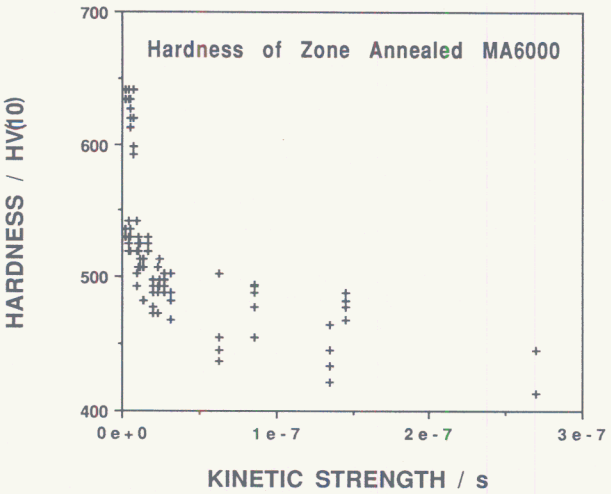
a 1·4 mm min^{-1}, 482 HV(10); *b* 3·2 mm min^{-1}, 503 HV(10); *c* 5·0 mm min^{-1}, 513 HV(10)

16 Microstructures and hardness obtained after cross-annealing at $T_p \approx 1300$°C

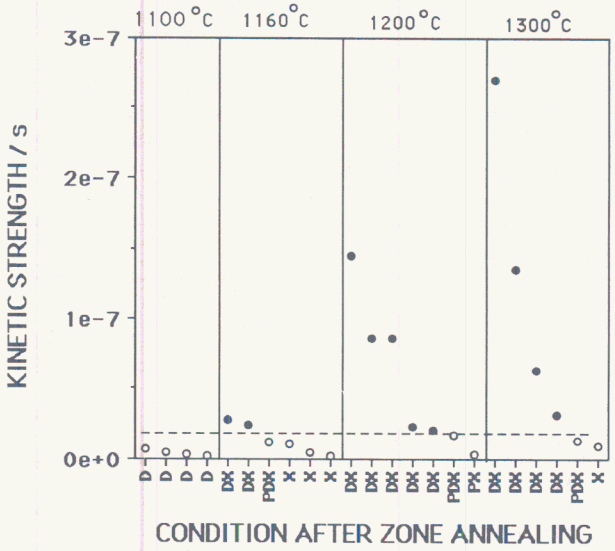


a $T_p \approx 1100$°C: curves a, b, c, and d represent specimen travel speeds of 0·8, 1·4, 3·2, and 5·0 mm min^{-1}, respectively; *b* $T_p \approx 1160$°C: curves a, b, c, d, e, and f represent specimen travel speeds of 0·8, 1·4, 3·2, 5·0, 7·7, and 10·0 mm min^{-1}, respectively; *c* $T_p \approx 1200$°C: curves a, b, c, d, e, f, and g represent specimen travel speeds of 0·4, 0·8, 1·4, 3·2, 5·0, 7·7, and 10·0 mm min^{-1}, respectively; *d* $T_p \approx 1300$°C: curves a, b, c, d, e, and f represent specimen travel speeds of 0·8, 1·4, 3·2, 5·0, 7·7, and 10·0 mm min^{-1}, respectively

17 Curves of $\exp(-Q/RT)$ versus t for each of experimentally measured thermal cycles presented in Fig. 2; area under each curve represents kinetic strength of heat treatment



18 Hardness of MA 6000 as obtained from large variety of zone annealing experiments, as function of kinetic strength: it is apparent that, in spite of the wide range of zone annealing conditions, data all fall within scatterband which varies smoothly with kinetic strength



19 Kinetic strength *v.* microstructural conditions after zone annealing: closed and open circles represent experiments in which directional recrystallisation did or did not occur, respectively; dashed line thus represents minimum kinetic strength required to ensure directional recrystallisation during zone annealing; temperatures are approximate peak temperatures experienced during zone annealing

strength is required before the specimen will undergo directional recrystallisation. This minimum value appears to be independent of, for example, the peak temperature or speed of zone annealing.

Summary and conclusions

The recrystallisation behaviour of a commercially available oxide dispersion strengthened superalloy, Inconel MA 6000, has been investigated. Consistent with published data, the microstructure of the alloy after hot rolling was found to contain submicrometre equiaxed grains which have a low defect density, and to show many of the microstructural features (such as annealing twins and sub-boundaries) common in recrystallised materials. This, combined with an analysis of the orientation relationships between adjacent grains, confirms that the hot rolled microstructure evolves by primary recrystallisation during the deformation process. Consequently, any subsequent change in microstructure when the hot rolled samples are annealed is best described as secondary recrystallisation, driven essentially by the energy stored in the material in the form of grain boundaries.

As expected, when the hot rolled alloy was zone annealed, a highly elongated, directionally recrystallised microstructure was produced, especially when the rate at which the specimen moved through the heat source was relatively low. At higher speeds, the recrystallised microstructure was equiaxed. The effect can be explained if it is assumed that the transition from directionally recrystallised to equiaxially recrystallised microstructure occurs when the maximum rate at which grain boundaries can move for specific annealing conditions is less than the sample travel speed. In these circumstances, the recrystallisation front cannot keep up with the motion of the sample, and the sample is only partially recrystallised on leaving the heat source. This theory was shown to predict that the transition should occur at higher specimen speeds as the peak temperature of the zone annealing treatment rises, as is observed experimentally.

In alloy MA 6000, the production of the directionally recrystallised microstructure has been shown to be a consequence of annealing in a temperature gradient, rather than of any anisotropic grain growth resulting from a non-uniform distribution of grain-boundary-pinning oxide particles. For example, it is possible to produce equiaxed grain structures during zone annealing at high specimen travel speeds. Furthermore, isothermal annealing always resulted in an equiaxed recrystallised microstructure. Directional recrystallisation could also be produced in an orientation normal to the hot rolling direction. Nevertheless, there is some evidence of a minor degree of particle alignment along the hot rolling direction, which has a correspondingly small effect on grain boundary mobility as a function of orientation with respect to the hot rolling direction.

The concept of kinetic strength, which quantifies the combined effect of time and temperature for isothermal or anisothermal heat treatments, has been found to be of use in modelling directional recrystallisation. For example, the hardness data for a large variety of anisothermal heat treatments can be rationalised onto a single curve as a function of kinetic strength. Furthermore, it has been demonstrated that the kinetic strength of any heat treatment must exceed a critical value if directional recrystallisation is to occur. As a corollary, the present results cannot be used to judge whether the dissolution of the γ'-particles is essential if directional recrystallisation is to occur, because althouth zone annealing with a peak temperature of 1100°C (below the γ'-solvus) did not result in directional recrystallisation, the kinetic strength in all the experiments done at that peak temperature turned out to be less than the required critical value. Experiments involving even slower specimen travel speeds (larger kinetic strengths) could not be carried out because of equipment limitations.

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