

RECRYSTALLIZATION CHARACTERISTICS OF OXIDE DISPERSION
STRENGTHENED NICKEL-BASE ALLOYS

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Electron microscopy was employed to study the process of recrystallization in two oxide dispersion strengthened (ODS) mechanically alloyed nickel-base alloys, MA 754 and MA 6000E.

MA 754 contained both fine uniformly dispersed particles and coarser oxides aligned along the working direction. Hot rolled MA 754 had a grain size of 0.5 μm and high dislocation densities. After partial primary recrystallization, the fine grains transform to large elongated grains via secondary (or abnormal) grain growth.

Extruded and rolled MA 6000E contained equiaxed grains of 0.2 μm dia. Primary recrystallization occurring during working eliminated virtually all dislocations. Conversion from fine to coarse grains was triggered by gamma prime (γ') dissolution; this was also a process of secondary or abnormal grain growth.

Comparisons were made to conventional and oxide dispersion strengthened nickel-base alloys.

INTRODUCTION

The formation of large elongated grains in oxide dispersion strengthened (ODS) alloys has long been recognized as a crucial step in the development of optimum high temperature properties. (1) However, the mechanisms by which these grain structures formed were not well understood. Further, it was observed that the transformations were sensitive to both prior thermal and mechanical history and varied greatly in characteristics depending on alloy composition. (2,3) For example those alloys known to precipitate large (>50%) quantities of γ' exhibited rapid coarse grain transformations and only at temperatures above their γ' solvus. (4,5) Alloys containing little or no γ' on the other hand, exhibited much slower transformations and showed evidence of large grain formation over broader time-temperature spectrums. (6,7) In the present study, an attempt was made to characterize the large grain transformations common to 2 ODS nickel-base alloys, MA 754 (8) and MA 6000E (9). These recently developed alloys contain YAlO₃ type dispersoids and are produced by mechanical alloying. MA 754 essentially represents a nickel-chromium based solid solution strengthened only by the presence of a dispersoid while the highly alloyed MA 6000E is strengthened by both a dispersoid and the precipitation of approximately 50% γ' . Attempts have been made to elucidate the role of grain size, thermomechanical working, oxide dispersion and γ' precipitation as related to the large grain transformation mechanisms.

PROCEDURE

Both the MA 6000E and the MA 754 employed in this investigation were produced by the International Nickel Company using mechanical alloying techniques. MA 6000E bars were fabricated from attrition milled powder which was consolidated by an 8:1 extrusion at 1010°C and subsequently hot-rolled 50% @1042°C using a 2 pass sequence. The MA 754 bars were also fabricated from attrition milled powder, however the specific extrusion and hot rolling parameters were proprietary. Chemical compositions for both alloys are provided in Table 1.

TABLE I - CHEMICAL COMPOSITIONS (Wt.%)

Alloy	Ni	Cr	Ti	Al	Y	O	N	C	Fe	Mo	W	Ta	B	Zr
MA 754	Bal	20.2	0.5	0.3	0.52	0.42	0.12	0.06	1.0	-	-	-	N.D.	N.D.
MA 6000E	Bal	14.0	2.3	4.6	0.74	0.57	0.15	0.07	0.2	2.0	4.0	2.1	0.03	0.07

All thermal treatments were carried out in argon atmospheres including gradient annealing which was performed in a stationary temperature gradient of $\approx 30^{\circ}\text{C}/\text{cm}$. Reported temperatures were controlled to within $\pm 2^{\circ}\text{C}$.

The γ' and coarse oxide dispersions were examined using platinum shadowed parlodion surface replicas while the fine grained structures were studied by means of transmission electron microscopy. Both grain and particle size determinations were obtained from micrographs recorded on plates. Lineal intercept methods were employed to determine grain sizes with a 1000 grain sample used for each measurement and the resulting sizes reported as average lineal intercept values - ' \bar{L} '. Volume fractions were determined using point counting techniques.

RESULTS

Transmission electron microscopy was used to characterize as-received extruded and hot-rolled MA 6000E and MA 754 microstructures. MA 6000E exhibits an extremely fine equiaxed structure (Figure 1 a) having an average grain size of $\approx 0.2 \mu\text{m}$ (\bar{L}). Similar grain morphologies were recorded for both transverse and longitudinal sections. Primary recrystallization was judged to have occurred dynamically during, or immediately following, the hot-working operations thus accounting for the extremely low dislocation density. The dispersoid appeared exceedingly fine and uniform with an average volumetric particle size of $\approx 240\text{\AA}$ and showed no evidence of any directional alignment (stringers). γ' particles appeared blocky and surface replicas revealed that their sizes were comparable to those of the matrix grains ($\approx 0.2 \mu\text{m}$), although an extremely fine γ' dispersion was also observed.

MA 754 in the extruded and hot-rolled condition (Figure 1 b) also exhibited an extremely fine grain morphology; however, in this case the average grain size appeared as $\approx 0.6 \mu\text{m}$ in the longitudinal section and $\approx 0.4 \mu\text{m}$ in the transverse section thereby indicating a slightly oblong grain shape. In contrast to the MA 6000E, the hot-rolled MA 754 microstructures revealed extremely high dislocation densities and a duplex dispersoid consisting of a uniform fine particle distribution (similar to that found in the MA 6000E) and a much coarser oxide ($0.5 - 4 \mu\text{m}$) strongly oriented along the primary working direction. Figure 2 illustrates the macrostructures developed in longitudinal bars of MA 754 and MA 6000E following 1 hour exposure to a stationary thermal gradient. The large grain

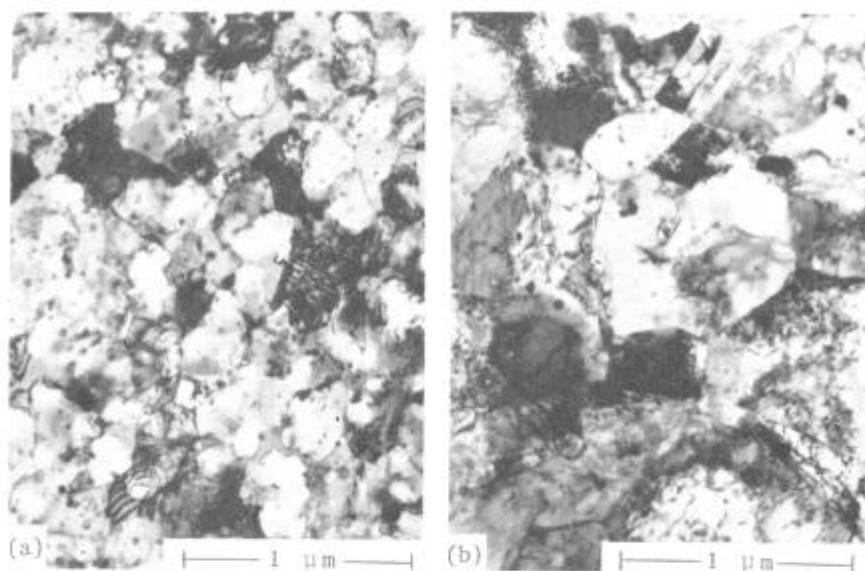


Figure 1 - (a) Transmission micrograph of MA 6000E following hot-rolling at 1042°C. (b) Transmission micrograph of MA 754 following hot-rolling.

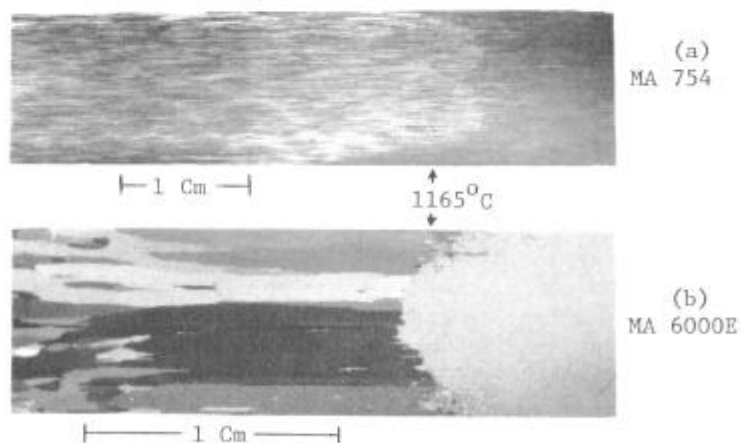


Figure 2 - (a & b) Gradient bar macrostructures of MA 754 and MA 6000E following one hour exposure to a stationary thermal gradient - longitudinal sections of rolled product.

formation observed in the MA 6000E was found to occur at all temperatures above $1165^{\circ}\text{C} \pm 3^{\circ}\text{C}$ as indicated by the position of the interface on the gradient bar. MA 754 displayed a less defined interface effect and much narrower elongated grains. Upon continued gradient exposure, the position of the original 1165°C interface remained unchanged in the MA 6000E and no further transformation was detected regardless of exposure time. In contrast, however, the MA 754 exhibited continued large grain formation down to temperatures of $\approx 1050^{\circ}\text{C}$ after 200 hours.

Secondary Recrystallization of MA 6000E

As primary recrystallization is essentially completed by a dynamic mechanism following extrusion and hot-rolling, the large grains which appear upon annealing above 1165°C must result from a secondary recrystallization (SRx) process. (10) To help elucidate the factors controlling this transformation, surface replicas were obtained from the interface regions of the gradient bar. Figure 3 illustrates the γ' morphologies revealed by these replicas. It appears that the areas which transformed to a coarse columnar grained structure had been heated to temperatures above the γ' solvus as the γ' in these regions has reprecipitated (upon cooling) into a much finer morphology than that exhibited by the hot-rolled structure. This structure is represented by the coarser, blocky γ' still prevailing in the untransformed or fine grained regions that were not heated above the solvus. Thus, the observed SRx transformation appears to be triggered by γ' dissolution and the critical reaction temperature controlled by the solvus. Independent measurements of the γ' solvus in this alloy have established it as $1175^{\circ}\text{C} \pm 5^{\circ}$ using DTA and 1160°C using metallographic techniques. (2) The transmission micrograph shown in Figure 4 (a) illustrates the microstructure common to those areas which have experienced SRx. The presence of cuboidal γ' particles is quite evident as is the uniformity of the dispersoid. X-ray diffraction of extracted dispersoids revealed the presence of mixed YAlO_3 - type oxides with nitrides and carbides ($\text{Ti}(\text{C},\text{N})$) appearing as small irregular particles having average sizes between 0.1 and 0.5 μm . Occasional 1 μm particles were observed; however, as they are randomly distributed, they only play a minor role in determining the resulting grain shape. This appeared to be primarily controlled by the thermal gradient as it was found that columnar grains could be grown transverse to the primary working directions by proper application of a thermal gradient.

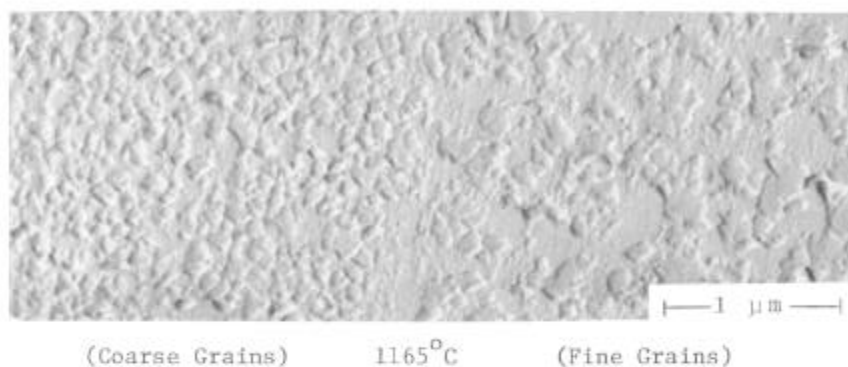


Figure 3 - Surface replica illustrating the γ' morphology common to the columnar grain - fine grain interface after one hour in a steep thermal gradient. Interface temperature - 1165 $^{\circ}\text{C}$.

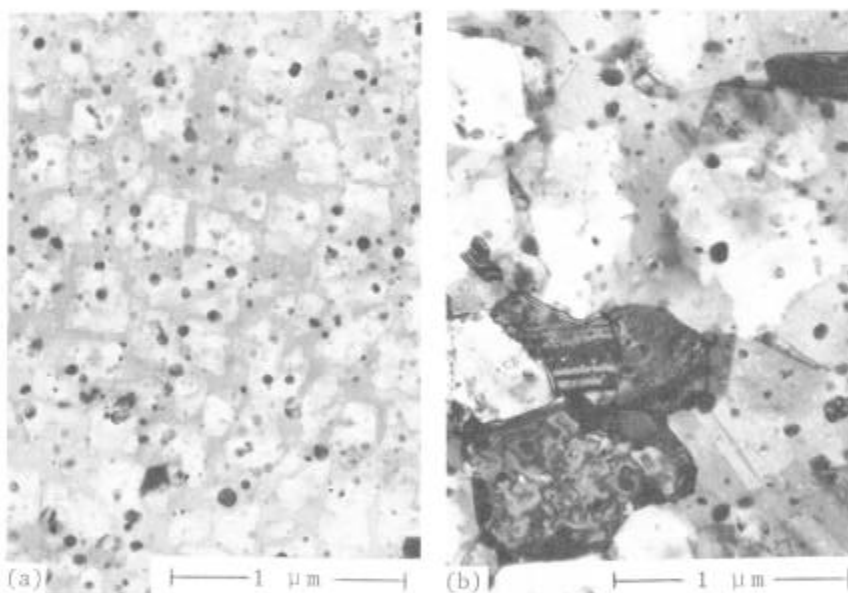


Figure 4 - (a) Transmission micrograph illustrating the substructure of MA 6000E following one hour at 1200 $^{\circ}\text{C}$ (AC).

(b) Transmission micrograph of hot-rolled MA 6000E following one hour at 1130 $^{\circ}\text{C}$. ($L = 0.42 \mu\text{m}$).

TEM conducted on hot-rolled product annealed below the 1165°C transformation temperature established that the fine grained structure exhibited normal grain growth behavior at all temperatures above 1000°C . Evidence is provided by the transmission micrograph shown in Figure 4 (b) for a sample annealed one hour at 1130°C . The average grain size in this sample was measured as $0.42\ \mu\text{m}$ (\bar{L}). Analysis of the grain growth data obtained revealed that standard ' t^n ' kinetics were operative ($n = \approx 0.2$). No evidence of grain boundary - dispersoid pinning was observed until the average grain size approached the $0.5\ \mu\text{m}$ range. Samples whose grain size increased to $0.5\ \mu\text{m}$ or greater during annealing below 1165°C , no longer exhibited SRx when subsequently heated above 1165°C . (11)

Secondary Recrystallization of MA 754

The high dislocation density and the partially recrystallized equiaxed grain shape common to hot-rolled MA 754 indicates that both deformation and primary recrystallization were occurring simultaneously. Upon exposure to elevated temperatures this deformed fine grain structure was observed to transform to large columnar grains (Figure 2 a). Their shape was influenced by the coarse oxide which severely restricted lateral grain growth. The transmission micrograph in Figure 5 illustrates the interface between fine grained hot-rolled structure and a transformed columnar grain region. This structure was observed in hot-rolled product following a one hour isothermal anneal at 1170°C . Because very few large grains replaced numerous relatively stable fine grains in a discontinuous manner, the transformation was determined to be one of secondary grain growth. The fine grained structure evident in Figure 5 showed an increase in grain size during annealing ($0.4 \rightarrow 0.56\ \mu\text{m}$ \bar{L}); however, this did not appear to restrict the transformation once initiated. Also, it can be noted that significant dislocation densities were still evident within the remaining fine grains despite the observed growth. Strain compliance at the advancing interface appears responsible for generation of the micro-twinned structure common to the SRx material.

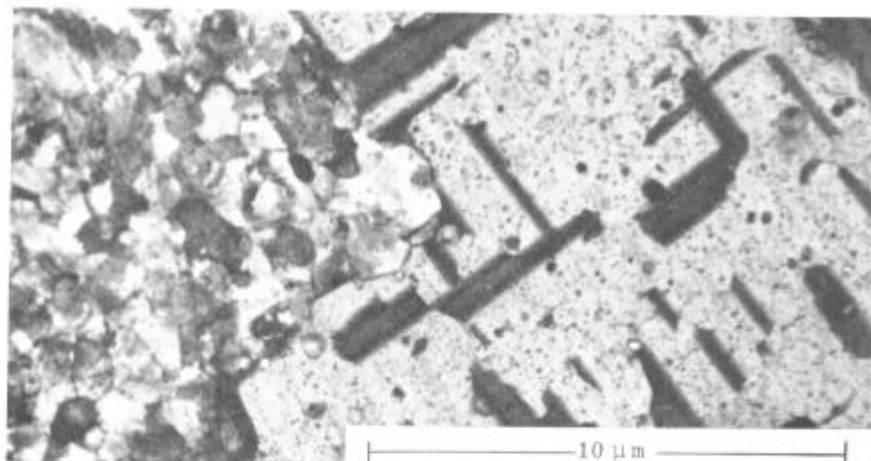


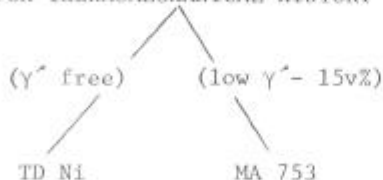
Figure 5 - Transmission micrograph of MA 754 illustrating the interface region between fine grained hot-rolled structure and a secondary recrystallized region.

DISCUSSION

The SRx behavior of MA 6000E and MA 754 has been compared with that previously reported for other Ni-base alloys both with and without oxide dispersions. The results can be summarized by the illustration below.

SECONDARY RECRYSTALLIZATION BEHAVIOR OF NICKEL-BASE ALLOYS

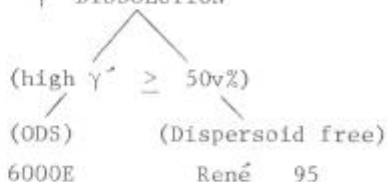
SRx RESPONSE DEPENDENT SOLELY
UPON THERMOMECHANICAL HISTORY



DS & TD NiCr

MA 754

SRx RESPONSE INITIATED BY
γ' DISSOLUTION



IN(MA) - 738

WAZ-D

IN - 713C

RSR Superalloys

MA 754 (γ' free) exhibits an SRx response which is entirely dependent upon the material's prior thermomechanical history and thus shares the behavior patterns observed for TD Ni, ⁽⁶⁾DS and TD Ni Cr. ⁽⁷⁾ MA 753, ⁽¹⁾ which contains small additions of titanium and aluminum, and develops about 15% γ' , also exhibits an SRx response which is dependent upon the alloy's prior thermomechanical history. In essence these materials do not adhere to one identifiable transformation temperature, but instead, exhibit a range of temperatures over which SRx occurs depending upon processing. Thus it appears that their SRx response is dependent purely upon the more classic grain growth mechanisms involving dislocation and grain boundary energy considerations.

In contrast, the high γ' ($\geq 50\text{v}\%$) alloys which exhibit SRx transformations all appear to have several common characteristics. The most important of which is that their SRx response is initiated or triggered by dissolution of γ' and therefore, the γ' solvus represents a critical transformation temperature. This applies to both the conventional superalloys 713C, ⁽⁵⁾ René 95 ⁽¹²⁾ and RSR superalloys ⁽¹³⁾ as well as the ODS alloys MA 6000E, ⁽¹¹⁾ IN (MA) - 738 ^(14,15) and WAZ-D. ⁽⁴⁾ The high γ' ODS alloys apparently share several other common characteristics including the SRx limiting processes by which thermal exposures below the SRx transformation temperature and/or low heating rates can eliminate the alloy's SRx response. As established by the work on MA 6000E, this effect results from a stabilization of the fine grained structure by both grain growth and the pinning of grain boundaries by coarse dispersoids. ⁽¹¹⁾

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