Congress Book
of
Heat & Surface '92

The 8th International Congress
on
Heat Treatment of Materials
held in
Kyoto, Japan,
November 17-20, 1992,

sponsored by
The International Federation of Heat Treatment and Surface Engineering

and hosted by
The Japan Society for Heat Treatment
Research Institute for Applied Sciences

Published by
Japan Technical Information Service
Tokyo

GRAIN BOUNDARY TOPOLOGY IN MECHANICALLY ALLOYED MA6000

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Synopsis: The roughness of grain boundaries in recrystallised MA6000, a mechanically alloyed oxide–dispersion strengthened nickel base superalloy, is studied as a function of the stored energy. It is found that coarser grains with smoother boundaries are developed when the stored energy is larger. The result can be understood in terms of recrystallisation theory.

Key Words: Recrystallisation; dispersion strengthened; mechanical alloying; nickel base superalloy; grain boundary pinning; grain boundary topology.

I. INTRODUCTION

Incoalloy MA6000 is a nickel base superalloy manufactured via a powder metallurgical route, using mechanical alloying [1] to introduce a yttria dispersion. After compaction, extrusion and hot–rolling, the microstructure is primary recrystallised with a submicron, equiaxed grain structure. Further heat treatment can induce secondary recrystallisation with coarse, elongated grains whose morphology is akin to that obtained by directional solidification in conventional superalloys. The purpose here was to investigate the role of stored energy in the primary recrystallised structure, on the grain boundary topology after secondary recrystallisation. There is no published research on the roughness of these boundaries, and the indications are that smoother boundaries lead to better properties. Surface tension effects normally oppose rough boundaries, but as stated earlier, MA6000 contains a dispersion of oxide particles which at low driving forces should lead to grain boundary pinning and hence to convoluted boundaries.

II. EXPERIMENTAL

The detailed alloy processing route is given elsewhere [2,3]; the chemical composition (wt.%) is:

$$Ni - 0.06C - 14.96Cr - 4.44Al - 2.28Ti - 1.97Ta - 1.96Mo - 3.91W - 0.13Zr - 0.08Si - 0.01Mn - 0.22Co - 0.01Cu - 1.49Fe - 0.2N - 0.05Nb - 0.01V - 0.006P - 1.08Y_2O_3$$

Metallographic samples were prepared on a plane normal to the extrusion direction, from differential scanning calorimetry (DSC) samples. They were etched using a mixture of 2 g $CuCl_2$ in 40 ml HCl and 80 ml ethanol. The stored energy was controlled by isothermal *preannealing* at a relatively low temperature (1160–1180 °C) where heat–treatment can be conducted without gross recrystallisation. The γ' solution temperature for this alloy is somewhat higher than 1160 °C, and gross recrystallisation occurs at temperatures in excess of 1180 °C during continuous heating

at 10 K min^{-1}. The heat–treatments were all carried out in a differential scanning calorimeter with any isothermal preannealing being followed immediately afterwards by continuous heating, using $5 \times 5 \times 2$ mm samples. One of the long edges of the sample was parallel to the extrusion direction, and all samples were extracted from the surface region of the extruded bar where the particle dispersion is anisotropically aligned along the extrusion direction [2].

Differential scanning calorimetry was carried out using a Netzsch DSC 404/3/413/D machine which is a specially designed high–temperature, heat flux DSC with computer control and data acquisition. It uses a computer controlled Pt/Rh furnace which has very low temperature gradient characteristics. Calorimetric measurements were carried out during continuous heating (2.5–40 K min^{-1}), the sample mass typically at about 200 mg. A reference of comparable mass was used, made of the same alloy as the sample, but in an already recrystallised condition. This led to a very significant improvement in accuracy, consistent with earlier research. All the experiments were carried out using an argon atmosphere in the DSC chamber, the argon flow rate being 50 K min^{-1}.

III. RESULTS AND DISCUSSION

The measured effect of the preannealing heat–treatment on the stored energy is illustrated in Fig. 1. As expected, the stored energy decreases with increasing time and temperature, although optical microscopy revealed significant changes in microstructure for those heat treatments in excess of 60 min at 1170 °C. These results need to be verified using detailed transmission electron microscopy.

Fig. 2 shows optical micrographs of representative preannealed samples which were then heated at 10 K min^{-1} to complete the recrystallisation process. It is clear that there are two main effects. Firstly, there is a general refinement of microstructure, together with a lesser degree of aniostropy, as the preannealing is increased, and this is confirmed by the quantitative data presented in Fig. 3. Secondly, the recrystallised grain boundaries become more ragged as the stored energy is reduced by preannealing (Fig. 2c). A representative, high magnification comparison of smooth and rough grain boundaries is presented in Fig. 4.

The increased raggedness of the grain boundaries at low stored energy is expected, since the driving force for grain boundary motion is reduced relative to any pinning force due to oxide particles. This should make the pinning effects more pronounced and hence obvious in the microstructure.

Fig. 3 shows that the grain width goes through a maximum as stronger preannealing treatments are applied. The effect of a short preanneal it to cause a sudden rise in grain width, because the preanneal induces a few recrystallisation nuclei without much change in stored energy. These few nuclei therefore grow rapidly during the high temperature treatment, giving the sudden rise in grain size. Further preannealing causes a reduction in grain size because decrease in stored energy reduces the grain growth rates, thus allowing more nuclei to develop independently, giving grain refinement and a more equiaxed microstructure.

Further data from the calorimetric experiments are presented in Fig. 5. They reveal that recrystallisation during continuous heating becomes easier as the amount of preannealing is increased. This is not surprising given that the elevated temperatures involved during preannealing initiate recrystallisation on a microscopic scale, and hence make further secondary recrystallisation easier during continuous heating. Initial transmission electron microscopy of a preannealed sample confirms this conclusion (Fig. 6), and further work is in progress to examine the detailed nucleation sites.

IV. CONCLUSIONS

A preannealing heat treatment below the temperature at which significant recrystallisation begins reduces the stored energy. However, the preannealing process does on a microscopic scale initiate the recrystallisation process, and hence subsequent gross recrystallisation is accelerated. There are two other consequences of the reduced stored energy. Firstly, the recrystallised grain boundary topology becomes relatively rough as the driving force for grain boundary motion is reduced relative to the pinning force due to oxide particles. Secondly, due to the associated reduced grain boundary

velocity, recrystallisation is able to develop from many different sites during subsequent continuous heating, giving a more refined ultimate microstructure. In contrast, at large stored energies the grain growth velocity is so large that the few grains which begin their growth first are able to swamp all others, giving a coarse grain structure.

VI. ACKNOWLEDGMENTS

The authors are grateful to Professor C. J. Humphreys for laboratory facilities at the University of Cambridge, and to Dr. M. Yamazaki for his help in facilitating this project. This work was carried out under the auspices of the "Atomic Arrangements: Design and Control Project", a collaborative effort between the University of Cambridge and the Research and Development Corporation of Japan. The alloy used was kindly supplied by Dr. Ian Elliott of INCO ALLOYS.

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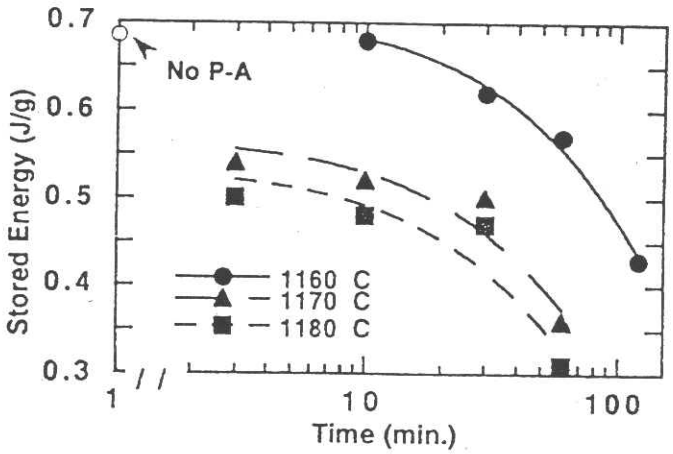

Fig.1 Stored energy as a function of pre-annealing temperature and time

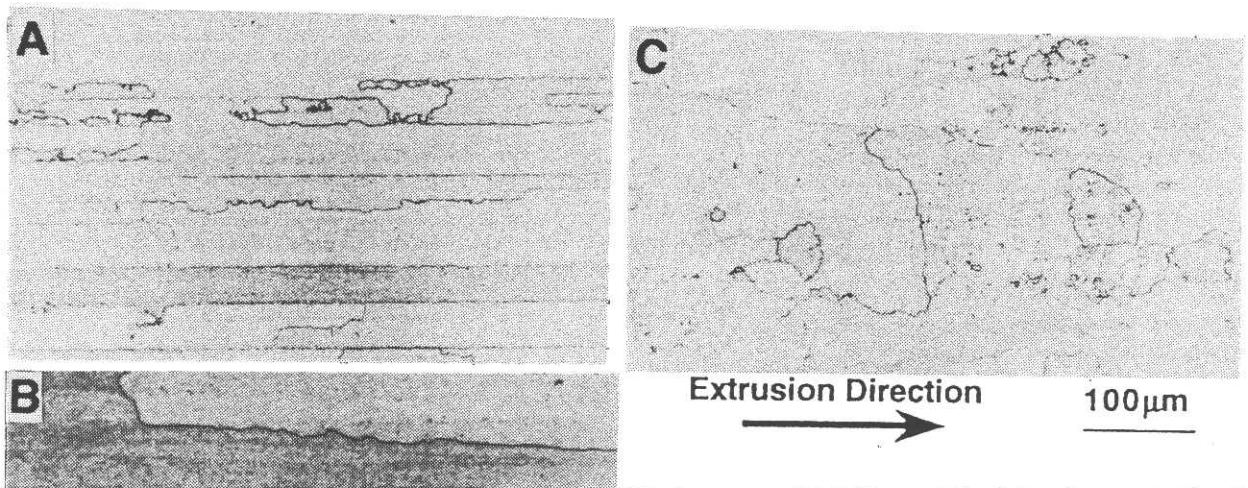

Fig.2 Metallographic data of recrystallised structures after various pre-annealing. dH shows stored energy. Heating rate is 10K/min.
A:without having pre-annealing: dH=0.69J/g
B: 1160℃,10 min. : dH=0.68J/g
C: 1180℃,60 min. : dH=0.36J/g

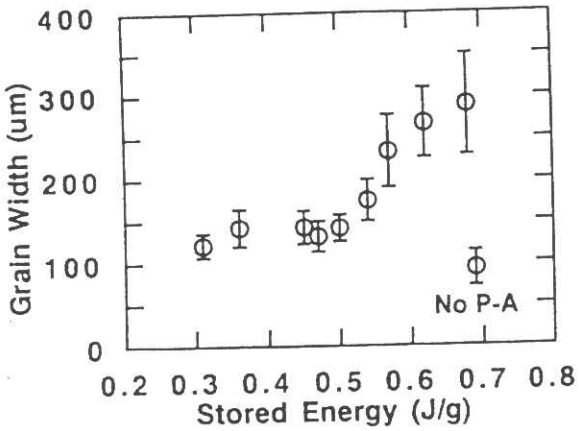

Fig.3 Plot of the recrystallised grain width as a function of stored energy

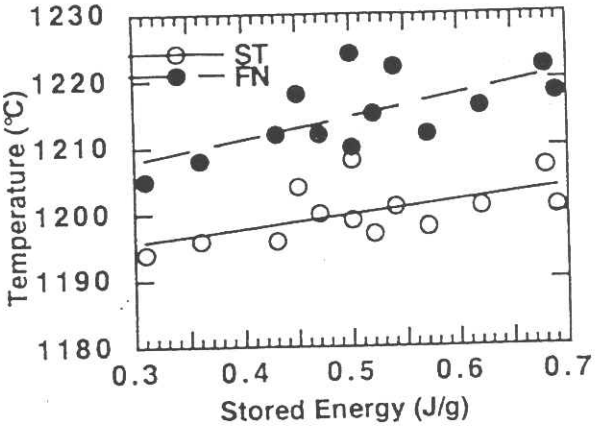

Fig.5 Recrystallisation start and finish temperature as a function of stored energy

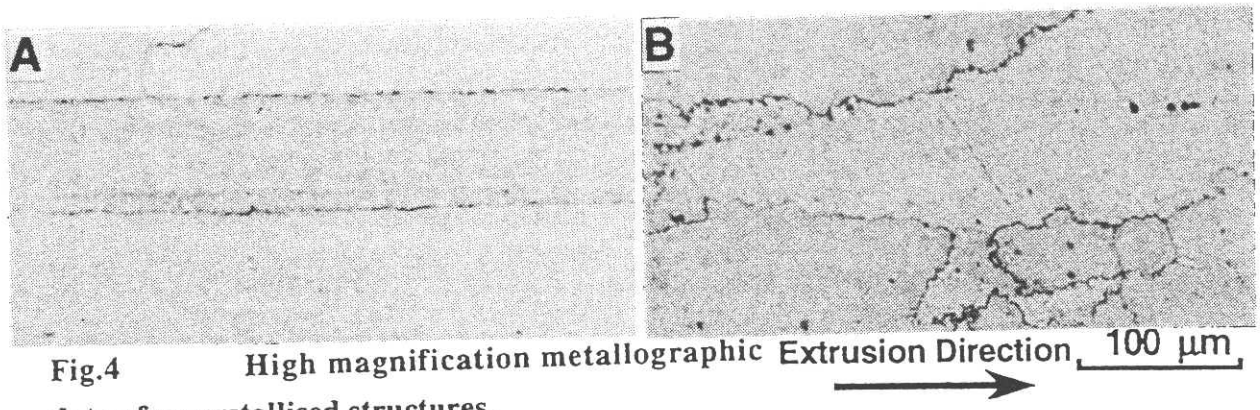

Fig.4 High magnification metallographic data of recrystallised structures.
A:without having pre-annealing: dH=0.69J/g
B: 1180℃,60 min. : dH=0.36J/g

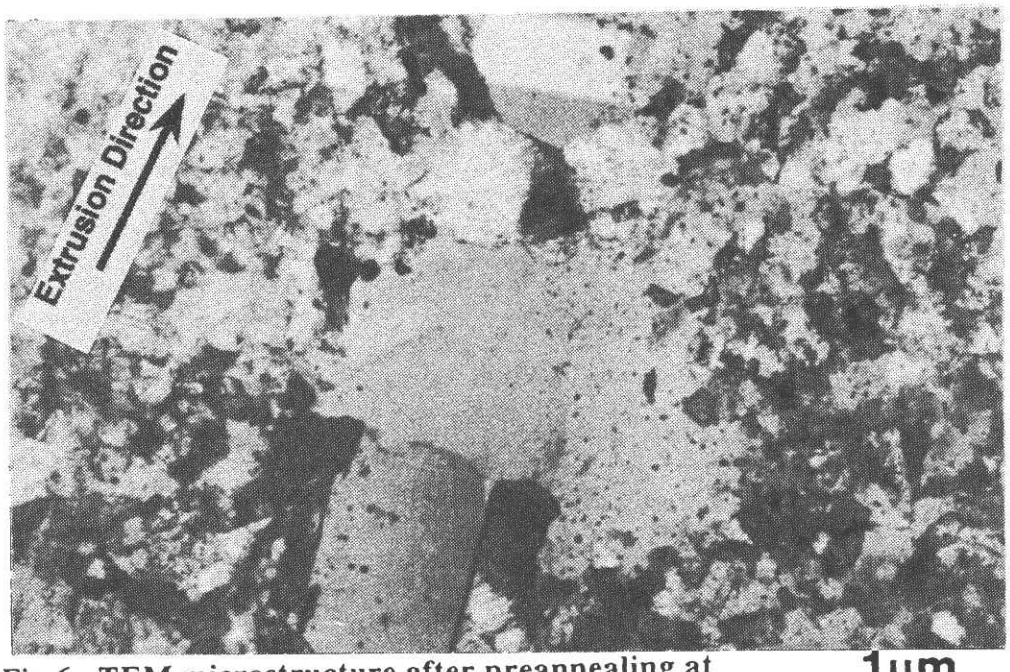

Fig.6 TEM microstructure after preannealing at 1160℃ for 30min.