Development of Equilibrium Submicrocrystalline Structure in Superalloy MA754

V. Valitov, R. Kaibyshev and N. Gajnutdinova

Institute for Metals Superplasticity Problems, Khalturina 39, RU-450001 Ufa, Russia

Abstract

The present work considers two methods for transformation of a submicrocrystalline structure into the equilibrium state in a MA754 superalloy. A specific feature of the superalloy initial structure is a presence of high internal elastic stresses. The grain boundaries exhibit a high-energy non-equilibrium configuration. TEM examinations and microhardness tests showed plastic deformation to result in a significant decrease in the internal elastic stresses due to recovery operating within the boundaries. This entails elimination of grain boundary defects. Hence the formation of recrystallized structure and microhardness reduction take place. The process is accompanied by minor grain growth. In contrast, the size of submicrocrystalline grains is essentially stable under annealing. The transformation mechanism of converting the non-equilibrium submicrocrystalline structure into a recrystallized one during annealing and hot plastic deformation is discussed.

Introduction

There is a considerable interest in producing an ultra-fine grain structure in bulk materials using intense plastic straining techniques. The major disadvantage of these bulk materials consists in that the submicrometer or nanometer grains exist in a non-equilibrium state [1-3]. These materials exhibit poor workability [3]. The best superplastic properties were demonstrated by the submicrocrystalline materials with equilibrium grain boundaries [4,5]. Recrystallization annealing can result in the conversion of the ultra-fine grain structure into the equilibrium state [4]. However, submicrocrystalline materials exhibit enhanced grain growth [4], and annealing results in essentially coarse grains that have the equilibrium boundaries. Introduction of ultra-fine dispersoids into a metallic matrix is an effective route to inhibit grain growth of in the submicrometer range [6].

The present investigation was initiated to evaluate the potential of producing equilibrium submicrocrystalline grain structure in the oxide dispersion strengthened alloy MA754. Nanoscale oxide particles provide considerable enhancement of the thermal stability [7]. As a result, recrystallization annealing may decrease the internal stress in the MA754 alloy and no significant grain growth would be occur. Hot plastic deformation is considered to be another attractive method of producing equilibrium structure in a superalloy. It is known [9] that plastic deformation results in decreased internal stress in nanocrystalline materials. A major objective of the present study is to find out the best production method for obtaining equilibrium submicrometer structure in the MA 754 alloy that is suitable for superplastic deformation.

Materials and Experimental Technique

The MA754 superalloy was produced by mechanical alloying with the subsequent hot extrusion being done at a starting temperature of 1100^{0}C. The chemical composition of this alloy in weight pct is 20%Cr, 0.3%Al, 0.5%Ti, 0.6% Y_2O_3, 0.05%C and balance Ni. Compression specimens 10 mm in diameter and 15 mm height were machined from an extruded rod. The samples were strained in compression at a constant crosshead speed up to a strain of 80% in the temperature interval 800-1150^{o}C; the strain rate interval was 10^{-4} and 10^{-1} s^{-1}. A Schenck RMS-100 testing machine was used. The tested specimens were air cooled. The strain rate sensitivity coefficient ''m'' was determined by a standard jump test. Static annealing was carried out in a muffle furnace in the temperature range 800-1150^{o}C for 2 hours. Microhardness measurements were made at a load of 200 g using a Vickers diamond pyramid intentor. For TEM examination, discs 3 mm in diameter were cut out of the samples, then electropolished to perforation in a Tenupol-3 twin-jet polishing unit with an electrolyte of 10pct perchloric acid in buthanol at ambient temperature and 40 mA current. Thin foils were examined with a Jeol-2000EX TEM and a double-tilt stage with an accelerating potential of 200 kV.

Results

Initial Microstructure

The TEM observation revealed the heterogeneity of the initial grain structure of the MA754 alloy (Fig.1). Similar observations were reported in [7]. Grains of two types were distinguished. The grain fraction with non-equilibrium boundaries exhibiting the specific diffuse contrast [1-4] was dominant. Remarkable continuous misorientation was observed inside these grains, which was evident from the shape of the diffraction spots elongating towards the azimuthal direction. This is indicative of the internal long range stress fields. Lattice dislocations were rarely observed inside the grains. Another structural fraction showed up as grains in the equilibrium state. Uniform diffraction contrast was observed within these grains. Their boundaries exhibited conventional extinction contours, and grain boundary dislocations were also found.

Figure 1. Microstructure of extruded MA754.

Static annealing

Static annealing resulted in no remarkable grain growth at all the examined temperatures (Table.1). A minor microhardness decrement occurred as the annealing temperature was risen (Fig.2).

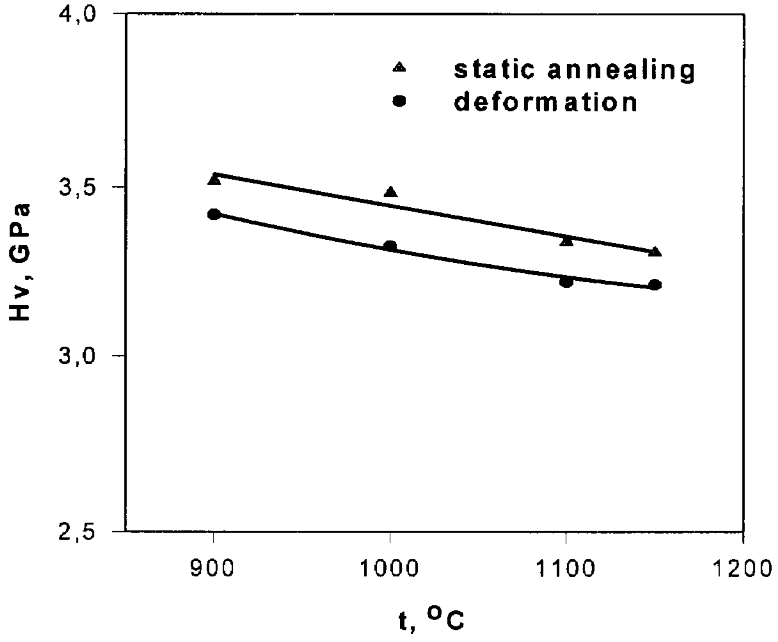

Figure 2. Microhardness *vs* static annealing temperature and deformation temperature for MA754.

The volume fraction of submicrocrystalline grains with boundaries exhibiting clearly defined extinction contours increased with temperature (fig.3). Enhanced lattice dislocation density was observed inside the grains. The oxide particles were rather effective in pinning grain boundaries to suppress grain growth. At least half of the boundaries exhibited the diffuse contrast even after annealing at t=1150^{0}C. However, from the diffraction spot shape, it was clear that a decrease in the grain body misorientation took place at annealing. Therefore, the static annealing of the MA754 superalloy provided a minor reduction in the internal elastic strain.

Table 1. Annealing temperature effect on the average grain size in the MA754 superalloy.

t,°C	800	900	1000	1150
d, μm	0.33	0.35	0.37	0.38

Hot Plastic Deformation

Typical true stress-strain curves for the MA754 alloy are shown in Fig. 4. A minor stress peak was observed at the initial stage of plastic flow. Stable stage of plastic flow was reached after ε=3-30%. A rise in temperature as well as strain rate led to an increase in the peak stress and strain at which the stable stage was reached. The strain rate sensitivity index "m" did not exceed 0.3. Moderate grain growth was observed during hot plastic deformation at t≥1000^{0}C (Table 2). After deformation at $t=1150^0C$ and $\dot{\varepsilon}=5.5\times10^{-3}\ s^{-1}$ the average grain size was greater by a factor of 1.5 than in the initial state. This is less than the reported data [7]. This gap was caused by a high temperature annealing of a MA754 rod just before plastic deformation in [7].

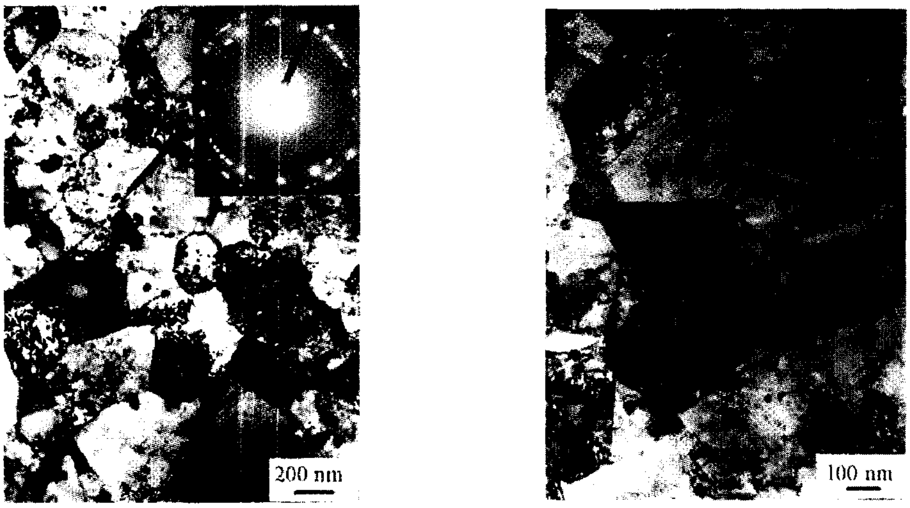

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Figure 3. Influence of static annealing at 2 hours on the microstructure of MA754 a) $t=800^oC$, b) $t=1150^oC$.

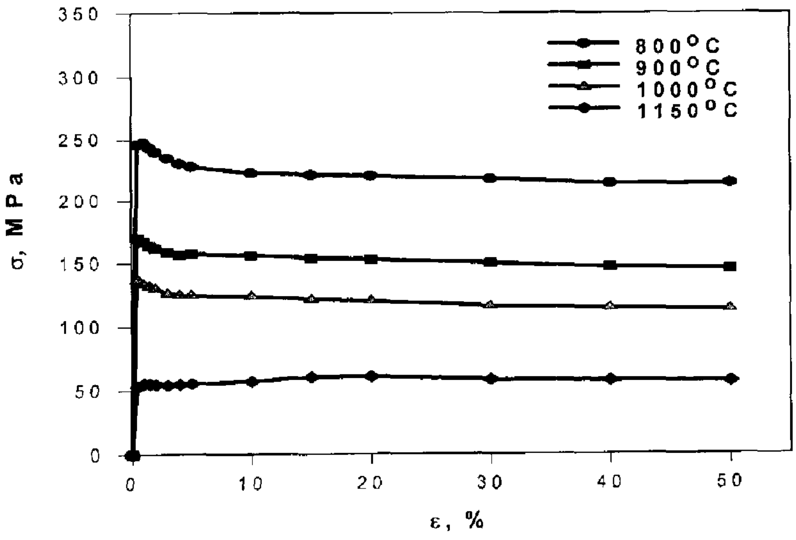

Figure 4. Typical true stress-strain curves in the MA754 alloy.

Table 2. Deformation temperature effect on the average grain size in the MA754 superalloy.

t,°C	900	1000	1100	1150
d, μm	0.40	0.45	0.5	0.53

A fully recrystallized structure was formed during the plastic deformation of the MA754 alloy (Fig.5). Almost all the grain boundaries exhibited conventional extinction contours. The diffraction contrast was uniform inside the grains, and diffraction spots took a round shape. This indicated a lack of internal stress fields.

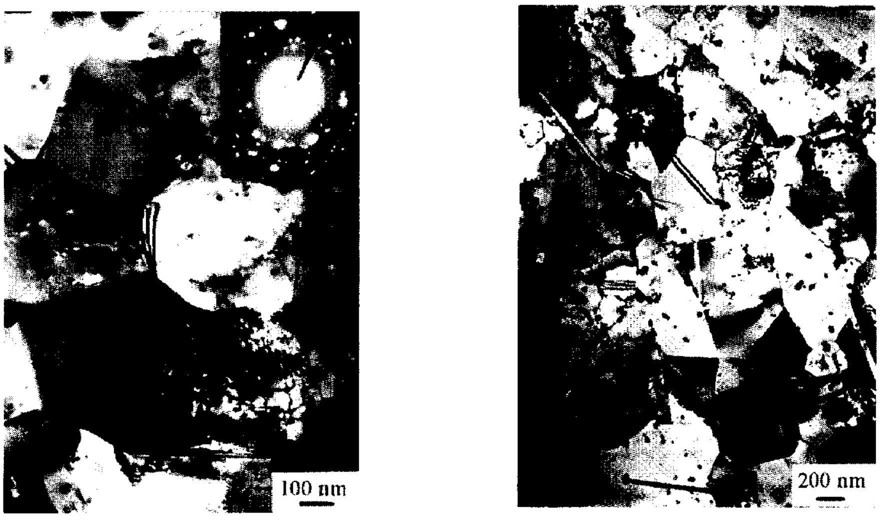

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Figure 5. Microstructures of MA754 strained up to ε=80% at a) t=1000°C, $\dot{\varepsilon}=5.5\times10^{-3}s^{-1}$, b) t=1100°C, $\dot{\varepsilon}=10^{-1}s^{-1}$

Dislocation network and low-angle boundaries were observed inside the grains. Significant number of annealing twins was found at the highest strain rate of 10^{-1} s^{-1}. Most of the grains were effectively free of lattice dislocations. At the same time, an enhanced lattice dislocation density of about $\rho\sim10^9$ cm^{-2} was observed at t=900°C and t=1150°C.

Discussion

It is known [1,3,8], that boundaries of strain induced ultra-fine grains contain pile-ups of dislocations and junction disclinations. These defects provide a significant distortion of the lattice planes in the vicinity of grain boundaries (Fig.6). The recovery in the boundaries of ultra-fine grains is due to the adsorption of the above defects. It is evident that static annealing as well as hot plastic deformation resulted in the recovery of a non-equilibrium structure of submicrometer grain boundaries in the MA754 superalloy. However, the recovery rate during static annealing was much less than that under hot plastic deformation. As a result, full transformation of the non-equilibrium boundaries into equilibrium ones did not take place (Fig.6). The grain boundary defects are partially recovered during static annealing. It provided higher thermal stability of the submicrometer grain structure. The intrinsic boundary defects inhibited the grain boundary migration.

A dramatic rise in the recovery rate during plastic deformation was caused by grain boundary sliding. Grain boundary sliding is known to step up effectively the adsorption of the grain boundary dislocations. As a result, most of the boundary defects are taken up by the grain boundaries during the plastic flow in the MA754 alloy (Fig.6). Such grain boundaries are able to migrate, and remarkable grain growth is observed during plastic flow. Annealing twins appear due to strain induced grain boundary migration. As is indicated by the ''m'' value, the dislocation glide makes the major contribution to the total elongation. Extensive dislocation slip leads to an enhanced lattice dislocation density. The formation of dislocation networks and low-angle boundaries inside the grains is caused by dislocation climb and cross-slip. The resulting structure is typical of dynamic recrystallization. It should be emphasized that at t=1150^{0}C and $\dot{\varepsilon}=10^{-1}$ s^{-1} the metadynamic recrystallization may occur. Therefore, plastic deformation is the most effective route to obtain an equilibrium submicrometer grain structure in the MA754 superalloy.

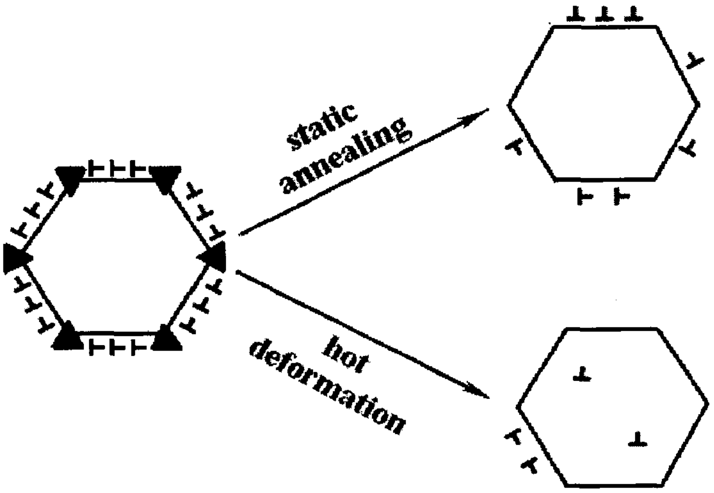

Figure 6. Scematic representation of recovery into a grain boundary during static annealing and hot plastic deformation.

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