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Strain induced abnormal grain growth in nickel base superalloys

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Abstract. Under certain circumstances abnormal grain growth occurs in Nickel base superalloys during thermomechanical forming. Second phase particles are involved in the phenomenon, since they obviously do not hinder the motion of some boundaries, but the key parameter is here the stored energy difference between adjacent grains. It induces an additional driving force for grain boundary migration that may be large enough to overcome the Zener pinning pressure. In addition, the abnormal grains have a high density of twins, which is likely due to the increased growth rate.

Introduction

Controlling grain size is necessary for nickel base superalloys, designed for high temperature applications. Under certain circumstances abnormal grain growth (AGG) occurs during the forming process [1], leading to bimodal grain size distributions that are detrimental for the in-use properties [1-3]. In the present paper, two examples of such a heterogeneous microstructural evolution are depicted and analyzed in order to identify the underlying mechanisms. The studied cases are the PER@72 and Inconel718 alloys where AGG was observed under dynamic and static conditions, respectively. Apart from their larger size these grains also exhibit a higher annealing twin density; this will also be discussed considering the microstructural parameters controlling annealing twin formation.

Dynamic strain induced abnormal grain growth in PER@72

Initial microstructure. The PER@72 alloy is a two phase (γ - γ') nickel-based superalloy (equivalent to the alloy 720 LI). It was provided by Aubert&Duval® in the form of a forged bar. The microstructure consists in fine ($\sim 7\mu\text{m}$) equiaxed grains of the γ matrix and round-shaped γ' precipitates ($\text{Ni}_3(\text{Al,Ti})$). With a typical size of 1-3 μm and a rounded shape, the latter are primary precipitates which formed during the alloy solidification. Secondary and tertiary ones ($< 0.5\mu\text{m}$) also form from the supersaturated solid solution state during cooling and aging treatment. These will not interfere with the studied mechanisms since they are dissolved at the temperature at which hot deformation was performed. Chemical segregations can not be avoided in such a heavily alloyed material and this will have to be taken into consideration when discussing the microstructural evolution mechanisms.

Thermomechanical processing and microstructure evolution. A cylindrical sample of the initial material was submitted to a hot-torsion test, up to a relatively low equivalent strain ($\epsilon = 0.12$), and quenched immediately after, while another one has been simply annealed, at the same temperature and for a time equivalent to the duration of the former torsion test. Under static annealing conditions, the microstructure is very stable (Fig. 1a), whereas large grains developed under dynamic conditions (Fig. 1b). If the torsion test is conducted to higher strains, then dynamic recrystallization is triggered and the microstructure becomes homogeneous again [4].

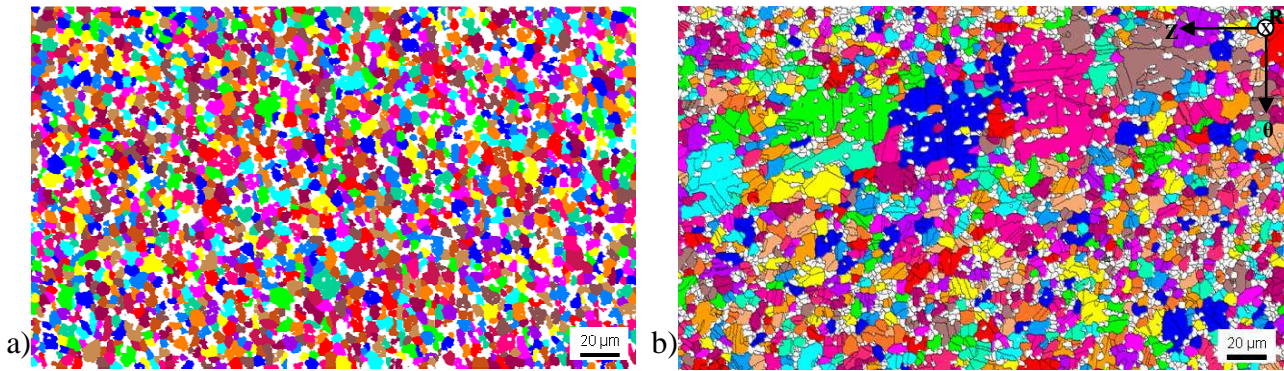


Figure 1. EBSD maps showing the microstructure of the PER@72 alloy after annealing (a), and the microstructure near the surface of the torsion test sample (radial direction normal to the map). Grains (defined as groups of neighbouring pixels being either disoriented by less than 10° or twin related) are randomly coloured. The white areas are the smallest grains, filtered out because considered to be more likely γ' precipitates than γ grains. Grain and twin boundaries are drawn in black in (b) only.

In the deformed sample (Fig. 1b), the density of secondary phase particles (SPPs) and accordingly the Zener pinning forces seem to be lower in the areas where the large grains have grown. However, these fluctuations directly arise from the chemical segregations inherent to such heavily alloyed materials. Similar chemical fluctuations exist in the other sample, which has only been statically annealed and did not develop abnormal grains. Strain has therefore an influence on the phenomenon onset. Our understanding of that case is that the stored energy gradients across grain boundaries induce an additional driving force for their migration, which allows overcoming the pinning forces, but only in the negatively segregated areas.

Static strain induced abnormal grain growth in Inconel 718

Initial microstructure. The Inconel718 alloy is a $(\gamma-\gamma'(-\gamma'')-\delta)$ nickel-based superalloy. The δ phase particles (typically $1 \mu\text{m}$ in size) are supposed to hinder grain boundary migration during thermomechanical processing. The γ' , and possibly γ'' , particles are much finer (few nm to few tens of nm typically) and they play a hardening role. Only the δ phase particles subsist at the annealing temperature which will be applied in the coming annealing experiments.

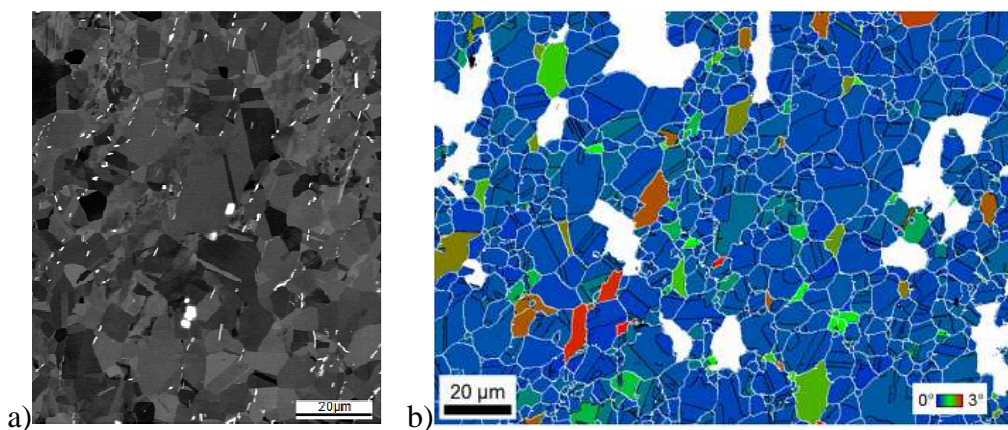


Figure 2. a) Backscattered electron micrograph of the initial Inconel718 microstructure showing white δ phase particles (and few carbides, larger). b) EBSD map coloured according to the intragranular misorientation (as quantified by the Grain Orientation Spread GOS, mean value of the misorientation angle of each grain pixel relative to the average orientation of the grain) in grains detected with a 5° threshold and ignoring twins. Grain boundaries in white, twin boundaries in black. White areas have GOS values above 3° .

The initial microstructure is shown in Fig. 2. The average grain size is 10 μ m and the GOS is very low in most of the grains. It is commonly admitted that since intragranular misorientations reveal the presence of geometrically necessary dislocations, they are indicative of the stored energy level. The actual microstructure can be interpreted as almost fully recrystallized. Only about 10% of the structure is made of hardened or recovered grains with a GOS higher than 3 $^\circ$.

Thermomechanical processing and microstructure evolution. A cylindrical sample of the latter material has been submitted to a hot-torsion test up to $\epsilon = 0.1$ in the δ -subsolvus domain. It results in a macroscopic stored-energy gradient along the radius (the strain being proportional to the distance to the neutral axis). The sample has subsequently been annealed, also below the δ phase solvus temperature, and a metallographic section has been prepared for EBSD analysis in the longitudinal plane after annealing (Fig. 3).

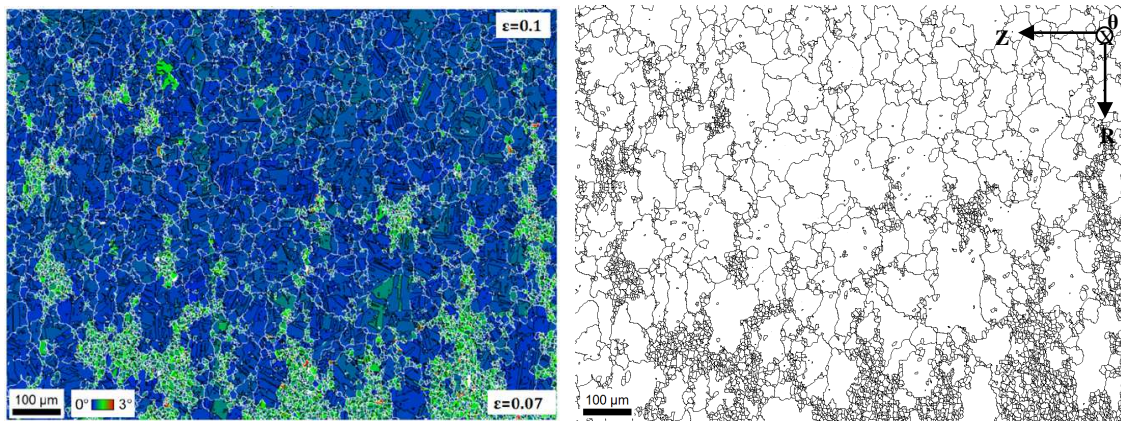


Figure 3. EBSD map of the Inconel718 sample after 10% torsion-straining and annealing. Left : same color codes as in Fig. 2. Right : only general grain boundaries (misorientation angle > 5 $^\circ$). The radial direction is vertical, the axial direction is horizontal. The cylindrical surface of the torsion test sample ($\epsilon = 0.1$) is on top, whereas the bottom of the map, located at some distance from the surface, was less deformed ($\epsilon = 0.07$).

The annealed sample shows a microstructure gradient that follows the strain gradient. In the less deformed area, large grains co-exist with small hardened one, while only large ones are obtained at larger strains. Furthermore, the large grain size is decreasing with increasing strain, as a result of a higher density of grains fulfilling the abnormal growth conditions. Not shown here, the strained microstructure was very similar to the bottom fine grain population, with a GOS level at ~ 1 to 2 $^\circ$. The abnormal grains have on the contrary low GOS values, consistent with their static development. More detailed observation of an abnormal grain (Fig. 4) reveals intragranular delta phase particles. Grains boundaries could therefore move despite the Zener pinning pressure.

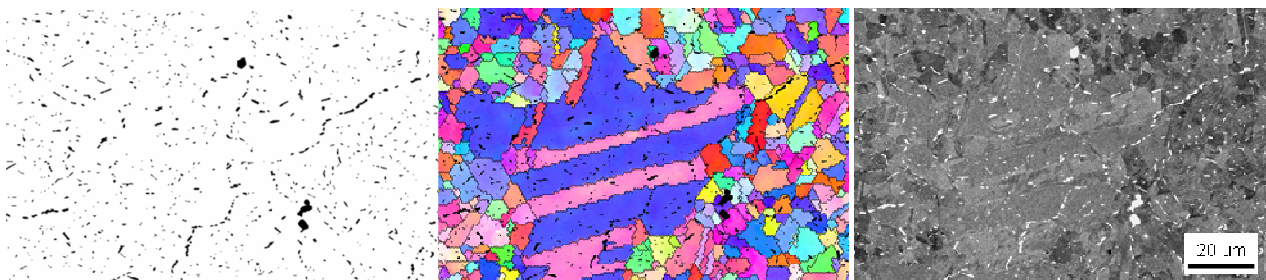


Figure 4. Middle : EBSD map (orientation colouring; twin and grain boundaries: thin black lines). Black particles are delta phase particles (left and middle), as detected on the backscattered electron image (right) micrograph.

The initial grain size was in the range of the limiting grain size that can be calculated based on the Zener model [5]. In this case, abnormal grain growth is due to grains having enough difference in stored energy with the neighbourhood, thus triggering strain induced grain boundary migration across secondary phase particles. The probability to fulfil this requirement is increasing with increasing strain, as shown by the abnormal grain size gradient.

High twin density in abnormal grains

Another feature of the abnormal grains in both alloys is a high density of annealing twins. According to Pande's model [6], the number of twins per grain is increasing with the distance over which grain boundaries have moved (i.e. grain size), and with the grain boundary velocity, which itself is usually decreasing when increasing grain size. For Inconel718, the twin density has been compared between a 40 μ m-grained microstructure obtained by normal grain growth at a supersolvus temperature ($T_{\delta}+25^{\circ}\text{C}$), and a microstructure with the same average grain size but resulting from abnormal grain growth in the subsolvus domain ($T_{\delta}-35^{\circ}\text{C}$). The abnormal grains have roughly two times more twins as compared to normal ones. This result is consistent with Pande's idea that the probability to form twins is increasing with increasing grain boundary velocity.

Summary

Abnormal grain growth occurs in two-phase Nickel base superalloys, both dynamically and statically, due to stored energy spatial heterogeneity. It could therefore also be described properly as a secondary recrystallization process. Similar phenomena have been reported in other materials as well, and referred to as "strain-induced selective grain growth" [7]. The reason for this is strain induced grain boundary migration across secondary phase particles, which occurs in low strain microstructures. In addition, in the present work, abnormal grains exhibit high twin densities, which are likely to be due to the increased grain boundary migration rate.

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