Recrystallization and the Development of Abnormally Large Grains After Small Strain Deformation in a Polycrystalline Nickel-Based Superalloy



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The formation of abnormally large grains has been investigated in the polycrystalline nickel-based superalloy René 88DT. Cylindrical specimens with a 15 μ m grain size were compressed to plastic strains up to 11.0 pct and subsequently rapidly heated to above the γ' solvus at 1423 K (1150 °C) and held for 60 seconds. All deformed samples partially recrystallized during the heat treatment, with the recrystallized grain size varying with the degree of deformation. The largest final grain size occurred in samples deformed to approximately 2 pct strain, with isolated grains as large as 700 μ m in diameter observed. It is proposed that abnormally large grains appear due to nucleation-limited recrystallization, not abnormal grain growth, based on the high boundary velocities measured and the observed reduction in grain orientation spread.

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I. INTRODUCTION

THE growth of abnormally large grains in superalloy disk components during thermomechanical processing causes a significant reduction in mechanical properties, particularly the fatigue life.^[1–4] These grains may be the result of primary recrystallization, abnormal grain growth (AGG), or some combination of the two. Understanding the source and formation processes of the abnormal grains is essential for prediction of the microstructural and the corresponding property changes during service. Ultimately, this understanding can then be used to design polycrystalline superalloys that have more optimal combinations of properties across a wide range of processing conditions.

Recovery, recrystallization, and grain growth occur in many polycrystalline materials in order to reduce the free energy of the system. Recovery involves the rearrangement and annihilation of deformation defects, typically dislocations, to lower-energy configurations. While it does not directly result in the formation of new grains, recovery can substantially influence the structure and mobility of moving boundaries, as well as in the deformed environment encountered by a recrystallization front. Recrystallization is the process by which new strain-free grains are

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formed from the deformed or recovered structure. All recrystallization processes considered in this study are discontinuous recrystallization mechanisms, where the recrystallized grains nucleate in discrete locations, then grow to consume the deformed structure.^[5,6]

Grain growth may occur after the impingement of recrystallized grains following deformation or independent of any deformation, in both cases achieving a reduction in grain boundary area upon coarsening.^[6] It has been shown that in material-specific ranges of strain, strain rate, and temperature, some grains may experience enhanced growth and will become significantly larger than the average grain size, often resulting in a bimodal grain size distribution. This phenomena is called AGG.^[6] However, it must be noted that some reported cases of AGG may actually be a recrystallization process, or "abnormal" recrystallization, in which only a few grains nucleate and grow through a deformed matrix, resulting in an exceptionally coarse grain size.^[5] This possibility has been considered in other Ni-based superal-loys by Bozzolo *et al.*,^[7,8] who proposed selective growth of the grains having the lowest stored energy, resulting in a microstructure composed of abnormally large grains. However, further evidence is required to definitely classify the abnormal grain formation process. As such, both recrystallization and grain growth should be considered as mechanisms contributing to the formation of abnormally large grains in the present study. This paper addresses the early stages of the formation of abnormally large grains in a nickel-based superalloy form the perspectives of both AGG and recrystallization. Details of each of these processes are discussed further below.

A. Abnormal Grain Growth

AGG has been observed in many different material systems, including steels, nickel alloys, and aluminum

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alloys.^[5,9,10] The proposed mechanisms for AGG generally invoke spatial or temporal variations in relative boundary mobility. Grain boundary mobility, in general, is determined by number interdependent factors, including boundary misorientation and boundary plane normal, the number and size of neighboring precipitates, dopant and impurity levels, and possibly the presence of grain boundary dislocations. Many of these factors have been specifically linked to cases of AGG, including:

- Dissolving and/or coarsening precipitates.^[11,12] As precipitates dissolve or coarsen during heat treatment, the Zener pinning force changes with time. This variable pinning force may cause larger grains to grow more easily, resulting in a broadening of the grain size distribution and thus AGG.^[5] As particles disappear, kinetics return to normal grain growth. This type of AGG has been modeled using Monte Carlo (*e.g.*, Reference 13), mean field (*e.g.*, References 14, 15), and phase field (*e.g.*, References 16, 17) approaches.
- Solute drag.^[18] Preferential segregation of solute atoms to grain boundaries occurs in many systems due to the extra free volume present at the boundary. The presence of solute at the boundary has been reported to reduce boundary mobility by orders of magnitude.^[19] In phase field simulations, solute drag has been observed to induce AGG.^[20]
- Boundary anisotropy and faceting.^[21-25] It is well understood that, in general, grain boundary energy and mobility are a function of the boundary misorientation and boundary plane normal. Monte Carlo simulations have demonstrated that AGG can be triggered by strong anisotropy of boundary properties^[24,25] The role of particular boundaries during AGG may be exacerbated by boundary faceting below a transition temperature. This phenomena has been observed in polycrystalline Ni^[21,23] and a model Ni-based superalloy.^[22]
 Textured material.^[5,26,27] Sharply textured materials
- *Textured material*.^[5,26,27] Sharply textured materials sometimes undergo AGG because of the limited mobility of boundaries between grains of similar orientations. Grains outside of the primary texture component will have a strong growth advantage.
- Deformation.^[7,28–31] Specific types of deformation pathways, particularly small deformations (a few percent plastic strain), have been reported to promote AGG in a range of materials, including the material employed in this study, René 88DT.^[28,31] However, as discussed further in this paper, it is possible that many of these are actually instances of primary recrystallization resulting in a large final grain size rather than conventional AGG.

Any or all of these driving forces may contribute to AGG. Nickel-based superalloy components are particularly vulnerable to AGG, likely due to the existence of several populations of precipitates of varying sizes, which known to affect boundary mobility, a known tendency to produce faceted grain boundaries, and gradients in the amount of plastic deformation induced during thermomechanical processing.

B. "Abnormal" Recrystallization

Recrystallization occurring in specific ranges of temperature, strain, and strain rate can cause a microstructure composed of "abnormal" grains of exceptionally large size, similar to those that result from AGG.^[7,8] This typically occurs when only a small number of recrystallization nuclei are able to form. These nuclei grow, driven by the difference in stored energy between the deformed and recrystallized structure, until impingement occurs. This is distinct from AGG, where abnormal grain formation is initiated by differences in boundary mobility and the growth advantage of a small number of grains.^[5]

The relatively low energetic driving force for recrystallization in comparison to other solid state transformations, such as precipitation, results in high sensitivity to nucleus formation. The driving force is typically two to three orders of magnitude smaller than the driving force for a phase transformation. Additionally, the energy penalty associated with the new high angle grain boundary created by the nucleus is quite large. As a result, the critical nucleus size for recrystallization is large, typically greater than 100nm even at moderate strains.^[32,33] At lower levels of deformation the driving force for recrystallization will be even lower, requiring an even larger critical nucleus size. Thus, nucleation events under low-strain conditions will become statistically more uncommon, so there will be fewer recrystallized grains when compared to a more heavily deformed sample. As such, the final recrystallized grain size tends to decrease with increasing strain beyond a critical minimum strain level required for recrystallization to occur. This trend has been well-documented, and was included in the "laws of recrystallization" proposed by Burke and Turnbull in 1952.^[32]

While abnormal recrystallization and AGG can result in similar microstructures, the underlying mechanisms are distinct. They must be considered separately in order to design alloys and processing schedules that are resistant to abnormal grain formation and the corresponding loss of desired mechanical properties. This study examines the formation of abnormal grains in René 88DT after small to moderate deformations during heat treatment above the γ' solvus temperature. Both AGG and abnormal recrystallization mechanisms are examined as potential sources of the abnormally large grains.

II. EXPERIMENT

The René 88DT material, with nominal composition shown in Table I, used in this study was received as a powder-extruded billet with a grain size of 1 to 2 μ m. Individual cylindrical specimens were cut from the as-received extruded bulk sample using electrical

 Table I.
 Nominal Composition of René 88DT in Weight Percent

Al	Ti	Cr	Co	Zr	Nb	Мо	W	С	В	Ni
2.1	3.7	16	13	0.04	0.7	4	4	0.07	0.015	bal

discharge machining (EDM). Samples measured 4 mm in diameter and approximately 4 mm in height, with the cylinder axis parallel to the extrusion direction. The EDM-affected layer was removed using silicon carbide paper. The samples were subsequently heat treated at 1423 K (1150 °C) for 30 minutes to achieve an average grain size of approximately 10 to 15 μ m, to facilitate direct comparison to similar experiments conducted in Reference 28. No abnormal grains were observed after this initial heat treatment.

The samples were uniformly compressed at room temperature to plastic strains up to 11.0 pct, based on the final length change, in an Instron Model 5582 system fitted with SiC dies. Both dies and specimens were lubricated with molybdenum disulfide powder.

Following deformation, the samples were heat treated above the γ' solvus, 1380 K (1107 °C), to initiate the formation of abnormally large grains. Samples were heat treated at 1423 K (1150 °C) for approximately 1 minute *via* induction heating at a nominal frequency of 250 kHz in air. The temperature was measured using a two-color pyrometer and the power was controlled manually to maintain a constant temperature. The sample reached the target temperature within 8 seconds and quickly stabilized. After 55 seconds at temperature, the power was instantaneously turned off and the sample was allowed to air cool. The measured temperature of the sample fell below 973 K (700 °C) (the minimum temperature able to be measured with the pyrometer) within 10s. Additional samples were heat treated in a box furnace in air at 1423 K (1150 °C) for 1 hour to examine the structure at longer exposure times.

After heat treatment, sections were cut from the central region of the compression specimens *via* EDM. These sections were hot mounted and polished *via* conventional metallographic techniques, with 0.25 μ m colloidal silica as the final polishing step.

Microstructural observation via backscattered electron imaging (BSE) and electron backscatter diffraction (EBSD) was conducted using an FEI XL30 scanning electron microscope (SEM) equipped with a Hikari XP EBSD camera from EDAX, Inc. The EBSD datasets presented in this work were analyzed using inverse pole figure (IPF) and grain orientation spread (GOS) maps. IPF maps provide information on the crystallographic orientation of each sampling point. The GOS of a grain is defined as the mean misorientation (in degrees) of each EBSD sampling point within a grain, compared to the grain average orientation. Grains that have high values of GOS contain higher densities of geometrically necessary dislocations and therefore a higher level of stored energy. Conversely, recrystallized grains will have low GOS values.

III. RESULTS

The pre-deformation René 88DT microstructure is shown in Figure 1. The average grain size is approximately 10 to 15 μ m, with a high proportion of annealing twins. The bright contrast particles are carbides and borides, while the smaller dark contrast cuboidal precipitates (see inset) are secondary γ' . The grain boundaries in this condition appear serrated or wavy.

Examination of specimens subjected to the short-duration heat treatments reveals information about the formation of the abnormal grains. Figure 2 shows BSE images for the compressive strain levels of 0, 1.8, 4.7, and 11.0 pct after the 1 minute heat treatment. Specimens at all deformation levels contained approximately 50 vol pct transformed grains, with average transformed grain sizes of approximately 25, 300, 75, and 25 μ m, respectively. Strong preferential nucleation is observed in the interior of the sample, as shown in Figure 3. No abnormal grains are initially observed to form in a 100 to 200 μ m layer at the surface of the specimen, whereas relatively uniform nucleation is observed in the interior. This is likely a result of non-uniform deformation near the specimen surface during compression.

Figure 4 illustrates the approximate maximum observed grain size after heat treatment as a function of the imposed compressive strain level. The maximum recrystallized grain detected was approximately 750 μ m in diameter, in a sample deformed to 1.8 pct. For larger deformations the recrystallized grain size decreased with increasing deformation. At 11.0 pct strain, the average diameter of the transformed grains was 25 μ m, close in size to the control sample that was not deformed.

An IPF map of an abnormal grain in a 1.9 pct deformed sample is shown in Figure 5. In this and all other IPF maps shown in this work, the reference direction is the page normal. Within the abnormal grain numerous Σ 3 boundaries are visible.

Figure 6 is an IPF map and the corresponding GOS map from a sample deformed to 1.7 pct strain and subsequently subjected to the 1 minute induction heat treatment. Again, twins are present both in the fine grained matrix and within the abnormal grains. No strongly preferred orientation or misorientation relationships are observed. In the GOS map, blue grains have the least internal misorientation, while red grains have greater amounts of internal misorientation. The transformed grains have the smallest amount of internal misorientation of any grains present in the microstructure despite their large size.

Additionally, the matrix grains clearly have non-uniform storage of dislocations during deformation; some matrix grains have minimal GOS while others display greater than 3 deg, sometimes in close physical proximity. A histogram showing the relative frequency of grain boundaries with given ΔGOS values, defined as the absolute value of the difference in GOS between a grain and its neighbor, is presented in Figure 7. All boundaries belonging to grains larger than five EBSD sampling points are considered. Approximately 0.5 pct of the grain boundaries present in the deformed microstructure have ΔGOS values greater than 1.5 deg.



Fig. 1—BSE images of the René $88 \mathrm{DT}$ microstructure prior to deformation.

The abnormal grain boundary velocity has been estimated for the largest grains, which are approximately 700 μ m in diameter. For these calculations, the recrystallized grains are assumed to grow with a constant velocity in all directions and growth is assumed to end when the induction coil is turned off. Growth is unlikely to continue substantially past that time, because the sample temperature drops rapidly, falling below 973 K (700 °C) in less than 10 seconds. Assuming as a



Fig. 3—Composite BSE image showing strong preferential nucleation in the interior of the compression specimen. Specimen was compressed to 1.8 pct plastic strain and heat treated for 75 s at 1423 K (1150 $^{\circ}$ C).



Fig. 2—SEM images for (a) 0 pct, (b) 1.8 pct, (c) 4.7 pct, and (d) 11.0 pct deformation after a 1 min heat treatment at 1423 K (1150 $^{\circ}$ C). Section normal is perpendicular to the compression direction.

lower bound immediate nucleation of the transformed grains upon reaching peak annealing temperature, the abnormal grain front velocity is at least 5.6 μ m/s. If the grain had instead nucleated after 30s of heat treatment, the velocity would be 11.2 μ m/s.

Figure 8 shows BSE images of René 88DT samples that were deformed to 0, 1.8, and 3.2 pct and subjected to the 1 hour heat treatment for nearly complete recrystallization. Again, the material with the lower plastic strain developed a substantially larger grain size after heat treatment than the material subjected to higher strains. The average abnormal grain sizes are approximately 400 and 200 μ m for the 1.8 and 3.2 pct



Fig. 4—Maximum observed grain diameter after 1 min heat treatment at 1423 K (1150 °C) as a function of compressive strain level.

samples, respectively. The maximum grain sizes are approximately 800 and 500 μ m, respectively. When a sample not subjected to any deformation was given the same long-duration heat treatment, 1 hour at 1423 K (1150 °C), no abnormal grains were observed to form. Only a modest increase in grain size, to an average grain diameter of approximately 20 μ m, is observed.

IV. DISCUSSION

In the present work, the formation of abnormal grains in René 88DT is observed within 1 minute of heat treatment above the γ' solvus in samples deformed at room temperature. The largest abnormal grains occur in material strained to approximately 2 pct, after which point abnormal grain size decreases with increasing deformation. These abnormal grains have exceptionally low levels of internal misorientation in comparison to the original matrix grains. These observations suggest that the abnormal grain formation under these conditions should be more properly considered a recrystallization process, *not* a result of AGG.

This does not imply that AGG is not possible in this system; on the contrary, it has been demonstrated that AGG will occur after long (50 hours) heat treatments in the absence of deformation.^[28] However, this remains a distinct process from the recrystallization observed here. Boundary velocities and driving forces are considered in more detail in the following sections.

A. Abnormal Grain Front Velocity

A comparison of the velocity of the abnormal grain front with AGG and recrystallization front velocities



Fig. 5—(a) IPF map of an abnormal grain in René 88DT. (b) Twin boundaries present in the EBSD map. Σ 3 boundaries are shown in red, Σ 9 boundaries are shown in blue, and all other grain boundaries are gray. For this and all other references to color, the reader is referred to the online version of this article. The compression direction is normal to the page.



Fig. 6—IPF map (left) and GOS map (right) illustrating the low-strain large recrystallized grains surrounded by smaller deformed grains. This sample was deformed 1.7 pct and heat treated for 60 s. Section normal is perpendicular to the compression direction.



Fig. 7—Relative frequency of ΔGOS values for grain boundaries present in the microstructure displayed in Fig. 6.

from studies on these phenomena from the literature is given in Table II. The abnormal grain front velocity observed here is an order of magnitude higher than velocities reported for AGG and instead falls within the ranges reported for recrystallization.

In both processes, the boundary velocity can be considered the product of the mobility and the driving force for boundary migration. Generally, the boundary mobility will be considered to be intrinsic to the boundary structure and chemistry, and therefore equal during AGG and recrystallization in a given system. As such, the boundary velocities will be more strongly correlated with the driving forces for boundary motion. The velocities for AGG tend to be one to two orders of magnitude lower than those for recrystallization processes because capillarity is the only driving force instead of including a reduction in stored deformation energy.

B. Mechanisms for the Formation of Abnormally Large Grains

In general, recrystallization will produce an exceptionally coarse grain size when the number of active nucleation sites is small in comparison to the number of grains in the assembly. In this work, the decreasing recrystallized grain size with increasing compressive strain suggests that additional strain creates or activates more nucleation sites. This increasing number of nucleation sites results in a smooth transition from "abnorrecrystallized grains to a conventional mal" recrystallized microstructure. After a compressive deformation of 11.0 pct the grain size is the effectively unchanged by recrystallization, suggesting that the number of active nucleation sites for is approximately equal to the original number of grains in the system. If the larger deformations were imposed, it is likely the



Fig. 8—Grain structures of René 88DT samples compressed (a) 0 pct, (b) 1.8 pct, and (c) 3.2 pct and then heat treated for 1 h at 1423 K (1150 °C). The compression direction is out of the plane of the page.

Table II. Recrystallization and AGG Front Velocities Measured in the Literature

	Velocity (µm/s)	T/T_m	Strain	Notes
Recrystallization Fronts				
René 88DT	5.6 to 11.2	0.87	1.8 pct	Present study
Al-0.05wt pctSi	1 to 25	0.6 to 0.7	30 to 70 pct	Reference 41
99.995 pctNi	0.5 to 4	0.4	25 pct	Reference 42
High purity Al	15 to 26	0.8	7.5 pct	SIBM in Reference 32
Abnormal Grain Growth Fr	onts		1	
Nanocrystalline Ni	up to 0.7	0.73	_	Reference 18
99.97wt pctNi	0.6 to 0.7	0.55 to 0.95		Reference 43
Pt (2D matrix)	0.6	0.86	_	Reference 10
Fe-2.5wt pctSi	0.6	0.65		Reference 10
99.999wt pctAg	0.9	0.87	—	Reference 10

final recrystallized grain size would decrease further as additional nucleation sites are created or activated.

This strong dependence on the formation of nuclei suggests that the mechanism of nucleation is crucial. As with all recrystallization processes, the nuclei must already exist at the end of deformation; the critical nucleus size for recrystallization processes is too large to form via random thermodynamic fluctuations.^[33] At these very low strains, the most likely nucleation mechanism is strain-induced boundary migration (SIBM). SIBM is a nucleation mechanism in which a part of a pre-existing high angle grain boundary "bulges" into an adjacent grain. The bulged region sweeps up local dislocations as it migrates, leaving behind a more perfect region of crystal.^[34,35] The formation or stabilization of this bulge is the critical step for SIBM, typically requiring an incubation time before recrystallization begins. However, the initial grain boundaries present in René 88DT are already wavy or serrated, which has been attributed to distortion by the γ' precipitates formed during cooling.^[28] The very short incubation time required for nucleus formation suggests that pre-existing grain boundary serrations may accelerate recrystallization via SIBM in this material.

The first model of the kinetics of SIBM was developed by Bailey and Hirsh.^[36] In their analysis, the critical radius (R_{crit}) for a nucleus is given by

$$R_{\rm crit} > \frac{2\gamma_b}{\Delta E}$$

where ΔE is the difference in stored energy between the two grains and γ_b is the energy of the high angle grain

boundary. Note that this analysis shows that as the difference in stored energy increases, the critical radius of the grain boundary bulge decreases, making nucleation easier. The GOS results presented in Figures 6 and 7 illustrate that for small deformations in this material, the strain is stored very inhomogeneously. Inhomogeneously reported in this material, both after moderate strains at elevated temperature^[37] and during the elasto-plastic transition and early stages of plastic deformation at room temperature.^[38]

This inhomogeneity of deformation after low strains creates a relatively small number of regions in the microstructure with relatively high ΔE values, where nucleation via SIBM is very favorable. The boundaries with ΔGOS above 1.5 deg identified in Figure 7 and comprising approximately 0.5 pct of the total number of boundaries would be particularly favorable nucleation sites. This relative scarcity of favorable nucleation sites would promote a very coarse recrystallized grain size. At higher levels of deformation a greater fraction of grains will accumulate enough deformation damage to serve as recrystallization nucleation sites and it is possible that other recrystallization mechanisms may also be activated. This would produce the observed trend of increased frequency of nucleation with increasing strain.

C. The Role of Boundary Mobility

While the availability of nucleation sites appears to be the determining factor for the recrystallized grain size, this does not preclude grain boundary mobility from playing an important role during nucleation. Grain boundary mobility is particularly important for nucleation *via* SIBM though the development and growth of a grain boundary bulge. Possible factors influencing boundary mobility include γ' precipitate dissolution, anisotropy of grain boundary structure and energy, and dislocation absorption.

During heat treatment above γ' solvus, the precipitates in René 88DT dissolve concurrently with recrystallization. This dissolution will alter grain boundary mobility and likely plays a role in the velocity of the recrystallization or AGG front. However, in this set of experiments samples with no deformation did not experience AGG. With no prior deformation, there is no driving force for recrystallization to occur, so no abnormal grains form. In this system, dissolution of precipitates alone does not appear to be sufficient to cause abnormal grain formation. However, it is possible that γ' dissolution and the accompanying increase in boundary mobility are necessary for recrystallization.

It has been clearly demonstrated in nickel that both boundary mobilities and energies are strongly anisotropic, leading to faceted growth during recrystalliza-tion.^[23,39] However, the faceted boundaries do not persist after impingement, indicating this faceting is primarily due to mobility differences. It has been suggested that the orientation dependence of grain boundary mobility is not due to the intrinsic structure of the boundary, but because of the orientation depen-dence of solute segregation^[5,40] With the simultaneous dissolution of the γ' precipitates, the effect of solute on boundary mobility is highly likely to influence boundary velocity. However, the present experiments do not provide sufficient evidence to examine this quantitatively.

Other authors have suggested that dislocation absorption at grain boundaries can dramatically enhance mobility, leading to instances of AGG. This mechanism is particularly important in systems where the boundaries are highly faceted and mobility is dependent on step growth or similar mechanisms^[28,29] It is possible that this may play a role in selecting sites for SIBM nucleation, but it is unlikely that this is the determining factor.

To summarize, this study illustrates the critical importance of nucleation behavior on the evolution of grain size during recrystallization of lightly deformed René 88DT. Increased deformation promotes the formation of additional nucleation sites for recrystallization, ultimately leading to a finer grain size. Additionally, it demonstrates that the formation of abnormal grains is not a case of AGG, but instead a special case of recrystallization with a low density of nuclei.

V. CONCLUSIONS

Recrystallization occurs during heat treatment above the γ' solvus at 1423 K (1150 °C) in René 88DT deformed to strains between 1.7 and 11.0 pct. The

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largest recrystallized grains observed were approximately 700 μ m in diameter after 60s of heat treatment and occurred in samples deformed to approximately 2 pct. Above this threshold in strain, the recrystallized grain size decreases, becoming equal to the initial grain size at compressive strains of approximately 11 pct. This is attributed to an increase in the number of active recrystallization nuclei with increasing deformation. Contrary to what has been previously reported in the literature, conventional AGG behavior (driven by a reduction in grain boundary energy and characterized by an average AGG front velocity less than 1 μ m/s) was not observed after small deformations in this study.

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