34.0 IN-SITU OBSERVATION OF PHASE AND TEXTURE EVOLUTION PRECEDING ABNORMAL GRAIN GROWTH IN NI-BASED AEROSPACE ALLOYS

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34.1 Project Overview and Industrial Relevance

Nickel-based superalloys are utilized extensively in the aerospace industry for their excellent high temperature strength, fatigue life, oxidation resistance and corrosion resistance. Turbine engine discs are flight critical components, and failure of these components risk loss of the entire plane. With the continuous push for more efficient commercial aviation, higher operating temperatures and pressures are desired. Complex Ni-based superalloys are being processed through novel methods to meet these stringent requirements.

The alloy utilized in the present study is RR1000, a γ-γ' disc alloy with approximately 45% volume fraction of γ' at room temperature. Processing steps include alloyed powderization, hot isostatic pressing and extrusion of a 4.5:1 reduction ratio [34.1]. These steps produce a fully densified and recrystallized billet with γ grain size of 1-5 μm diameter and a distribution of primary γ' (1-3 μm) and secondary γ' (20-50 nm). Two material conditions provided are shown in Figure 34.1; note the significant difference in fraction of primary γ'. Subsequent isothermal deformation processing of slices of material is performed near the γ' solvus temperature; secondary γ' is dissolved, while primary γ' pins the γ grain boundaries and superplastic deformation keeps flow stresses low. Super-solvus heat treatment (SSHT) allows for γ growth to approximately 50 μm for increased creep resistance during service [34.1]. Abnormal grain growth (AGG) has been shown to occur during the SSHT, based upon processing parameters in the isothermal deformation (ε, $\dot{\varepsilon}$, T) and SSHT heating rate. The AGG results in γ grains up to 3 mm that reduce mechanical performance, while being difficult to detect through non-destructive testing (NDT) techniques. The objective of this project is to better understand the microstructural mechanisms that cause AGG in these materials by in-situ experimental techniques.

34.2 Previous Work

Prior research into AGG has been most successful in exploring the processing parameters required to produce AGG, in an effort to prevent the phenomena from occurring in industrial components. Huron et al. [34.2] performed double cone isothermal compression testing on a similar alloy (René 88DT) and found a range of strain rates and deformation temperatures that produce AGG; increasing deformation temperature required higher strain rates. Parr et al. [34.3] did similar testing on RR1000 and found AGG conditions to occur at near-γ'-solvus deformation temperatures, low strain rates, and low strains, similar to those explored in the present study. In-depth work performed by Payton [34.4] explored characterization techniques in an effort to understand the microstructural mechanism behind AGG. Results indicated stored energy within the γ grains was a likely contribution to AGG, however combined contributions from γ' coherency changes and redistribution are important as well.

Work further exploring AGG mechanisms has been performed by Charpagne et al. [34.5], and proposes that stored energy is the driving force for AGG, with static recrystallization of grains being the initiation of the event. Interestingly, the γ grain boundaries appeared to pass through large, primary γ' with relatively low Zener pinning influence. Charpagne's work demonstrated continued growth of grains within critical regions until impingement limited growth, indicating nucleation limited events of grains that grow at the expense of unrecrystallized grains. The nucleation limited growth may be explained by inhomogenous distributions of stored energy that is a precursor to static recrystallization. Tu et al.'s work [34.6] supports this through strain mapping characterization techniques, demonstrating significant grain-to-grain variations in plastic strain accumulation, as well as changes in deformation mechanisms near the critical strain rates required for AGG. Based upon prior research, it appears that stored energy, accumulated inhomogeneously during isothermal forging, creates the precursor requirements for AGG.

34.3 Recent Progress

34.3.1 Thermomechanical Processing to Produce Abnormal Grain Growth

Isothermal compression of RR1000 specimens prepared via wire electron discharge machining was performed in a Gleeble® thermomechanical simulator. This allowed for control of deformation temperature, strain, and strain rate, as well as providing load-displacement data. Post-deformation SSHT of the material utilized a TA Instruments quenching dilatometer to maintain precise temperature and heating rate control, as well as measure qualitative γ' dissolution and γ grain growth behavior through changes in length. The deformation temperature, strain rate, and strain utilized in the Gleeble® to produce AGG were 1110°C, 0.0008 ε/s and 0.16 ε, respectively. Utilizing the dilatometer, a low heating rate (0.12°C/s) up to the SSHT temperature promoted AGG occurrence. Unfortunately, thermal gradients in the samples created a grain size and strain gradient.

Counterintuitively, the lower temperature side of the specimen accumulated more strain during compression. These results are corroborated by isothermal compression testing at various temperatures, showing a higher flow stress at higher temperatures for this strain and strain rate. Interpretation of preliminary results indicates one of two mechanisms are contributing to this irregular flow stress response; both involve a grain size gradient caused by dissolution of primary γ' that allows for increased γ growth in the higher temperature region. Strain localization to the small grain region may occur through dynamic recrystallization, occurring at lower temperatures than the large grain region due to grain size effects on recrystallization temperature. This results in flow softening behavior that continues strain localization, but assumes the grain size effect on reducing flow stress is stronger than the temperature effect. Furthermore, serrations typically associated with dynamic recrystallization were observed in load-displacement data for the temperatures tested. Alternatively, Coble creep may be a different mechanism that could explain the anomalous strain localization in the lower temperature, smaller grain region; however, this makes the same assumption of flow stress behavior with grain size and temperature.

The growth of abnormally large grains is sensitive to the heating rate during SSHT, particularly in the temperature regime approaching the γ' solvus temperature (1135°C). Decreasing the heating rate (to 0.02°C/s) reduced the degree of AGG and appears to promote more static recrystallization nucleation events that impinge upon each other. Increased heating rates above the critical regime decreases the degree of AGG, likely due to lower accumulated strain energy neighboring grains reaching the static recrystallization temperature. Decreased SSHT temperature shifts the location within the specimen to one of lower deformation temperature, smaller initial γ grain size, and higher strain. This supports the theory of static recrystallization driven by stored energy, with increased stored energy required for lower temperature.

Based upon experimental work performed in the present and prior studies, as well as theories presented in prior literature, it appears that AGG occurs through inhomogeneous distribution of stored energy that triggers static recrystallization during SSHT. Adjacent regions of different strains, strain rates, deformation temperatures and γ grain sizes are theorized to deform under conditions of superplasticity or dynamic recrystallization, both resulting in lower stored energy post-deformation. Due to the abnormal flow stress response to temperature, it is difficult to state with certainty at this point what deformation mechanism the neighboring regions are undergoing. Continued super-solvus temperature holding allows for AGG to consume adjacent γ grains in the higher temperature, lower strain region. Note that there exists a significant gradient in γ size in this region post-SSHT, transitioning from millimeter size to roughly 50 μm, as shown in Figure 34.2. Interestingly, regions of higher strain (relative to the AGG region) show a slow transition from the extreme AGG region down. These may be due to dynamic recrystallization and concurrent flow softening behavior, with a thermal gradient inducing a strain gradient that drives progressive static recrystallization with increasing temperature. Further experimental work is necessary to provide reliable data for generating a mechanistic theory behind AGG.

34.4 Plans for Next Reporting Period

Near term work will predominantly focus on controlling thermal and strain gradients within the Gleeble® compression testing to deconvolute effects of γ grain size and strain accumulation, however, the two appear to be intertwined with each other and stored energy gradients. Further work is clearly necessary to further develop or disprove the theory of deformation mechanism driven stored energy gradients resulting in AGG. To perform this, characterization and qualitative comparisons of stored energy in the deformed state of the material through micro-spot X-ray diffraction will compare statistically stored dislocation densities. Additionally, electron back scatter diffraction will be used to compare geometrically necessary dislocations within the material through mapping of local misorientations within each grain. Transmission electron diffraction is another technique to support the prior testing, as well as determine the distribution of dislocation structure within the γ and γ'.

34.5 References

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[34.6] W. Tu, T. M. Pollock. Grain Scale Straining Processes during High Temperature Compression of a PM Disk Alloy, Superalloys 395 (2008).

34.6 Figures and Tables

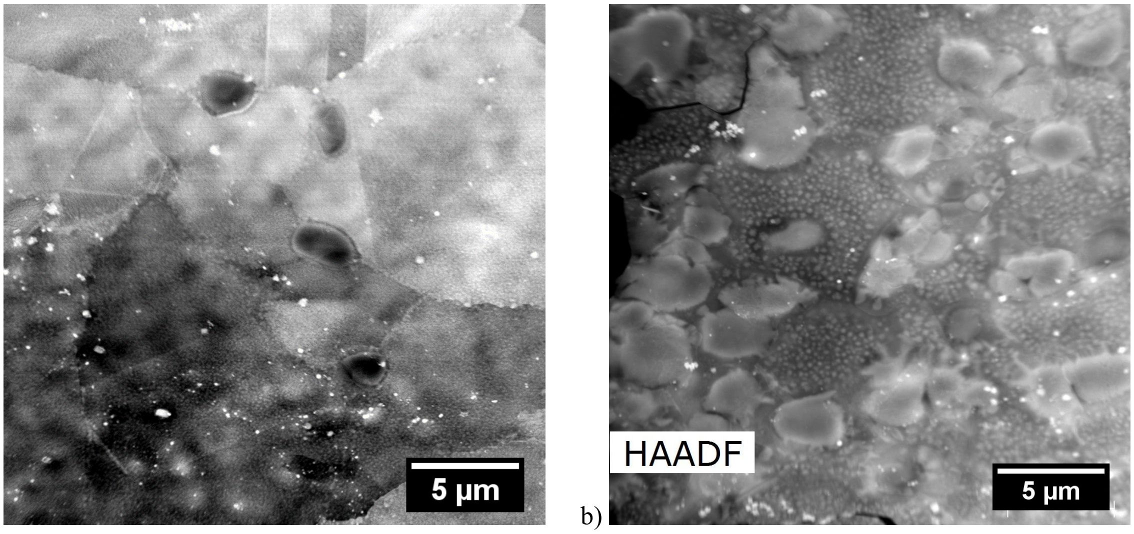
a)  b)

Figure 34.1: TEM micrographs of starting material conditions for 'Slice' 1 (a) and 2 (b). Slice 1 has primary γ' shown in darker regions, with secondary γ' dispersed throughout the γ grains. Slice 2 shows higher volume fraction of primary γ' and larger secondary γ'. Thanks to Yaofeng Gao for operating the TEM and producing these images.

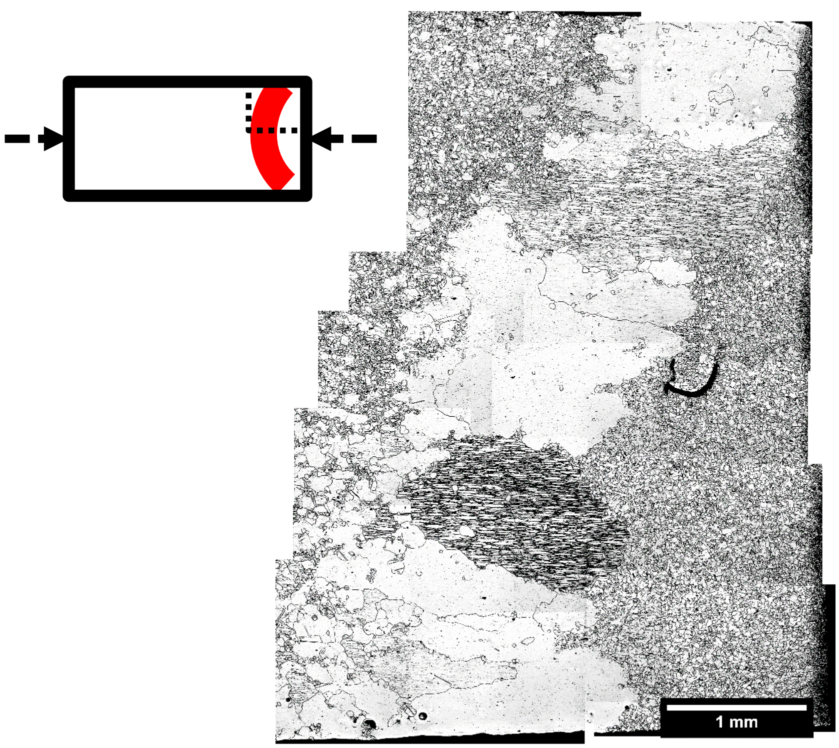


Figure 34.2: Micrograph of abnormal grain growth near the corner of an isothermal compression specimen. Note the band of abnormal grains following a specific strain and strain rate regime. Schematic with dashed box showing location of micrograph on isothermal compression specimen.