## 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 play a critical role in modern turbine engines. Their high-temperature strength, oxidation and corrosion resistance, and creep-rupture properties allow for more efficient, powerful, and reliable turbine engines for aerospace applications. Newer generations of materials have enabled higher efficiency by increasing operating temperatures and thus requiring less redirected airflow. The high pressure turbine disc materials in turbine engines require high creep-rupture strength and damage tolerance, as turbine disc failure can lead to loss of the entire aircraft. To achieve these properties, disc alloys are typically composed of roughly 50% γ' phase fraction within a γ matrix. Key microstructural variables that influence the mechanical properties of these alloys are the γ and γ' size. The alloy currently used in this application is RR1000. The γ matrix grain size is initially controlled through sub-solvus forging operations, while the γ' size is controlled by aging time and temperature (primary and secondary precipitates), as well as cooling rates (secondary and tertiary).

Manufacture and processing of nickel-based superalloys requires significant expense, due to difficulties in alloying, high flow stresses during forging, and environmental control to meet the tight requirements of the aerospace industry. Elemental segregation in nickel alloys due to solute rejection during solidification is a common issue. To remove the possibility for macroscopic segregation,RR1000 is commonly produced by argon gas atomization, powder metallurgy, and hot isostatic pressing (HIP). The HIP stage is useful for densification of the material and to reduce and/or eliminate internal voids. The HIPed billet is then extruded at a reduction ratio greater than 4.5:1 to produce a billet of appropriate size for turbine discs. This extrusion step serves to break up any micro-/meso-structure from the powder interfaces and reduce the γ matrix grain size. Isothermal forging is performed to get near net shape through sub-solvus temperatures and low strain rates ($5x10^{-2}$ to $1x10^{-3}$/s). A super-solvus heat treatment after isothermal forging allows for growth of γ grains from approximately 3μm to 40μm [34.1].

Abnormal grain growth (AGG; also known as secondary recrystallization, inhomogeneous grain growth, or critical grain growth) is a phenomena that has been observed in nickel-based superalloys and is detrimental to the mechanical properties at operating temperatures and stresses. It has been demonstrated that forging parameters during the isothermal forging operation of the material is influential in AGG and the creation of grains that are orders of magnitude larger than neighboring grains during the post-forging super-solvus heat treatment. Varying degrees of disparity occur in the growth of the γ grains; critical grain growth (CGG) has been attributed to the isothermal forging strain rate, temperature, and super-solvus heating rate, while inhomogeneous grain growth (IGG) has been attributed to isothermal forging strain and temperature. The resulting differences between CGG and IGG are the degree of grain size variation; CGG results in select grains orders of magnitude larger than neighboring grains, while IGG results in several factors larger than neighboring grains [34.2]. Despite this, Parr et al. [34.2] have agreed there is likely a connection in the mechanism behind the phenomena.

### 34.2 Previous Work

Prior research performed explored the processing conditions and possible microstructural mechanisms contributing to AGG in turbine disc superalloys such as René 88DT, René 104, René 95, Udimet 720(Li), N18, and RR1000. These materials have varying degrees of γ' phase fraction, γ' solvus temperature, γ-γ' misfits, and solid solution strengthening; however, their microstructure, processing, and response to thermomechanical processing (TMP) are comparable. The current study will focus primarily on the processing and mechanisms to produce CGG, instead of the less severe IGG. The isothermal forging temperatures and strain rates resulting in AGG were explored by Huron et. al [34.1], and occurred at forging temperatures and strain rates between superplastic deformation and higher strain rate deformation accommodated by dislocation climb, grain boundary sliding, and significant stored energy. Payton et al. [34.3] performed similar processing on René 104 and characterized the microstructure throughout different processing steps. Payton's results indicated that key microstructural contributions for AGG to occur are γ' coherency (and their Zener pinning strength), stored energy differences in γ grains from isothermal forging, and twin boundaries that likely play a combined role. In a similar study, Tu and Pollock supported this with Grain Reference Orientation

Deviation (GROD) mapping, demonstrating inhomogeneous local strain accumulation at critical strain rates in the critical region. Further testing was performed by Charpagne et al. [34.4] utilizing a similar technique to GROD, demonstrating static recrystallization.

The strain rate during hot isothermal forging appears to be influential on the deformation mechanism, which in turn allows for AGG. The previous literature described here suggests that the underlying mechanism is either (or both) stored energy differences in γ grains (in the form of dislocations or twins) or loss of γ' coherency with the γ matrix (before or during super-solvus heat treatment).

## 34.3 Recent Progress

### 34.3.1 Isothermal Compression Testing

Mults, or plates of RR1000 material have been wire electric discharge machined (EDM) and final machined on a lathe (to remove the recast and heat affected zone) to produce Gleeble compression specimens 10 mm in diameter by 15 mm in height. A Gleeble thermomechanical simulator will allow for TMP of the material at controlled strains, strain rates, temperatures, heating/cooling rates, and environments.

Scanning electron microscopy with electron back scatter diffraction (SEM-EBSD) characterization of the two plates of RR1000 demonstrated (**Fig. 32.1**) a low degree of texture in the γ specimens, and transmission electron microscopy (TEM) analysis reveals differences in γ' precipitate sizes within the γ matrix. Quench dilatometry of the as-received material re-iterated two important characteristics of the material; **Fig. 34.2** illustrates the strong γ' pinning effect on γ grains and **Fig. 34.3** shows the rapid γ grain size stabilization within two minutes of super-solvus temperature hold.

## 34.4 Plans for Next Reporting Period

The experimental portion of the project will aim at initially recreating abnormal grain growth in the γ-γ' nickel-based superalloy RR1000 and characterizing the material post-forging and post-heat treatment. Once the critical forging strain rate and temperature have been established, in-situ HEDM during isothermal forging will be performed to observe abnormal grain growth at a later time. The processing and ex-situ characterization portion of the experimental process is planned as follows:

- Perform a two variable test matrix of isothermal forging to an ideal strain of 0.3 at:
    - Temperatures of 1035-1110 °C (1895-2030°F)
    - Strain rates of 0.003 – 0.03 $s^{-1}$
- Model experiments in DEFORM® software to estimate local strains and strain rates
    - Utilize Gleeble flow curves and existing physical/mechanical property databases
- Section specimens for SEM-EBSD and TEM characterization
- Use quench dilatometer to perform sub- and super-solvus heat treatments of isothermally forged material at various heating rates
    - Characterize specimens using SEM-EBSD and TEM

The SEM-EBSD characterization will be primarily investigating local strain accumulation gradients in grain-to-grain scale, changes in microstructural texture, grain size distributions, and twinning densities. TEM analysis will be performed to analyze the γ' coherency with the matrix at various processing conditions, as well as stages in the TMP operation.

## 34.5 References

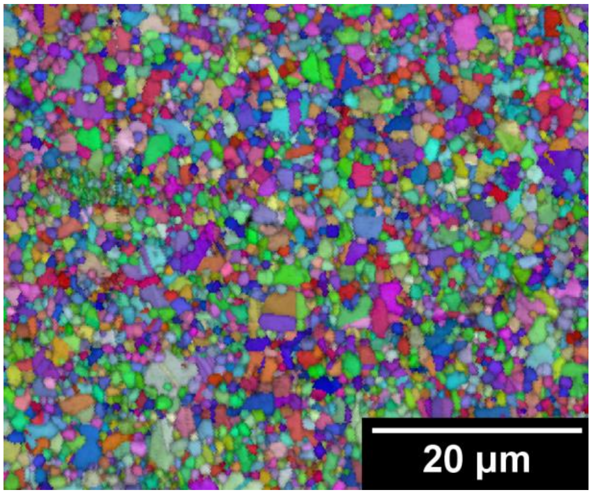

[1] E. Raymond, E. Huron, and S. Srivasta, "Control of Grain Size Via Forging Strain Rate Limits for R'88DT," *Superalloys*, pp. 49–58, 2000.

[2] I. M. D. Parr *et al.*, "Inhomogeneous Grain Coarsening Behavior in Supersolvus Heat Treated Nickel-Based Superalloys RR1000," *Superalloys*, pp. 447–456, 2016.

[3] E. J. Payton, "Characterization and Modeling of Grain Coarsening in Powder Metallurgical Nickel-Based Superalloys," The Ohio State University, 2009.

[4] M. A. Charpagne, J. M. Franchet, and N. Bozzolo, "Overgrown grains appearing during sub-solvus heat treatment in a polycrystalline γ-γ' Nickel-based superalloy," *Mater. Des.*, vol. 144, pp. 353–360, 2018.

## 34.6 Figures

a) 

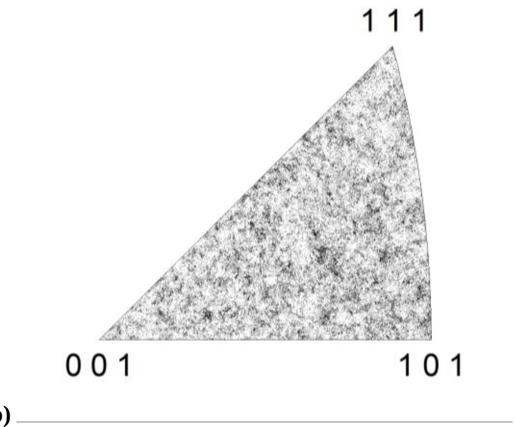
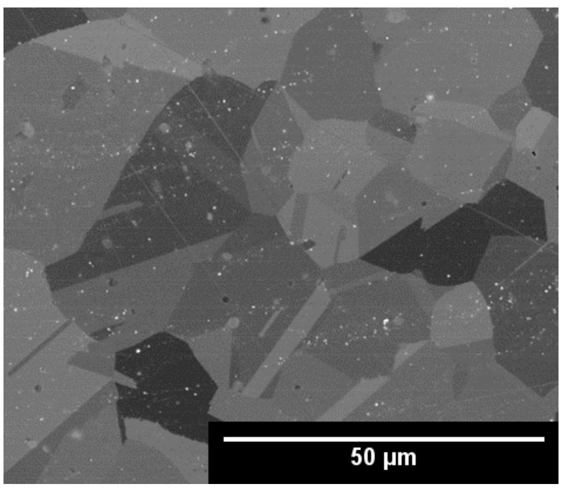

b) 


Figure 34.1: (a) Orientation map for as-received RR1000 material, and (b) inverse pole figure showing the extrusion direction.

a) 

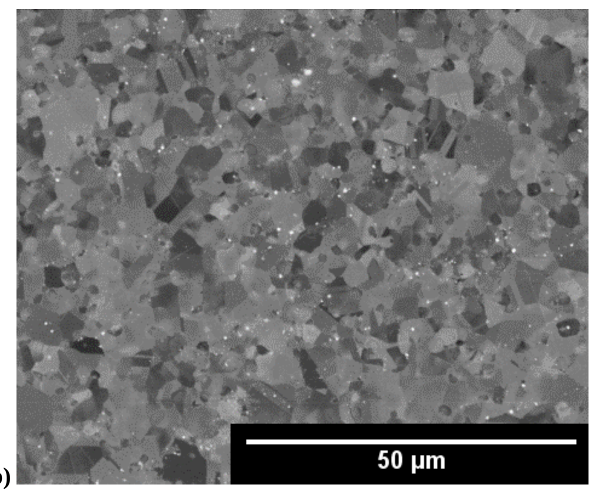

b) 


Figure 34.1: RR1000 microstructure after holding for 5 minutes at a temperature 20°C (a) above and (b) below the solvus temperature.

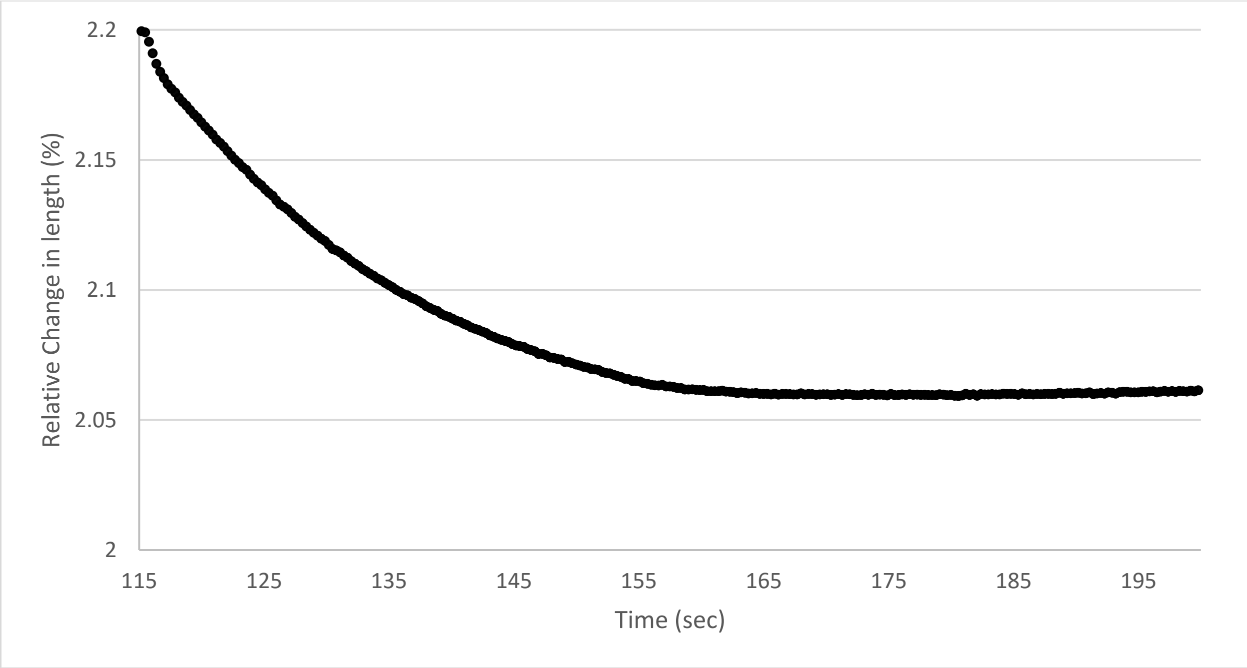



Figure 34.2: Relative change in length due to gamma grain growth during isothermal hold above super-solvus temperature. Note the rapid stabilization in length.