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# Effect of thermomechanical processing on grain boundary character distribution of a Ni-based superalloy

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**Abstract**

Grain boundary engineering (GBE) was performed on an INCONEL alloy 617 by means of thermomechanical processing (TMP) to optimize the grain boundary character distribution (GBCD) with an increased fraction of low-$\Sigma$ coincidence site lattice boundaries (CSLBs). Samples with a 5% thickness reduction followed by annealing at 1100 °C for 90 min promoted more low-$\Sigma$ CSLBs than those with greater thickness reductions. The fraction of the $\Sigma 3^n$ ($\Sigma 3$, $\Sigma 9$, and $\Sigma 27$) boundaries was promoted preferentially by TMP, which is desirable for the improvement of material's properties. The thermal stability of the GBE-optimized GBCD was verified to be stable at 850 °C for 672 h without significant degradation.


## 1. Introduction

The Ni-based superalloy, INCONEL 617 (UNS N06617), has been broadly utilized in gas turbines for combustion cans and ducts as well as industrial furnace components and applications where high-temperature corrosion resistance is generally important. Based on its excellent resistance to oxidation in a wide range of corrosive media and excellent high temperature strength, alloy 617 had been selected as a potential candidate alloy for Generation IV nuclear plant designs [1]. Alloy 617 is an austenitic solid-solution alloy with a composition Ni–22Cr–13Co–10Mo (wt%). It is normally used in a solution-annealed condition which provides a coarse grain structure for the best creep-rupture strength. However, to withstand the high temperature and pressure environments in modern and future power plant system, the properties of alloy 617 could benefit from further improvement for extending its service lifetime and thus maintaining system stability.

Grain boundary engineering (GBE), proposed by Watanabe [2] in the early 1980s, has been extensively investigated as an approach to improve properties such as creep [3] and stress corrosion cracking resistance [4], by promoting a high proportion of low-$\Sigma$ ($\Sigma \leqslant 29$) coincidence site lattice boundaries (CSLBs) in materials. In the nomenclature of the CSL model, the newly formed superlattice is denoted by a value of $\Sigma$, where the $\Sigma$ value is defined as the reciprocal of the fraction of lattice points in the boundaries that coincide between the two adjoining grains. Thus, there is a low distortion of atomic bonds and relatively small free volume for CSLBs and consequently result in low boundary

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energies. Detailed information about the CSL model and CSL effect on GBE may be found in Ref. [5].

Based on our previous work on GBE of INCOLOY alloy 800H, which demonstrated significant improvement on oxide stability by GBE during exposure to supercritical water [6], the GBE approach was investigated for alloy 617 with the goal of understanding the effect of thermomechanical processing on grain boundary character distribution (GBCD).

## 2. Experiment

Commercial alloy 617, which was solution annealed at a temperature of 1176 °C for a time commensurate with section size followed by water quenching, was procured from special metals for this study. Its chemical composition is shown in Table 1. Various combinations of cold rolling and annealing were employed as the thermomechanical processes to achieve GBE effects on the as-received samples. Based on previous experience with GBE of an austenitic alloy 800H [7], an annealing temperature, 1100 °C, followed by water quenching was used to ensure a single phase austenite. Three levels of thickness reduction (5%, 9%, and 13%), different annealing times (15, 30, 45, 60, and 90 min), and multiple cycles of thermomechanical processing were employed in this study to investigate their effect on GBCD.

Electron backscatter diffraction (EBSD) was employed for the determination of grain boundary misorientations using the TSL orientation imaging microscopy (OIM) system MSC2200 attached to a field-emission scanning electron microscope LEO 1530 FESEM. To obtain good quality EBSD patterns, the samples were mechanically polished with SiC abrasive paper down to 1200 grit followed by sequential polishing with 1 μm diamond paste, alpha alumina and colloidal silica solutions. The SEM was operated at 20 kV, and the automatic EBSD area scan was performed over an area of $500 \times 1000\ \mu m^2$ using a hexagonal grid with a step size of 5 μm. The number of scanned grains was about 1100 for each of the samples, which provided statistically significant results. To allow for the identification of phases with the EBSD analytical software, a crystallographic data file was established based on X-ray diffraction analysis. For phase analysis with EBSD, a voting scheme and the confidence index (CI) established by TSL were used [8]. A high number of votes and higher CI values indicate a higher possibility that the phase is correctly identified. The character of grain boundaries in all samples were classified according to the Brandon criterion [9] for the maximum allowable misorientation ($\Delta\theta$) from the exact CSL as given by: $\Delta\theta = \Delta\theta_m \Sigma^{-1/2}$, where $\theta_m$ is the maximum misorientation angle for a low-angle boundary (typically, 15°).

## 3. Results and discussion

As-received samples were subjected to a variety of thickness reductions and thermomechanical processing (TMP) cycles. The average grain size of as-received and GBE-treated samples is 22 μm and 25 μm, respectively. The minor enlargement of the grain size is reasonable because some random boundaries were transformed to coherent $\Sigma 3$ boundaries. Because coherent $\Sigma 3$ boundaries are immobile and not a constituent of the intergranular transport network, they were not counted as grain boundaries during grain size measurement by defining a 60° rotation about the $\langle 111 \rangle$ crystal direction with a 5° misorientation tolerance.

### 3.1. GBCD of as-received and GBE-treated samples

The EBSD analyzed GBCD of the GBE-treated samples, as well as as-received sample, are shown in Fig. 1. GBCD is composed of three categories of boundaries: $\Sigma 1$, low-$\Sigma$ CSLBs ($3 \leqslant \Sigma \leqslant 29$), and random boundaries. $\Sigma 1$ boundaries may be introduced by deformation-induced texture, which increases the probability that grains having almost the same orientation will reside as neighbors. In addition, the presence of many sub-boundaries in the material [5] and the recombination of two like boundaries impinging on each other [10] will also increase the $\Sigma 1$ fraction. Thus, the fraction of low-$\Sigma$ CSLBs does not include the contribution from

Table 1
Composition of the as-received INCONEL alloy 617 (wt%)

| Ni | Cr | Co | Mo | Al | Fe | Ti | Si | Mn | C | Cu | S | B |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| >51.4 | 21.7 | 12.4 | 10.9 | 1.2 | 1.2 | .3 | .2 | .05 | ∼.1 | <.05 | <.015 | <.006 |