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Grain Boundary Engineering of Allvac 718Plus® for Aerospace Engine Applications

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Abstract

Grain Boundary Engineering (GBE®) and GBE surface treatments (GBEST®) are patent- and trademark-protected thermo-mechanical processing technologies that are applicable to a broad range of metals and alloys. The main objective of this study was to extend the applicability of the GBE processing technologies to a more advanced superalloy, Allvac®718Plus®, which is a new derivative of Alloy 718. The specific technical objectives of the study were to: (1) develop and optimize GBE processing strategies for the bulk microstructure of the 718Plus® and (2) develop a processing methodology for locally optimizing the GBE microstructural characteristics at the near-surface region of the alloy. In this paper, the microstructural characteristics achieved from the GBE processing of the 718Plus® alloy are presented. The effect of the GBE processing on the high temperature mechanical properties of the 718Plus® alloy is presented in a separate paper in this symposium proceedings.

Introduction

The significant benefits of the GBE processing on the resistance to creep deformation and crack growth on the Alloy 718 were demonstrated in a previous study [1]. In that study it was shown that the optimized GBE microstructure resulted in a four-fold improvement in the alloy creep resistance when compared to the non-GBE counterpart. In this follow-on study the GBE processing methodology has been applied to Allvac®718Plus® nickel-based superalloy. Allvac®718Plus® alloy (a newly developed derivative of 718 alloy) has been shown to have a higher temperature capability by about 55^{o}C than the 718 parent alloy [2] that is largely due modifications to its chemistry. Relative to the parent alloy, 718Plus® contains a higher Al/Ti ratio together with higher W and Co contents. Therefore, the main objective of the present study was to extend the applicability of the GBE® processing technologies to Allvac®718Plus®, with an intent to seek further mechanical properties improvement using an approach based on grain boundary structure optimization rather than chemistry modification.

Grain Boundary Engineering (GBE®) and GBE surface treatment (GBEST®), are metallurgical processing technologies that were developed and patented by Integran [3]. These technologies involve the optimization of an alloy microstructure via the strategic application of thermo-mechanical metallurgical processing and fabrication steps that are designed to increase the fraction of special, low-energy, and degradation-resistant grain boundaries (i.e., structurally ordered low Σ grain boundaries) in the alloy microstructure. By elevating the fraction of special grain boundaries in an alloy, a commensurate improvement in the bulk material properties is achieved owing to the intrinsic degradation-resistance (sliding, cracking, corrosion) of the "special" grain boundaries. For metallic structural components that require specific treatment to improve their resistance to surface crack initiation at elevated temperatures and for metallic materials with limited room temperature ductility, GBE processing at the near-surface region is particularly relevant since it is ideally suited for these types of applications. As mentioned above, the specific technical objectives of this study were: (1) to develop and optimize GBE processing strategies for optimizing the bulk microstructure of 718Plus® alloys; (2) to develop a GBE surface treatment strategy for locally optimizing the microstructure at the near-surface region, and (3) to fabricate test sample coupons for high temperature mechanical tests using the optimized corresponding GBE processing parameters. This paper summarizes the key microstructure features obtained in the 718Plus® alloy resulting from the bulk GBE and the GBE surface treatment processing. Sample coupons obtained from six different sets of processing conditions were provided to NASA for high temperature mechanical testing, including creep, tensile strength, and fatigue tests. The influence of the GBE processing on the high temperature mechanical properties of the GBE optimized 718Plus® alloy is presented in a separate paper in the proceedings of this symposium.

Experimental

Starting Material

The Allvac®718Plus® alloy used in this study was sourced from ATI Allvac in the rod form with a 1.375" diameter and the heat number for this particular lot of material is Heat B02-J1. The actual chemical composition in weight percent is listed in Table 1 below.

Table 1: Chemical Composition of Allvac®718Plus® (wt%)

C	Mn	Fe	S	Si	Cu	Ni	Cr	Al	Ti	Co	Mo	CB+Ta	V	W	B
0.21	0.05	9.53	0.003	0.06	0.01	51.8	17.79	1.46	0.75	8.99	2.69	5.47	0.03	1.02	0.005

GBE Bulk Process Development

In an effort to maximize the fraction of special grain boundary (Fsp) content in Allvac®718Plus®, a series of thermo-mechanical processing steps were conducted in accordance with the general multi-step processing principles outlined in Integran's US Patents 5,702,543 and 6,129,795. The specific heat treatment temperatures were 950°C, 1000°C, 1025°C and 1050°C. The range of deformation examined were 5%, 10%, 20% 30% and 50%, respectively. The heat treatment times were respectively 2 and 5 minutes. Pre-straining was applied to the sample coupons by cold rolling at room temperature and the heat treatments were carried out in an air furnace. Thermocouples were mounted on the surface of each sample to ensure that the sample desired temperature was reached and that the sample was in thermal equilibrium during the short exporure time at temperature. All the heat treatments were conducted in air and followed by air cooling. All samples were slow cooled (~ 60-70°C per minute) from the target heat treatment temperature down to ~800°C by removing the sample from the middle of the hot zone to the edge of the insulating brick of the tube furnace before completely exiting the furnace.

GBE Surface Treatment Development

GBE surface treatment (as taught in Integran's US Patents #6,344,097) utilizes conventional high intensity shot peening (carbon steel shot MI-330) coupled with recrystallization heat treatment to selectively achieve an increased frequency of special grain boundaries at the near-surface region of the sample. The residual stress and the depth of the affected layer as a result of applied shot peening intensities and coverage were monitored by means of micro-hardness measurements along the cross section of the surface treated samples. Three different shot penning intensities were used (4A, 12A and 18A) corresponding to coverages of 100%, 400% and 800%, respectively. The shot peened samples were then heat treated utilizing the optimized temperature and exposure time that were determined from the corresponding GBE bulk optimization processing. All the GBE surface heat treated samples were cooled using the same procedure employed for the GBE bulk-processed samples.

Microstructural Characterization

For grain boundary structural characterization, all sample surfaces were mechanically polished to a 1 µm finish and subsequently electropolished in a solution of 10% perchloric acid in methanol at -40°C using an applied voltage of 40V for 10 seconds rendering the surface suitable for electron backscattered diffraction pattern analysis (EBSP). Specimens (positioned at a tilt angle of 70 degrees) were analyzed using a JEOL-840 scanning electron microscope equipped with an automated electron backscattered diffraction system, i.e., Orientation Imaging Microscopy (OIM) [4]. With respect to the OIM scanning parameters, the step size and scanned areas were varied according to the average grain size observed in the microstructure in order to maximize the number of grains analyzed per specimen. The crystallographic orientations determined at each step (from the electron backscattered diffraction patterns) were calibrated to be accurate within ±0.5°. All crystallographic misorientations were determined from changes in the electron diffraction pattern as the incident electron beam traverses the grain boundaries. All grain boundaries were classified in accordance with the Coincidence Site Lattice (CSL) model [5] using the Brandon's criterion [6] for allowed angular deviation,

$$\Delta\theta = 15°\Sigma^{-1/2} \qquad (1)$$

An upper limit of Σ=29 was applied in this analysis after the earlier work of Watanabe [7].

Fabrication of Mechanical Testing Coupons

In an effort to minimize the effects of cooling rates on the mechanical properties, (1) all of the base-line non-GBE processed samples also received the same final heat treatment as the GBE-processed samples, and (2) all the samples were machined into the same final dimensions prior the standard 2-stage aging treatment to minimize the effects of different thermal masses on the resulting microstructure. The dimensions of all the GBE bulk processed mechanical test coupons were 1.375" wide x 2" long x 0.6" thick; the same dimensions were also used for the non-GBE processed sample coupon counterparts. GBE bulk processed samples in six different conditions were prepared and then sent to NASA for mechanical testing evaluation. The key variations in the sample conditions (NG, GI GIP and GIIP) were the difference in the GBE processing routes (i.e., GBE I and GBE II) and the incorporation of Post Solution Deformation (PSD) step prior to the aging treatment as taught in Integran's US patent #6,129,795. The other two conditions, NGD and GIPD, were the only two groups of samples subjected to the delta-heat treatment (exposure at 930°C for 4 hours) to promote formation of delta phases. The sample nomenclatures as well as corresponding specific processing conditions are summarized in Table 2 below.

Table 2: A summary of nomenclature and processing history for samples submitted for mechanical testing

Sample Condition	Processing Sequence and Description
NG	AR → GFHT → AHT
GI	GBE I → AHT
GIP	GBE I → PD → AHT
GIIP	GBE II → PD → AHT
NGD	AR → GFHT → DHT → AHT
GIPD	GBE I → PD → DHT → AHT

AR-Material in the as-received condition, GBE I - Fsp >60%, GBE II - GBE has the highest Fsp >70%, GFHT - GBE final Heat Treatment (1025°C for 5 minutes), PD - Post Solution Deformation, DHT- Delta Heat Treatment (940°C for 4 hours), AHT - 2-stage 718 Aging Treatment (788°C for 8 hours, furnace cooled at 38°C per hour to 649°C and held for 8 hours)

Results and Discussion

GBE Bulk Processing Development

As previously mentioned, GBE processing involves repetitive deformation-annealing cycles and the methodology primarily relies on the formation of annealing twins during early stage of recrystallization to facilitate the formation of other low Σ special grain boundaries. The selection of the thermo-mechanical processing parameters (i.e., for each cycle, the appropriate combination of degree of deformation and short exposure time at an elevated temperature) is chosen to promote the formation of annealing twins. Table 3 summarizes the nomenclature, processing sequence and the final microstructural characteristics of the samples processed for mechanical testing. As shown in this table, the grain sizes of the GBE processed samples were kept in the similar range as the non-GBE sample in order to minimize the influence of grain size on the mechanical properties. However, the difference in the Fsp content was maximized and there is a ~2-fold increase in the Fsp content between the GBE and non-GBE processed samples. With respect to the applied post solution deformation (PD) step as well as the delta heat treatment (DHT) which were intended to promote formation of γ/γ' and δ phases during each perspective heat treatments, no significant changes to the microstructural characteristics (grain size and Fsp) were expected because the grain boundaries are thermally stable at these treatment temperatures.

Table 3: A summary of nomenclature, processing sequence and the final microstructural characteristics for the samples submitted for mechanical testing.

Sample Condition	Processing Sequence and Description	Grain Size (μm)	Twin Density (%)	Fsp(%)
NG	AR → GFHT → AHT	~20-35	~17	~35
GI	GBEI → AHT	~25	~45	~60
GIP	GBEI →PD →AHT	~25	~45	~60
GIIP	GBEII → PD → AHT	~25-35	~60	~71
NGD	AR → GFHT → DHT → AHT	~20-35	~17	~35
GIPD	GBEI → PD → DHT → AHT	~25-35	~45	~60

Figure 1 illustrates the grain boundary character distribution (GBCD) of 718Plus® samples in NG, GI and GIIP conditions. A general prevalence of $\sum 3^n$ (where n=1, 2 and 3) type of grain boundaries is observed for the GBE processed samples. This is indicative that the GBE processing was successful in enhancing the fraction of the special grain boundaries through multiple twinning events. The non-GBE sample displayed a much lower twin density when compared with the GBE processed counterparts, the fraction of the twin densities in the 718Plus® material was increased from 17% to as high as ~60% by using different GBE processing routes.

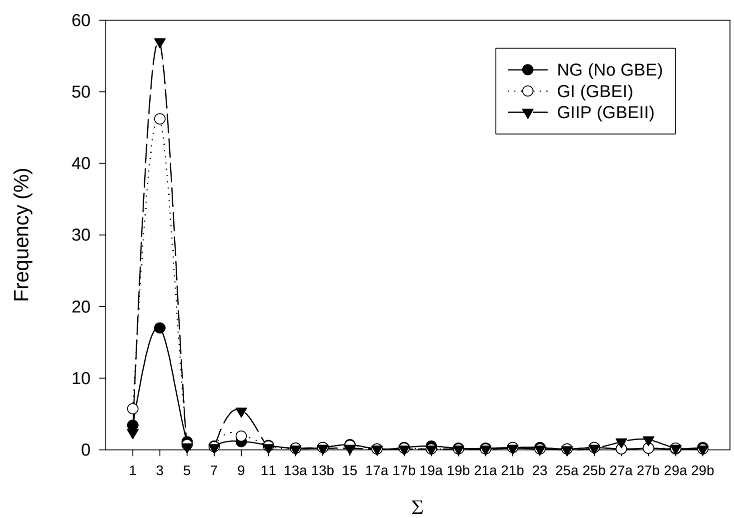
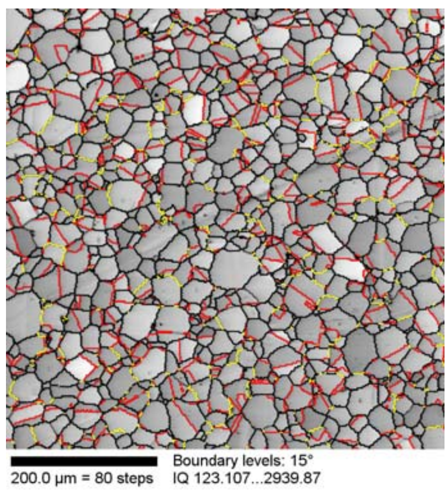


Figure 1: Illustration of Grain Boundary Character Distribution for samples in NG, GI and GIIP conditions.

Figure 2 illustrates the representative microstructures of the 718Plus® alloy in NG (Non-GBE), GI (GBE I) and GIIP (GBEII) conditions showing the increase in the special grain boundaries content following the applied GBE treatments. In the OIM micrographs, the colored lines indicate the specific grain boundary character of individual grain boundary segments in the sample areas analyzed. The boundary segments are classified as follows: red-colored lines denote low energy Σ3 grain boundaries, yellow-colored lines denotes other low Σ (i.e., 3<Σ≤29) grain boundaries, thick black lines represent general high angle grain boundaries and thin black lines show the positions of the low angle grain boundaries (i.e., Σ1). Compared to the material in the non-GBE processed condition, the microstructural characteristics of the GBE optimized samples can be summarized as follows: (1) a grain size of ≈25 μm and (2) an enhanced 'special' (i.e., ∑≤29) grain boundary content in the range of ~60%, whereas the GBE II optimized samples achieved an enhanced 'special' (i.e., ∑≤29) grain boundary content in the range of ~70% accompanied by a slight increase in grain size.

NG


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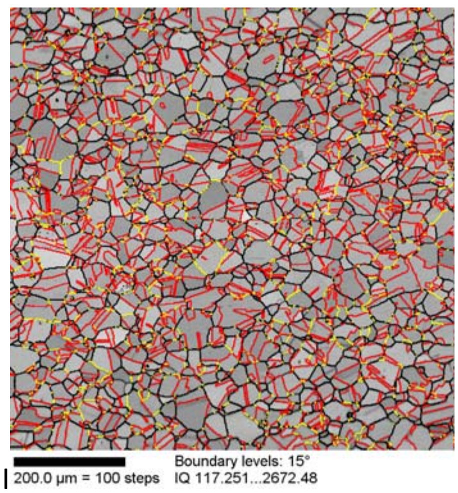


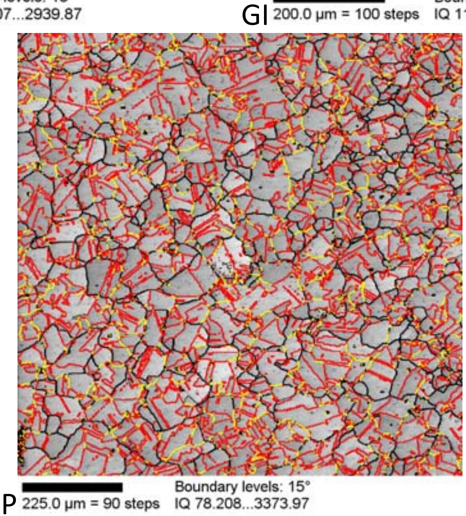
GIIP


Figure 2: OIM images of the 718Plus® Alloy in NG (No GBE), GI (GBE I) and GIIP (GBEII) conditions showing the increase in the special grain boundaries content following the GBE processing. The boundary segments are classified as follows: red lines denote low energy Σ3 grain boundaries, yellow denotes other low Σ (i.e., 3<Σ≤29) grain boundaries, thick black lines

represent general high angle grain boundaries and thin black lines show the positions of the low angle grain boundaries (i.e., Σ1).

Figure 3 summarizes the results obtained from the discrete triple junctions analysis (approximately 200 junctions from each alloy sample were included in the statistical analysis results); each triple junction was classified on the basis of their specific 'special' grain boundary character (i.e., 0, 1, 2 or 3-special at intersection of special grain boundaries) at triple junctions. As shown in this figure, the GBE and the GBE II processed alloy samples displayed fewer triple junctions comprised entirely of random grain boundary segments; also, the number of triple junctions comprised entirely of special grain boundaries was increased significantly as result of the GBE processing. The enhancement in the grain boundary network connectivity has been previously shown to create a more tortuous path for crack propagation [1] and this is expected to lead to an increased overall cracking resistance in the GBE processed material.

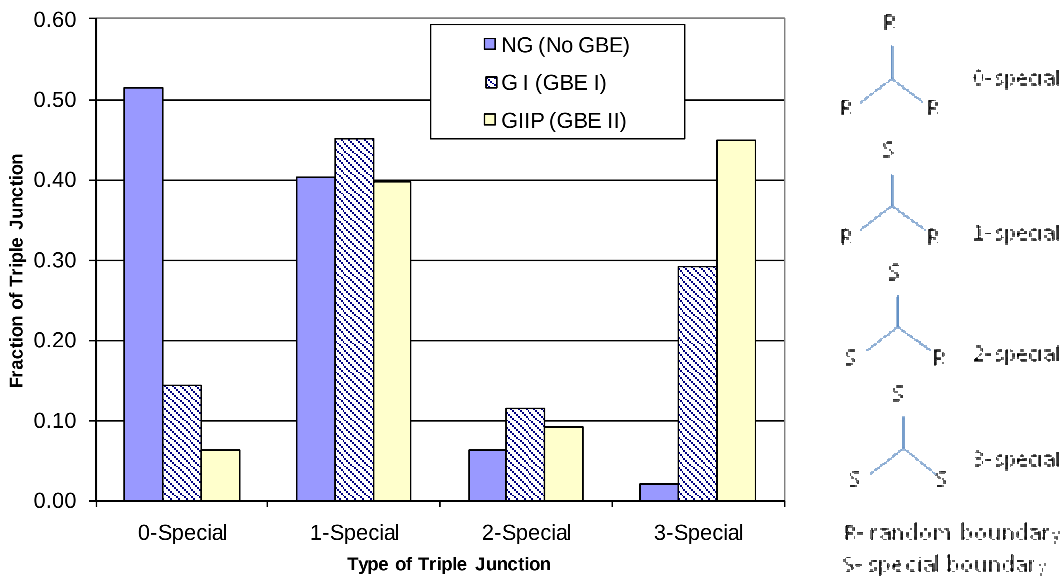


Figure 3: Discrete triple junction character analysis of specific 'special' grain boundary frequency (i.e., the junction comprised of 0-, 1-, 2-, 3-special) from OIM micrographs of the NG (No GBE), GI (GBEI) and GIIP (GBE II) processed 718Plus®.

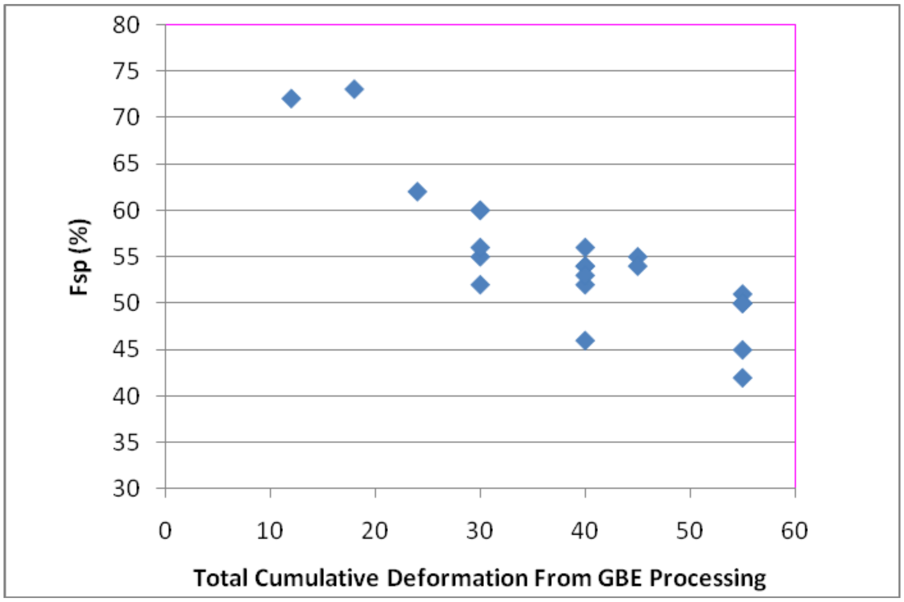


Figure 4: Illustration of the frequency of special grain boundaries (Fsp) as a function of the total cumulative deforamtion from GBE processing for 718Plus® alloy.

Figure 4 summarizes the evolution of the fraction of special grain boundary as a function of the total cumulative deformation for the GBE processed 718Plus® alloy. These data collectively show that the applied deformation during the GBE processing has a significant impact on the final special grain boundary content (Fsp); the Fsp tends to increase with decreasing total cumulative deformation.

GBE Surface Treatment Development

Figure 5 illustrates the evolution of the surface microstructure following three iterations of the applied GBE surface treatment. The applied treatment involving three iterations of shot-peening and heat treatment cycle was successful in increasing the Fsp content from ~30-35% to approximately 55%-60% in the near-surface region of the sample. Multiple cycles of surface treatment was shown to be effective in promoting and retaining a relatively high Fsp content in the near-surface region of the 718Plus® alloy samples. However, evidence of grain growth is noted in the layer residing between the unaffected base material and the thin layer just below the shot-pinned-GBE modified surface. Given that the achieved microstructure is fully recrystallized and free of any residual deformation, from the OIM images (see Figure 5), it is reasonable to assume that the GBE surface-modified samples do not retain any significant residual stress, following the combination of shot peening and heat treatment.

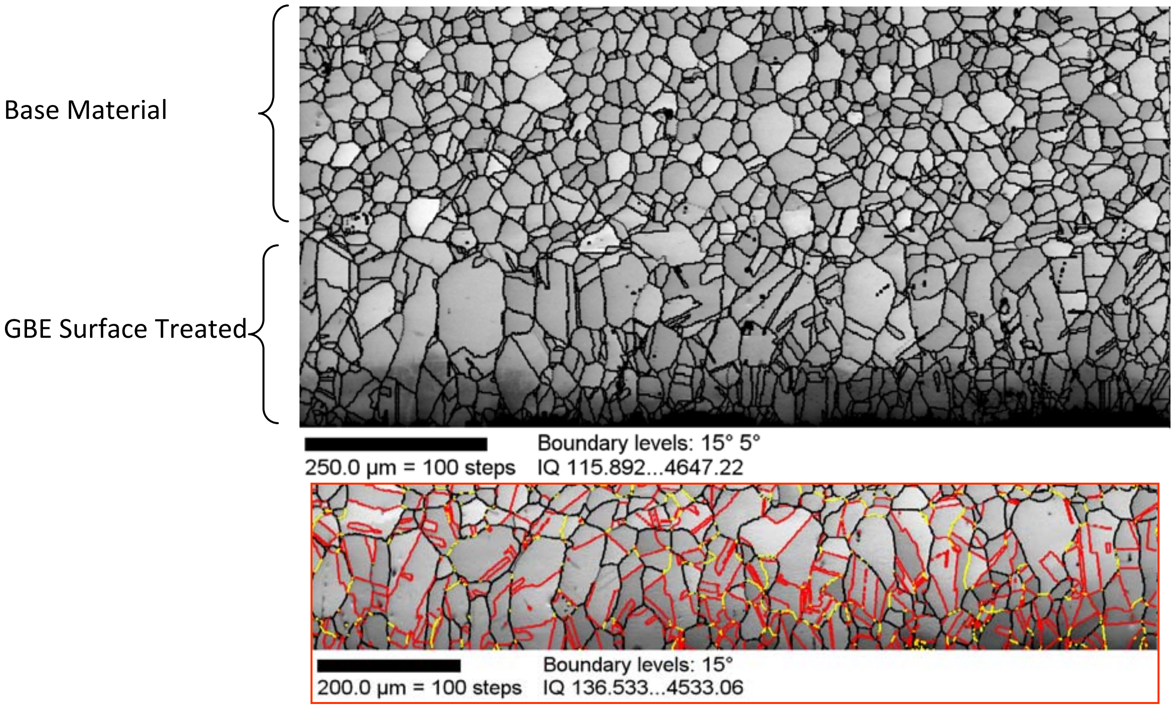


Figure 5: OIM images of the GBE surface treated 718Plus® sample showing the enhanced Fsp content near the surface region following three cycles of surface treatments.

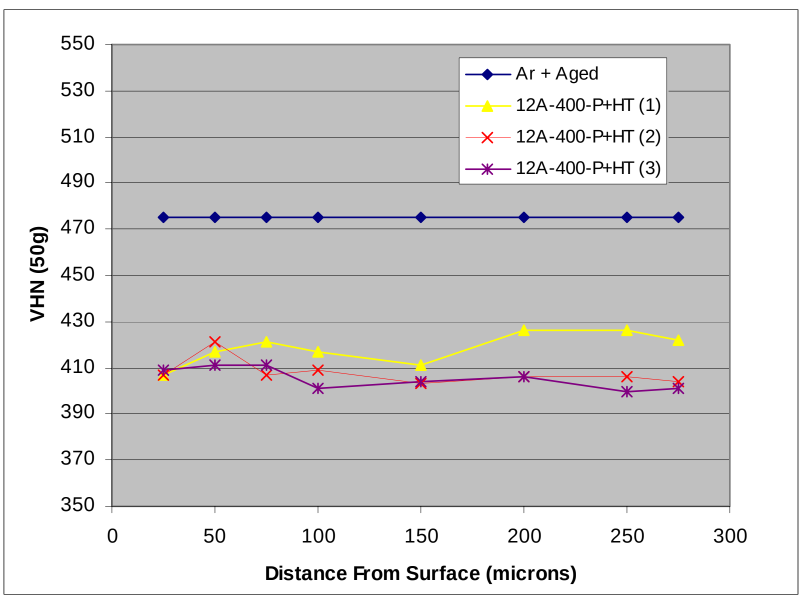


Figure 6: Illustration of the changes in the surface hardness following the applied GBE surface treatment on 718Plus® alloy.

The assessment of residual stresses in the GBE surface treated samples was subsequently determined by micro-hardness measurements and the result are summarized in Figure 6. In 718Plus® alloy, compared with the starting sample in the fully aged condition, a general decrease in the surface hardness in the near-surface region was observed on the samples that had been subjected to the GBE surface treatments. This decrease in hardness can be attributed to the relatively high GBE heat treatment temperature (1025°C) that was applied which is above the solvus temperature of the γ/′γ. The standard 2-stage aging treatment needs to be reapplied in order to restore the attendant mechanical properties

Preliminary Mechanical Testing Result

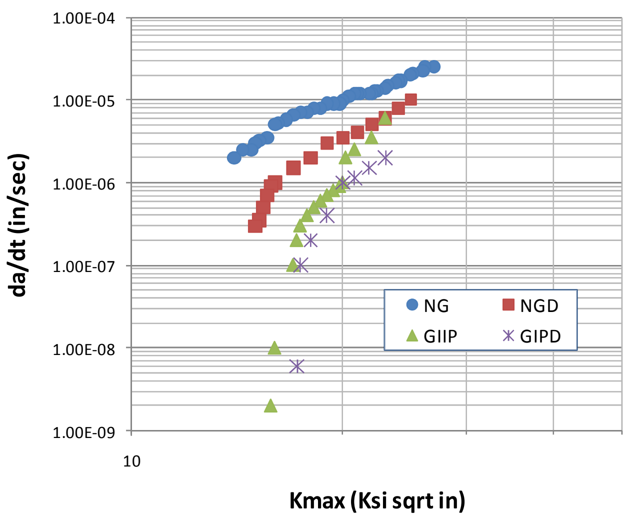


Figure 7: The effect of Grain Boundary Engineering on hold time crack growth; 649ºC (1200°F), 90 sec hold (preliminary test data provided by NASA).

Preliminary mechanical test data obtained at NASA indicate that the GBE processing has a substantial benefit on the hold-time crack growth behavior and the test data that illustrate this benefit is presented in Figure 7. The crack growth rate (da/dt) is shown as a function of applied stress intensity (kmax), which is one of the most important limiting properties for many high temperature disk applications. As shown in this figure, the GBE processing can achieve a significant reduction in the dwell fatigue crack growth rate (GIIP vs. NG and GIPD vs. NGD).

Summary

The optimization efforts carried out in this study clearly demonstrated that the GBE® processing methodologies can be successfully applied to the 718Plus® alloy for both bulk and surface processing. These treatments significantly increase the population of special grain boundaries content as well as the grain boundary connectivity in the alloy microstructure that are expected to have a beneficial effect on the alloy elevated temperature mechanical properties. The optimized GBE processing was shown to be effective in increasing the Fsp content from ~35% (in the non-GBE processed samples) in the range of 60% (i.e., for the GBE protocol) and 70% (i.e., for the GBEII protocol). The nearly 2-fold increase in the Fsp content and the improved grain boundary network connectivity are expected to have a positive impact on the creep and high temperature dwell fatigue performance of the alloy.

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