MICROSTRUCTURE EVALUATION OF SERVICE AGED AND REJUVENATED NICKEL SUPER ALLOYS USING HOT ISOSTATIC PRESSING TREATMENT

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Abstract. The design life of gas turbine blades is normally 48,000 equivalent operating hours (EOH) or 72,000 EOH based on the original equipment manufacturer (OEM) recommendation. Extending the service life of gas turbine blades is a huge advantage in the aspect of capital cost-saving in the power plant industries. The coarsening and shape transformation of γ′ precipitates are one of the main life-limiting factors for gas turbine blades. Conventional heat treatment could not restore or recover γ′-phase precipitates to their original morphology. In this study, hot isostatic pressing (HIP) treatment was used to improve the material properties of the blades to nearly zero-hour operation and prolong the life of gas turbine blades. HIP rejuvenation applies high temperature and pressure to close the micropores, retransform the microphases (i.e., γ′-phase precipitates), and heal the material properties. For this case study, three HIP parameters were applied on nickel-based superalloy (GTD-111). Microstructure characterization was carried out on new, aged, and HIP-treated GTD-111 alloys using field emission scanning electron microscopy (FESEM). The microstructure after HIP treatment exhibited significant improvement and recovery in the morphology of γ′ precipitates. The hardness of HIPed blade samples appeared consistent at 440 HV and did not deviate significantly from new materials with an average value of 439 HV. The average size of γ′ precipitates was successfully reduced from 1.3 µm (degraded state) to 0.3 µm (near-zero state). The experimental results suggested that HIP treatment at 1,220 °C produced the best performance in microstructure transformation to the near-original state for the exposed blade of GTD-111 alloys as cast EQX.

Keywords: hot isostatic pressing, nickel superalloy, turbine blade

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Introduction

Nickel-based superalloy is a precipitation-strengthened alloy with a large amount of γ′ precipitates, $Ni_3(AlTi)$ systematically placed in the austenitic matrix (γ), and the superalloy has the ability to withstand extreme temperatures that would normally destroy conventional metals [1-3]. The main characteristics of nickel-based superalloy are high-temperature strength, toughness, resistant to degradation, and good creep resistance [4, 5]. Thus, due to its excellent characteristics, nickel-based superalloy has been used in various applications, such as aircraft, power generation turbines, rocket engines, chemical processing industry, and other applications with high-temperature exposure (800-1,000 °C) [4]. However, the formation of defects (i.e., porosity, segregation, and coarsening in γ′ precipitates) is inevitable due to long service exposure in a high-temperature environment [5].

Nickel-based superalloy GTD-111 is normally used in gas turbine blades for thermal power plants [3]. This superalloy consists of multiphase microstructures, such as γ-matrix, γ′-phase, primary carbides (MC), and secondary carbides ($M_{23}C_6$) [6]. Long term exposure to extreme temperatures and stress conditions results in the merging of γ′ precipitates, forming $M_{23}C_6$ at the grain boundary, separation of MC, and formation of sigma phase [6]. These can lead to cracking and degradation of the mechanical properties of the material.

Until the mid-1970s, most turbines were replaced upon the lifespan expiry recommended by the original equipment manufacturer (OEM). Operation and maintenance costs generally play a significant part in the life-cycle cost of a turbine engine. The replacement cost is more than one-third of the repair, refurbishment, or maintenance cost due to expensive capital or asset cost. High replacement cost has created urgency for researchers to explore more for a new initiative. Therefore, as an initiative to save capital costs, studies on rejuvenating technology have been explored. Conventional and alternative heat treatment on degraded material has been done to recover its mechanical properties in order to extend its service life [1]. Epishin *et al.* reported that conventional heat treatment could only partially recover the degraded materials, while full recovery is possible by hot isostatic pressing (HIP) treatment [7].

HIP technology is a process to heat-treat the component at elevated temperature and isostatic gas pressure in a high-pressure containment vessel [5, 8]. The application of both heat and pressure simultaneously on all surfaces of a material helps to eliminate any small gaps (pores) in the material, which consequently increases density and promotes uniform composition [8]. Hence, this leads to the improvement of the internal microstructure, mechanical properties, and material reliability [5].

This method is usually used in two cases: to eliminate closed porosity in the workpiece and powder consolidation in a closed metal container [9]. The basic operation of HIP treatment involves the use of a HIP furnace. Figure 1 shows an example of a HIP furnace. HIP usually occurs at pressures of 100–200 MPa and temperatures of 900–2,250 °C [9]. HIP allows the sample to achieve the final shape in manufacturing and to repair complex parts from various metals and alloys. Equal pressure in all directions leads to isotropic properties. The high-pressure application provides the final density of the material and eliminates defects, such as porosity and shrinkage shells, thus may prolong the material life [10]. Nevertheless, the HIP treatment procedure is unique and proprietary to each manufacturer and utility.

Figure 1. HIP furnace (photo courtesy from Bodycote ltd.)

The purpose of this study is to restore the life of used or aging gas turbine blades to near zero-hour condition, which is basically the life of new material. The received samples were repaired and heat-treated through conventional heat treatment and HIP treatment. The characteristics of equiaxed (EQX) GTD-111 after HIP treatment were studied through microstructure analysis, microhardness testing, and elemental analysis.

Materials and Methods

Materials. The material used was nickel-based superalloy (EQX GTD-111). The used or aged sample was obtained from a gas turbine blade with approximately 72,000 equivalent operating hours (EOH). Figure 2 shows the used blade of EQX GTD-111. The new material of EQX GTD-111 in rod form was purchased from Korea Lost Wax Ltd., South Korea. Figure 3 shows the samples of the new material with a diameter of 3 cm and a length of 25 cm. Table 1 presents the material composition of standard nickel-based superalloy (GTD-111).

Figure 2. Used blade of GTD 111 EQX.

Figure 3. New materials of GTD 111 EQX (rod form).

Table 1. Material composition of standard Nickel-based superalloy (GTD 111).

Sample ID	Cr	Co	W	Mo	Ti	Al	C	B	Ta	Ni
GTD 111 (source: General Electric)	14	9.5	3.8	1.5	4.9	3	0.1	0.01	2.8	Bal.

Sample conditions. All samples were subjected to fluorescent penetrant inspection to check the presence of defects and blockages in cooling holes. Then, all the samples were heat-treated using conventional heat treatment at 1,200 °C ± 5 °C for 2 h ± 15 min in vacuum with argon atmosphere.

After conventional heat treatment, the used blade samples were rejuvenated via HIP treatment. The used blade of GTD-111 underwent HIP treatment with three parameters. Table 2 lists the parameters of rejuvenation conditions applied to each sample. Only temperature was varied to obtain optimum properties, whereas pressure and soaking time were fixed. Due to cost and time constraints, limited HIP-treated samples could be produced.

Table 2. Detailed of sample conditions.

Sample ID	Heat treatment (ºC/hours)	HIP (ºC/hours/MPa)	Post weld heat treatment (ºC/hours)	Aging (ºC/hours)
Used Blade As-R	-	-	-	-
NM	-	-	-	-
Used Blade	1200/2	-	-	-
NM HT	1200/2	-	-	-
Used Blade Par.1	1200/2	1191/4/102	1200/2	845/24
Used Blade Par.2	1200/2	1200/4/102	1200/2	845/24
Used Blade Par.3	1200/2	1220/4/102	1200/2	845/24

<u>Note</u>: R-Received, NM- New Material, Par- Parameter, HIP-Hot Isostatic Pressing

Analysis. All samples underwent microstructure analysis, microhardness testing, and material composition analysis. Microstructure analysis was carried out using field emission scanning electron microscopy (FESEM, Hitachi SU8020) to analyze phase transformation and degradation signs, such as precipitate coarsening and grain growth [11]. The blade samples and rods were cut across the transverse plane. The samples were then mounted in hardened resin using the cold mounting method. The embedded metal surfaces were ground, polished, and etched with a chemical solution of 61% lactic acid, 36% HNO_3, and 3% HF to reveal the microstructure. The microstructure samples were taken at a location about 10% from the tip and in the transverse section. The microstructure was examined using a scanning electron microscope up to 5,000× magnification to examine the morphology changes of γ' precipitates. Microhardness properties were measured using the Vickers hardness test instrument (Future-Tech, FM1) in accordance to ASTM E384-2017. The applied load was set as 500 gf, and the average readings were reported from a total of five indentations per sample.

A glow discharge spectrometer (Spectruma-GDA 750) was used to analyze the material composition of the blade and new material. Meanwhile, energy-dispersive X-ray (EDX) spectroscopy was conducted to analyze carbide composition using a Bruker XFlash 6/60 detector fitted in a FESEM machine (Hitachi SU8020). An accelerating voltage of 15 kV was used at all locations. EDX analysis was performed on mounted samples.

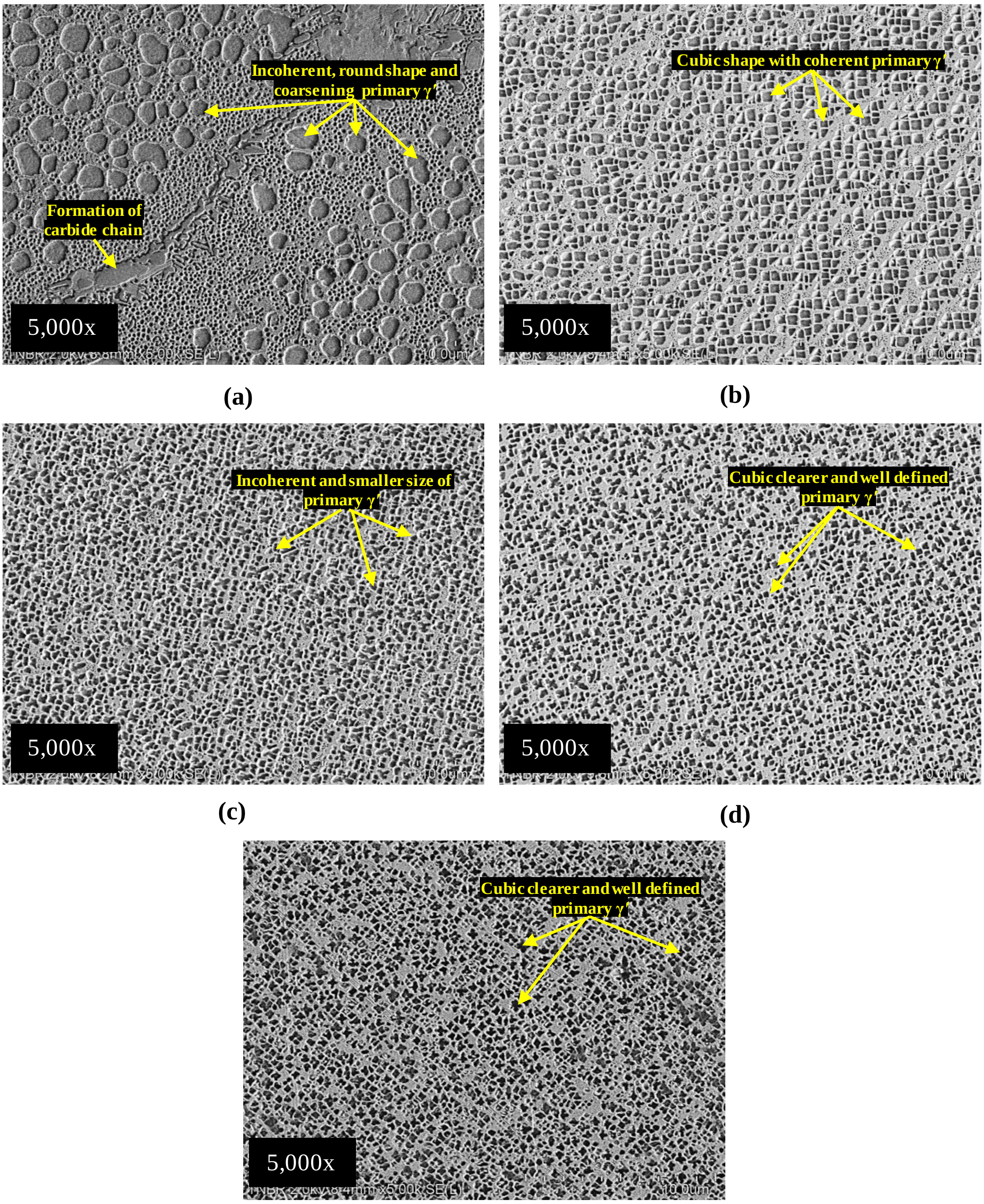
Results and Discussion

Microstructure analysis. Figure 4(a) presents the image of the sample taken from the as-received used blade (service-exposed GTD-111 turbine blade of EQX type with 72,000 EOH). The microstructure consists of incoherent, round shape, and coarsening primary γ', which are commonly found in materials with prolonged thermal exposure.

Microstructural changes in a material due to thermal exposure can also cause the decomposition of MC and the formation of $M_{23}C_6$ along the boundary, as depicted in Figure 5 [12, 13]. Figure 5 shows the EDX results for the second-phase precipitates of the as-received used blade. Primary carbide and continuous carbide chains along the grain boundaries were detected using EDX. The microstructural changes may happen due to carbide reaction, where carbon is released during service exposure. The principal carbide reaction leads to the formation of $M_{23}C_6$, as presented in Equation 1 [4]:

$$MC + \gamma \rightarrow M_{23}C_6 + \gamma' \qquad (1)$$

The microstructure of the new material consists of cubic shape with coherent γ' precipitates, as shown in Figure 4(b). Figure 4(c) shows the microstructure image of the HIPed used blade using parameter 1, which exhibited incoherent and smaller size of γ' as compared to the as-received used blade. As the HIP temperature increased, it was observed that the shape of primary γ' became clearer and well defined, as presented in Figure 4(d) and (e). The microstructure image of the HIPed used blade using parameter 2 indicated a more defined irregular γ' shape as compared to that of parameter 1, as well as a smaller size of γ' as compared to the as-received used blade. However, it could be observed that used blades treated at 1,220 °C (parameter 3) showed the most optimum conditions in terms of size and shape of microphases, with uniform and aligned γ' in nearly cubical shape as a new material and a smaller size compared to the as-received used blade. Nevertheless, all rejuvenated blades at 1,191 °C, 1,200 °C, and 1,220 °C were considered successful in the recovery of γ′ precipitates to the near-original state, based on the visual appearance under the microscope.



(a) (b) (c) (d) (e)

Figure 4. Microstructure images of (a) used blades before HIP treatment, (b) new materials, (c) Microstructure from used blades by applying HIP treatment using parameter 1, (d) parameter 2 and (e) parameter 3.

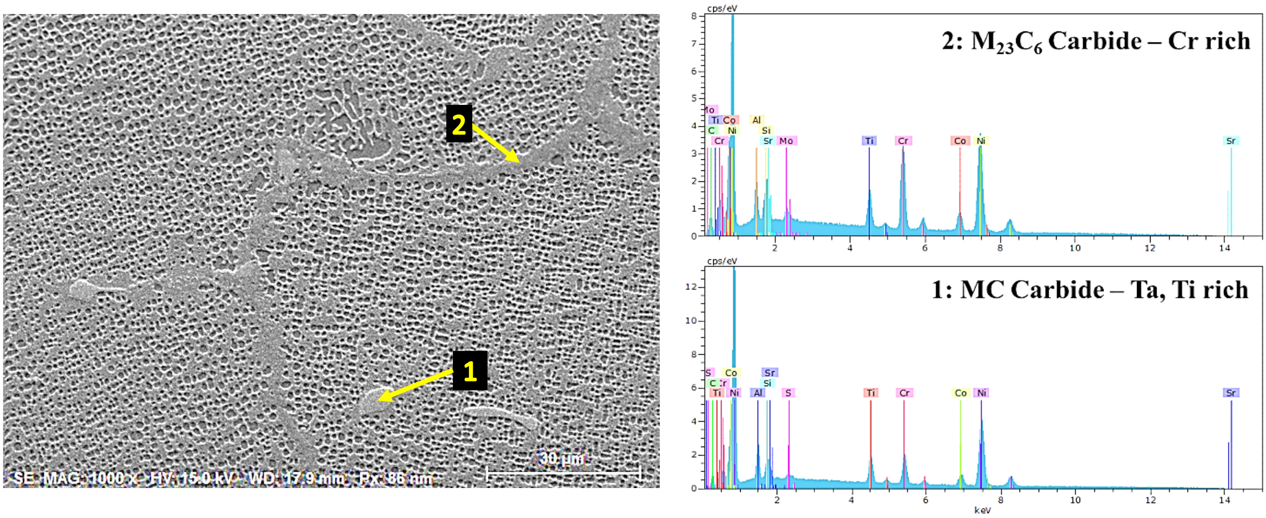


Figure 5. EDX result for second phase precipitates of used blade as received.

The function of γ′ precipitates is to strengthen and enhance the creep property of nickel super alloys [2]. The optimum creep property is obtained from fine and uniform γ′ precipitates in the γ matrix. Over the exposure time, γ′ precipitates tend to grow and coarsen, consequently losing the ability in the strengthening mechanism [12]. γ′ precipitates are one of the essential key measurement parameters of material degradation [12].

Figure 6 shows the results of quantitative measurement of the γ′ phase size for the as-received blade (without any heat treatment), the new material after conventional heat treatment, the used blade after heat treatment, and the used blade after HIP (refer to Table 2). The γ′ min and γ′ max represent the smallest and biggest sizes of γ′ phase, respectively. The γ′ phase size of the used blade grew and coarsened due to service exposure. It was observed that the heat treatment and HIP treatment affected γ′ phase size significantly, as the average γ′ phase size of the used blade reduced significantly from 1.297 µm to 0.603 µm after heat treatment. The average size of γ′ precipitates in the new sample (the rod after heat treatment) is approximately 0.4 µm. Subsequent to conventional treatment, the HIP treatment reduced the size of γ′ precipitates from 0.6 µm to 0.3 µm. It was found that the HIP-treated blades using three temperatures at 1,191 °C, 1,200 °C, and 1,220 °C exhibited an average γ′ size of 0.340 µm, 0.327 µm, and 0.324 µm, respectively. Previous studies suggested that the optimal primary γ′ phase size is between 0.2 and 0.5 µm [12]. Thus, the experiment data of this study indicate a successful recovery of γ’ precipitates to near the original state as all HIPed blades exhibited an average γ′ size between 0.2 and 0.5 µm. It was observed that parameter 3 with the temperature of 1,220 °C produced the finest γ′ precipitates of 0.32 µm, suggesting 1,220 °C as an optimum temperature to obtain the optimal primary γ′ phase size. It is important to obtain the optimal primary γ′ phase size in order to strengthen and enhance the creep property of the materials.

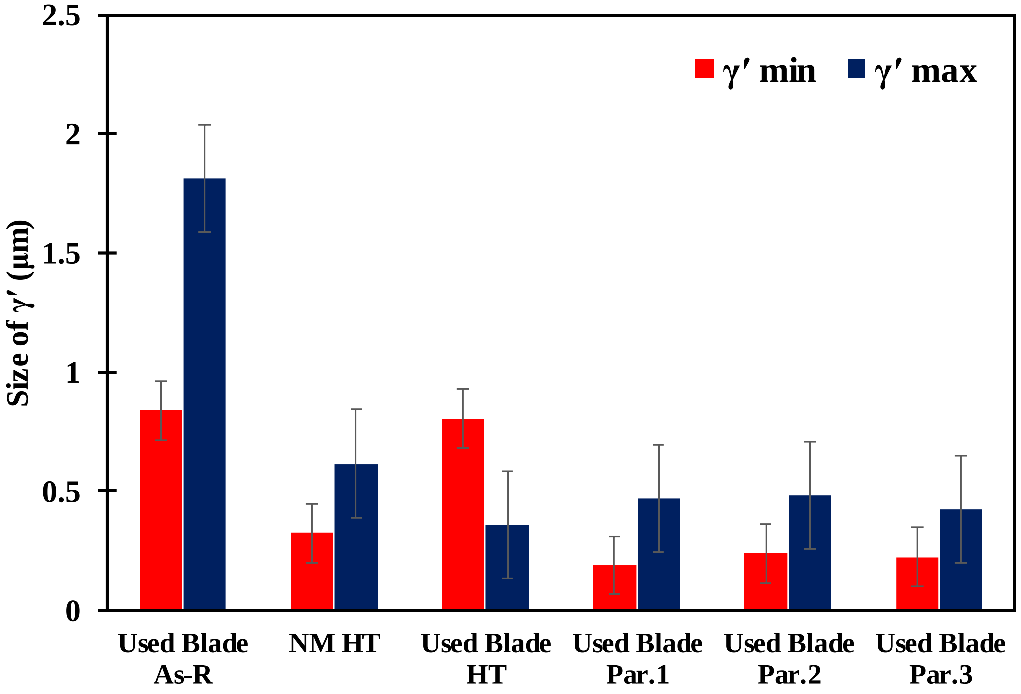


Figure 6. Quantitative measurements of γ′ phase size.

Microhardness testing. Figure 7 presents the effect of rejuvenation conditions on microhardness changes in the samples. It was observed that HIP rejuvenation increased the hardness property of the used gas turbine blade for all parameters. The hardness property of all HIPed blade samples appeared to have a consistent value at ca. 440 HV and did not deviate significantly from new materials, which has a value of 439 HV. This may occur due to its fine and uniform distribution of γ′ precipitates in the γ matrix, as shown in Figure 4. On the other hand, the microstructure of the as-received used blade exhibited a coarse and degraded γ′ phase, thus lowering its hardness value. The microhardness results suggested that the variation in hardness value is likely related to the type, shape, size, and morphology of microphases, as all samples besides the as-received used blade exhibited similar uniform and defined shape of γ′ phase (Figure 4) [13]. Thus, it is suggested that HIP treatment has successfully increased the hardness property of the used gas turbine blade near its zero-hour condition.

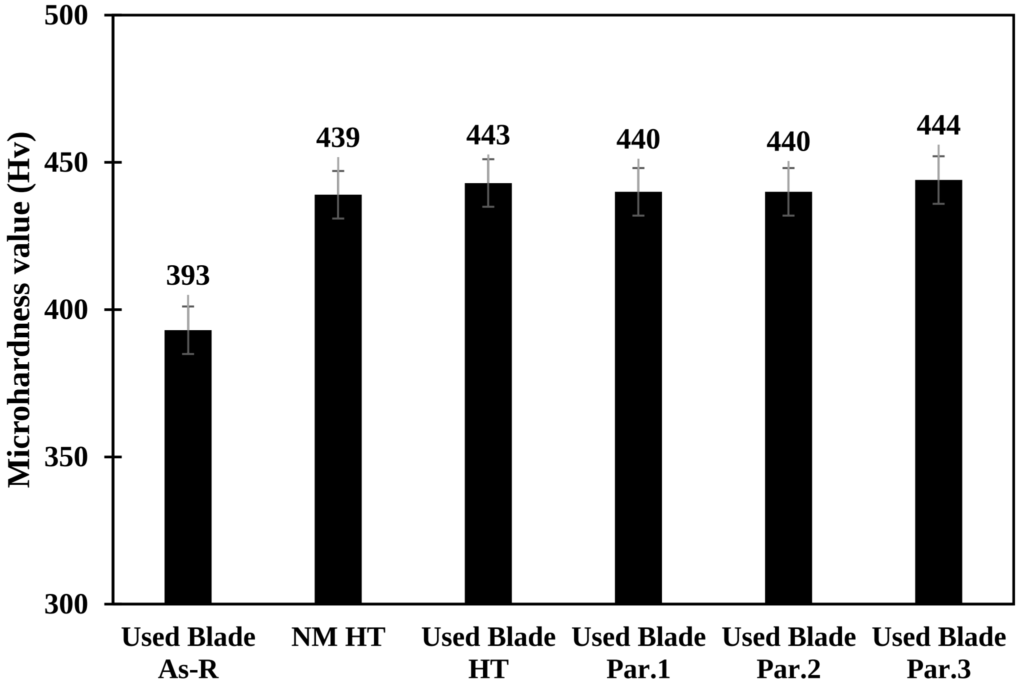


Figure 7. Hardness Value

Elemental analysis. Table 3 shows the material composition analysis of each sample using glow discharge spectrometry. Chromium (Cr), cobalt (Co), tungsten (W), molybdenum (Mo), titanium (Ti), aluminum (Al), carbon (C), boron (B), tantalum (Ta), and nickel (Ni) were detected with concentrations of approximately 13.1%, 10.6%, 2.9%, 1.6%, 5.3%, 3.0%, 0.1%, 0.01%, 2.8%, and 60.1%, respectively. The elemental composition for all samples appeared similar and did not change after HIP heat treatment. Thus, it can be suggested that HIP treatment does not affect the elemental composition of the materials and is suitable as a rejuvenating technology for nickel-based superalloy (GTD-111).

Table 3. Material composition analysis.

Sample ID	Cr	Co	W	Mo	Ti	Al	C	B	Ta	Ni
Used Blade As-R	13.1	10.6	2.9	1.6	5.26	3.09	0.1	0.01	2.84	60.1
NM HT	12.9	10.7	2.9	1.57	5.24	3.18	0.15	0.01	2.78	60.3
Used Blade HT	13.3	10.5	2.9	1.6	5.36	3.05	0.1	0.01	2.85	60
Used Blade Par.1	13.3	10.6	2.8	1.6	5.34	3.06	0.1	0.01	2.84	60.1
Used Blade Par.2	13.1	10.6	2.9	1.59	5.28	3.04	0.1	0.01	2.81	60.1
Used Blade Par.3	13.3	10.6	2.9	1.59	5.29	3.03	0.1	0.01	2.81	60.1

Note: R-Received, NM- new material, Par-Parameter

Conclusion

The used gas turbine blades of EQX GTD-111 with 72,000 EOH were successfully rejuvenated using HIP treatment to the near-original state. Microstructural characterization revealed that the used blade consists of coarse γ′, MC, and continuous carbide chain ($M_{23}C_6$) along the grain boundaries. After HIP treatment, these microstructures were transformed to finely dispersed γ′, forming a more diminutive and more defined γ′ shape. The HIPed used blade with a temperature of 1,220 °C (parameter 3) shows the finest and aligned γ′ with a nearly cubical shape closest to a new material. HIP treatment has successfully reduced the size of γ′ precipitates from 0.6 µm to 0.3 µm. The hardness property of all HIPed blade samples appeared consistent at ca. 440 HV and did not deviate significantly from new materials, which have a value of ca. 439 HV. It is suggested that HIP treatment can recover the microstructure and improve the hardness property. HIP rejuvenation does not affect the chemical compositions of the alloy. Based on the microstructure, microhardness, and elemental analyses, it was found that HIP treatment at 1,220 °C produced the best performance in microstructure transformation to the near-original state for the exposed blade of GTD-111 alloys as cast EQX.

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Author Contributions

All authors contributed toward data analysis, drafting and critically revising the paper and agree to be accountable for all aspects of the work.

Disclosure of Conflict of Interest

The authors have no disclosures to declare

Compliance with Ethical Standards

The work is compliant with ethical standards

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