EFFECT OF HEAT TREATMENT CYCLE TO THE MICROSTRUCTURAL ANALYSIS OF SERVICE-EXPOSED NI-CR ALLOY TURBINE BLADE

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ABSTRACT Ni-Cr superalloys has been widely used in various applications particularly at high temperature and aggressive atmosphere especially in turbomachinery equipments. The turbine blade was made from Ni-Cr based superalloys experience the effect of high temperature and stress during service which certainly cause various microstructural changes. The turbine blade was operated by a PETRONAS Gas Berhad, where it has been exposed for 100,000 running hours at 830 ℃ operating temperature. The study has attempt to obtain the most suitable and practicable repair condition, which could provide the desired microstructural characteristics by rejuvenation method of various heat treatment cycles. In this study, changes in microstructural in the Ni-Cr superalloys after exposed to different heat treatment cycles are reported. During solution treatment, the coarse carbides and gamma prime precipitates formed at the grain boundaries during service by creep mechanism, would dissolve into the matrix. However, the MC carbides will remain and transformed into $M_{23}C_6$ carbides. The specimens then processed through a series of precipitation aging, which re-precipitates the strengthening phase to form a proper morphology in distribution of shape and size that is almost similar as the new alloy. Metallurgical examination of the microstructure had been performed by using electron microscopy after the heat treatment cycles to evaluate the specimens' microstructural changes.

1. INTRODUCTION

Ni-based superalloys are a special class of metallic materials with an exceptional combination of high-temperature strength, toughness and resistance to degradation in corrosive and oxidizing environments. Ni-based superalloys exhibits microstructural stability, excellent strength and high corrosion resistance at high temperature simultaneously. It is well known that these alloys possess high temperature strength from the fine particles of γ' phase precipitated coherently with the matrix (Moshatghin and Asgari, 2003).

The microstructure of Ni-Cr based superalloy under standard heat treatment (SHT) condition mainly consists of two forms of cuboids and spheres precipitates. In Ni-Cr microstructure system, Cr is a key alloying element in Ni-based alloys for providing corrosion protection through the formation of a passive Cr_2O_3 surface oxide. The difference in crystal structure between Cr (BCC) and Ni (FCC) result in binary phase diagram with terminal solid-solution phase fields (Lippold *et al.*, 2009). The exposure of high temperature on the alloys show a high tendency of coarsening the gamma prime (γ') phase. This phenomenon impacted the grain boundaries and carbide precipitate which can leads to decrease the strength of the material.

One way to extend the service lifespan of the service-exposed turbine blade is through heat treatment process which is known as rejuvenating process. The practice of rejuvenating reheat treatments, with and without hot isostatic pressing (HIP), has been successful in restoring severely overages gas turbine blade microstructure and allot properties to a nearly as-new condition (Lvov and Norsworthy, 2000). Extensive research has been carried out related issues on the rejuvenation of the Ni based superalloys (Koul et al., 1988; Lvov and Norsworthy, 2000; Kim et al., 2007; Kim and Oh, 2008; Zhou et al., 2013; and Zhou et al., 2012). From the studies, the rejuvenation process recover the microstructural restoration (through re-solution and re-precipitation aging), sintering of cavities (by vacancy diffusion under the action of surface tension and/or supplied pressure), inhibition of growth in grain boundary cavities (via isolating them within the gain interiors through recrystallization or grain growth) and changes in dislocations structure. However, the improper heat treatment may cause damaging effects, such as formation of undesirable phases, incipient melting, crack initiation and oxidation during service (S.S. Hosseini *et al*., 2012).

The rejuvenation technique requires a proper control of the temperatures and pressures. By developing an optimum heat treatment parameters, the microstructure of the alloys can be restored to as-new conditions and thus the alloys are viable to be back in service. This investigation concerns on the changes of the microstructural aspects of the service-exposed turbine blade during different heat treatment cycles.

2. EXPERIMENT

The experimental work in this study has mainly based on the exposure of exposed part (leading edge area) gas turbine blade specimens to the heat treatment process. The research material, a service-exposed gas turbine blade (type DR61) was provided by Petronas Gas Berhad. The blade is an uncoated blade and exposed to a temperature of 830℃ during operation. The rotor have been in service for 100,000 running hours. The blade is a first stage blade of In-738LC, where the blade consist the major elements of Ni-Cr superalloys. The composition of this material measured using optical emission spectrometer (OES) where (wt%) was: 15.86Cr, 2.17Mo, 2.81Al, 6.68Ti, 0.09Si, 1..39W, 0.03Cu, 8.35Co, 2.81Nb, 0.05Mn, 0.24C and balance Ni. The blade is vacuum casting blade with dimension of 230mm x 10mm and 1,123.5g weight. Fig. 1(a) shows the first stage service-exposed turbine blade.

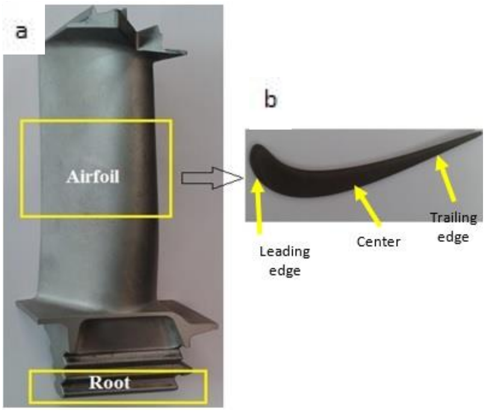


Fig. 1 (a) Turbine blade and, (b) blade sectioning area.

The specimens were sectioned by the wire electrical discharge machining (EDM) process at two locations: the root (shank) and the airfoil. Fig. 1 (b) shows the blade sectioning area. The root (shank) metal served as a reference of the initial metal condition, since the service temperature at the root area is relatively low and does not cause degradation of the microstructure during service. The specimens were section approximately to a dimension of 10mm x 10mm x 1.4mm using electric wire-cut machine and abrasive cutter machine.

The heat treatment was applied to the alloys to alter the microstructure of the specimens. The heat treatment schedule and designation of the specimens for this study is shown in Table 1. Rejuvenation heat treatments consisting of two main stages, solution and precipitate aging treatments, where the target is to achieve maximum re-establishment of the original microstructure and initial properties (S.S. Hosseini *et al*., 2012). Specimens were undergone solution treatment at 1 hour soaking at three (3) different temperatures, namely, 1145 ºC, 1195 ºC and 1245 ºC and subsequent air cooling to room temperature. Standard heat treatment for Ni-Cr based superalloys consist of primary and secondary aging at 1120ºC/2hrs and 845ºC/24hrs followed by air cooling to the room temperature.

For microstructural examination, the specimens were mount and polished to a mirror-like surface by using metallographic grinding and polishing respectively. The polished samples then were etched using 10ml of HNO_3 and 30ml of HCl for about 5-10s. Microstructural examination and characterization involved using field emission scanning electron microscope (FESEM) at low and high magnification.

Table 1: Designation of Specimens from Leading Edge Area

Specimen	Solution Treatment Temperature (℃)			Aging temperature (℃)	
	1145 (1)	1195 (2)	1245 (3)	1120 (1)	845 (2)
A010	-	-	-	X	
A012	-	-	-	X	x
A112	x	-	-	X	x
A212	-	x	-	X	x
A312	-	-	x	X	x

3. ANALYSIS

The backscattered electron (BSE) imaging mode was used to analyze the microstructure of the specimens since this method is capable of differentiating microstructural constituents based on the average atomic weight, without interference of the etching. The as-received service-exposed turbine blade alloys are shown in Fig. 2. FESEM micrographs revealed that the cuboidal primary γ' precipitates became rounded and coarsened considerably at the expand of secondary γ'. The $M_{23}C_6$ grain boundary carbides coarsened and coalesced, overloading the grain boundaries and forming continuous films at some areas while the primary MC carbides degenerated losing their sharp edges.

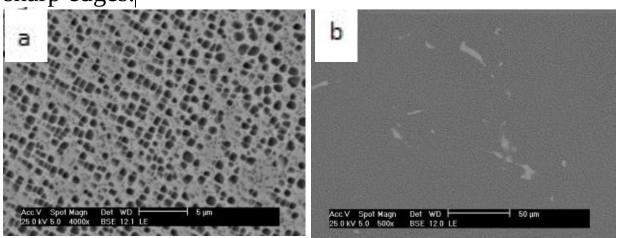


Fig. 2. FESEM micrographs of service exposed turbine blade: (a) primary and secondary γ' precipitates; (b) primary MC carbides

Solution treatment temperatures were chosen at three different temperatures to examine the influence of temperature on the microstructure of the service-exposed turbine blade. Fig. 3(a) exhibits the microstructure after

1120℃/2 h/AC primary aging (specimen A010). Fig. 3(a) shows that the primary aging produced a spheroidal γ' microstructure. This indicates that the primary aging temperature at 1120℃ is a sub-solvus temperature for γ' to dissolved, partial dissolved for the coarsened γ' precipitates and a large number of residue precipitates were remained within the matrix. The microstructural examination revealed that, apart from γ' precipitates, the grain boundary films were also affected by the primary aging temperature. Fig. 3(b) shows the continuous films observed along the grain boundaries after aging at 1120℃.

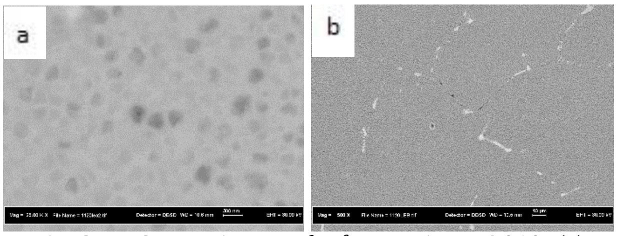


Fig. 3. FESEM micrographs for specimen A010: (a) spheroidal γ' microstructure; (b) grain boundary region.

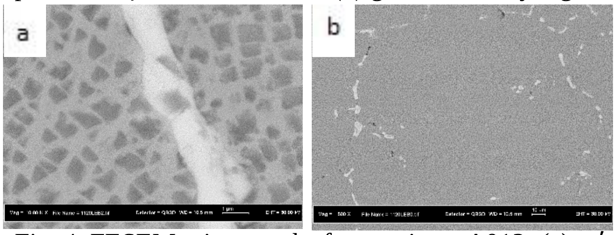


Fig. 4. FESEM micrographs for specimen A012: (a) γ' precipitate and MC carbides; (b) grain boundary region.

Fig. 4 reveals the microstructure obtained for specimen A012. Specimen A012 is indeed undergone the standard heat treatment cycle for IN738LC alloy where it is consist of 1120℃/2 h/AC primary aging and subsequent secondary aging at 845℃/24 h/AC. Specimen A012 produced a large number of remnant fine secondary γ' precipitates formed in the space during the aging treatment. The influence of primary aging temperature on MC carbides also noticeable in Fig. 4(a) where a blocky MC carbide shows in the microstructure after the primary aging and the MC carbides degenerated losing its sharp edges.

Fig. 5 shows the microstructure obtained for specimen A112. After the solution treatment at 1145℃/1h/AC, followed by primary and secondary aging treatment, it is shown that the microstructure contains coarsened γ' precipitates and merged with neighboring precipitates. It is presume that the unique microstructure was due to the diffusion of solute atoms and addition to the growing precipitates during the cooling period. Fig. 5 (b) shows the continuous grain boundary area even though after exposed to the full heat treatment cycle.

Fig. 6 shows the microstructure for specimen A212. Specimen A212 been exposed to a complete solution treatment at 1145℃/1h/AC, followed by primary and secondary aging treatment. Fig. 6(a) reveals the very fine cuboidal primary γ' precipitates. However, the primary MC carbides transformed to $M_{23}C_6$ and overloading at the grain boundaries and form continuous films at some areas (Fig. 6(b)). The $M_{23}C_6$ carbides formed at the MC periphery which was confirmed by EDX analysis (Fig. 8). The aging treatment resulted in the formation of fine cuboidal secondary γ' precipitates which performed as per specimen A212. Formation of fine secondary γ' precipitates appear to be related to the solute absorption from the supersaturated matrix during the aging. With respect to the MC carbides and grain boundary regions, the microstructures gained after the heat treatment cycles are comparable. Primary solution treatment caused the continuous films partially dissolved and no transition zone observed around MC carbides. However, after secondary aging, it can be seen that there is different at the grain boundary regions. The grain boundary were covered with irregular shaped precipitates after the aging treatment.

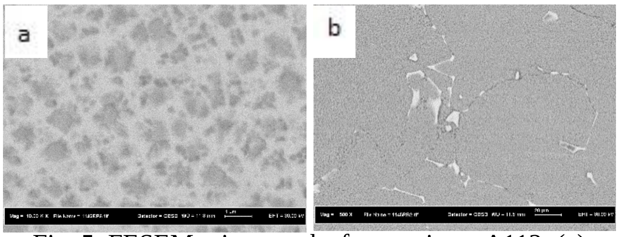


Fig. 5. FESEM micrographs for specimen A112: (a) intragranular γ' precipitate; (b) grain boundary region.

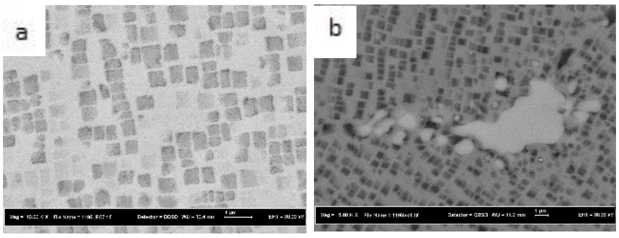


Fig. 6. FESEM micrographs for specimen A212: (a) fine cuboidal γ' precipitate; (b) $M_{23}C_6$ carbides.

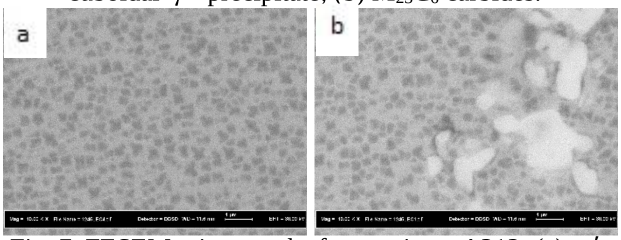
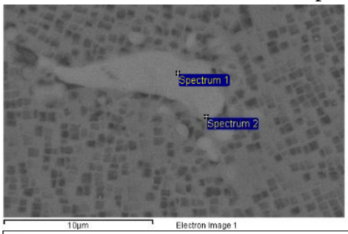


Fig. 7. FESEM micrographs for specimen A312: (a) γ' precipitate; (b) $M_{23}C_6$ carbides.

The microstructure of specimen A312 after solution treatment at 1245℃/1 h/AC is shown in Fig. 7. It has been reported that the melting temperature for In-738 range at 1230℃ - 1300℃, thus at this temperature it can cause complete dissolution for the γ' phase. The microstructure of specimen A312 shows large and small cubes after the complete heat treatment cycle. The effect of high temperature solution treatment on the grain boundary films and MC carbides is another aspect which should be taken into account. Fig. 7(b) shows the grain boundary areas and MC carbides for specimen A312. As it is evident, no grain boundary film is observed along the grain boundaries. It seems that the grain boundaries were depleted from continuous films. Moreover, the primary MC carbides have been reported to transform to $M_{23}C_6$ carbides precipitate after the heat treatment cycle. It is known that even high temperature solution treatments were not able to dissolve the primary MC carbides.



Element	Weight% $M_{23}C_6$	Weight% MC Carbide
C K	35.32	30.91
Ti K	12.48	17.18
Cr K	4.46	1.48
Ni K	11.96	4.79
Ta L	18.93	26.06

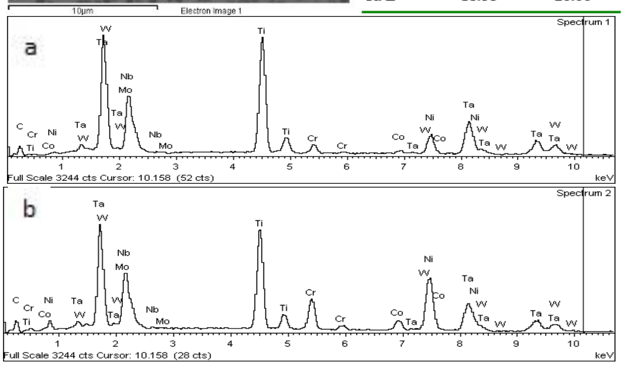


Fig. 8: EDX analysis: (a) Spectrum 1 is MC carbides; (b) Spectrum 2 is $M_{23}C_6$ carbides.

Comparing the microstructure for specimen A112, A212 and A312 in Fig. 5, 6 and 7. Specimen A212 shows the microstructure has successfully been restored back to the as-new condition of the alloys eventhough the MC carbides did not dissolved during the heat treatment cycle.

CONCLUSION

The service-exposed Ni-Cr based superalloys turbine blade was subjected to heat treatment cycles at different temperature to restore the degraded microstructure from the service-exposed turbine blade. The changes of the microstructural features during the different stages of heat treatment cycles was studied. Based on the findings of this study, the conclusion could be summarized as the followings:

(1) Among all heat treatment cycles investigated, specimen A212 led to dissolution of nearly all coarsened γ' precipitates, continuous grain boundary films and transition zone around MC carbides.

(2) Solution treatment at higher temperature resulted in smaller size and fine γ 'precipitates, no grain boundary films and yielded MC carbides free from surrounding transition zone but unable to dissolve the MC carbides.

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