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Comparison of Different Sample Preparation Techniques in TEM Observation of Microstructure of INCONEL alloy 783 Subjected to Prolonged Isothermal Exposure

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Recently developed INCONEL alloy 783 (nominal composition of Ni-34Co-25Fe-5.4Al-3Nb-3Cr) is a low coefficient of thermal expansion (CTE) superalloy [1]. Due to high aluminum content (5.4wt%), in addition to formation of coherent γ' (Ni_3Al-type) phase to provide alloy strength, alloy 783 precipitates incoherent β (NiAl-type) phase in an austenite matrix [2]. Due to its low CTE, high strength, and good SAGBO (Stress Accelerated Grain Boundary Oxidation) resistance, alloy 783 has been specified for use in aircraft gas turbine components such as rings, casings, shrouds and seals. Gas turbine components usually experience thermal exposure of up to 600°C up to 30,000 hours or more at condition of service. Therefore, the engine designers have concerned the influence of prolonged thermal exposure on the properties of superalloys for the over past decade. In this study, commercially produced alloy 783 was annealed and aged following the standard heat treatment procedure. One set of specimens was then isothermally exposed at 500°C for 3,000 hours. Mechanical properties were measured at room temperature and 650°C, and the results showed the prolonged exposure increased the strength and decreased elongation of alloy 783. The microstructures of as-produced and exposed materials were examined with optical microscope, scanning electron microscope (SEM) and transmission electron microscope (TEM) respectively. Isothermal exposure could not modify the morphology of microstructure of alloy 783. The optical microscope observation indicated that isothermally exposed and as-produced materials exhibited an isotropic microstructure with a similar grain size to that specified by ASTM 5-7. The SEM photographs showed an identical morphology of β phase for as-produced and exposed samples.

In order to understand the variation of mechanical properties of alloy 783 due to isothermal exposure, TEM was employed to investigate the internal microstructure of as-produced and isothermally exposed alloy 783. To gather true information about material structure and exclude artifact effect, the selection of sample preparation technique is very important for the TEM analysis. The modern material science requires the TEM sample preparation technique with sub-micron-level precision, high sample yield, and minimum of artifact. Since sample preparation techniques are very material dependent, there are currently three major methods, jet electro-polishing, ion milling technique, and FIB (Focus Ion Beam) technique, to be applied for the different materials such as metallic material, composite, and semiconductor. In this study, three techniques, jet electro-polishing, ion milling, and FIB, were utilized to prepare the TEM samples to observe the internal microstructure of alloy 783 subjected to different heat treatments. TEM observation of samples prepared by different methods was conducted. The results showed that the jet electro-polishing technique allowed the detail microstructure of alloy 783 subjected to standard heat treatment and isothermal exposure to be well revealed. Based on TEM imaging of samples prepared by jet electro-polishing technique as shown in FIG. 1, it was found that the internal precipitation of ordered γ' phase within β phase occurred during prolonged term isothermal exposure, FIG. 1 (a). In contrast, the internal precipitation within β phase did not take place in as-produced material, and β phase was clean as seen in FIG. 1 (b). The internal alteration of β phase due to precipitation of γ ′ phase was thought to be responsible for the enhancement of strength and reduction of elongation of isothermally exposed alloy 783. Ion milling technique induced artifacts on the austenite matrix of both as-produced and exposed alloy 783 so that the morphology of γ′ phase could not be well revealed, but the structure of large plate-like β phase was observable still. Also, this study showed FIB caused the extensive surface damage and amorphous structure for the as-produced and exposed samples. Therefore, the TEM images of samples prepared by FIB cannot reflect the microstructure of as-produced and exposed alloy 783. In summary, all results of TEM imaging for samples prepared by jet electro-polishing, ion milling, and FIB suggest that the jet electro-polishing technique is the best method to prepare the TEM sample of superalloy 783.

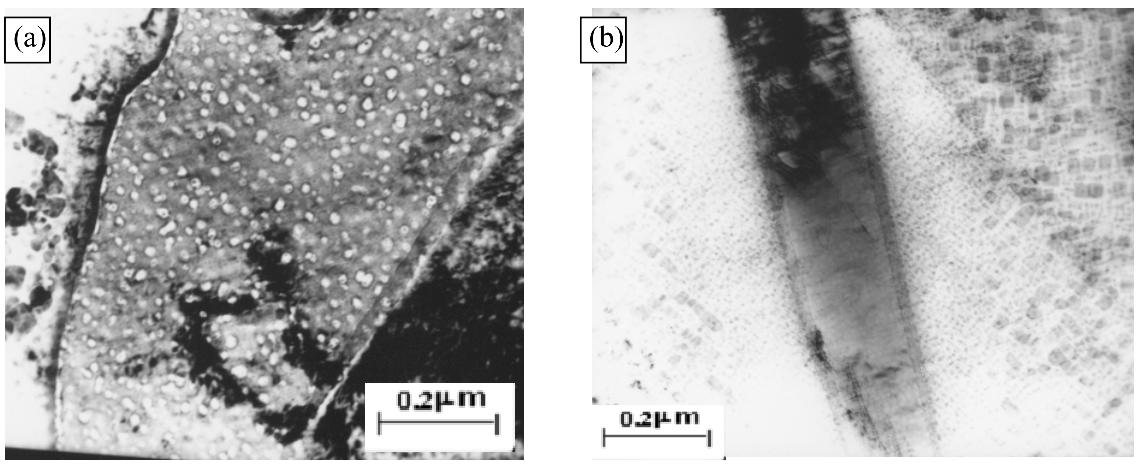

FIG. 1. TEM photograph of β phase in as-produced and isothermally exposed sample prepared by jet electro-polishing

(a). Internal structure of β phase in isothermally exposed sample showing the internal precipitation of γ′ within β phase.
(b) Internal structure β phase in as-produced sample showing the lack of internal precipitation

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