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Intermediate temperature brittleness of Ni based superalloy Nimonic263

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ABSTRACT

Ni based superalloy Nimonic263 offers excellent resistance to corrosion and high temperatures, and as such can be used under an A-USC environment, which has extremely high temperatures and high pressure. To investigate the intermediate temperature brittleness that is the temperature dependence of this alloy, the tensile test was carried out at a constant cross head speed of 1 mm/min in the temperature range of room temperature to 900 °C. As the temperature increased during the test, the constant yield strength decreased sharply at 800 °C while the ductility began to decrease from 500 °C and then recovered after showing a minimum value at 800 °C. The results of the OM, SEM and TEM observations indicate that the deformation mechanism changed from γ′ shearing to by-pass as the temperature increased, and the minimum ductility resulted from decohesion of the glide plane.

1. Introduction

To adhere to CO_2 emissions regulations, the power generation efficiency of coal fired power plants, which is affected by the depletion and degradation of coal fuel, needs to improve. Thus, Ni-based superalloys for power plants have drawn attention for use in extreme environments with high temperatures and high pressures [1–4] and contain a higher content of Cr and Co than conventional superalloys. The superalloys have outstanding resistance to corrosion and high temperatures [5]. Therefore, these are core materials for boilers and turbines with A-USC (advanced-ultra supercritical) conditions with steam temperatures ranging from 700 to 760 °C. The tensile test is a commonly used method to determine the mechanical properties of materials that appear at temperatures from room temperature to high temperatures. It is very common for the strength and ductility to have opposite tendencies. However, regardless of the strength change as the tensile test temperature, the temperature dependence of the ductility at an intermediate temperature between 500 and 800 °C is a feature of Ni-based superalloys [6–19]. This phenomenon is referred to as intermediate temperature brittleness (ITB), and the point at which the elongation minimum appears is referred to as the ductility minimum. This unexpected phenomenon is uncommon and can be a risk factor for materials that expose various temperatures. Therefore, the ITB temperature and its cause have to be investigated.

Many researchers studied the ITB of Ni-based superalloys according to variations in the alloy compositions and heat treatment conditions. L. Z. He et al. [6] studied the ITB phenomena of an M963 alloy that was precipitation strengthened at various temperature ranges from 20 °C to 1100 °C, with an ITB at 800 °C caused by decohesion of the glide plane (DGP) from a pile up of dislocations at the γ/γ′ interface. Also, the major cause of ITB at 750 °C of GTD-111 alloy, which is a precipitation strengthened directionally solidified material was DGP [7–10]. I. S. Kim et al. investigated the mechanical behavior on various temperatures of B1900 [11] and CM247LC [12,13] that are a precipitation strengthened casting material, the ITB temperatures are 871 °C and 650 °C, respectively. The results indicate that the DGP is the main cause of ITB regardless of casting method, whether conventionally cast or through directional solidification. Cast materials and forged materials were also investigated [14–19]. According to A. K. Roy et al. [14], the ITB of Waspaloy results from dynamic strain aging (DSA) that emerges as serrated flow from the strain-stress curve. However, the results of experiments in nitrogen to replicate the environment of a nuclear reactor environment are hardly the same as in an air condition. Another cause of ITB of forged materials reported on DGP [15–17] and intergranular precipitates (IP) [19]. The ITB of heavy-duty forged materials for power plants was also reported [18], but the cause has not been clarified. In particular, Nimonic263, which is an improvement over Nimonic80A with a high ductility, showed that the volume fraction of γ′ contributes to an increase in the elongation of the alternative heat treated sample [20]. However, the high temperature tensile properties should be carefully studied because these have only been tested at room temperature. In the case of forged materials, post heat treatment processing is required for the formation, and the brittleness at a specific temperature is extremely lethal. Therefore, it is necessary to study the deformation behavior at various temperatures.

N. F. Fiore [21] indicated that the causes of the ductility minimum

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in the case of Ni-based superalloys were (1) grain boundary embrittlement through the formation of brittle phases such as carbides or oxides, (2) inhomogeneous deformation mechanisms caused by localized strain, (3) γ' coarsening. However, grain boundary embrittlement is observed for the intergranular fracture mode in the tensile test, while deformation mechanisms appeared for the transgranular fracture mode due to a localized strain from the interaction of the matrix and the γ/γ' interface. Therefore, these conflict. Since the time that the specimen is exposed to a high temperature during the tensile test is short compared to that, phase stability was secured from the alloy design and heat treatment, and there is an insignificant influence of coarsening, not the growth of γ'. In order to clarify the cause of uncertain ductility minimum, it is necessary to conduct a microstructure analysis.

In this study, the cause of ITB in heavy duty forged Ni based superalloy Nimonic263, which is suitable for use under an A-USC condition, was investigated through observations of the microstructure. Tensile specimens were tested at various temperatures from room temperature to high temperature with various microscopes.

2. Experimental Procedure

The Nimonic263 alloy used in this experiment is a wrought Ni-based superalloy with a high content of Cr and Co of up to 20 wt%. The chemical composition of Nimonic263 alloy was given in Table 1. The material was provided from Doosan Heavy Industries & Construction Co. The alloy was subjected to standard heat treatment at 1150 °C, followed by water quenching and aging at 800 °C for 8 h.

The tensile tests were carried out in air at room temperature at up to 900 °C using a sub-size cylinder shape specimen following ASTM E-08 [22] with a Hounsfield Universal Testing Machine. The cross head speed was 1 mm/min, and the corresponding strain rate was $5.5 * 10^{-4}$. In the case of the high-temperature tensile test, a thermocouple was attached to the surface of the gauge section of the specimen, and the tensile test was performed after the specimen reached the test temperature. The microstructure was observed at a gauge section in a plane perpendicular to the direction of the tensile load. For the optical microscope (OM) and scanning electron microscope (SEM) observations, the specimens were electro-polished with a solution of 10% perchloric acid +90% methanol at −25 °C, flow 14, 20 V after ground using SiC paper, subsequently etched with a solution of 10% HCl + 90% ethanol for a few seconds. SEM observations were conducted using Inspect F (FEI Company, FEI Japan). For the transmission electron microscope (TEM) observation, thin foil specimens were prepared using a twin jet polishing technique with a solution of 20% perchloric acid +80% methanol at −30 °C, 20 V condition. An observation of the thin foil specimens was carried out using a TECNAI F20 (FEI Company, FEI Japan) transmission electron microscope operating at 200 kV.

3. Results and Discussion

3.1. Tensile Properties

Fig. 1 shows the yield strength and elongation of Nimonic263 with temperatures up to 900 °C. The variation in the yield strength was small until the temperature reached 800 °C and then drastically decreased to about 312 MPa at 900 °C. On the other hand, the elongation slightly increased with the temperature at a temperature range from room temperature to 500 °C. It then decreased rapidly, showing a minimum value of 19% at 800 °C, and then recovered.

Table 1
The chemical composition of Nimonic263 (wt%).

Ni	Cr	Mo	Fe	Co	Ti	Al	Mn	Si
Bal.	20.3	5.5	0.410	19.7	2.1	0.478	0.205	0.068

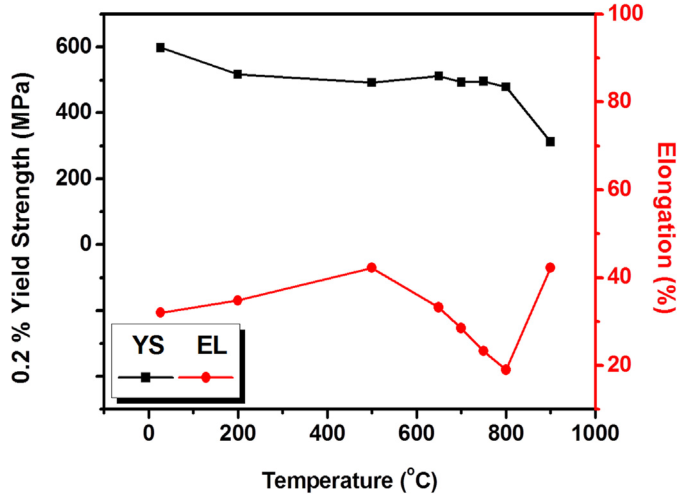

Fig. 1. Yield Strength and elongation for Nimonic263 as a function of the temperature from the tensile test at room temperature and a high temperature.

The polycrystalline Ni-based superalloys are known to be strengthened by the grain size hardening, solid solution strengthening of the γ matrix and precipitation strengthening due to an interaction of the dislocation with γ' [23]. Since, the Ni-based superalloy is suitable for long term use due to its excellent thermal stability, it is not necessary to consider the changes in grain sizes due to each tensile test temperature. Also, solid solution strengthening, which is determined by the alloy composition [24], depends heavily on the size of the alloying element and strain rate [25,26], not the temperature. In addition, unstable solute atoms can migrate due to high temperatures [27]. For this reason, the increment of solid solution strengthening was decreased with increase of temperatures. Therefore, the temperature dependence of the tensile properties, shown in Fig. 1, is related to the interaction of the dislocation with γ'. γ' precipitates are not only a main cause of ITB [21], but they also play an important role as a strengthening mechanism, as mentioned earlier, by an interaction with the dislocation like to pair coupling, cross slip induced hardening, bowing for resistance to antiphase boundary formation from γ' shearing [23], so it is necessary to consider precipitation hardening. The most important strengthening mechanism of the Ni-based superalloy strengthened with γ' which has an $L1_2$ ordered structure [28], and this is discussed in detail in Section 3.2.2.

3.2. Microstructure

3.2.1. Initial Microstructure

Fig. 2 shows the SEM and TEM images of the initial microstructure taken from the aged specimen. MC, indicated with arrows, and $M_{23}C_6$ precipitated along the grain boundary were observed (Fig. 2(a)). Fig. 2(b) shows a bright field image taken from aged specimen together with the inserted selected area diffraction pattern. The zone axis is $[100]_\gamma$ in two beam conditions of $[002]_\gamma$. The spherical precipitates observed in matrix were γ' having ordered $L1_2$ crystal structure, and their sizes ranged from 20 to 30 nm. γ' with double arc contrast arises from the strain field around coherent γ' precipitates, and the initial microstructure of Nimonic263 after the standard heat treatment was consistent with the results proposed in the literature [20].

Fig. 3 presents the equilibrium phase fraction calculated for Nimonic263 using the ThermoCalc. software with the TCNI6 data base. When calculation the equilibrium phase, only the phases observed in the initial microstructures (Fig. 2) were set to be output. The equilibrium η and μ phases of this alloy have been reported to have slow precipitation kinetics compared to γ' [29]. Solvus temperature of $M_{23}C_6$ and γ' is 840 and 1026 °C, respectively. The equilibrium phases at