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Characterization of alloy 718 subjected to different thermomechanical treatments☆

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ABSTRACT

Chemical phase and microstructural investigations of alloy 718 solution-annealed and age-hardened were performed in this study. Focus was made in the effects of solution annealing temperature, aging temperature and holding time, and the amount of intermediate cold work on the alloy. The formation of secondary phases such as γ'-phase, γ"-phase, and δ-phase, grain sizes, and any deformations of the microstructure with respect to the processed conditions have been studied. Statistics such as size and number densities of these precipitates with respect to the processing conditions were evaluated and a discussion on optimum conditions in obtaining finer and higher density of γ'- and γ"-phase precipitates is also presented.

1. Introduction

Inconel or alloy 718 has been widely used in a number of industries such as in spacecraft, gas turbines, pumps, and as well as in nuclear reactors. This material has considerably high corrosion resistance, weld stability, and microstructural stability at high temperatures (~650 °C) [1]. It is an alloy of Ni (~53 wt%), Fe (~19 wt%), Cr (~19 wt%), Nb (~5 wt%), and Mo (~3 wt%) with small amounts of Ti and Al and impurity amounts of Mn, Si, Co, Cu, C, P, Ta, and B. These are combined with Ni in a vacuum induction melting process followed by vacuum arc remelting to obtain this 718 superalloy specification. Ingots are subsequent forged for homogenization before final thermal mechanical heat treating of finished parts.

Over the past years, behavior of alloy 718 has been studied under various environmental conditions. These include testing of toughness, mechanical strength, and most importantly stress corrosion cracking (SCC) or irradiation assisted stress corrosion cracking (IASCC), which has been seen in pressurized water reactors (PWR).

Depending on the chemical and physical processes used in fabricating alloy 718, it could include secondary phases such as γ'-, γ"- and δ-phases, and minor secondary phases such as Laves phases and carbides and/or nitrides. The γ"-phase is generally considered as Ni_3Nb with a tetragonal unit cell (space group: I4/mmm). But in the presence of constituent elements in alloy 718, Ni_3(Nb, Ti, Al) has also been proposed as the general chemical formula for the γ"-phase [ii] since this phase can retain other elements too. Similarly, γ'-phase has been given a general chemical formula of Ni_3(Ti, Al, Nb) opposed to Ni_3(Al,Ti) or Ni_3Al with a cubic unit cell (space group: Pm-3m). The δ-Ni_3Nb phase has an orthorhombic unit cell with a space group: Pmmn.

The γ'- and γ"-phases harden alloy 718 by precipitating into the metal matrix, therefore, its strength attributed to the presence of these secondary phases, especially the γ"-phase in alloy 718. The uniformly distributed fine precipitates of γ'- and γ"-phases are important in order to obtain good mechanical properties of alloy 718 materials [2]. However, the metastable γ"-phase can undergo a slow transformation into δ-phase if the material operating temperature is > 650 °C, leading to limit the use of alloy 718 at higher operating temperatures. The δ- and Laves phases were reported to influence the alloy corrosion behavior in deleterious ways [3]. Ni-based superalloys have a tendency to fail at high temperatures (~600 °C or higher) due to its intergranular structure, which leads to cracking phenomena such as from oxygen diffusion followed by decohesion at grain boundaries [4]. Hydrogen embrittlement can also occur in this material [5]. The stress rupture and any loss in ductility of the alloy are believed to be reduced with the addition of small amounts of phosphorus and boron to the solid-solution since segregation of P and B to the grain boundary reduces grain boundary diffusion reactions [6–9]. As one of the remedies to

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Table 1
Chemical compositions of the samples.

Element	Chemical composition (wt%)		Comp. #1 – Comp. #2
	Comp. #1	Comp. #2	
Ni	52.77	52.73	0.04
Fe	18.95	19.18	−0.23
Cr	18.46	18.49	−0.03
Nb	5.20	5.01	0.19
Mo	2.90	2.90	0
Ti	0.96	0.76	0.20
Al	0.61	0.78	−0.17
C	0.36	0.36	0
Si	0.02	0.02	0
Ta	0.01	0.02	−0.1
Cu, Mn, Co	< 0.01	< 0.01	–
P	< 0.005	< 0.005	–
S	< 0.0005	< 0.0005	–

such cracking problems, grain boundary engineering, which involves processes such as nanocrystal surface modifications, microstructural deformation, and different types of annealing such as strain annealing in modifying surface microstructure [10], have been studied.

In overall, microstructure of alloy 718 also plays a major role in leading the alloy's better behavior under nuclear reactor operating conditions, especially considering its optimum IASCC performance [11]. In this study, changes in the microstructure of alloy 718 samples processed at different experimental conditions have been examined using electron microscopy with X-ray diffractometry as a supporting technique.

2. Experimental details

2.1. Chemical compositions and processing conditions of the alloy 718 samples

The alloy 718 used in this work consists of two different chemical compositions (Table 1), which were made intentionally. In these samples, the major differences in elemental composition can be observed in elements Fe, Nb, Ti, and Al, while Al and Ti are the most important because of their presence in the γ'- and γ"-phases. Less Nb and more Fe are present in composition #2. Ti amount is greater in the composition #1, while composition #2 has more Al.

The alloy 718 sample names and different processed conditions are given in Table 2. These various experimental conditions include three different solution annealing temperatures, two different cold work conditions, and aging at few different temperatures. A solution annealing temperature of 945 °C was used on three samples (first three in Table 2). These samples were subjected to cold work at 0%, 10%, and 20% followed by two aging processes at 718 °C and 621 °C for 8 h. at each temperature. The two samples solution-annealed at 1065 °C were subjected to aging at 760 and 649 °C for 10 and 20 h., respectively. One of these samples was 10% cold worked. Out of the four samples (last four in Table 2) solution-annealed at 1093 °C, three samples were aged at 718 and 621 °C for 8 h. at each temperature, while the fourth sample was aged at 788 and 718 °C for 16 and 50 h., respectively. This sample was subjected to an additional aging step at 663 °C for 50 h. Only one sample (eighth sample in Table 2) was cold worked (10%) out of these four samples annealed at 1093 °C.

2.2. Sample characterization

2.2.1. X-ray diffractometry

X-ray diffraction (XRD) patterns of the samples were acquired using two diffractometers. A Scintag XRD operated at 40 kV and 40 mA and a benchtop D2 Phaser XRD operated at 30 kV and 10 mA. A range

Table 2
Processing conditions used on alloy 718 samples. Chemical composition of sample #7 is indicated by comp. #2 in Table 1. Sample names are denoted by solution-annealed temperature followed by the amount of cold work. For samples #6 and #7 in the table, the cold work amount is followed by a number to denote their difference in the chemical compositions reported in Table 1. The cold work is followed by '663' in sample #9 to denote its extra aging step. AC, WC, SA, and RT denote air-cooled, water-cooled, solution-annealed, and room temperature, respectively.

Sample Name	Solution Anneal (° C/h)	Cooling	Cold Work After SA (%)	Aging #1 (° C/h)	Cooling rate to Aging #2 (°C/h)	Aging #2 (° C/h)	Cooling rate to Aging #3 or RT (° C/h)
945-0	945/1	AC	0	718/8	55	621/8	AC
945-10	945/1	AC	10	718/8	55	621/8	AC
945-20	945/1	AC	20	718/8	55	621/8	AC
1065-0	1065/0.5	WC	0	760/10	55	649/20	AC
1065-10	1065/0.5	WC	10	760/10	55	649/20	AC
1093-0-1	1093/1	WC	0	718/8	55	621/8	AC
1093-0-2	1093/1	WC	0	718/8	55	621/8	AC
1093-10	1093/1	WC	10	718/8	55	621/8	AC
1093-0-663[a]	1093/1	WC	0	788/16	55	718/50	55

[a] An additional aging at 663 °C for 50 h. was done.

of 10 – 110° 2Theta with a step size of 0.004–0.005 were used in acquiring the XRD patterns. A NIST silicon standard (Si SRM640b or SRM640d) was also used with the samples when acquiring XRD patterns to accurately refine lattice parameters using Reitveld refinement using GSAS [12] software. The results obtained from these XRD full profile fits were also utilized in determining crystallite size and microstrain using Williamson-Hall relationship, $\beta Cos\theta=(K.\lambda/\tau)+4\varepsilon Sin\theta$, where β is the peak broadening due to crystallite size while microstrain, shape factor, wave length, and crystallite size are represented by ε, K, λ, and τ, respectively. A shape factor of 0.94 was used assuming spherical crystallites. The value of θ is equal to half the value of the reflection angle. The value of βt.Cosθ was plotted against Sinθ, and microstrain of the sample was determined using the slope of the plot. Crystallite size can be determined using its intercept of the plot or using individual peaks using Schrrer formula (crystallite size: $\tau=K\lambda/(\beta.Cos\theta)$).

2.2.2. Electron microscopy

Microstructural investigation of the samples was performed using a JEOL 6500 FEG-SEM (scanning electron microscopy) equipped with energy dispersive spectroscopy (EDS) and electron backscatter diffraction (EBSD) detectors. Specimen preparation for TEM (transmission electron microscopy) imaging was carried out using a Struers TenuPol-5 electropolisher. A 3 mm disk mechanically polished up to ~70 μm thickness was used in the electropolishing, which was done in a solution of 10% perchloric acid in 90% methanol at −30 °C temperature. A couple of TEM specimens were prepared using a focused ion beam (FIB) system (Quanta™ 3D FEG). A 2100 F JEOL TEM was used to image the specimens.

3. Results

XRD studies indicated mainly the presence of a Ni-based chemical phase with a face-centered cubic (fcc) unit cell and Fm-3 m space group. One of the typical XRD patterns together with its full profile fit performed using Rietveld method is shown in Fig. 1a. Since some undeveloped minor peaks were observed in this XRD pattern, comparatively high-resolution XRD patterns were obtained using increased scanned time for each step. These high-resolution scans showed the