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Microstructural and DSC studies of Alloy 718^{TM} Plus

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ABSTRACT

Thermal stability of the microstructure of Alloy 718^{TM} Plus has been investigated using the non-isothermal differential scanning calorimetry (DSC) technique. Scanning electron microscopy (SEM) and X-ray diffraction (XRD) techniques are used for microstructural analysis. Samples are heat treated at different temperatures before performing DSC tests. The DSC curves exhibited endothermic and exothermic peaks reflecting *in situ* changes in the microstructure. The ratio of endothermic to exothermic peak area increased as the preheating temperature increased.

1. Introduction

In recent decades, wide spread interest has been developed in nickel-base superalloys due mainly to their superior mechanical properties at higher temperatures [1]. These materials are relatively affordable, and are used in modern jet engines, power generation industries, petrochemical industry, etc. [1,2]. Alloy 718^{TM} Plus is one such recently developed alloy exhibiting higher temperature capabilities than its predecessor Alloy 718^{TM} (704 °C vs. 649 °C). It has also been reported that Alloy 718^{TM} Plus has good fatigue crack propagation resistance, superior tensile and stress rupture properties [3,4]. In this alloy, precipitation hardening is achieved by manipulating Al/Ti ratio and increasing the (Al + Ti) content, resulting in dominant γ' phase [5]. Fine γ' precipitates have been shown to be stable up to 760 °C [6]. As temperature increases, γ' phase in the alloy can transform to a new δ phase. Such change in the microstructure can influence the mechanical and thermal properties of the alloy due to the nature, morphology and formation kinetics of δ phase [7]. These thermal properties can be evaluated by using various thermal analysis techniques, such as differential thermal analysis (DTA) and differential scanning calorimetry (DSC). DTA and DSC were carried out on Inconel 718 [8] and other nickel-base superalloys [9,10], but no detailed information is available on the DSC experiments of Alloy 718^{TM} Plus.

In this paper, we investigate the microstructural evolution characteristics of Alloy 718^{TM} Plus in both as received and preheated conditions (600–1350 °C) using the DSC technique and complemented by SEM and XRD.

2. Experimental details

2.1. Material

The Alloy 718^{TM} Plus was received in the form of a forged bar of 1.25 in. diameter from ATI Allvac, Monroe, NC, USA. Chemical composition (in wt.%) of the as received material is as following: C, 0.025; Cr, 18.0; Mo, 2.70; W, 1.00; Co, 9.00; Fe, 10.00; Nb, 5.40; Ti, 0.70; Al, 1.45; P, 0.007; B, 0.004 and balance, Ni. For the DSC study, the bar was cut into small pieces with masses of ~50–60 mg. Some samples were heat treated (i.e. preheated) at 700 °C, 800 °C, 900 °C, 1040 °C, 1275 °C, and 1350 °C for several hours in a heavy duty furnace prior to the DSC tests. All heat treated samples were quenched in water to room temperature immediately after taking out of the furnace.

2.2. Microstructural studies

For microstructural studies, standard metallographic procedures were used. SEM and XRD were used to characterize the microstructure of the samples: (i) as received and (ii) heat treated sample at 1275 °C for 168 h. For SEM analysis, samples were etched in Kalling's reagent: 2 g $CuCl_2$, 40 ml HCl, and 80 ml methanol. Images were captured using a FEI Sirion 200 FESEM (Field Emission Gun Scanning Electron Microscope) operating in backscattered electron (BSE) imaging mode. Limited energy dispersive X-ray spectroscopy (EDXS) studies of some phases in FESEM were carried out. XRD experiments were performed to study the different phases. The asymmetric scan time for the samples was 1 h with an incident Cu Kα X-ray beam of 5.00° incidence angle and scan axis 2θ (20–80°).

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2.3. DSC measurements

DSC technique was used for examining the microstructural evolution of the alloy. DSC involves measurement of the heat absorbed/released during a phase transformation in the sample during heating or cooling depending on whether the reaction is endothermic or exothermic. A NETZSCH STA (simultaneous thermal analyzer) model 409 PC was used to perform the DSC tests. In a DSC test, the sample can provide an understanding of thermal behavior because it is sensitive to the reaction of the main phases determined as a change in microstructure at higher temperatures. The DSC chamber was evacuated and purged several times with highly pure (99.9999%) argon gas. The sample masses ranging between 50 and 60 mg was found suitable for an acceptable signal to noise ratio. The sensitivity curves for scan rate of 20 °C/min were calibrated using the melting point of pure indium, tin, zinc, aluminum and gold under research grade argon atmosphere. Baseline calibrations were performed by using a pair of empty crucibles corresponding to three heating rates as mentioned above. The flow rate of argon was 85 cm^3/min and was kept constant for all measurements to prevent contamination. All samples were cleaned with methanol before each DSC experiment.

Non-isothermal DSC measurements were accomplished on preheated samples (i.e. heat treated at 700 °C, 800 °C, 900 °C, 1040 °C, 1275 °C, and 1350 °C for various times), and the as received material. The DSC furnace temperature was raised from 25 °C to 1150 °C and then the temperature was lowered at the same rate down to 400 °C.

3. Results and discussion

The microstructure of the as received Alloy 718^{TM} Plus is shown in Fig. 1. A BSE SEM microscopic view (Fig. 1a) reveals the presence of an average grain size of ~34 μm (i.e. average spatial grain size = 1.78 × mean linear intercept grain size). A few large Nb-rich MC carbides can be observed. A higher magnification micrograph in

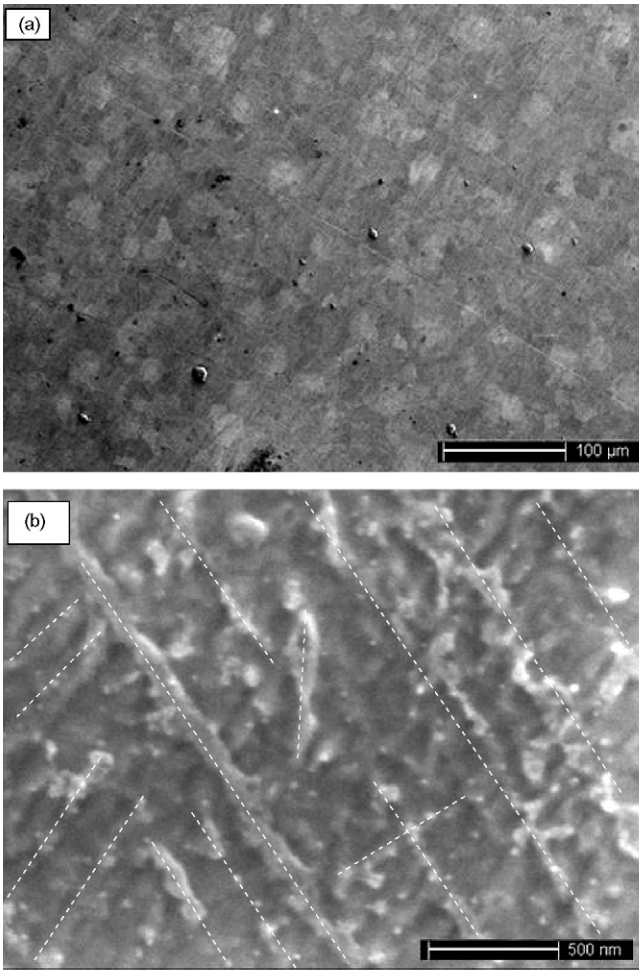

Fig. 1. (a) Lower and (b) higher magnification SEM BSE images of the as received Alloy 718^{TM} Plus.

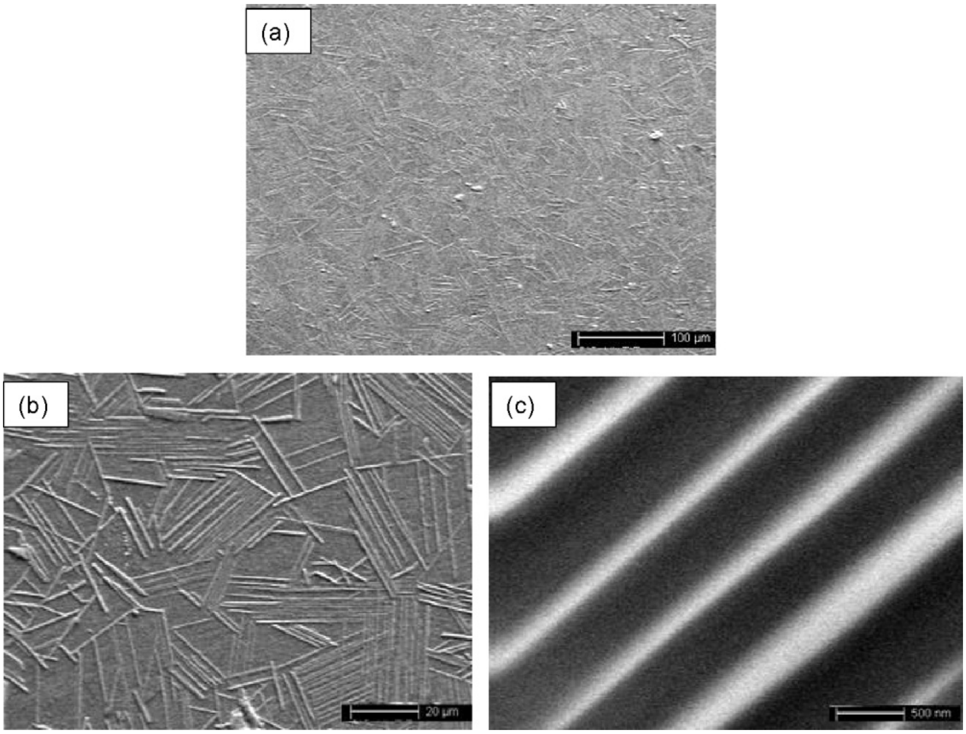

Fig. 2. SEM images of Alloy 718^{TM} Plus heat heated at 1275 °C for 168 h at various magnifications.

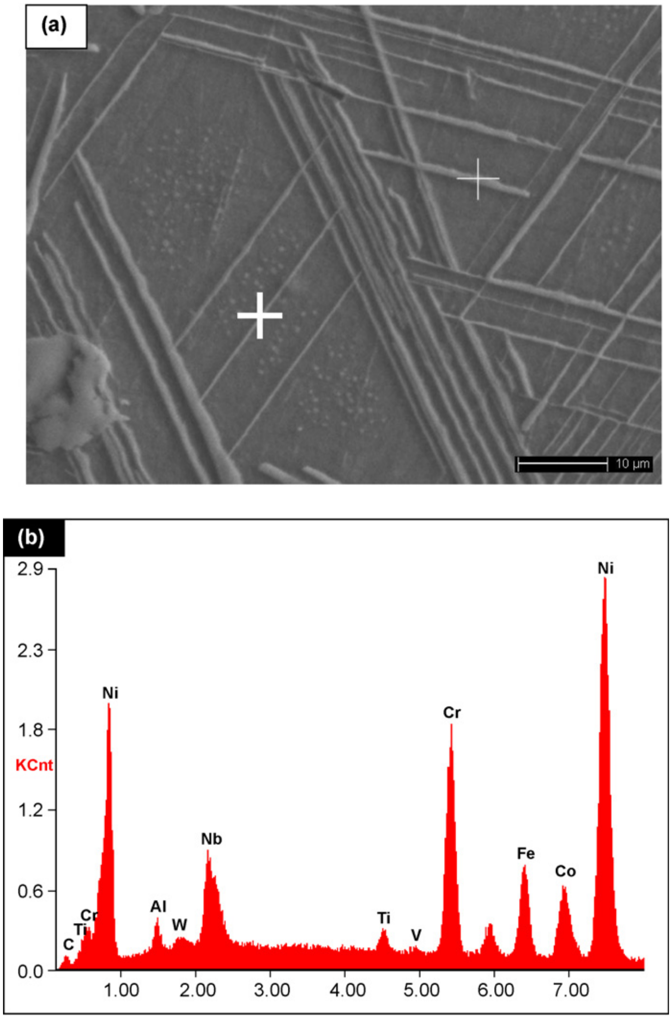

Fig. 3. EDXS spectrum of preheated (1275 °C/168 h) Alloy 718^{TM} Plus.

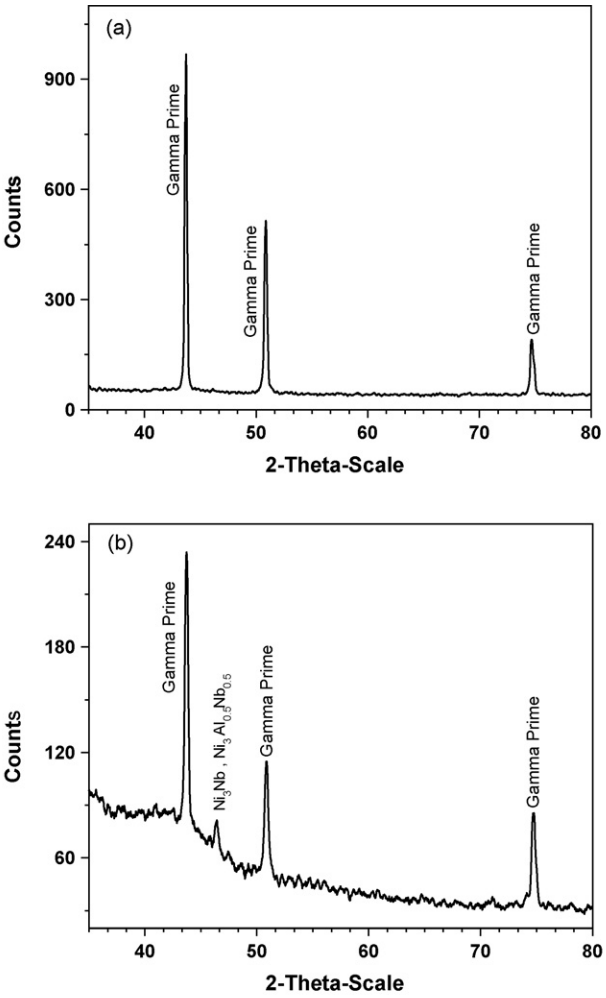

Fig. 4. XRD scan on (a) as received and (b) preheated (1275 °C/168 h) Alloy 718^{TM} Plus.

Fig. 1b shows microstructure in finer detail. Two kinds of precipitate morphologies can be detected in this microstructure. The majority of the precipitates are arranged in the form of irregular edged plates as delineated by dotted lines. Smaller precipitates (diameter < 20 nm) are randomly dispersed in the rest of the γ matrix.

The microstructure of the preheated (1275 °C for 7 days) material was also examined. Fig. 2a shows the presence of well defined relatively straight edged plates and some MC carbides. The plate-type precipitates have variable thickness (most being <1 µm) and are distributed in the form of groups as shown in Fig. 2b. The number of plates in the group varies. Fig. 2c shows BSE images of the plate-type shaped precipitates in finer detail.

The nominal chemical composition of these plate-like precipitates was determined by the EDS technique at different positions, such as two positions as shown in Fig. 3a. The EDS spectrum shown in Fig. 3b reveals close chemical compositions at both positions. Spectrum showed the presence of all major alloying elements of Alloy 718^{TM} Plus in plate-like precipitates. It is suggested that when the alloy was heated at higher temperature (1275 °C for 7 days), elements present in irregular edged plates become active and went through extensive redistribution of atoms to form well defined plates. The high temperature microstructure was preserved as the sample was quenched in water at room temperature.

X-ray diffraction (XRD) experiments on both as received and preheated (1275 °C for 7 days) sample were conducted for phase identification. XRD pattern of the as received material is shown in Fig. 4. No δ phase was detected in the XRD pattern, only γ' phase was present as shown in Fig. 4a. This observation reveals that second phases present in the as received alloy (Fig. 1a) are mainly γ' phase [5]. XRD pattern of the preheated sample is shown in Fig. 4b. Additional phases, such as Ni_3Nb (δ phase) and $Ni_3Nb_{0.5}$ $Al_{0.5}$ (η phase) are thought to be present. But their peaks are present at the same position and that is why they appear superimposed.

It is known that γ' phase can be transformed to δ phase, but the rate is much slower in Alloy 718^{TM} Plus. Previously, it has been reported [7] that the volume fraction of δ phase decreases at higher temperatures and finally dissolve into the γ matrix at 1018 °C at its solvus temperature, but our XRD results (Fig. 3b) showed the presence of δ phase. It means that δ phase may not completely dissolve in the γ matrix.

DSC measurements were carried out on the as received alloy and other preheated samples at 700 °C, 800 °C, 900 °C, 1275 °C and 1350 °C at constant heating and cooling rate of 20 °C/min. There was no endothermic or exothermic peak observed for as received Allvac 718^{TM} Plus sample during the heating and cooling between 25 °C and 920 °C. This suggested that either there is no or relatively insignificant phase transformations occurred within 25–920 °C. For the as received sample, DSC heating (with the endothermic peak) and cooling (with the exothermic peak) curves were sharp as compared to the preheated sample as shown in Fig. 5. The endothermic peak onset temperature for the as received sample is observed at 942 °C while for the preheated sample (1275 °C for 7 days) it was 711 °C. The peak endothermic temperature decreases with increas-

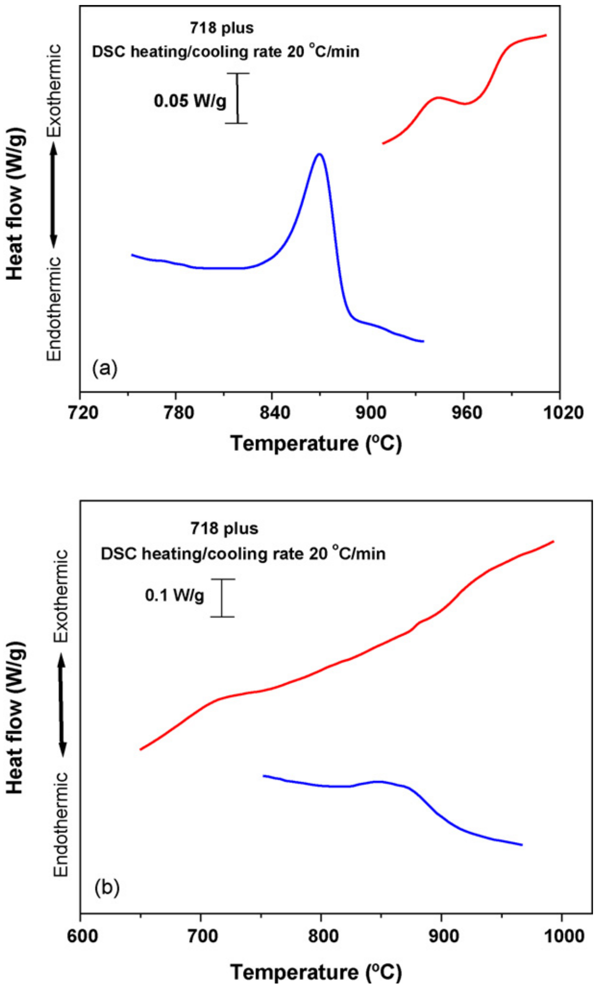

Fig. 5. Heat and cooling curves of (a) the as received 718^{TM} Plus and (b) the sample heat treated at 1275 °C for 7 days (168 h).

ing preheating temperature. The endothermic peak energies were smaller compared to the exothermic ones and increased with preheating temperatures as shown in Table 1. Fig. 6 shows that the ratio of the endothermic to the exothermic peak area increases with increasing preheating temperature with a dramatic increase observed at 1275 °C and 1350 °C.

In studies of alloy phase transformation, DSC performed at constant rates is often considered a key technique. It is possible to track the phase transformation by virtue of endothermic or exothermic peaks. On heating different phases may dissolve slowly as for the γ′ dissolution in this case. For the as received Alloy 718^{TM} Plus, the heat flow effect due to the dissolution is a sharp endothermic peak between 942 °C and 989 °C, and at constant cooling rate, the heat flow effect observed is a sharp exothermic peak between 901 °C and 869 °C.

Table 1
Endothermic and exothermic peak behavior at different temperature.

No.	Heat treated temperature (°C)	Heat treatment (h)	Endo peak area (J/g)	Exo peak area (J/g)
1	As received sample	–	2.625	21.05
2	700	72	3.553	10.37
3	800	72	4.134	10.58
4	1350	72	24.46	12.71
5	900	24	4.744	11.36
6	1040	24	5.673	10.9
7	1275	24	8.549	3.988
8	1275	168	14.86	13.46

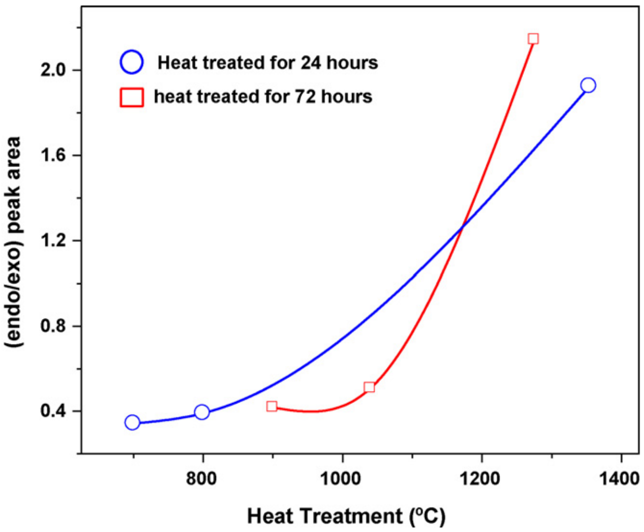

Fig. 6. The peak area ratios increase with temperature.

What was particularly unusual in the present results was the energy values obtained on heating were lower than those for cooling within preheating temperatures of 700–1040 °C, and the situation reversed for preheated samples at 1275 °C and 1350 °C. The decrease in the endothermic onset temperatures of preheated samples is due to the change in the microstructure. When a sample is heated to higher temperatures, more phases dissolve affecting the endothermic reaction during the DSC scan. Further targeted experiments are required to fully explain the origin of phase transformations observed in this study.

4. Concluding remarks

This study focused on the thermal stability of Alloy 718^{TM} Plus using differential scanning calorimetry and other characterization techniques. The as received material showed the presence of irregular shaped γ′ phases. However, upon heat treatment at 1275 °C for 7 days, there appear certain changes in the shape of those phases and it appeared to have undergone phase transformation. A combination of DSC, SEM/EDXS and XRD techniques was employed to elucidate the behavior.

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References

[1] X.Z. Qin, J.T. Guo, C. Yuan, C.L. Chen, H.Q. Ye, Metall. Mater. Trans. 38A (2007) 3014–3022.
[2] K. Schreiber, K. Loehnert, R.F. Singer, Rolls Royce Deutschland Ltd. & Co. KG, Eschenweg 11, 15827, Blankenfelde-Mahlow, Germany.
[3] X. Liu, J. Xu, N. Deem, K.-M. Chang, E. Barbero, W.D. Cao, R.L. Kennedy, in: E.A. Loria (Ed.), Tadeu Carneiro, Superalloys 718 706 and Derivatives 2005, TMS (The Minerals, Metals & Materials Society), 2005.
[4] W.D. Cao, R.L. Kennedy, Acta Metall. Sinica 18 (2005) 39–46.
[5] W.D. Cao, R. Kennedy, in: K.A. Green, T.M. Pollock, H. Harada, T.E. Howson, R.C. Reed, J.J. Schirra, S. Walston (Eds.), Superalloys 2004, TMS (The Minerals, Metals & Material Society), 2004.

[6] X. Xie, G. Wang, J. Dong, C. Xu, W.D. Cao, R. Kennedy, in: E.A. Loria (Ed.), Superalloys 718, 625, 706 and Derivatives, TMS (The Minerals, Metals & Materials Society), 2005.
[7] W.D. Cao, in: E.A. Loria (Ed.), Superalloys 718, 625, 706 and Derivatives 2005, TMS (The Minerals, Metals & Materials), 2005.
[8] T. Antosson, H. Fredriksson, Metall. Mater. Trans. 36B (2005) 85–96.
[9] L.A. Chapman, J. Mater. Sci. 39 (2004) 7229–7236.
[10] S. Raju, N.S.A. Kumar, B. Jeyaganesh, E. Mohandas, U.K. Mudali, J. Alloys Compd. 440 (2007) 173–177.