OXIDATION CHARACTERISTICS AND THERMAL EXPANSION OF

INCONEL ALLOY 718 FROM 300 K TO 1273 K

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Abstract

The measurements of the coefficient of thermal expansion (CTE) of Inconel alloy 718 are reported for the temperature range of 300 K to 1273 K and compared with the CTE of Ni. For Ni, CTE (in units of 10^{-6} K^{-1}) varies from 12 at room temperature to 19 at 1273 K with a sharp peak at the magnetic transition T_c = 630 K. For the 718 alloy, CTE increases monotonically with temperature from 11 at 300 K to a value of 16 at 925 K. Above 925 K, CTE increases sharply reaching a peak value of 28 at 1150 K above which temperature, CTE again falls, matching the value of Ni at the higher temperatures. X-ray diffraction studies of alloy 718 at various stages of annealing, aging and CTE measurements show that the above heat treatments oxidize the sample, producing $FeNbO_4$ and $(Cr,Fe)_2O_3$ as surface oxides, contracting the lattice of the austinitic fcc phase and depleting the δ-Ni_3Nb phase. Thermogravimetric measurements show that weight gain due to oxidation begins near 700 K, with the maximum rate of weight gain occurring between 925 K and 1075 K and somewhat lower rate at the higher temperatures. The sharp increase in the CTE of alloy 718 above 925 K is therefore most likely associated with its oxidation and the resulting degradation of its austinite and δ-Ni_3Nb phases.

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Introduction

INCONEL alloy 718, a commercial nickel-base superalloy, is widely used for high temperature applications for temperatures up to 650°C.[1] It is know to consist of solid solution austinitic fcc matrix (γ phase), strengthening γ' phase of precipitated $Ni_3(Al,Ti)$, the orthorhombic δ-Ni_3Nb, the body-centered-tetragonal γ'' phase of Ni_3Nb, and other minor phases (carbides, Laves).[1, 2] Recently, we have reported [3] on the magnetic behavior of alloy 718 at cryogenic temperatures since this alloy is also receiving increasing attention for cryogenic applications [1]. In this paper, we report on the temperature dependence of the coefficient of thermal expansion (CTE) of alloy 718 in the temperature range of 300 K to 1273 K. It is observed that above about 650°C (= 923K), CTE of alloy 718 increases rather sharply by nearly 70% and sample surface gets oxidized. X-ray diffraction measurements of the oxidized surface show that the products of the oxidation are the oxides $(Cr,Fe)_2O_3$ and somewhat smaller amounts of $FeNbO_4$. Details of these results are reported in the following, along with their implications.

Pertinent Experimental Details

The sample of alloy 718, provided by INCO Alloys International, has the following nominal weight percentage composition: Ni – 52.5; Cr – 19.0; Fe – 18.5; Nb – 5.1; Mo – 3.0; Ti – 0.9; Al – 0.5; Cu – 0.15; and C – 0.08. X-ray diffraction (XRD) studies

reported here were carried out with a Rigaku (D/Max) diffractometer using CuK_α radiation with average λ = 1.54185 Å. The thermogravimetric measurements were carried out on a Mettler 3000 system. The measurements of thermal expansion above room temperature were carried out using a computer-controlled fused-quartz push-rod dilatometer and a linear variable differential transformer (Lucas-Schwevitz). In these measurements, some details of which have been reported recently [4], the sample zone is evacuated to a pressure of about 10^{-3} Torr and then filled with a positive pressure of argon gas (which does contain some oxygen as an impurity). The system was calibrated using the known thermal expansion of oxygen-free high purity copper [5]. The data of thermal expansion $\Delta\ell/\ell$ reported here are the average of three independent measurements on the same sample. CTE values are computed from $\Delta\ell/\ell$ versus temperature data by differentiation i.e. computer-fitting a set number of consecutive data points to a polynominal, the slope at the median temperature yielding the CTE and then sliding the polynominal to neighboring set of points.

CTE measurements were done in the as-received 718 alloy and the same sample aged using the following standard procedure:[3] (i) annealing in air at 982°C for 1 hr; (ii) etching and polishing the annealed sample; (ii) annealing in air at 718°C for 8 hr, furnace cooling to 621°C followed by aging for 18 hr then air cooing to room temperature; (iv) etching and polishing the aged sample. After each step, room temperature XRD measurements of the samples were done when possible, to determine any crystallographic changes.

Results and Discussion

The measured $\Delta\ell/\ell$ values for the as-received and the aged 718 alloy samples are plotted against temperature (from 300 K to 1273 K) in Fig. 1 where ℓ

refers to the value at room temperature ($\approx$300 K). The aged sample has higher CTE at all temperatures, indicating that aging treatments described above are producing significant changes. However, significant increases in CTE are evident in Fig.1 between 1000 K and 1200 K for both samples, as discussed in some detail later.

In order to determine the crystallographic changes at different stages, we show the representative XRD patterns. For example, before and after annealing at 982°C for 1 hr, (the patterns in Figs. 2 and 3 respectively), we note that annealing has produced $(Cr,Fe)_2O_3$ and small amounts of $FeNbO_4$ whereas before annealing, lines only due to fcc austinitic phase and δ-Ni_3Nb are present (Fig 2).
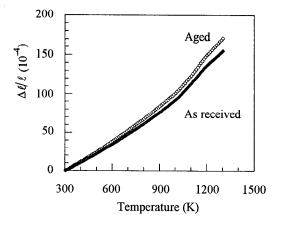

Fig. 1 The variation of $\Delta\ell/\ell$ with temperature for as-received and the aged Inconel 718 alloy.

After completing the aging process, and etching and polishing, the XRD pattern obtained is shown in Fig.4. This shows that lines due to δ-Ni_3Nb are considerably weakened and lines due to the fcc phase are broadened.
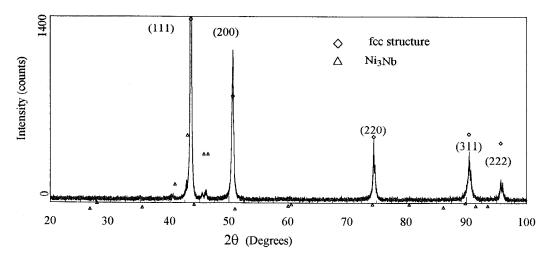

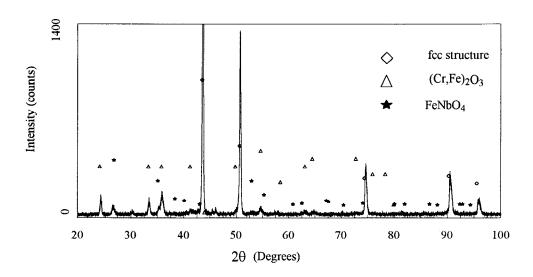Fig. 2 X-ray diffractogram of as-received Inconel 718 alloy

690

Fig. 3 X-ray diffractogram of annealed Inconel 718 alloy

Reasons for this broadening are not clear. After completing the CTE runs to 1273 K, the surface of the sample surface is severally oxidized as very strong lines due to $(Cr,Fe)_2O_3$ and $FeNbO_4$ are clearly identified, and the δ-Ni_3Nb lines are completely missing. This oxidation is presumably due to the high temperatures and residual oxygen present in the sample chamber otherwise filled with argon gas. In Table 1, we list the lattice constant a of the fcc phase and the status of the δ-Ni_3Nb phase at the different steps noted above. The measured lattice constants of the tetragonal $FeNbO_4$ phase are: a = 4.725(5), b = 4.725(4) and c = 3.033(4) Å to be compared with the reported values [6] of a = b = 4.700 and c = 3.046 Å. For $(Cr,Fe)_2O_3$, the measured rhombohedral parameters are a = 5.387(5) Å and α = 55.2°(1), to be compared with the reported values of a = 5.387 Å and α = 55.44°. We note that both Cr_2O_3 and α-Fe_2O_3 also crystallize in the rhombohedral structure [7] with a = 5.350 Å and α = 55.9° for Cr_2O_3 and a = 5.4135 Å and α = 55.17° for α-Fe_2O_3. The lattice constant

a for $(Cr,Fe)_2O_3$ is nearly the average of those of Cr_2O_3 and Fe_2O_3, a completely understandable result.

The results given in Table 1 show that upon surface oxidation, the lattice constant of the fcc phase decreases and the δ-Ni_3Nb phase containing Nb essentially disappears. These results can be understood if oxidation removes Fe and Cr from the fcc austinite phase thereby contracting the lattice and Nb is removed from the δ-Ni_3Nb phase, making the intensity of the latter negligible at the surface layer detected by x-ray diffraction. α-Fe_2O_3 and Cr_2O_3 are the highest oxidized states of Fe and Cr respectively so that the formation of $(Fe,Cr)_2O_3$ is understandable and desirable in regards to providing a protective layer inhibiting further oxidation. The reasons for the formation of $FeNbO_4$ requires further investigation. In this connection, the accelerated oxidation mechanism of the intermetallic $NbAl_3$ at 727°C may be relevant [8]. In this case Nb oxidizes to Nb_2O_5 and Al to Al_2O_3, the two oxides then combining to form

Table 1. The variation of lattice constant of the fcc phase and the intensity of the δ-Ni_3Nb phase, with different heat treatment steps.

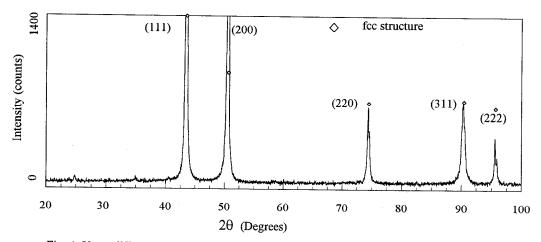| Sample	lattice parameter of fcc phase (Å)	Intensity of δ-Ni_3Nb phase
as-received	3.5987 (1)	normal
Annealed at 983 °C for 1 hr.	3.5962 (1)	decreased intensity due to surface oxides
Annealed and etched	3.5987 (2)	no change from as-received alloy
Aged with oxide	3.5962 (3)	absent
Aged and etched	3.5979 (2)	very low in intensity

Fig. 4 X-ray diffractogram of the aged and etched Inconel 718 alloy

AlNbO4. A similar reaction here between Nb2O5 and Fe2O3 could produce FeNbO4, as observed experimentally. The temperature variation of CTE of the aged alloy 718 from 300 K to 1273 K is shown in Fig. 5 where the data on pure Ni is also shown for comparison. The peak in CTE for Ni near 630 K is due to magnetic ordering and it is a well understood phenomena.[9]. The interesting part of this comparison is that below 900 K, CTE of alloy 718 nearly equals that of pure Ni except for the difference from the magnetic contribution near T_C of Ni. Above about 925 K, CTE of alloy 718 increases dramatically, approaching a peak value near 1150 K whereas CTE for pure Ni has the expected smooth temperature dependence in the same temperature range. This increase in CTE of alloy 718 above 925 K is most likely due to the result of severe oxidation discussed above which makes the major fcc austinite phase of alloy 718 soft for lattice expansion. This degradation of alloy 718 above 925 K is perhaps what limits its
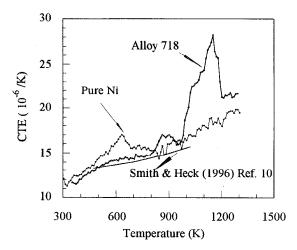
usefulness above this temperature. In Fig. 5, we also show the recent data of Heck and Smith [10] on alloy 718 in a limited temperature range which compares favorably with our measurements.

To further investigate the high temperature oxidation behavior of alloy 718, the data of mass change versus temperature obtained from thermogravimetric measurements in flowing argon gas (with some oxygen impurity) are shown in Fig. 6.
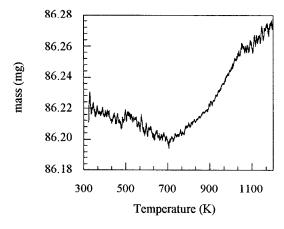

Fig. 6 The variation of mass with temperature for as-received Inconel 718 alloy.

The mass increase begins to occur above 700 K, with the rate of mass change being the highest in the 925 K to 1075 K range. The largest increase in CTE also occurs in this temperature range, clearly suggesting a strong correlation between changes in CTE and oxidation. In-situ x-ray diffraction experiments at high temperatures are now planned to further investigate the process of oxidation of alloy 718.

Summary and Conclusions

The results presented here show that alloy 718 undergoes severe oxidation above 925 K, resulting in the formation of the surface oxides (Cr,Fe)2O3 and

Fig. 5 The variation of the CTE of Inconel 718 alloy with temperature. The Data for Ni is shown for comparision purposes.

$FeNbO_4$. This oxidation results in the contraction of the fcc austinite phase, the disappearance of the δ-Ni_3Nb phase and nearly 70% increase in the CTE of this alloy beginning around 925 K. This oxidation and the resulting softening of the fcc phase for rapid lattice expansion limits the usefulness of this alloy to temperatures below 925 K.

Acknowledgements

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