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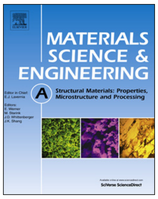

Rapid communication

Brittle failure of Alloy 693 at elevated temperatures

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ABSTRACT

This reports transgranular brittle failure during high temperature deformation of a wrought Alloy 693 received in solution annealed condition. This has been attributed to the inability to suppress the formation of γ' precipitates during quenching after the solution annealing treatment. The fine γ' precipitates severely strained the matrix and inhibited plasticity during subsequent deformation.

1. Introduction

Alloy 693 is a newly developed nickel-base superalloy based on Alloy 690 with Al and Ti additions. The alloy is a disordered solid-solution single-phase alloy and supplied in annealed condition. The matrix has a disordered face centred cubic (fcc) austenite (γ phase) phase with lattice parameter $a = 3.524$ Å. Nominal composition of Alloy 693 is given in Table 1 [1]. Since there is limited peer reviewed literature on Alloy 693, its typical properties are described on the basis of technical Bulletin of Inconel 693 of Special Metals Corporations [1]. The alloy exhibits excellent mechanical strength, rupture strength and creep resistance at elevated temperatures. Alloy's high chromium content imparts excellent resistance to oxidation and sulfidation while the addition of Al vastly improves its resistance to other forms of high temperature corrosion as well. Inconel 693 can precipitate second phase when exposed to intermediate temperatures (538–760 °C). The presence of these phases can decrease the ductility and impact properties of the alloy. Though it is not clear if the said second precipitating phase refers to the well known gamma prime (γ') phase or other precipitates such as carbides or sigma phase, it is assumed to be the γ' phase as additions of Al and Ti in nickel-base alloys promotes its formation. The γ' phase is a coherent and stable ordered intermetallic fcc phase with $Ni_3(Al,Ti)$ stoichiometry ($a = 3.568$ Å for $Ni_3(Al_{0.5},Ti_{0.5})$ [2]). This makes Alloy 693 similar to other γ' phase precipitation hardenable nickel-base superalloys such as Nimonics, Alloy 740, etc. [2]. Inconel 693 exhibits lowest ductility of about 6% as total elongation in tension at temperature about 800 °C. The Inconel alloy always exhibits yield strength (Y.S.) of about 400 MPa in the temperature range from room temperature to 700 °C, while it increases marginally above 400 MPa in the temperature range from 700 °C to 800 °C after which it falls drastically. The Inconel 693 can be annealed by heating it to 1010–1066 °C and holding for a time commensurate with section thickness followed by rapid cooling in air or water quenching [1].

In general, nickel base superalloys are known to be readily fabricated by standard hot deformation forming techniques like extrusion, forging, etc. Typically, the normal hot-working temperature range for the Alloy 690 is 870–1230 °C. Heavy hot working is done in the 1040–1230 °C temperature range, while light working can be continued down to 870 °C [3]. Hot working of the alloy is avoided between 650 and 870 °C because of their low ductility in that temperature range [3]. The rate of cooling following hot working is not critical with respect to thermal cracking in this alloy. The alloy, however, is cooled rapidly through the temperature range of 540–760 °C if subsequent use dictates freedom from sensitization [3]. Likewise, the Alloy 693 is also expected to possess forming and machining characteristics similar to those of Alloy 690. However, the studied Alloy 693 remained hard up to about 900 °C even after the purported solution treatment, which was at variance with properties of the Inconel alloy [1]. The studied alloy failed in a brittle manner at elevated temperatures even with the slowest strain rate employed. The work reported here is a part a systematic study that has been

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initiated in our laboratory to study the precipitation and deformation behaviour of the Alloy 693 in question.

The objective of this study is to identify the cause of brittle failure of the alloy at elevated temperatures and to understand if the solution treatment given was proper or not.

2. Experimental

Samples for the study were taken from an as-received wrought commercial Alloy 693 (10 mm thick sheet), which was received in the solution annealed condition. Table 2 gives the chemical composition of the alloy studied. Chemical analysis of major alloying elements was carried out using inductively coupled plasma optical emission spectrometry (ICP-OES) technique, while the carbon was analysed using the combustion analysis. Average composition was calculated on the basis of three measurements. This analysis confirmed that the composition of the alloy was well within the nominal range of Alloy 693. Since the as-received alloy exhibited a brittle behaviour, all the tests were repeated after giving the alloy a solution treatment of 1150 °C for 2 h (in laboratory conditions) followed by water quenching to verify the solution annealed state of the as-received alloy. A solution treatment temperature higher than that recommended in [1] was chosen on the basis of the solution treatments typically given to γ' strengthened alloys [2] to ensure complete dissolution of all the precipitates that are known to form in nickel-base superalloys during ageing.

Deformation studies were carried out in a BAHR DIL 805A/D dilatometer in the deformation mode using programmable heating-holding-deformation-cooling cycles. Cylindrical shape solid samples of 4 mm diameter and 10 mm length held between two alumina plungers were heated up to the desired temperature at a rate of 5 °C/s by an induction coil, held for 10 min, deformed at elevated temperatures using strain rates of $0.002\ s^{-1}$ and higher, and quenched by helium gas. The sample temperature was measured by a platinum/platinum–10% rhodium thermocouple spot-welded at the centre of cylindrical surfaces of samples. Temperature of the dilatometer was calibrated on the basis of phase transformation temperatures of pure metal samples, see, e.g., reference [4]. All experiments were carried out in a vacuum better than 10^{-5} mbar. The same dilatometer, in the quenching mode, was used to generate continuous cooling transformation (CCT) curve of the alloy using a hollow cylindrical sample of length 10 mm, outer diameter of 4 mm and wall thickness of about 0.5 mm to ensure temperature homogeneity across the radial direction of the sample [4]. The hollow sample was held between two quartz push-rods attached to a linear variable differential transformer (LVDT), with one of the rod fixed and the other attached to the sample. A detailed description of the dilatometer is given in reference [4].

Microstructure was studied using optical, scanning and transmission electron microscopy. Specimens for transmission electron microscopy (TEM) were prepared by a dual jet electropolishing unit, using an electrolyte containing 20% perchloric acid in ethanol. The electrolyte temperature was maintained at around −40 °C and a voltage of about 20 V was used for thinning. TEM thin foils were examined in a transmission electron microscope operated at 160 kV.

3. Results and discussion

Optical microscopy examination of the as-received alloy showed a microstructure typical of a wrought nickel base superalloys having about 120 μm size grains with a number of globular shape carbide particles (Fig. 1). Grain boundaries were devoid of carbide precipitates along them. Sparsely distributed globular shape particles, visible in Fig. 1, were primary carbides which form shortly below the solidification temperature. These particles were distributed homogeneously throughout the alloy at intergranular and transgranular positions. Presence of primary carbides is usually found to be not detrimental to mechanical properties [2].

The as-received alloy showed room temperature hardness of about 265 VHN, which was equivalent to about 850 MPa tensile strength. This strength is typical of precipitation hardened nickel-base superalloys [2]. The alloy failed when deformed under compression at elevated temperatures up to 900 °C even with a strain rate as low as $0.002\ s^{-1}$. The alloy exhibited a little ductility as evidenced in small bulging and curvature of the sample around the failed surfaces. Fig. 2a shows a photograph of a failed compression sample at 900 °C. It is evident that the sample failed in a brittle manner by shearing at angle of about 45° with respect to the compression axis. The fractured surface had a faceted texture. The fractured surface was devoid of "dimples", "microvoids" or any appreciable gross plastic deformation (like twisting, tearing, etc) usually observed for a ductile fracture. It was characterised by ploughing lines or ridges, which

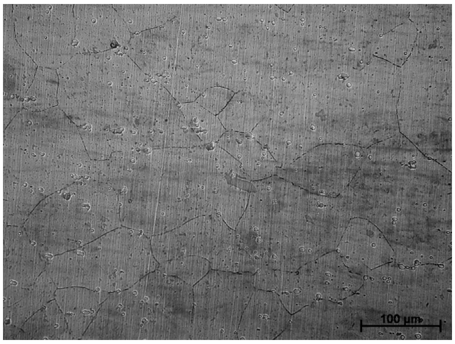

Fig. 1. Optical micrograph of the as-received wrought Alloy 693.

Table 1
Nominal composition (in wt%) of Alloy 693 [1].

Ni	Cr	Fe	Al	Ti	Nb	Mn	Cu	Si	S	C
Bal.	27.0–31.0	2.5–6.0	2.5–4.0	1.0 max	0.5–2.5	1.0 max	0.5 max	0.5 max	0.01 max	0.15 max

Table 2
Measured chemical composition (in wt%) of the Alloy 693 under study.

Ni	Cr	Fe	Al	Ti	Nb	Mn	S	C
58 ± 2	27 ± 1	3.6 ± 0.1	2.9 ± 0.2	0.33 ± 0.02	1.90 ± 0.07	0.12 ± 0.02	0.0017	0.023 ± 0.002