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Microstructural studies on Alloy 693

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ABSTRACT

Superalloy 693, is a newly identified 'high-temperature corrosion resistant alloy'. Present study focuses on microstructure and mechanical properties of the alloy prepared by double 'vacuum melting' route. In general, the alloy contains ordered Ni_3Al precipitates distributed within austenitic matrix. M_6C primary carbide, $M_{23}C_6$ type secondary carbide and NbC particles are also found to be present. Heat treatment of the alloy at 1373 K for 30 min followed by water quenching (WQ) brings about a microstructure that is free from secondary carbides and Ni_3Al type precipitates but contains primary carbides. Tensile property of Alloy 693 materials was measured with as received and solution annealed (1323 K, 60 min, WQ) and (1373 K, 30 min, WQ) conditions. Yield strength, ultimate tensile strength (UTS) and hardness of the alloy are found to drop with annealing. It is noted that in annealed condition, considerable cold working of the alloy can be performed.

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1. Introduction

Immobilization of high level nuclear wastes within inert glass matrices is considered to be the best available options today [1–5]. In general, (a) induction heated metallic melter pot and (b) Joule heated ceramic melter pot are used for this purpose [6,7]. However these technologies suffer from faster degradation of Superalloy 690 (high Cr, low Fe containing Ni based austenitic alloy) components, e.g. thermowells, feeders, electrodes, pour spout assemblies, metallic process pot, etc., during service. Experimental studies under partially simulated service conditions [8–16] suggest that the following physicochemical changes at the Alloy 690/borosilicate melt interfaces might play crucial roles in the material failures;

(i) secondary Cr carbide ($Cr_{23}C_6$ type) precipitation along grain boundaries and at triple point junctions,
(ii) Cr depletion within austenitic matrix close to interface,
(iii) intergranular attack and incorporation of waste glass melt pool components through diffusion and redistribution along these openings,
(iv) formation of Cr_2O_3 and glassy layers (containing needle shaped Ni_2CrO_4, and cubic $NiCr_2O_4$ phases) at the interface.

Such pre-mature failures not only cause unprecedented shut down of vitrification plant operations but also generate huge pile-up of hazardous non-compactable radioactive metallic wastes. To avoid such situations, 'preventive management strategy', either through development of diffusion barrier coating on Alloy 690 or through its compositional modification appear to be more appropriate ones. Towards the first option, the authors have already successfully developed (i) Ni-YSZ composite [17] and (ii) Ni-aluminide [18] coatings on the laboratory scale specimens. However, considering the harsh environment experienced by the Superalloy within vitrification furnaces, it is thought worthwhile to explore the alloy modification route as well [19,20].

Superalloy 693, is a newly developed 'high-temperature corrosion resistant alloy' which contains more than about 2.5 wt% Al than its predecessor, i.e. Alloy 690 (Table 1). The present study describes the microstructure and mechanical properties of Superalloy 693 prepared through a combination of 'vacuum induction melting' and 'vacuum arc melting' routes.

Gallium (Ga) isotopes are commonly produced within spent nuclear fuels during neutron irradiation and subsequent decay of radio-nuclides such as U^{235}, U^{238}, Th^{232}, Pu^{239}, Am^{241} and Np^{237} [21]. Since Ga is known to cause stress associated failures in aluminium alloys [21] and as Alloy 693 contains ~2.5 wt% of Al it was felt prudent to examine the possible effect of low energy Ga ion irradiation on the alloy. Further, as during immobilization of high level nuclear wastes, the vitrification furnace components are being exposed to 'ionic damage mechanisms' so Ga ion irradiation is also used for simulating the expected service conditions.

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Table 1
Bulk composition (in wt%) of Superalloys 690 and 693.

Element (wt%)	Cr	Fe	Al	Cu	Si	Mn	S	C	Nb	Ti	N	Ni
Alloy 690	27.0–31.0	7.0–11.0	0.50 max	0.50 max	0.5 max	0.5 max	0.01 max	0.05 max	–			Rest
Alloy 693 (minimum)	27.0	2.5	2.5	–	–	–	–	–	0.5	–	–	Rest
(maximum)	31.0	6.0	4.0	0.5	0.5	1.0	0.01	0.15	2.5	1.0	–	Rest
Used Alloy (XRF analyses)	29.32	3.96	3.19	<0.02	0.04	0.09	<0.002	0.097	1.86	0.42	130 ppm	Rest

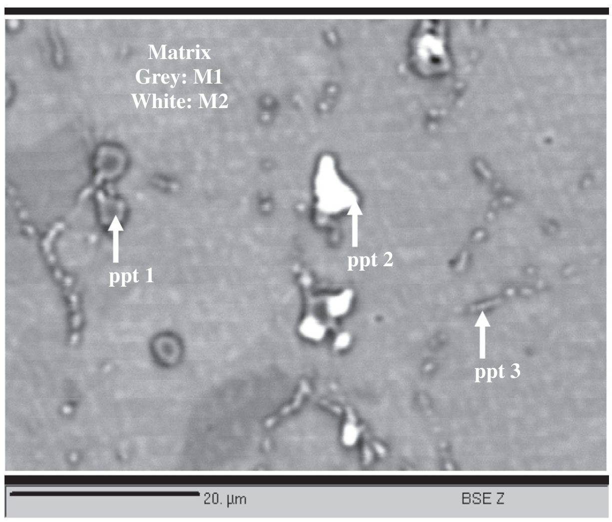

Fig. 1. Back scattered electron image (with atomic number contrast) showing the three types of precipitates, designated as precipitate 1 (ppt 1), precipitate 2 (ppt 2) and precipitate 3 (ppt 3) within the alloy. Also note that the matrix is constituted of two phases namely grey phase (M1) and white phase (M2).

2. Experimental studies

Microchemical analysis of as-received Alloy 693 was carried out using CAMECA SX-100 Electron Probe Micro-Analyser (EPMA). Small coupons were cut from Alloy 693 sheet, ground with emery paper and polished with diamond paste of 1 µm grain size. An acceleration voltage of 20 keV and stabilized beam current of 4 nA and 20 nA were used for electron imaging (secondary electron; SE and back scattered; BSE) and qualitative analysis respectively.

Samples for examination under transmission electron microscopy (TEM) were prepared by jet electropolishing technique using an electrolyte containing 90% methanol and 10% perchloric acid by volume. A temperature of about 238 K was maintained during electropolishing with the applied voltage of around 20 V. The electropolishing of each specimen was continued until a tiny hole was formed at the centre of 3 mm disc.

To understand the nature of grain-orientation and its size distributions, electron backscatter diffraction patterns (EBSD) of the samples were obtained using FEI Quanta 3D scanning electron microscope system. The scans were taken over an area of $250 \times 400\ \mu m^2$, using an accelerating voltage at 30 kV and probe current 16 nA. Kikuchi bands were obtained for each of the different phases present within the alloy. In order to obtain some preliminary idea on the response of the alloy to ionic damage mechanisms, small coupons were exposed to 30 keV Gallium (Ga) ions with ion current 0.1 nA for 10 min at ambient temperature under high vacuum, using a focussed ion beam (FIB) gun attached to the system. To study the "Ga ion damage on microstructure" detailed grain boundary mapping of the austenitic matrix ($70 \times 125\ \mu m^2$) was carried out. The net fluence and dose were found out to be 4.29×10^{15} ions/cm^2 and 2.878 KGy respectively; assuming that all ions fell on the surface where fluence was being calculated. As the experiment was carried out at high vacuum, attenuation through scattering in the medium was neglected.

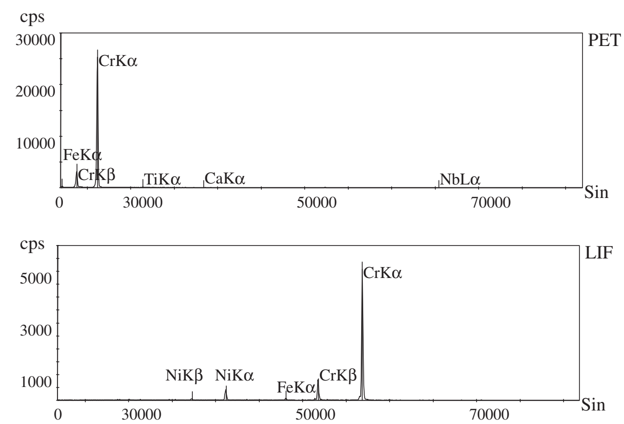

Fig. 2. X-ray spectrum showing the presence of various elements within precipitate 1.

Tensile property measurements were done in as received and solution annealed (1323 K, 60 min, WQ) and (1373 K, 30 min, WQ) conditions each with 20 mm gauge length. Experiments were carried out at ambient temperature using test velocity of 0.25 mm/min (i.e. $\sim 10^{-4}$/s).

Hardness of as received and solution annealed alloy coupons was measured using a load of 200 gf and 10 s as dwell time. Micro-hardness values quoted here were obtained over an average of 10 readings.

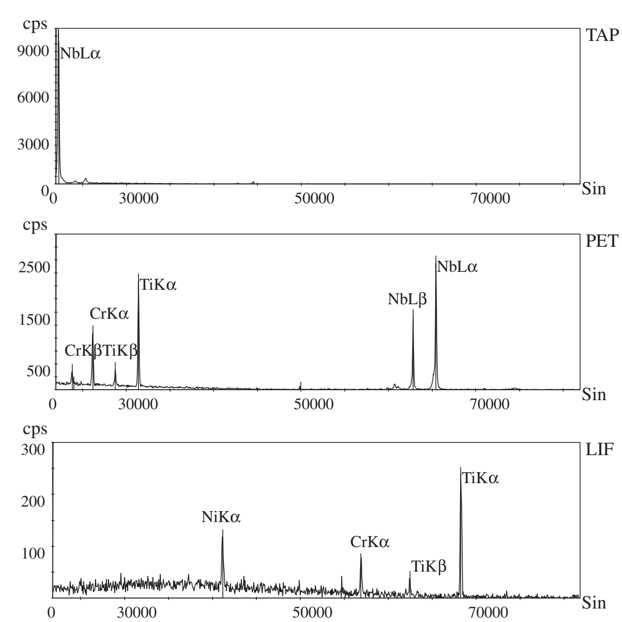

Fig. 3. X-ray spectrum showing the presence of various elements within precipitate 2.

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