

Investigation of the secondary phases of Alloy 617 by Scanning Kelvin Probe Force Microscope

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Abstract

Alloy 617 (nickel based austenitic alloy) is a candidate structural material for next generation high temperature nuclear reactor. The secondary phases present in the Alloy 617 has been identified as TiN and $M_{23}C_6$ by optical microscopy, scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDAX). Surface potentials of these phases have been evaluated by Scanning Kelvin Probe Force Microscope (SKPFM). SKPFM investigation showed higher surface potential of the secondary phases than that of the austenite matrix. The surface potential of the TiN was nobler than that of $M_{23}C_6$ indicating that these precipitates could act as cathodic sites during oxidation reaction. Difference in the local chemistry of these phases results in variation of the chemical potentials, which in turn could lead to enhanced oxidation at localized areas in the vicinity of the secondary phase precipitates that act as cathodic sites.

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1. Introduction

Alloy 617 is a Ni–22Cr–9Mo–12Co–Al–Ti austenitic alloy that shows exceptional creep strength at temperatures above 800 °C, because of solution strengthening by molybdenum and cobalt additions, and also shows improved oxidation and carburization resistance at high temperatures from aluminum and chromium additions [1]. Microstructural evaluation of Alloy 617 after long term exposures to temperatures in the range of 649 °C to 1093 °C showed predominantly $M_{23}C_6$ and a small volume fraction of TiN [2]. Formation of γ' phase was observed at temperatures below 760 °C. Kimball et al. reported that $M_{23}C_6$ was present as a discontinuous network and also as discrete particles at intragranular regions in mill-annealed condition [3]. After aging for 8000 h at 648 °C, the carbide network in the grain boundary became continuous throughout the specimen and the intragranular carbides increased in sizes [3]. The major phases present after a long time exposure of Alloy 617 at 1000 °C was identified as $M_{23}C_6$, Ti(C,N) and also

a small amount of γ' [4]. Recently Gariboldi et al. reported that a new phase δ started to form on the grain boundary at 700 °C and disappeared at 800 °C [5].

Considerable research has been carried out on Inconel 617 alloy to evaluate its corrosion behavior in different environments, even though the alloy was developed for high temperature applications [1]. Yilbas et al. [6] observed pitting corrosion in Inconel 617 near the chromium depleted region of carbide precipitates in 0.1 N H_2SO_4 +0.05 N NaCl solution at room temperature. Kewther et al. observed that the corrosion rate of Inconel 617 in 0.1 N H_2SO_4 +0.005 M NaCl solution accelerated near the Cr and Mo depleted region of the specimen surface [7]. Further, high temperature oxidation resistance of Alloy 617 was observed to be affected by the presence of $M_{23}C_6$ carbides and partial pressure of the oxygen in the environment. At low oxygen partial pressures, decarburization and formation of elemental Cr have been reported by Christ et al. [8] because of the following reaction:



Depending on the temperature and the activity of carburizing environment, the reaction (1) can be driven from right to

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left also to form a stable oxide film. It should be noted that both carburization as well decarburization affect the intended mechanical properties of the alloy. Further, formation of chromium rich carbides and chromium rich protective oxide deplete the Cr in the austenite matrix and deteriorate the solid solution strengthening and lead to formation of η' carbide [9]. It is well known that the difference in the chemical potential between two phases could act as driving force for phase transformation. The phases will be closer to the equilibrium when the difference in the chemical potential is smaller. The chemical potential can be manifested in to the electrochemical potential and in turn can be correlated to the work functions of individual phases. The variations in the work functions of the microscopic phases can be measured using a reference probe by Scanning Kelvin Probe Microscopy (SKPFM). This technique has a very high lateral resolution in the sub-micron level since it employs a conducting probe of an atomic force microscope [10,11]. The variation of work functions between the secondary phases/precipitates and the matrix has been reported for various aluminum base alloys and stainless steels [12,13].

The present work investigates the work function variations of Alloy 617 in solution annealed condition using SKPFM technique due to the formation of different secondary phase precipitates. The work function variation was expressed as surface potential variation. The variation in surface potential map will be helpful to understand the corrosion and oxidation behavior of Alloy 617.

2. Experimental

12.5 mm diameter rods of Alloy 617 material (ASTM B 166/UNS N06617) was acquired in mill-annealed condition. Solution annealing treatment was carried out at 1100 °C for 30 min in flowing argon and air cooled. The chemical composition of the alloy is given in Table 1. The specimen was metallographically polished down to 0.05 μm surface finish with alumina powder and etched with *aqua regia* before observation under the microscope.

The microstructure of Alloy 617 was investigated with a FESEM (Hitachi, model S-4700) and Energy Dispersive X-ray Analyzer (EDAX, Oxford Instruments). The micrographs and EDAX spectra were collected with beam energy of 20 keV. The main focus of SEM and EDAX analysis was on the second phases and also on the grain boundary to identify the second phases in the alloy.

A Scanning Kelvin Probe Force Microscope with iridium coated reference probe (Pacific Nanotechnology, model: nanoR2) which can simultaneously map the topography and potentials was used for SKPFM analysis. The SKPFM analysis was performed on the metallographically polished samples at

room temperature in air with a pixel density of 256×256 and at a scan rate of 1 Hz. The samples were not etched. The surface potential was measured for different second phase precipitates using a line scan analysis along the matrix and second phase. In addition, the surface potential variation was also measured along the grain boundaries.

3. Results and discussion

3.1. SEM and EDAX analysis

The specimens were first observed under optical microscope to identify the precipitates. Shiny golden precipitates with faceted morphology were identified as TiN and the size of this precipitates was about 4–6 μm . Most of the TiN precipitates were distributed intragranularly along with a low volume fraction of intergranular precipitates. The globular type smaller size (2–4 μm) precipitates were identified as Mo and Cr rich carbides that were distributed both intra and intergranularly. The SEM micrograph of polished and etched Alloy 617 is shown in Fig. 1(a). Fig. 1(b) shows the elemental profile across a TiN precipitate. Fig. 1(c) shows the EDAX line scan across a Mo rich carbide precipitate and Fig. 1(d) shows that of Cr rich precipitate. EDAX line scans performed along the grain boundary also indicated intergranular precipitation of M_{23}C_6 (results not shown). Presence of carbide precipitates in the solution annealed condition has been observed by other researches also. Takahashi et al. reported that these intergranular M_{23}C_6 precipitates were stable at 1177 °C [4]. Further, it was observed that the carbide was enriched with Mo even though it contained chromium and other alloying elements.

3.2. SKPFM analysis

The topographical image of the specimen is shown in Fig. 2(a). It showed a variation in surface topography for different precipitates present in the matrix. The grain boundary also showed a change in surface topography. Fig. 2(b) shows the concurrent surface potential mapping of the specimen. The map showed a change in measured surface potential by exhibiting a contrast between the second phases and the matrix. Since an absolute value strongly depends on the careful data acquisition and also sensitive to many factors such as relative humidity and adsorbed layers (moisture, oxygen etc.) on the specimen surface, only change in surface potential with respect to the matrix is primarily studied [10]. The lighter areas showed a higher measured potential and this represent the secondary phases. As it was confirmed from the EDAX and SEM observations that larger size precipitates were TiN and the smaller size precipitates were M_{23}C_6 , line scan was performed along these precipitates to observe the change in surface potential. Moreover the change in the surface potential along the grain boundary was also performed and was compared with the measured surface potential in the matrix.

Fig. 3 shows a plot of the measured surface potential of all the scans. It has been observed that TiN has the highest surface potential compared to the matrix. The second highest potential was measured for the M_{23}C_6 precipitates. Similar amplitude of surface potential was measured in the grain boundary which suggested M_{23}C_6 precipitates in the grain boundary. The change in the surface potential was due to the change in the composition of the precipitates relative to the matrix. The TiN and M_{23}C_6 both showed more positive surface potential suggesting that they were nobler than the matrix. It was also evident that TiN precipitates were nobler than M_{23}C_6 with a more positive

Table 1
Chemical composition of as-received Alloy 617 (wt.%)

Ni	Cr	Co	Mo	Al	Fe	Ti	Si	Mn	C	Cu
Bal.	21.5	12.0	9.3	1.0	1.2	0.36	0.12	.06	.08	.06

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