

Oxidation of Inconel alloy 617 at the Elevated Temperature Air Environment

Daejin Lee, Sanggyu Lee, In Sup Kim, and Changheui Jang

Department of Nuclear and Quantum Engineering, KAIST, 373-1 Guseung-dong, Yuseong-gu, Daejeon 305-701,
Republic of Korea
daejinlee@kaist.ac.kr

1. Introduction

The VHTR (Very High Temperature Reactor) is one of the generation-IV reactor, which is a helium-cooled, graphite moderated thermal neutron spectrum reactor. Since its operating temperature is very high (950°C or higher), VHTR has high thermal efficiency and this can be also used for hydrogen production.

In spite of such great advantages, in realizing this type reactor there are many difficulties related to structural material issues. The high temperature application requires much more stable structural materials in view of strength and oxidation/corrosion.[1] Especially, VHTR requires intermediate heat exchanger (IHX) to transfer heat to power conversion system and hydrogen production facility. IHXs have to be engineered to withstand the coolant gas pressure of 8MPa and high temperature, and to transfer heat as effectively as possible. Nickel base superalloys have excellent oxidation resistance and high temperature strength.[2] Some of nickel base superalloys are considered to be the candidate materials for VHTR.

In the present work, tensile and oxidation tests were performed at the elevated temperature on one of the candidate materials, Inconel 617 alloy. The relation between the high temperature oxidation and strength was investigated. This was discussed based on the microstructural features.

2. Experiment

2.1 Test material and Specimen

Test material is Inconel alloy 617 (UNS N06617 / W.Nr. 2.4663a) produced by Special Metals Company. Chemical compositions are shown in Table 1. This material was heat treated at 1175°C for an hour followed by water quench. As the supplier's information, average ASTM grain size is 4.6. Tensile properties of as-received condition at room temperature are 364MPa in Y.S., 823MPa in UTS, 53.3% in total elongation, and 55.9% in reduction area.[3]

Table 1. Chemical composition of Inconel alloy 617

	C	Mn	Fe	S	Si	Cu	Ni
w/o	.09	.06	1.16	.001	.08	.05	53.94
	Cr	Al	Ti	Co	Mo	P	B
w/o	21.68	1.14	.52	11.53	9.74	.006	.002

Tensile test specimen used in this study is a round bar type, 25 mm in length and 6.0 mm in diameter.

2.2 Test system and Test condition

Tensile tests were carried out at 1100 ± 10 , and $700 \pm 5^\circ\text{C}$ and strain rate of $5 \times 10^{-4}/\text{s}$ in air environment. Since the samples were heated by induction heating system, heating rate was relatively high, $90^\circ\text{C}/\text{min}$.

The tensile tests were started after obtaining temperature stability. Two LVDTs were used to measure displacement of sample. As another test set, oxidation test was conducted with same equipment and similar method. The elevating time to reach a target temperature was 12 min. and heated 5 and 10 hours. Oxidation rate was calculated using weight gain. The test samples were characterized by OM and SEM.

3. Results and Discussions

3.1 Oxidation

Weight gains and oxide layer thickness were measured and shown in table 2.

Table 2. Weight gain and oxide layer thickness

	Weight gain	Oxide thickness
950 °C 5h.	2.61 g/m ²	1.6 μm
1050 °C 5h.	7.69 g/m ²	10 μm

These oxide layers reflected that more oxidation occurs at higher temperature and there was no passivation behavior up to this temperature.

At 1050 °C, oxide thickness was not much affected by the test time, as shown in figure 1. (a), (c). However, in the 10 hour test, oxide layer became more porous and more internal oxidation was observed. According to the previous study [4], external oxide layer was mainly composed with Cr_2O_3 , and internal oxidation was related to Al_2O_3 formation. Since above 1000 °C, $\text{Cr}_2\text{O}_3(\text{s})$ became unstable [5], the internal oxidation could be more increased.

As showed in Figure 1, grain boundary carbides were removed by electrolytic etching but below the oxide layer, approximately 60 μm, carbide removed traces were not found. That's carbide depletion zone. At high temperature, and low carbon environment, decarburization occurs easily and it is known that decarburization makes micro-crack and degrades the creep property.[6]

Grain size No. was increased with oxidation time and temperature, when the grain size of each sample was measured far away from surface and shown in table 3.

Table 3. Grain size of tested specimen.

	Grain size No. G	Grain diameter
950 °C 5 h.	6.5	37.8 μm
1050 °C 5 h.	6.3	40.6 μm
1050 °C 10 h.	5.4	55.4 μm

It should be noted that oxidation test sample showed smaller grain size than as-received. It seems to be a recrystallization and grain growth stage of annealing. [7]

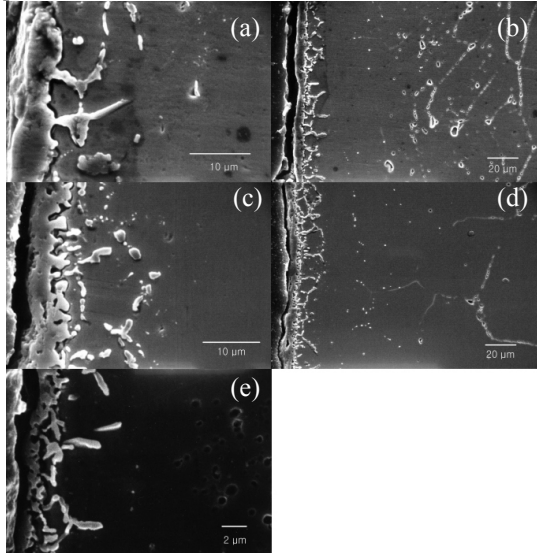


Figure 1. Microstructure of the oxidation test sample (a), (b) 1050 °C 5hour, (c), (d) 1050 °C 10hour, (e) 950 °C 5hour in air.

3.2 Tensile property

There was abrupt decrease in tensile strength between 700 °C and 1100 °C. At 700 °C, Y.S. and UTS were 215.6 MPa and 460.4 MPa, respectively. At 1100 °C Y.S. and UTS were 44.7 MPa and 45.1 MPa, respectively. Uniform elongation at 700 °C was about 55%; while, at 1100 °C it decreased to 5.5%.

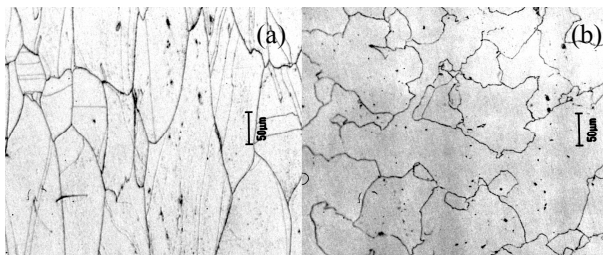


Figure 2. Microstructure of tensile test sample, (a) 700 °C, (b) 1100 °C. Tensile direction is vertical.

Figure 2 is grain morphology of the tensile tested specimen. Figure 2(a) shows smooth and elongated grains along tensile direction. To contrast, Figure 2(b) shows serrated grains. Serration should be related to dynamic recrystallization. This behavior was reported to be common at low and intermediate stacking fault energy alloys around 1000 °C. [8]

Fracture surface of the tensile specimen tested at 700°C was observed by SEM (Figure 3). Figure 3(b) is the representative shape of this sample, mixture of ductile and brittle fracture. Brittle fracture is not good at structural application because of no leak before break but sudden break down. The phenomenon called stress-assisted grain-boundary oxidation (SAGBO) involves brittle fracture of a bulk oxide in similar composition alloy, Inconel 718 at 650°C.[9] More observations are needed to understand the brittle fracture behavior, including SAGBO and other mechanisms, of alloy 617 at the elevated temperature.

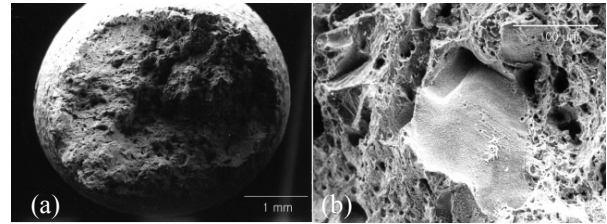


Figure 3. Fracture surface of 700°C tensile test specimen

3. Summary

Oxidation and tensile test results of inconel alloy 617 in air environment can be summarized as follows.

1. From oxidation tests, the higher temperature showed the more oxidation, and the internal oxidation enhanced with longer time.
2. Carbide depletion zone was observed, which would be occurred at the low carbon gas potential and high temperature. This depletion may degrade creep property.
3. Tensile test shows elongating of grains at intermediate temperature and serrated grains at high temperature. Fracture surface at 700°C reveals both ductile and brittle fracture.

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