A study on the oxidation behavior of nickel alloys at elevated temperatures

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Abstract. This study was conducted with the aim to investigate the high temperature oxidation behavior of Inconel 750 alloy, which has excellent high temperature corrosion resistance among Ni-based super alloys. In the study, the temperature was set at 700°C, 900°C and 1100°C in the atmospheric environment; the heat treatment holding time was increased to 1 hour, 6 hours and 12 hours at each temperature to investigate the weigh increase behavior at each condition; and the shape of the oxide layer formed on the surface and the distribution of the elements were analyzed by scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS).

1. Introduction
Ni-based superalloys have excellent corrosion resistance, oxidation resistance, strength and ductility and weldability at high temperature, so they are widely used to manufacture the parts requiring high temperature corrosion resistance and high temperature strength [1]. With the industrial development, materials are used in the gas atmosphere which is highly corrosive at high temperature such as coal gasification, petrochemical industry, or gas turbine. In this harsh atmosphere, Inconel 750 alloy among Ni-based super alloys is used [2].

Inconel is a heat-resistant Ni-based alloy containing 14-17% of chromium, 5-9% of iron, 2.5% of titanium, and less than 1% of aluminum, manganese, silicon and niobium. The Ni-based superalloy containing high Cr and Al forms a protective oxide film such as Cr2O3 and Al2O3 to maintain corrosion resistance even at high temperature [3, 4]. It also has excellent heat resistance, has oxidation resistance in high temperature oxidation stream, and is not immersed in an atmosphere containing sulfur. Its tensile strength, yield point and other properties do not change significantly until the temperature reaches about 600°C, indicating excellent mechanical properties. In addition, it is also resistant to organic and salt solutions. The inconel alloys are largely classified into four types: martensitic, ferritic, austenitic and precipitation strengthening alloy depending on the structure [5].

The Inconel 750 alloy, the subject of this study, is an age-hardening alloy with excellent corrosion resistance and oxidation resistance of Ni + Co > 70%, Cr 14 ~ 17% and Fe 5 ~ 9%. With high tensile strength and returning ability at high temperature, it is widely used for making springs, bolts and parts for turbine generator, plate spring, pressure tube, gas turbine, etc [5].

Although studies on the change of physical properties due to high temperature oxidation of Ni superalloy Inconel have conducted continuously, there have been few studies conducted on the recovery and recycling of the oxide scale generated by the oxidation of Inconel [6, 7, 8, 9, 10, 11].
Inconel is very high value metal among the metals used in the industry. Therefore, if the scraps from the inconel products can be recycled, it will yield very high returns. The recycling of Ni-based alloys is conducted mostly through wet process and there are very few studies [12].

This study, a base study on the possibility of dry recycling of the inconel among Ni-based alloys, investigated the high temperature oxidation behavior of Inconel 750 alloy in the atmosphere. When the Inconel 750 alloy was used at high temperature of 700°C, 900°C and 1100°C, the formation and deterioration process of the coating protecting the alloy base were observed according to the reaction with oxygen depending on the holding time. Scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX) were used to observe the oxide scale formation behavior and the composition and structure of the oxide layer were investigated, was examined.

2. Materials

Table 1 below shows the chemical composition of general Inconel 750 and the material used in this test was a commercially-available Inconel 750 alloy (Figure 1). Inconel 750 alloy was cut to a size of 20×30×0.5 (mm³) and the cross-section in the thickness direction was observed using SEM.

| Inconel | Ni   | Cr    | Fe    | Nb   | Co  | Mn  | Cu  | Al    | Ti   | Si   | C   | S    |
|---------|------|-------|-------|------|-----|-----|-----|-------|------|------|-----|------|
| X-750   | 70.0 | 14.0-17.0 | 5.0-9.0 | 0.7-1.2 | 1.0 | 1.0 | 0.5 | 0.4-1.0 | 2.25-2.75 | 0.5 | 0.08 | 0.01 |

Figure 1. Cross section of Inconel 750 alloy.

Figure 2 is a schematic diagram of the electric furnace used for testing atmospheric high temperature oxidation of Inconel 750 alloy. The electric furnace was designed not to be blocked from the outside to maintain an air atmosphere of 1 atm. An alumina crucible was placed inside the electric furnace and Inconel 750 alloy was put into the crucible for oxidation test. The heater rod was connected to the outside of the furnace to maintain high temperature, and the test conditions of 700°C, 900°C and 1100°C were maintained by adjusting the voltage using a controller.

Figure 2. Schematic diagram of electric furnace used in this study.
Inconel 750 alloy was put into the electric furnace maintained at each test temperature, and the oxidation was conducted for 1 hour, 6 hours, and 12 hours. The weight increase due to oxidation was measured and the behavior of the formed oxide was observed and analyzed using scanning electron microscopy (SEM / EDS). In order to observe the thickness direction during scanning electron microscope observation, the sample was observed after fine mounting with a 0.1 μm diamond paste after cold mounting.

3. Results and discussion

Figure 3 shows the heat treatment temperature and the weight increase with time of Inconel 750 alloy. The temperature conditions were 700°C, 900°C and 1100°C, and the weight change at 1, 6 and 12 hours of holding time at each temperature were expressed. As the temperature and holding time increased, the weight increase became significant due to oxidation. When the holding time was 12 hours at 700°C, the weight increase was greater than that measured when the holding time was 6 hours at 1100°C, which indicates the oxidation behavior is greatly dependent on the holding time from 700°C to 1100°C rather than the temperature.

**Figure 3.** Changes in the weight increase of Inconel 750 alloy according to temperature and holding time.

Figure 4 shows the oxide layer shape and compositional change of the Inconel 750 alloy oxidized at 700°C for 12 hours through SEM / EDS. On the surface of cross-section, it was observed that the oxide layer was formed to a thickness of about 9 μm. Looking at the line profile and the EDS results of the oxide layer, the first oxide layer having a high Cr content was formed from the non-oxidized Ni base and then the second oxide layer having a high Ti content was formed above it. It was confirmed that Cr oxide and Ti oxide were formed on the surface in order to prevent oxidation at a high temperature of 700°C where the inconel alloy is mainly used.

**Figure 4.** Inconel 750 alloy oxidized at 700°C for 12 hours.
Figure 5 shows the oxide layer shape and compositional change of the Inconel 750 alloy oxidized at 900°C for 12 hours through SEM / EDS. It was confirmed that the oxide layer was formed with a thickness of about 5 μm on the surface of cross-section. Looking at the line profile and the EDS results of oxide layer, the Cr oxide layer exists from the Ni base but the Ti oxide layer is not remarkable, differently from the test results at 700°C. Ti oxide layer was peeled off and removed at high temperature, so it seems further researches are needed.

Figure 5. Inconel 750 alloy oxidized at 900°C for 12 hours.

Figure 6 shows the oxide layer shape and compositional change of the Inconel 750 alloy oxidized at 1100°C for 12 hours through SEM / EDS. On the surface of the cross-section, oxide layer is formed to a thickness of about 14 μm. The line profile and the EDS results of the oxide layer are also similar to the test results at 900°C, showing that the Cr oxide layer exists but the Ti oxide layer almost does not exists. At a higher temperature of 900°C and above, the diffusion of Cr from the inside of the alloy to the surface gradually occurs and the Cr oxide layer which became thick has brittleness. This indicates that the oxide layer can be removed from the internal base.

Figure 6. Inconel 750 alloy oxidized at 1100°C for 12 hours.
4. Conclusions
In this study, heat treatment was conducted at 700°C, 900°C and 1100°C for 1 hour, 6 hours and 12 hours, respectively, to investigate the high temperature oxidation behavior of Inconel 750 alloy.

1. As oxidation temperature and holding time increased, the weight increased significantly. It was confirmed that the oxidation behavior was more remarkable according to the change of the holding time than of the temperature.

2. The high-temperature oxidation behavior of Inconel 750 alloy was observed through SEM / EDS. It was confirmed that Cr oxide layer and Ti oxide layer were formed from the Ni base to resist oxidation.

3. The Ti oxide layer was not formed at 900°C and 1100°C, and the thickness of the oxide layer was found to increase with temperature rise.

4. At a high temperature of 900°C and above, the Cr diffusion was gradually increased to the surface and the possibility of removing the Cr oxide layer with high brittleness formed on the surface was confirmed.

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