



Development and oxidation resistance evaluation of Al-rich surface layer on Alloy 617

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ABSTRACT

Surface treatment was carried out for Alloy 617 to improve the high temperature oxidation resistance. After high purity Al was pre-deposited on the surface, electron beam (EB) was applied to form Al-rich micro-alloying zone with the depth of ~50 μm. In the micro-alloying zone, cellular and dendritic structures with very fine arm spacing were developed. In the oxidation test performed in air at 900 °C up to 1000 h, a transient oxidation behavior was observed for the surface-treated Alloy 617. During the short initial oxidation stage (<1 h), Al-rich external oxide layers with the outermost spinel and inner alumina were quickly formed. However, during the steady-state oxidation stage, the oxidation rate of EB surface-treated Alloy 617 was substantially lower than that of the as-received Alloy 617.

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1. Introduction

A very high temperature gas-cooled reactor (VHTR) is one of the leading reactor concepts for the next generation nuclear systems. The operating temperature of a VHTR is in the range of 850 °C or higher with a coolant gas pressure of up to 9 MPa. Among various structure components in a VHTR, the hot gas duct and the intermediate heat exchangers (IHXs) would be exposed to the highest temperature during operation [1]. For IHXs, to achieve high heat transfer efficiency and lower design stress, compact-type heat exchangers, such as a printed circuit heat exchanger (PCHE) or a plate fin heat exchanger (PFHE), have been considered [1–3]. The PCHE-type heat exchanger features semi-circular flow channels, which are stacked and joined by the diffusion bonding [2] or the brazing [3] methods.

Considering the oxidation mechanisms of Ni–Cr–Al alloys discussed by Giggins and Pettit [4], the external chromia (Cr₂O₃) forming alloys were regarded as the promising candidates for the IHX applications [5,6]. Among them, Alloy 617 (Ni–22Cr–11Co–9Mo) is known for the good oxidation resistance and high creep-rupture strength due to the solid solution hardening by Co and Mo [7]. However, the external chromia layer would not be protective in the various high temperature coolant environments, such as a helium gas with small amounts of gaseous impurities [8–11] and a steam with and without hydrogen [12]. Although the level of impurities, such as CO, CO₂, CH₄, H₂O, and H₂, is low in a few decades of ppm [8], the influence of the environments on the mechanical properties is not negligible [11]. More specifically, Nakanishi and Kawakami reported that the crack growth rate of Ni-base alloys increased

in helium environment due to the lack of the oxide films on the crack surface [13]. Also, Schnaas and Grabke observed the accelerated creep strain rate of Ni–Cr–Fe alloys under oxidizing and carburizing conditions [14]. Therefore, materials with better high temperature oxidation resistance would be preferred for IHX applications.

To improve high temperature oxidation resistance, Al could be added to Ni–Cr alloys to promote the formation of an external alumina (Al₂O₃) [4]. Also, the application of aluminide coating to protect Ni-base alloys in the carburizing environments was discussed by Chin et al. [15] and Singheiser et al. [16]. Alternatively, alumina could be directly deposited on top of the substrate alloy. However, there are two drawbacks for IHX applications. Firstly, as ceramics are not easy to be joined, alumina coating should be applied after the IHX is manufactured. Then, as the size of the semi-circular flow channels is small (radius of ~1 mm) and geometric shape is complex, the alumina coating with uniform thickness throughout the flow channels seems questionable. Secondly, provided that the coated alumina layer was damaged or broken, the substrate alloy would be exposed to the environments and oxidized without the protection of the coated alumina.

To avoid mentioned problems above, an Al-rich micro-alloying zone on top of substrate alloy could be considered. By doing so, the authors thought that there would be three advantages. First of all, if sufficient Al is present in the micro-alloying zone, the external alumina would form and provide sufficient protection. Second, the joining of the plates by diffusion bonding [17], which is an essential process for PCHE type IHX manufacturing, would be more feasible as the mating surfaces still have metallic properties. Most importantly, the damage in surface alumina or surface cracking, which is expected under tensile stress (e.g., creep or creep-fatigue) during high temperature operation, could be healed by forming alumina layer around the propagating cracks when sufficient Al is present in the micro-alloying zone.

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In this study, an Al-rich micro-alloying zone on top of Alloy 617 was developed through applying electron beam (EB) on the pre-deposited Al layer. The microstructural characteristics of the surface Al-rich micro-alloying zone will be presented, and the oxidation kinetics and the formation of oxide layers of EB surface-treated Alloy 617 were discussed.

2. Experimental methods

A solid solution strengthening Ni-base superalloy, Alloy 617, was used in this study. The chemical compositions were analyzed by inductively coupled plasma mass spectroscopy (ICP-MS) at Korea Testing and Research Institute and are listed in Table 1. In the as-received Alloy 617, Cr-rich $M_{23}C_6$ type carbides are present on the grain boundaries and stringers of Mo-rich M_6C type carbides are aligned along the rolling direction. Specimens for EB surface treatment and subsequent oxidation test were prepared as 12 mm in diameter and 1 mm in thickness. All specimens were ground down to 1200 grit silicon carbide grinding paper, and ultra-sonically cleaned in ethanol before Al deposition. Fig. 1 schematically illustrates the sequence of the surface treatment process. To deposit high purity Al (99.999%) on Alloy 617, a magnetron direct current sputtering system was used. Before deposition process, the pressure in chamber was lowered to 1.33×10^{-4} Pa and then Ar gas pressure of 3.99×10^{-1} Pa was maintained during Al deposition. The thickness of pre-deposited Al was about 4–5 μm . Then, the Al-deposited specimens were exposed to EB in vacuum of 1.33×10^{-2} Pa to form the surface micro-alloying zone. For the EB treatment, accelerating voltage of 60 kV, working current of 13 mA, and feeding speed of 600 mm/min were used. To treat the specimens in a single pass, a diffuse-focused beam size (~14 mm) was used.

Isothermal oxidation test was performed in air at 900 °C up to 1000 h. After the as-received and the surface-treated Alloy 617 were oxidized in a pre-heated box furnace, the specimens were air cooled to the room temperature and the weight changes were measured by a microbalance with a resolution of 10^{-5} g. Characteristics of the oxide layers were investigated using an X-ray diffraction (XRD, Rigaku D/max-RC) using a filtered Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$), scanning electron microscope (SEM, FEI Sirion) equipped with energy dispersive X-ray spectroscopy (EDS), and electron probe micro-analyzer (EPMA, Shimadzu-1610). Specimens for SEM and EPMA analysis were chemically etched using a solution of HCl:HNO₃ = 3:1 to reveal the microstructure more clearly. Dual beam focused ion beam (FEI Helios NanoLab) specimens for transmission electron microscope (TEM, JEOL JEM-2100F) were extracted from the micro-alloying zone before and after oxidation test.

3. Results and discussion

3.1. Characteristics of the micro-alloying zone

Fig. 2 shows the cross-sectional SEM micrograph of the EB surface-treated specimens. As shown in the figure, a fine cellular and dendritic structure is developed in the micro-alloying zone, which are similar to the microstructure observed in the previous studies on the laser [18,19] and electron [20,21] beam surface treatment of metallic coatings. The carbides observed in the as-received Alloy 617 are not present in the micro-alloying zone, because they were dissolved by the EB treatment. Defects such as solidification cracking and porosity are observed neither in the micro-alloying zone nor at the interface of micro-alloying zone and the substrate (insets in Fig. 2).

Table 1

Chemical compositions of the as-received Alloy 617 (in wt.%).

Ni	Cr	Co	Mo	Al	Fe	Mn	Ti	W	Si	C
Bal.	22.1	11.6	9.57	1.41	1.8	0.12	0.35	0.07	0.42	0.09

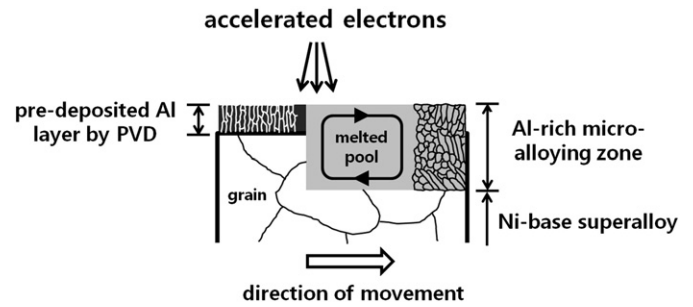


Fig. 1. Schematic diagram of the EB surface treatment process (not drawn to scale).

Fig. 3 shows the SEM micrograph of the uniformly developed micro-alloying zone of the depth of ~50 μm with the results of EPMA line scanning. It is clear from the results of EPMA line scanning that the counts of Cr, Co and Mo in the micro-alloying zone are rather uniform and similar to those of the substrate. Meanwhile, a large fluctuation in Ni counts is observed because of the micro-segregation in cellular and dendritic structures in the micro-alloying zone. Though the intensity of Al is about three times of that in the substrate, Al content in the micro-alloying zone varies 2.3–5.3 wt.% according to EDS point detection analysis results.

Due to the large fluctuations in Ni counts observed in EPMA analysis, an XRD analysis was carried out to check the existence of precipitates, which might be resulted from the micro-segregation. The peak angles of the surface-treated specimen are same as those of the as-received specimen, which indicate that other than γ (Ni–Cr–Co–Mo) substrate, no precipitates were developed in large quantity in the micro-alloying zone. Compared with the previous studies with relatively large amounts of Al (8–10 wt.%) showing the existence of precipitates, such as β (NiAl) [18,20] and γ' (Ni₃Al) [21] in the surface melted area, the amount of Al in the micro-alloying zone (up to ~5 wt.%) seems not enough to form such precipitates. Additionally, TEM/SADP (selected area diffraction pattern) analysis was performed to identify the presence of precipitates more in detail. Like the insets in Fig. 2, cellular structure with arm spacing of about 2 μm is observed. SADP analysis on the boundary of the cellular structure was performed to identify the crystal structure. Since γ' is an ordered FCC ($L1_2$) with the space group of $Pm\bar{3}m$ (space group number 221), it would be readily distinguishable from γ , which is an FCC (A1) with the space group of $Fm\bar{3}m$ (space group number 225). However, the superlattice reflection, which is an evidence of γ' , was not observed in two zone axes such as [112] and [123]. The XRD and SADP results described above correspond with the previous study performed by Giggins and Pettit; while two phases (γ and γ') were

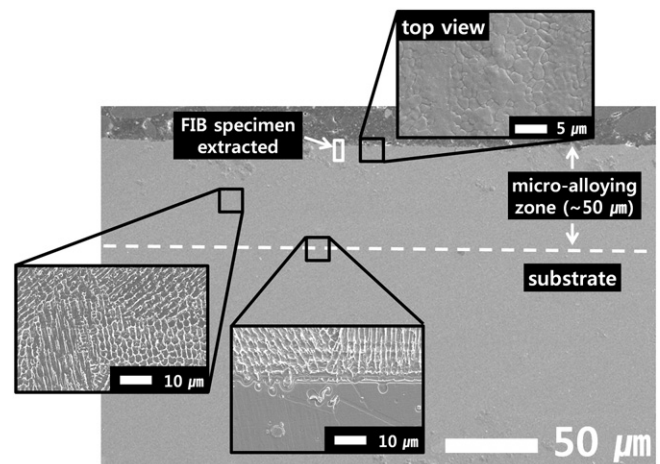


Fig. 2. Cross-sectional SEM micrograph of the surface-treated Alloy 617.

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