Evaluation of thermal ageing behavior of an alumina-forming duplex stainless steel (ADSS) alloy at 375 ºC and 400 ºC

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1. Introduction

Accident tolerant fuel (ATF) cladding materials have been actively studied in recent years to replace current Zr-based cladding materials [1,2]. One of promising candidate materials is Fe-based alloys, which have shown superior oxidation resistance and high strength compared to Zr-based alloys. However, Fe-based ATF cladding materials are susceptible to thermal embrittlement, which is a significant limitation for their application in nuclear reactors. Recently, authors developed alumina-forming duplex stainless steel (ADSS) alloys with high strength (about 1 GPa) and reasonable ductility [3]. ADSS alloys showed duplex microstructure with B2 precipitates, which provide the alloys with high strength. In addition, it is expected the austenite phases helped to lessen severe thermal ageing embrittlement at operating temperatures.

Therefore, in this study, an ADSS alloy was exposed to 375 ºC and 400 ºC for up to 6,000 h to evaluate thermal ageing embrittlement. Then, ageing embrittlement was measured by micro-hardness test and nano-indentation test on austenite, ferrite, and B2, respectively. Finally, thermal ageing behavior of ADSS will be discussed using microstructural observation at the conference.

2. Experimental methods

A 100-kg cast ingot of ADSS #B51 was made by vacuum induction melting (VIM). The ingot was homogenized at 1200 ºC for 4 h, then further processed by hot rolling at 1200 ºC with a thickness reduction of 90 % followed by heat treatment at 900 ºC. Cold-rolling was applied to the ADSS plate with a thickness reduction of 40 %, then final annealing was conducted at 900 ºC for 2 h. The chemical composition of the ADSS was analyzed by inductively coupled plasma atomic emission spectroscopy (ICP-AES) as Fe–18.89Ni–16.48Cr–6.38Al–1.06Mn–0.21Nb–0.13Si–0.11C in wt.%. The cold-rolled and final-annealed, which is called to as-CRFAed, ADSS plates were thermally aged at 375ºC and 400ºC for up to 6,000 h.

For micro-hardness testing, as-CRFAed and thermally-aged ADSS plates were fabricated into a square specimen with a side of 10 mm and a thickness of 0.5 mm by an electro-discharge machine (EDM). The specimens were ground with SiC paper and finally polished with 0.04-μm colloidal silica. Vickers micro-hardness testing was performed at room temperature with a load of 500 g. At least ten measurements were conducted to obtain the average micro-hardness values. Nano-indentation testing was performed on each austenite, ferrite, and B2 with a force of 800 μm kept for up to 2 s by utilizing a Bruker HYSITRON TI Premier. Then, the locations of nano-indents were confirmed with in-situ scanning probe microscopy (SPM), which was equipped in the nano-indenter. For nano-indentation results, at least five measurements were conducted to obtain the average hardness values.

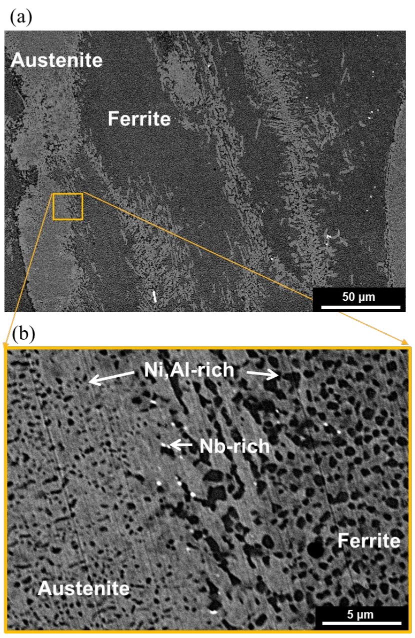

Fig. 1. BS-SEM images of as-CRFAed ADSS at (a) low magnification, and (b) high magnification

3. Results and discussion

Figure 1 shows back-scattered (BS) scanning electron microscope (SEM) images of as-CRFAed ADSS. As shown in Fig. 1(a), as-CRFAed ADSS exhibited austenite (light-shaded) and ferrite (dark-shaded) phases. Ni,Al-rich precipitates were also observed in each austenite and ferrite phase as black dots in Fig. 1(b). The Ni,Al-rich precipitates were revealed to B2 phase in the previous publication [3]

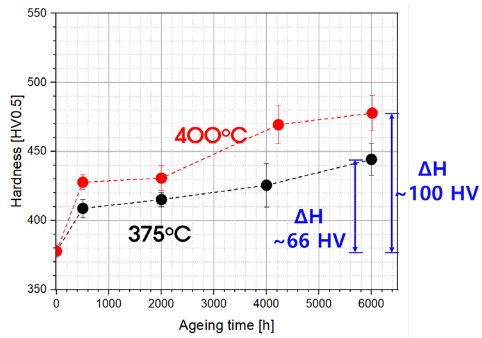

Fig. 2. Vickers micro-hardness results of the ADSS alloy after thermal ageing at 375 °C and 400 °C for up to 6,000 h

To evaluate the entire ageing embrittlement behavior of ADSS, micro-hardness testing was performed on as-CRFAed and thermally-aged ADSS with a load of 500 g. The measured micro-hardness values as a function of ageing time are plotted in Fig. 2. The hardness results of ADSS continuously increased at both temperatures, 375 °C and 400 °C. The higher increase in hardness (~26.7 %) was observed at 400 °C than that (~17.6 %) at 375 °C. Compared with austenitic stainless steel welds (ASSWs), ADSS showed more hardness increase than 316L ASSW aged at 375°C and 400 °C (~8 % increase for both ageing temperatures) [4]. However, while 316L ASSW had approximately 90 % of soft austenite phase, as-CRFAed ADSS had about 40 % of the austenite region including B2 precipitates. Considering the fraction of austenite, it seems that the degree of thermal ageing embrittlement in ferrite is not significant as that in ferrite of 316L ASSW

To check the hardness increase in each phase, nano-indentation testing was performed, and the results as a function of ageing time are plotted in Fig. 3. As reported in lots of literature, the austenite phase of ADSS also did not present the hardness increase along the ageing time at both temperatures. For the ferrite phase, the hardness increases were saturated at the early ageing time for both ageing temperatures, and the degree of ageing embrittlement at both temperatures looks similar. The hardness increase was about 20 % for both ageing temperatures. As compared with ageing behavior for ferrite phase of cast austenitic stainless steels (CASSs) and 2205 [5], ADSS showed much less ageing embrittlement in ferrite region than CASSs (~64 % hardness increase for CF8M aged 400°C for 3,000 h, ~115 % hardness increase for Z3CN20.09M aged at 400 °C for 6,000 h) and 2205 (~69 % hardness increase aged at 400 °C for 4,744 h.

To evaluate thermal ageing behavior of B2 precipitates, nano-indentation testing was conducted on large B2 precipitates (~ 300 nm in diameter) in ferrite region shown in Fig. 1(b). As shown in Fig. 3(c), continuous hardness increases were observed at both ageing temperatures. higher hardness increase was detected at 400 °C (~60 % increase) than 375 °C (~21% increase). Because austenite phase was not ageing-hardened and ferrite was ageing hardened at the early stage, the entire ageing behavior of ADSS would be mostly caused by B2 phase.

4. Summary and Future works

An alumina-forming duplex stainless steel (ADSS) was thermally aged at 375 °C and 400 °C for up to 6,000h. After thermal ageing, hardness increases were observed at each thermal ageing temperature. Through nano-indentation test, it was revealed that the hardness increase was mostly caused by B2 precipitates in ADSS, rather than the ferrite phase. To better understand the thermal ageing embrittlement of ADSS, a detailed microstructure observation will be conducted, and the ageing mechanism of ADSS will be discussed.

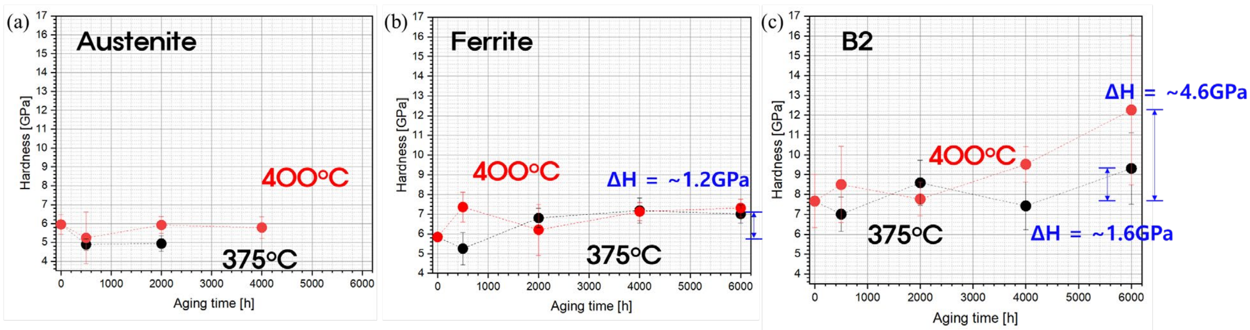

Fig. 3. Nano-indentation results for (a) austenite, (b) ferrite, and (c) B2 of the ADSS alloy after thermal ageing at 375 °C and 400 °C for up to 6,000 h

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