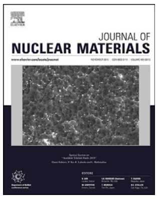
Journal of Nuclear Materials 466 (2015) 201–207

Contents lists available at ScienceDirect

Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat



Irradiation response of delta ferrite in as-cast and thermally aged cast stainless steel

Zhangbo Li [a], Wei-Yang Lo [a], Yiren Chen [b], Janne Pakarinen [c], Yaqiao Wu [d, e], Todd Allen [f, g], Yong Yang [a, *]

[a] *Department of Materials Science and Engineering, Nuclear Engineering Program, University of Florida, Gainesville, FL 32611, USA*
[b] *Nuclear Engineering Division, Argonne National Laboratory, Lemont, IL 60439, USA*
[c] *Belgian Nuclear Research Center (SCK•CEN), Boeretang 200, B-2400 Mol, Belgium*
[d] *Department of Materials Science and Engineering, Boise State University, Boise, ID 83715, USA*
[e] *Center for Advanced Energy Studies, Idaho Falls, ID 83401, USA*
[f] *Engineering Physics Department, University of Wisconsin, Madison, WI 53706, USA*
[g] *Idaho National Laboratory, Idaho Falls, ID 83415, USA*

ARTICLE INFO

Article history:
Received 28 August 2014
Received in revised form
27 May 2015
Accepted 5 August 2015
Available online 8 August 2015

Keywords:
Thermal aging
Duplex stainless steel
Neutron irradiation
Atom probe tomography
Delta ferrite

ABSTRACT

To enable the life extension of Light Water Reactors (LWRs) beyond 60 years, it is critical to gain adequate knowledge for making conclusive predictions to assure the integrity of duplex stainless steel reactor components, e.g. primary pressure boundary and reactor vessel internal. Microstructural changes in the ferrite of thermally aged, neutron irradiated only, and neutron irradiated after being thermally aged cast austenitic stainless steels (CASS) were investigated using atom probe tomography. The thermal aging was performed at 400 °C for 10,000 h and the irradiation was conducted in the Halden reactor at ~315 °C to 0.08 dpa (5.6×10^{19} n/cm^2, $E > 1$ MeV). Low dose neutron irradiation at a dose rate of 5×10^{-9} dpa/s was found to induce spinodal decomposition in the ferrite of as-cast microstructure, and further to enhance the spinodal decomposition in the thermally aged cast alloys. Regarding the G-phase precipitates, the neutron irradiation dramatically increases the precipitate size, and alters the composition of the precipitates with increased, Mn, Ni, Si and Mo and reduced Fe and Cr contents. The results have shown that low dose neutron irradiation can further accelerate the degradation of ferrite in a duplex stainless steel at the LWR relevant condition.

1. Introduction

To extend the service lifetime of Light Water Reactors (LWRs) beyond 60 years, the integrity of reactor components and the performance of structural materials must be evaluated adequately. The accurate assessment and prediction of materials performance under anticipated operation conditions are of particular importance for ensuring the safe operation of nuclear power plants over their extended lifetime. Both the cast austenitic stainless steels (CASS) and welds of austenitic stainless steel in LWRs consist of a duplex phase structure that contains austenite and delta ferrite phases. While the CASS alloys are mainly used in LWRs primary pressure boundary components, such as, coolant pipes, elbows, valves bodies, and pump casings, the welds for austenitic stainless steels can be found throughout the primary pressure boundary and reactor vessel internal components. The common cast stainless steels in service include the CF-3 and CF-8 series of alloys with a ferrite content up to 30%, and for the welds, e.g. 308 type, ferrite contents are limited to less than 20%. The ferrite phase improves the tensile strength, castability, weldability and stress-corrosion cracking resistance. In general, duplex stainless steel components perform properly with relatively few degradation modes, however, it has been long known that potentially significant embrittlement can be present due to the precipitation of fine second phases and spinodal decomposition of ferrite phase upon extended long-term thermal aging at temperatures of 280–350 °C [1,2].

Earlier studies on CASS by Chung and Chopra [3–5] concluded that the spinodal decomposition into Fe-rich α and Cr-rich α' phases and nucleation and growth of G, γ_2, and $M_{23}C_6$ carbide phases are the metallurgical processes contributing to the thermal-

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[*] Corresponding author.
E-mail address: yongyang@ufl.edu (Y. Yang).

http://dx.doi.org/10.1016/j.jnucmat.2015.08.006
0022-3115/Published by Elsevier B.V.

aging embrittlement of the ferrite phase. However, the primary embrittlement mechanism of aged CASS is the formation of Cr-rich α' phase. The effects of thermal aging on mechanical properties include increases in tensile strength and hardness but decreases in ductility, fracture toughness and impact strength [6–8]. Austenitic stainless steel welds share a very similar embrittlement mechanism with CASS upon thermal aging [9–12]. As an example, studies on thermally aged, unirradiated 316 SS welds consistently showed that the spinodal decomposition is the primary aging mechanism of the duplex weld materials [13]. Long-term thermal aging resulted in changes in fracture toughness that initially increased, then decreased with increasing aging time.

In addition to thermal aging, it is well known that the irradiation-enhanced diffusion can induce or accelerate phase transformations and hardening in stainless steels. Although CASS materials are not typically subjected to high neutron fluence, some CASS components, such as core support columns and mixing devices, may reach fluence level on the order of 10^{20} n/cm^2 ($E > 1$ *MeV*). Austenitic stainless steel welds used as PWR internals (e.g. core barrel welds and flux thimble tube plugs) can, as noted in the ERPI technical reports [14,15], be located in the high fluence regions with anticipated neutron fluence on the order of 10^{22} n/cm^2. Although the thermal aging induced embrittlement mechanism in duplex stainless steels has been well studied and understood, the irradiation effects and the potential combined effects of thermal aging and neutron embrittlement are yet to be fully investigated. There have been several studies on the weld overlay cladding of reactor pressure vessels [16], F–32Cr% model alloy [17] and ion-irradiated CF8M duplex stainless steel [18], but the results are inconclusive and even contradictory. Therefore, more systematic studies are needed.

In this paper, microstructural comparisons of ferrite in a cast stainless steel CF-3 are made among the conditions of as-cast, thermally aged, neutron irradiated, and neutron irradiated after thermal aging. It is aimed to elucidate whether the irradiation will further exaggerate the thermal aging degradation.

2. Experiments

The chemical composition of cast stainless steel CF-3 used in this study is listed in Table 1. The material contains 24% volumetric fraction of ferrite. Some of the as-cast samples were aged at 400 °C for 10,000 h prior irradiation, and the neutron irradiation on the un-aged and aged samples was conducted in the Halden reactor at 315 °C to 0.08 dpa (5.6×10^{19} *n/cm*^{2}, $E > 1$ *MeV with a dose rate of* 5×10^{-9}*dpa/s*). The optical image of Fig. 1 shows a general grain and phase structure of the as-cast stainless steel. No obvious change of this structure was identified after thermal aging or neutron irradiation.

In this paper, the characterizations were solely focused on the spinodal decomposition and nano-sized G-phase precipitation in ferrite using atom probe tomography (APT) technique. The APT probe specimens were fabricated using a focused ion beam (FEI 3D Quanta FEG FIB), and the samples were finished using ion beam imaging for 1 min at beam energy of 2 kV and current of 27 pA to ensure a minimal Ga implantation and radiation damage from ion beam. For each condition, a minimum of nine tips was fabricated while averagely four specimens tips were fractured before a good size of dataset was achieved. The 3-D reconstruction and analysis were performed using the Interactive VisuAlization Software (IVAS 3.6.8). The 3-D reconstructions were conducted by following the standard procedure of the Recon Wizard in the software, and SEM tip images were used for defining tip profiles. The evaporation field value was 33.0 V/nm and k factor of 3.30 was used. Both the FIB and APT equipment used for this study are located at the Microscopy and Characterization Suite (MaCS), Center for Advanced Energy Studies (CAES) in Idaho Falls, ID.

Table 1
Chemical composition (wt%) of the cast stainless steel CF3.

Ni	Si	P	S	Mn	C	N	Cr	Mo	Fe
8.59	1.13	0.015	0.005	0.63	0.023	0.028	20.18	0.34	Bal.

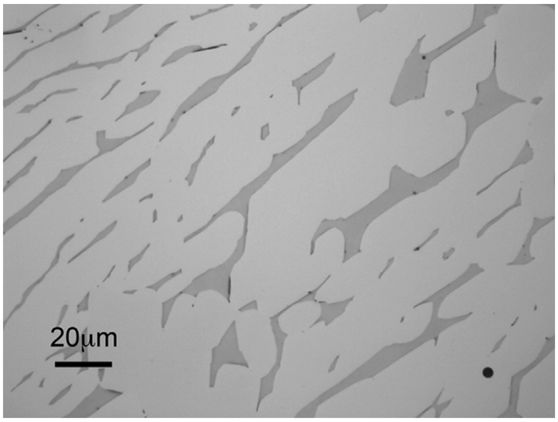

Fig. 1. Optical image of the as-cast CF-3 stainless steel etched using a Viella's agent for 30 s. The dark areas represent ferrite phase, and the grey areas are the austenite matrix.

3. Results and discussion

Overall, the delta ferrites in this cast stainless steel show a good chemical homogeneity. Table 2 lists the APT measured chemical compositions of the ferrites at four different conditions, and the standard deviations (sd) are calculated over all the measured specimens. For the major alloying elements, Fe, Cr, Ni, Mn and Si, the concentrations show a great consistence, and even for the elements of P, S and C with very low concentrations, the measured values vary insignificantly by considering a relative large measurement error for the minor alloying elements.

3.1. Spinodal decomposition in ferrite

Fig. 2 shows a direct comparison of Cr atom maps in the as-cast, aged, irradiated, and aged and irradiated ferrite phases. The atomic maps were constructed from a slice of 5 nm in thickness at the Y-middle plane of each APT tip. The atom maps clearly illustrate that both thermal aging and irradiation can individually induce spinodal decomposition in ferrite, while the neutron irradiation after thermal aging shows an exaggerated effect by further increasing the extent of Fe–Cr separation.

As shown in Fig. 3, to quantify the spinodal decomposition, the Cr–Fe concentration distributions are plotted by using the function of “Create Frequency Distribution Analysis” in IVAS software. The atoms in solute clusters were excluded from the analysis, and the bin size of ions is 100. For comparison, theoretical normal distributions of Cr and Fe were also calculated based on the measured average concentrations in the examined APT specimens. Apparently, the as-cast ferrite has nearly perfect normal distributions of Cr and Fe, and the profile peaks are located at 25.24 and 65.88 at.%, respectively. This is consistent with the Cr atom map shown in Fig. 2 that there is no spinodal decomposition occurring in the as-cast ferrite. Coincidently, the 10,000 h annealing at 400 °C produced a nearly identical Cr–Fe concentration profile with that of