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Irradiation resistance of a nanostructured 316 austenitic stainless steel

P B Revathy Rajan[1], I Monnet[2], E Hug[3], A Etienne[1], N Enikeev[4], C Keller[1], X Sauvage[1], R Valiev[4] and B Radiguet[1]
[1] Groupe de Physique des Matériaux, UMR CNRS 6634, Université et INSA de Rouen, Saint Etienne
du Rouvray Cedex, France
[2] CIMAP-ENSICAEN-CEA-CNRS-Université de Caen, Caen, France
[3] Laboratoire de Cristallographie et Sciences des Matériaux, ENSICAEN, Université de Caen, CNRS,
Caen, France
[4] Institute of Physics of Advanced Materials, Ufa, Russian Federation
E-mail: auriane.etienne@univ-rouen.fr

Abstract. The reduction of grain size down to several tens or hundreds of nanometers leads to the enhancement of radiation resistance of metals. Based on this approach, the aim of the Labex EMC3 (Energy Materials and Clean Combustion Center) project "Naninox" is (1) to study the stability of the microstructure of a nanostructured 316 stainless steel under ion irradiation and (2) to link between this microstructure and the properties (corrosion resistance and the microhardness) of the steel (thanks to a better irradiation resistance, a better corrosion resistance and higher mechanical properties after irradiation are expected in the ultra-fine grained stainless steel). Ultra-fine grained 316L austenitic stainless steel samples have been produced by high pressure torsion (HPT) at 430˚C and then ion irradiated in Jannus facilities (CEA Saclay) at 450°C and 5 displacements per atoms (dpa). Their microstructure is characterized before and after irradiation by atom probe tomography, X-ray diffraction and transmission electron microscopy. Corrosion behavior in NaCl solution is tested and nano-indentation tests are performed. The first results obtained by atom probe tomography described in this paper indicate that the microstructure of ultrafine grain 316 austenitic stainless steel is more stable under irradiation than the microstructure of a coarse grain 316 austenitic stainless steel.

Keywords: High pressure torsion, austenitic steel, irradiation resistance, corrosion resistance, radiation-induced segregation

1. Introduction

Austenitic 316L type stainless steel is used as baffle bolts to join the baffle plates and former frames in the pressurized water reactor (PWR) internal structures. These bolts undergo severe environmental conditions of stress, temperature and irradiation during the service. During periodic maintenance of the reactor internal structures, it is occasionally observed that some of these bolts have developed cracks due to the irradiation assisted stress corrosion cracking (IASCC). This degradation process is assisted by the radiation induced segregation or depletion of solutes at various defect structures including grain boundaries, dislocations, etc... In addition to the segregation of solutes, the formation of crystal defects (dislocation loops) due to the agglomeration of point defect produced by irradiation hardens the material and plays a role in the initiation and propagation of IASCC.

Grain boundaries are effective sinks for annihilating the point defects generated during the neutron irradiation. Since these point defects are at the origin of the evolution of the microstructure, the reduction of grain size is a promising method to limit radiation damage, as evidenced in literature [1-3]. Indeed, reducing the grain size provides more sinks for the point defects and the mean free path for the point defects to reach any grain boundary will be shorter. Also as the total grain boundary surface area increases, there will be less number of point defects annihilating per unit area of the grain boundary.

Historically severe plastic deformation techniques were considered as effective ways of producing ultrafine grained microstructure by imposing very high strains on the work piece. High pressure torsion is one of the techniques which can be designed such that the work piece dimension does not change significantly after processing. Ultra fine grained (UFG) samples for this study were made by HPT technique in Ufa state aviation laboratory, Russia. The UFG samples were annealed to remove excessive defect structures produced during deformation. The annealed UFG samples were irradiated at JANNUS facility of CEA, Saclay, France. The irradiated and non-irradiated samples were characterized by atom probe tomography. The details of experiments and results were discussed in the subsequent sections.

2. Experimental methods

316L Samples with UFG microstructure were developed using the severe plastic deformation technique High Pressure Torsion (HPT). The samples were disks of 10mm in diameter and approximately 1mm in thickness. The samples were loaded in compression with 6GPa and for 10 turns. They were maintained at 430˚C during the deformation. After deformation they were maintained at 450°C during 5hours under vacuum in order to make sure that the effects observed after irradiation (performed at 450°C) are only due to the irradiation and not to temperature. Irradiation experiments were performed in the Jannus facility of CEA, Saclay. $^{56}Fe^{5+}$ ions with energy of 10MeV bombarding on the sample surface with an angle of incidence of 15° were used. The samples were irradiated for a total average fluence of 1.4×10^{16} ions/cm^2. With the SRIM [4] simulation, using the "full damage calculation", the average dose in the first micrometer below the surface is estimated to 5 displacements per atoms (dpa).

Irradiations were performed with 10MeV Fe ions. Even if the primary damage productions from neutron and ion irradiations are not exactly the same, making difficult the direct transposition of the results. The use of heavy ions allows to identify and understand the effect displacement damages on the microstructure with the advantages of reaching high doses in short time without any activation of the samples. Heavy ions, such as Fe ions produce relatively large displacements cascades [5], that are more representative of the ones produced by fast neutrons, compared to light ions irradiations. The dose rates between ion and neutron irradiations are also very different (respectively 10^{-4} and 10^{-8} dpa/s). Several microstuctural studies [6, 7] indicated that the effect of the dose rate on the microstructure evolution can be compensated by increasing the irradiation temperature (roughly 40-50°C per decade of dpa/s). Here the ion irradiations were performed at 450°C to limit dose rate effect in comparison to neutron irradiation at about 300°C (light water reactor operating temperature).

Transmission electron microscopy (TEM) was performed on JEOL 2010F, to estimate the grain size of non irradiated samples.

Atom probe tomography (APT) analysis was carried out in Cameca FlexTAP with Field evaporation pulses provided by femtosecond UV laser. A laser energy giving an equivalent pulse fraction of 20% of the standing voltage was used, with the samples maintained at 50K during the data acquisition. The samples for APT were prepared by microloop electropolishing technique for non-irradiated samples. For the irradiated samples, focused ion beam milling and micromanipulator lift out techniques were used in Zeiss NVision40, as the uniform damage layer is approx 1µm in depth.

It has to be noticed that APT and TEM samples were prepared in disk areas where Vickers microhardness tests performed before irradiation reveal an uniform hardness and thus a uniform microstructure.

3. Results and discussion

3.1. Annealed samples

TEM dark field images were used to estimate the grain size. Since the deformed grains were observed to be elongated possibly towards the tangential direction of the disc (at the direction of material flow due to rotation), the calculation of grain size is done from the area of each grain. The diameter given here is a spherical equivalent diameter. It is equal to about 85nm.

From the atom probe tomography studies of the annealed sample, the grain boundaries were found to be enriched with Cr, Si, P, C and Mo. Cr levels at various grain boundaries range from 23 to 30%. One such APT reconstruction is shown in figure 1a, which shows the level of grain boundary enrichment of these elements. Grain boundary enrichment of Cr was also observed in literature with annealing [8]. No other features are observed by APT in this material.

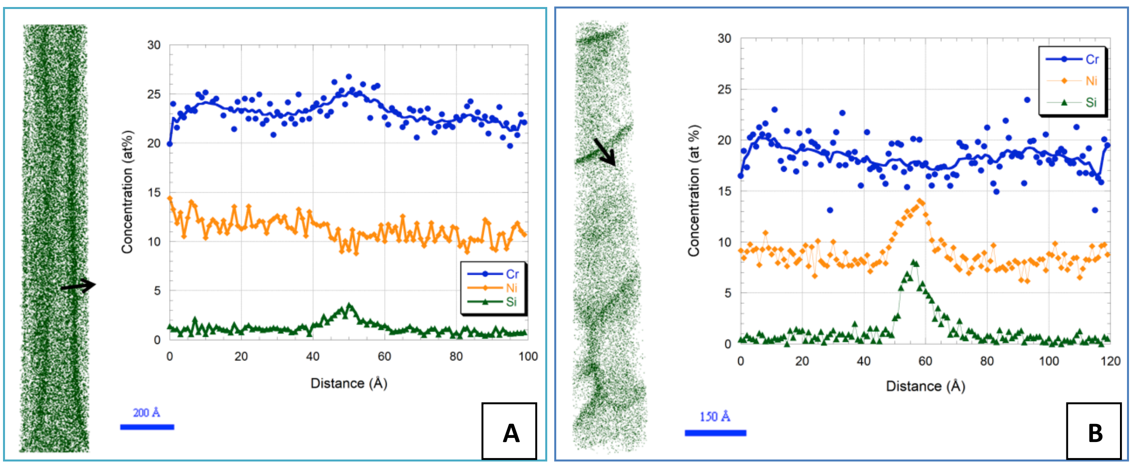

Figure 1 Reconstructed analyzed volumes of (a) annealed UFG sample and (b) irradiated UFG sample showing distribution of Si. Line profiles of elemental distribution across grain boundaries as indicated for Si, Cr and Ni.

3.2. Effect of irradiation

An example of APT analyzed volume from UFG annealed irradiated sample is shown in figure 1b. It shows the grain boundary segregation of solutes, but no segregation inside the grains. Such observations were obtained on all the irradiated samples.

After irradiation, APT analysis showed that the grain boundaries were enriched with Ni and Si. Cr was found to be depleted in some of the grain boundaries and there were numbers of grain boundaries which showed similar levels to that of the grain interiors. This is probably a result of the different enrichment levels of Cr after annealing. In conventional coarse grained samples, the level of Cr at grain boundaries was about 14 at.% for the same level of damage [9]. Compared to this value, the level of Cr observed in grain boundaries of the irradiated sample in the present study (ranges from 16% to 20%) is higher. Apart from the segregation at grain boundaries, there were no other segregations in the grain interiors. This is most possibly the effect of nanostructuration, as the CG material irradiated in similar conditions (5 dpa, 450°C) were exhibiting solute segregated loops and clusters in the grain interiors [10].

4. Conclusion

An UFG 316 austenitic stainless steel was elaborated by HPT followed by annealing at 450°C. It was irradiated by Fe ions at 450°C up to 5 dpa. Contrary to what is usually observed in coarse grain austenitic stainless steels, the microstructure of UFG 316 steel does not exhibit any radiation induced intragranular segregation. The only effect of irradiation is to decrease the Cr level at some of the grain boundaries, but in smaller extend than in coarse grain steel. These results are good evidence for the improved potential of the 316L material in the UFG state.

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References

[1] Radiguet B, Etienne A, Pareige P, Sauvage X and Valiev R, 2008, Irradiation behavior of nanostructured 316 austenitic stainless steel *Journal of Materials Science* **43** 7338-43
[2] Nita N, Schaeublin R and Victoria M, 2004, Impact of irradiation on the microstructure of nanocrystalline materials *J. Nucl. Mater.* **329–333, Part B** 953-7
[3] Samaras M, Derlet P M, Van Swygenhoven H and Victoria M, 2002, Computer simulation of displacement cascades in nanocrystalline Ni *Phys. Rev. Lett.* **88**
[4] Ziegler J F, Ziegler M D and Biersack J P, 2010, SRIM – The stopping and range of ions in matter *Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms* **268** 1818-23
[5] Was G S, Fundamentals of Radiation Materials Science Metals and Alloys, Springer Eds, 2007
[6] Was G S, Allen T, 1999, *J. Nucl. Mater.* **205** 332-6

[7] Borodin O V, Bryk V V, Kalchenko A S, Parkhomenko A A, Shilyaev B A, Tolstolutskaya G D, Voyevodin V N, 2009, J. Nucl. Mater. **385** 325-3

[8] Cole J I, Allen T R, Was G S, Dropek R B and Kenik E A 2002 The influence of Pre-irradiation heat treatments on thermal non-equilibrium and radiation-induced segregation behaviour in model austenitic stainless steel alloys. In: *21st symposium on the effect of radiation on materials,* ed M L Grossbeck*, et al.*: ASTM International) p 540

[9] Pareige P, Etienne A and Radiguet B 2009 Experimental atomic scale investigation of irradiation effects in CW 316SS and UFG-CW 316SS *J. Nucl. Mater.* **389** 259-64

[10] Volgin A 2012 Characterization and understanding of ion irradiation effect on the microstructure of austenitic stainless steel. In: *Group of Physics of Materials,* (Rouen: University of Rouen) p 245