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Microstructure and microhardness of CLAM steel irradiated up to 20.8 dpa in STIP-V

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ABSTRACT

Specimens of China low activation martensitic (CLAM) steel were irradiated in the fifth experiment of SINQ target irradiation program (STIP-V) up to 20.8 dpa/1564 appm He. Microhardness measurements and transmission electron microscope (TEM) observations have been performed to investigate irradiation induced hardening effects. The results of CLAM steel specimens show similar trend in microhardness and microstructure changes with irradiation dose, compared to F82H/Optimax-A steels irradiated in STIP-I/II. Defects and helium bubbles were observed in all specimens, even at a very low dose of 5.4 dpa. For defects and bubbles, the mean size and number density increased with increasing irradiation dose to 13 dpa, and then the mean size increased and number density decreased with the increasing irradiation dose to 20.8 dpa.

1. Introduction

In future fusion reactors, structural materials will undergo high displacement damage and transmutation helium/hydrogen production by an intense flux of high energy neutrons. As candidate structural materials, various reduced activation ferritic/martensitic (RAFM) steels [1,2] have been widely irradiated in spallation neutron source facilities to investigate irradiation effects including high transmutation. The China low activation martensitic (CLAM) steel has been selected as the candidate structure material for the Chinese Test Blanket Module (TBM) for ITER [3] and the liquid blanket of the China Fusion Engineering Test Reactor (CFETR) [2]. It exhibited equivalent mechanical properties compared to other RAFM steels, such as Eurofer97, Mod-F82H, JLF-1 and Optifer, in unirradiated conditions [4,5]. There have been several irradiation experiments carried out for the CLAM irradiated in the SINQ (Swiss Spallation Neutron Source) target irradiation program (STIP-V/VI/VII) to investigate its irradiation effects [6]. Tensile specimens of the CLAM steel irradiated up to 20.5 dpa (displacement per atom) were tested to investigate irradiation induced hardening and embrittlement effects [7]. In this work, we investigated microhardness and microstructure of the CLAM steel specimens irradiated in STIP-V at 5.4–20.8 dpa and 248–1564 appm He in a temperature range of 56–328 °C. And the effects of defects and helium bubbles on hardening were analyzed for CLAM.

2. Experimental

The steel used in this study was HEAT 0408B of the CLAM steel with main chemical compositions of 8.91Cr-1.44W-0.2V-0.15Ta-0.49Mn-0.11Si-0.12C. It was manufactured from a 20 kg ingot, hot-forged and rolled into a 12 mm thickness plate. The plate was normalized at 980 °C for 30 min followed by air-cooling, then tempered at 760 °C for 1.5 h and air-cooled. Miniature type of tensile specimens with 0.4 mm thickness were irradiated in STIP-V. The grip sections of three tensile specimens were used for hardness and TEM investigations.

Irradiation parameters of the grip sections of tensile specimens are given in Table 1, including calculated dose, helium concentration and irradiation temperature (T_{irr}). More detailed information about these tensile specimens irradiated in the STIP-V was reported in Ref. [7]. It should be noted that the temperature presented is averaged values and the variation is about ±10% during irradiation

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Vickers hardness (HV0.2) was measured with a 0.2 kg force and 15 s full-loading time and five measurements were conducted on each grip section. After grinding the grip sections from 0.4 mm to ~0.2 mm, two discs with a 0.8 mm diameter were punched out from each grip section. The discs were embedded into 3 mm diameter discs with holes of 0.8 mm diameter at the center. These 3 mm diameter discs were twin-jet electro-polished for transmission electron microscope (TEM) investigations. The TEM observations were performed in a JEOL 2010 type microscope operating at 200 kV. Micrographs of weak beam dark field (WBDF) (g, 5g), g = 110 were used to quantify the size and density of defect clusters and dislocation loops for all the specimens and micrographs taken in two beam bright field (TBBF) (g = 110) and under-focus conditions were used for quantifying the size and density of helium bubbles. Thicknesses of the observation areas were deduced from the number of thickness fringes in micrographs of WBDF (g, 5g), g = 110.

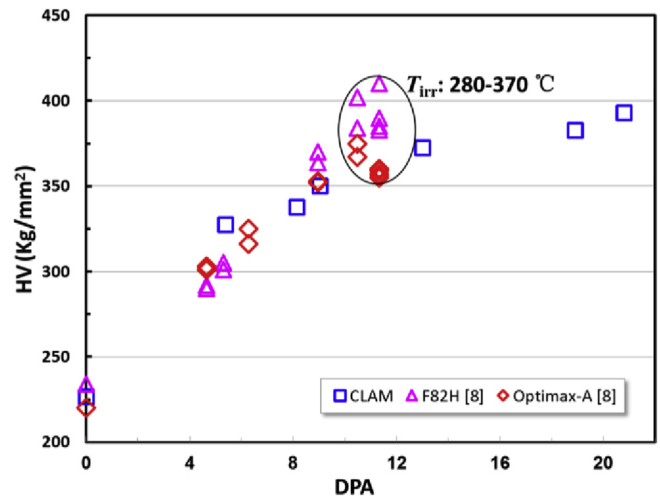

Fig. 1. Microhardness versus irradiation dose of CLAM and other RAFM steels [8] irradiated in STIPs.

3. Results and discussion

3.1. Microhardness

The mean microhardness values (HV0.2) measured from grip sections of the tensile specimens after performing tensile tests are presented in Fig. 1 and Table 1, which could be correlated to the tensile strength [7]. It is shown that the microhardness of specimens increases with increasing irradiation dose. At doses lower than 10 dpa, the trend of hardening is similar to the previous results of F82H and Optimax-A steels irradiated to 4.6–9 dpa in a temperature range of 115–260 °C in STIP-I [8]. The hardening seems to reach a saturation level at doses higher than 12 dpa. This is probably due to irradiation temperature which shows a stronger impact on microstructure and hardening at irradiation temperatures above ~250 °C, compared to irradiation dose [9,10].

3.2. Defect cluster and loop structure

Microstructures of the grip sections of the tensile specimens after performing tensile tests were observed, so that the microstructural data could be directly correlated to the microhardness data. Fig. 2 shows WBDF images of defect clusters and dislocation loops in the CLAM steel specimens irradiated to 5.4–20.8 dpa. At doses lower than ~13 dpa, a large number of small defect clusters were visible. Whereas at higher doses, fewer small defect clusters and more large dislocation loops were visible; few defect could be observed in the specimen irradiated to 20.8 dpa. This could be attributed to high irradiation temperature effect on the microstructure. It should be noted that the very small white/black dots homogeneously distributed in the specimens irradiated to 18.9 dpa and 20.8 dpa are helium bubbles, rather than defect clusters. The results agree with previous observations on the F82H/Optimax-A irradiated in STIP-I/STIP-II at similar doses lower than 20 dpa [8,9].

Fig. 3 shows TBBF images of bubbles in the CLAM steel irradiated to 5.4–20.8 dpa. The interesting finding is that bubbles were visible in specimens irradiated at low doses, such as 5.4 dpa/248 appm He/56 °C. The previous work showed that no helium bubbles could be observed in specimens irradiated at ~5.8 dpa with ~350 appm He at ~150 °C in F82H/Optimax-A/T91 in STIP-I [8,10], at ~7.2 dpa with ~550 appm He at ~145 °C in F82H/Eurofer97 in STIP-II [9,11] and at ~7.4 dpa with ~500 appm He at ~130 °C in F82H/Eurofer97/T91 in STIP-III [12]. The possible reason for this disagreement could be the bad resolution of small bubbles and the temperature variation during irradiation at low doses. More investigations on helium bubble observation in the specimens irradiated to low doses at low temperatures are ongoing.

As shown in Table 1, specimens irradiated to higher doses also experienced higher temperatures. The quantitative results of defect clusters and loops are presented in Table 1 and plotted in Fig. 4, For the CLAM steel specimens irradiated up to ~13 dpa and 200 °C, the mean size and number density of defect clusters and loops increase with increasing irradiation dose. After irradiation at high doses >18 dpa and high temperatures >280 °C, the mean size shows a large increase, while the number density shows a large decrease. And few defect could be observed in the specimen irradiated to 20.8 dpa. The reason is probably due to the strong impact of increased temperature, above ~250 °C, on defect structure [9,10]. The CLAM steel results can be seen more clearly in the size distributions of irradiation defects in these irradiation conditions, which are plotted in Fig. 5 (left column). These are supposed to be similar to the results of the F82H steel irradiated to 5–20 dpa at 115–400 °C [8,9]. For the disagreement on defects in the specimens irradiated to ~20 dpa, the reason could be the disappearance of

Table 1
Irradiation conditions, microhardness and TEM measurements of grip sections of CLAM tensile specimens in STIP-V.

Specimen ID	Dose (dpa)	He (appm)	Tirr (°C)	HV0.2	Defect size (nm)	Defect density (10^{22} m^{-3})	Bubble size (nm)	Bubble density (10^{24} m^{-3})
5-ST-J10	–	–	–	227	–	–	–	–
5-ST-J01L	5.4	248	56	328	3.52	1.44	0.75	1.10
5-ST-J01H	8.1	469	99	338	–	–	–	–
5-ST-J04L	9.0	530	113	350	5.19	1.65	1.08	1.23
5-ST-J04H	13.0	811	178	373	5.35	2.25	1.10	1.69
5-ST-J13L	18.9	1362	287	383	13.50	0.35	1.79	0.63
5-ST-J13H	20.8	1564	328	393	–	–	1.65	0.72

5-ST-J**L and 5-ST-J**H mean grip sections at low and high doses of a tensile specimen. Specimen 5-ST-01H was not observed for microstructure.