Microstructure and tensile properties in reduced activation 8-9% Cr steels at fusion relevant He/dpa ratios, dpa rates and irradiation temperatures

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Abstract: 9 – 12%Cr steels are successful materials for the use at higher temperatures. The universal application in turbines and power plants promised an expedient material for fission and future fusion reactors. The advantages are sufficient high-temperature strength, corrosion resistance, good thermo-physical behavior, and fast decay of radioactive inventory. In addition to good irradiation behavior and other physical advantages, these hardenable Cr steels are attractive low-activation materials and have been optimized further towards long-term activation. Such reduced activation ferritic/martensitic (RAFM) 8-10%Cr-WTaV steels not only offer favorable radiological properties. A critical effect of low temperature irradiation-induced hardening and embrittlement occurs in the range of 250 – 350 °C. One reason was found in the irradiation induced microstructural changes in the material, especially the production of He by the $^{10}B(n,\alpha)^{7}Li$ – reaction. While in fission reactors without B or Ni doping the He to displacement damage ratio is typically 0.1 – 0.3 appm He/dpa it amounts to ~10 appm He/dpa in a first wall of a fusion reactor. Former investigations of irradiated steels and in a recent past results of accelerator He implantations have shown the importance of He effects on microstructure. By this background, test alloys with different ^{nat}B and ^{10}B were melted, neutron irradiated, tensile tested, and microstructural characterized to show the hardening effect by He.

1. Introduction

Advanced martensitic 8-10%Cr-WTaV steels are leading candidates for fusion structural components. They are interesting due to their good irradiation behavior as e.g. minimal swelling, and good different physical as mechanical properties [1 – 7]. After a long period of developing and optimizing work the fabrication of an industrial 3.5 tons batch of 8-10%Cr-WTaV-RAFM steel, called EUROFER97 had been produced. This material was irradiated together with further three test alloys with different B contents. Not just B-content in the test-alloys were different, furthermore a variation of ^{nat}B and ^{10}B. This program was started to investigate and determine B or rather He influences on material properties during and after irradiation. Most interesting temperatures were lower irradiation temperatures between 250 and 350 °C, where the highest strengthening is anticipated.

2. Experimental procedure

Material

Within the framework of the European Fusion Program, three alloys on the basis of the RAFM-steel EUROFER97 were melted in 25 kg-heats each with different ^{nat}B - and ^{10}B -contents and compared with EUROFER97 reference heat. The chemical composition is given table 1. The materials were heat treated at 1040 °C 0.5 h + 760 °C 1.5 h, and circular tensile specimens were fabricated with a gauge length of 18 mm and 3 mm diameter. The investigation program contained un-irradiated and irradiated specimens to compare their properties.

Table 1: Chemical composition of EUROFER97.

Alloying	C	Si	Mn	P	S	Cr	Mo	Ni	Al	Nb	Ti	V	W	N	Ta
Elements	0.12	0.04	0.48	0.005	0.004	8.91	<0.001	0.02	0.009	0.0017	0.001	0.2	1.08	0.02	0.14

Irradiation

A special wrapper, with irradiation capsules accommodating the tensile- and further impact and fatigue specimens, was inserted in the central part of the reactor core of HFR (High Flux Reactor, Petten, Netherlands) [8 – 10]. The irradiation was set between 250 and 450 °C. The thermal neutron irradiation was carried out in 771 FPD (full power days) up to 16.3 dpa. The achieved He concentration by $^{10}B(n,\alpha)^{7}Li$ generation is shown in table 2. After the irradiation the specimens were transported to Forschungszentrum Karlsruhe, Fusion Material Laboratories to carry out the mechanical tests.

Table 2: Alloyed boron-content and achieved helium-content of the test alloys.

Heat	EUROFER97	Alloy1	Alloy2	Alloy3
B content	<1 ppm ^{nat}B	82 ppm ^{nat}B	83 ppm ^{10}B	1160 ppm ^{10}B
He production	<10 appm	~80 appm	~415 appm	~5800 appm

3. Results and discussion

Tensile tests

EUROFER97, Alloy1, and Alloy2 had the highest strength at 300 °C. At higher irradiation and test temperatures up to 450 °C, the data reached the level of un-irradiated materials, but still with a reduction in elongation, due to the limitation of the thermal stability of interstitial type defects.

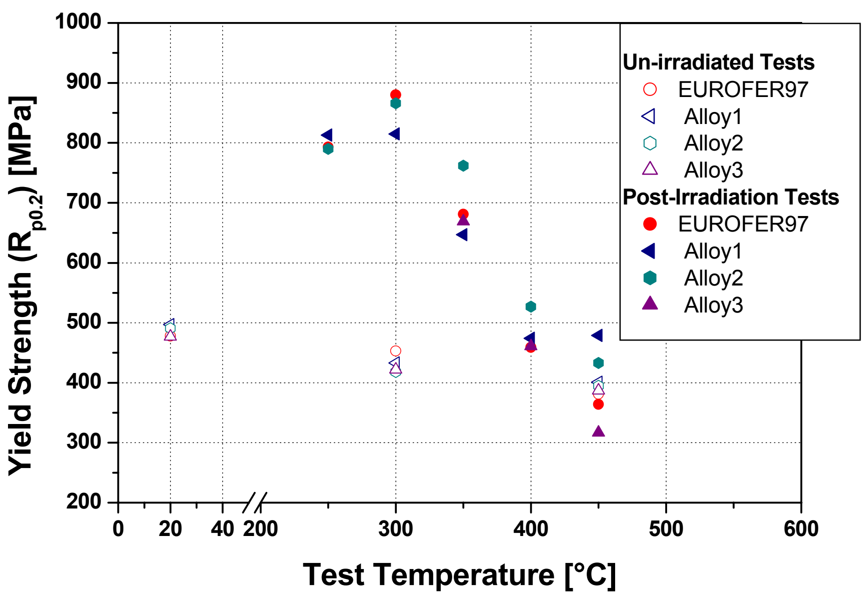

Fig.1: Yield strength before and after irradiation.

The strength of Alloy2 was systematically higher than of Alloy1. As in the ^{10}B doped alloy all B is transmuted to He after 1 dpa has reached. The additional induced strength increase of Alloy2 compared to Alloy1 can be interpreted as a direct proof, how He contributes to irradiation induced hardening at different irradiation temperatures, Figure 1. The yield strength values of EUROFER97 can only be compared in a relative manner to these test heats, because it has been produced as a large batch. Alloy3 with the extremely high ^{10}B induced He content of ~5800 appm had a similar strength behavior, but at lower temperatures, the specimens broke brittle. The higher concentration of He bubbles caused a ~10 % higher hardness resulting in a higher strength. He dependence could be seen in the comparison of Alloy1 and Alloy2; the ^{10}B containing Alloy2 had always higher strength than the ^{nat}B-containing Alloy1.

Fracture behavior

EUROFER97, Alloy1, and Alloy2 broke during tensile tests always ductile with typical dimple formations in the fractures. In Alloy1, small brittle areas could be found around former B-precipitates, which remained to "fish eyes" of H induced fractures. Our effect is irradiation induced as a "He eye", and could be observed much more in Alloy2; Fig. 2. The fractures of Alloy3 occurred much more brittle, mainly at grain boundaries; Fig. 3.

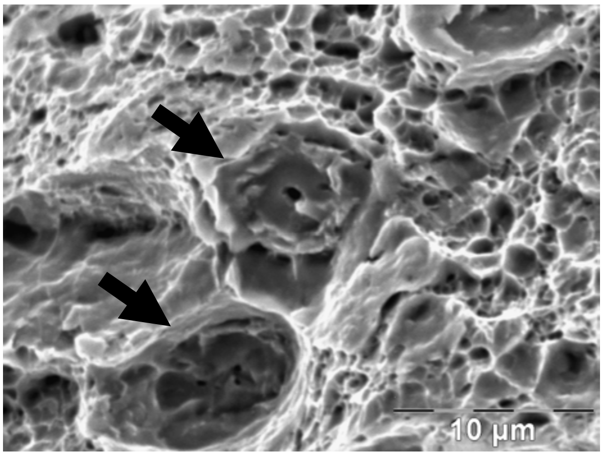

Fig.2: Fracture surface of Alloy2 after $T_{irrad.}$ And T_{Test}= 300°C with "He eyes", SEM picture.

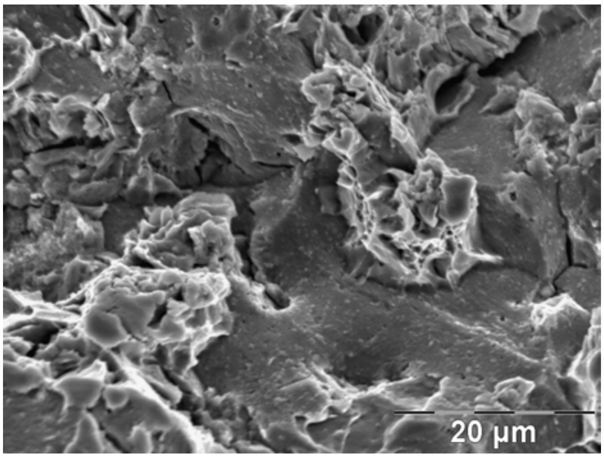

Fig.3: Fracture surface of Alloy3 after $T_{irrad.}$ and T_{Test}= 300°C, SEM picture.

Microstructure

Before irradiation, the distribution of the alloyed boron was controlled in the structure of the heats. In Alloy1 and Alloy2 some B inclusion could be analyzed. Much more B inclusions and further precipitates as (Cr,Fe)B and B(C,N) were found in Alloy3. In heat EUROFER97, just few inclusions of MnS and oxides were detected. Prior austenitic grain sizes of ASTM 6 - 7 were determined in the metallographic cuts.

Microstructures of the heats were investigated in TEM in the un-irradiated and irradiated state after 250, 300 and 450 °C test temperatures. The un-irradiated structure of these martensitic 8-10%Cr-WTaV steels is characterized by martensitic lath structures in between prior austenitic grains. The laths boundaries are decorated with carbides of the type $M_{23}C_6$. Only few primary carbides TaC are distributed homogeneously in the matrix. This structure does not change during the irradiation. At the lower irradiation temperature, the microstructure was characterized by dislocation loops and α`-precipitates. The dislocation loops of EUROFER97 were often oriented in a very pronounced manner as lines along to the gliding directions of the bcc crystal structure; Fig 4. At irradiation temperature of 450 °C, there was just recovered matrix; Fig.5. In EUROFER97 only few individual He bubbles were detected in the matrix, while in Alloy1 small clusters of He bubbles have been observed.

In contrast to EUROFER97 and Alloy1, an almost homogenous distribution of He bubbles was shown up in Alloy2. Alloy3 had the highest concentration of irradiation induced cavities. As expected, in all heats the He bubbles size increases with irradiation temperature.
One example of irradiation induced He bubbles in Alloy2 is shown in Fig. 6, this is the microstructure after the irradiation temperature of 250 °C. In comparison is shown Fig. 7, after irradiation at 450 °C. The observed He bubbles are fixed in dislocations.

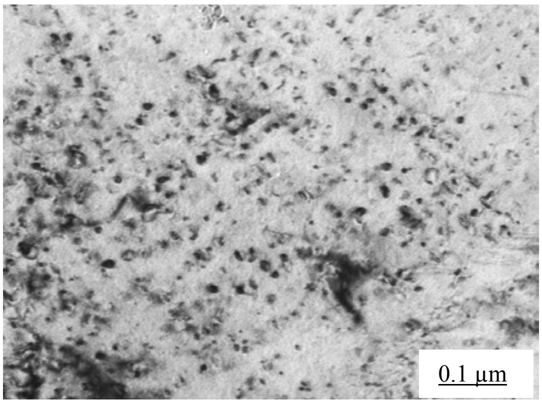

Fig.4: EUROFER97 after $T_{irrad.}$= 300° °C; TEM picture.

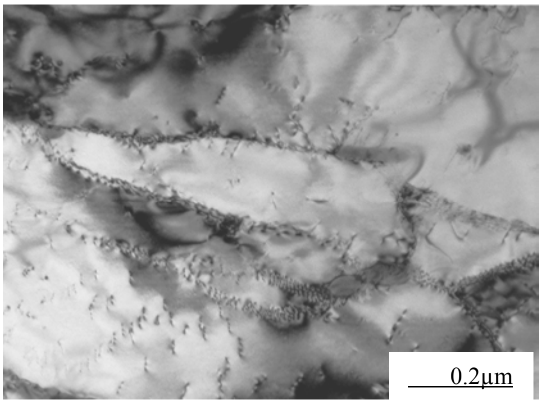

Fig.5: EUROFER97 after $T_{irrad.}$= 450° °C; TEM picture.

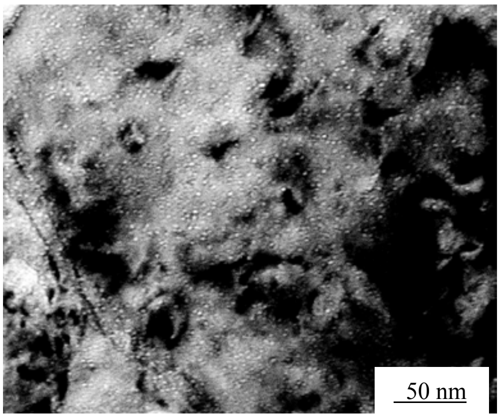

Fig.6: Alloy2 after $T_{irrad.}$= 250° °C; TEM picture.

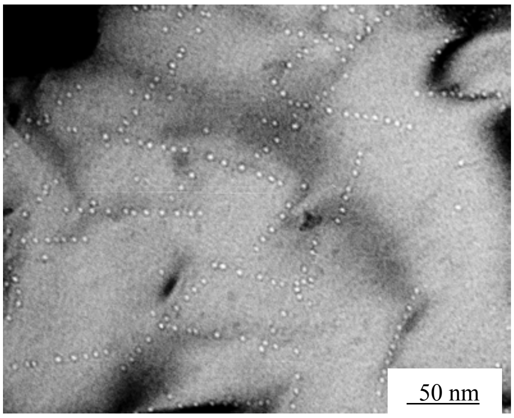

Fig.7: Alloy2 after $T_{irrad.}$= 450° °C; TEM picture.

4. Conclusion

- The expected strengthening at lower temperatures at 250 – 300 °C irradiation and test temperature were confirmed.
- The hardening and strengthening in the test alloys were generated by dislocation loops, α`-precipitates, and to a less extend be He.
- After tensile tests of EUROFER97 at 300 °C, very pronounced lines of dislocation loops have been observed with a typical line distance of ~20 nm.
- Alloy1 and Alloy2 with the same B contents, one with ^{nat}B and the other with ^{10}B alloyed, showed the clear difference irradiation induced strengthening due to the generated different He concentrations.
- The thermally induced recovery was observed by TEM in specimens of irradiation temperature > 400 °C. The hardening effect of dislocation loops and α`-precipitates was dissolved.

The significant implications of these results are obvious. Even at higher He levels of 400 appm, despite moderate uniform elongations, good levels of tensile ductility have been found in the entire temperature range of irradiation hardening (<400 °C). In addition, the results supported the hypothesis that at least up to ~400 appm He, doping with ^{10}B can be used to simulate He embrittlement effects, at least as long IFMIF with fusion relevant neutron spectra is not yet available.

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