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Tensile properties of ODS-14%Cr ferritic alloy irradiated in a spallation environment

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A B S T R A C T

The tensile properties of mechanically-alloyed oxide dispersion strengthened MA957 steel were measured at room temperature following irradiation in the SINQ spallation target up to almost 20 dpa corresponding to an accumulated helium content of about 1750 appm, with an average irradiation temperature range of 100–360 °C. In contrast to the behaviour of 9Cr–1Mo martensitic steel samples subjected to identical irradiation conditions and which were drastically embrittled at high dose, all tested MA957 specimens displayed a ductile fracture mode as shown by the measured values of uniform and total elongations and by the results of fracture surface examinations. This good mechanical behaviour is a new evidence that this type of material may be able to sustain high displacement damage and helium levels and is thus particularly well suited for fusion applications.

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1. Introduction

Oxide dispersion strengthened (ODS) ferritic alloys are being developed for fission applications, in particular as candidate materials for fuel cladding of Gen IV reactors [1]. They are also considered as blanket material for future fusion reactors [2]. They exhibit better high temperature strength and creep properties than conventional Ferritic/Martensitic steels while maintaining the attractive properties of this class of alloy such as a low thermal expansion coefficient, high thermal conductivity, and excellent void swelling resistance. Furthermore it was discovered that some experimental or industrially produced mechanically-alloyed steels such as the MA957 14%Cr ferritic steel, contain a high number density of Y–Ti–O nanoclusters (NCs) [3]. It is hoped that these NCs may prove highly efficient in trapping the high quantities of gas atoms, in particular helium, produced by transmutation reactions in fusion or spallation environments, which could provide a way of mitigating helium embrittlement. Indeed, preliminary transmission electron microscopy (TEM) studies of MA957 irradiated to 9 dpa and 380 appm He [4] indicate that the NCs probably trap the helium in ultra-fine bubbles. However, the impact of simultaneous irradiation and helium production on the mechanical properties of nanostructured ferritic alloys has not been investigated so far. The purpose of the present paper is to report results of tensile tests carried out on MA957 specimens irradiated in a spallation environment.

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2. Experimental details

The MA957 alloy, of nominal composition in weight% Fe–14Cr–1Ti–0.3Mo–0.25Y_2O_3, was manufactured by INCO. The as-received bar was subsequently hot-extruded at 1100 °C and 25% cold-worked. Miniature tensile specimens of 12 mm total length, 5 mm gauge length and 0.4 mm thickness were machined from the obtained 8 mm diameter bar, with specimen axis parallel to the extrusion direction. These specimens were placed in dedicated specimen holders which were enclosed in tubes and irradiated together with several other materials in the SINQ spallation target at PSI as part of the STIP II irradiation experiment. A detailed description of this experiment and of the procedures for the determination/calculation of proton and neutron fluxes, dpa, irradiation temperatures and gas contents can be found in [5–7]. It must be recalled however that the irradiation temperature was not constant during irradiation due to proton beam current fluctuations and beam trips. In addition a slight overfocussing of the proton beam occurred for 22 h resulting in a temperature excursion [5]. In order to reflect the complex irradiation temperature history of each specimen, the average temperature, the lower-bound and upper-bound temperatures during normal operation (i.e. excluding beam trips) and the highest temperature reached during the period of beam overfocussing were calculated. These temperatures are indicated in Table 1, together with values of dpa and gas contents. Reported helium contents are calculated values which were corrected based on measurements of helium concentrations in specimens irradiated in the STIP I and STIP II experiments [5,6,8], and should give a good indication of the actual helium contents in the irradiated specimens. In the case of hydrogen however, the calculated values

Table 1
Irradiation conditions and tensile properties.

Rod	ID mark	T_{av}[a] (°C)	T_h[a] (°C)	T_l[a] (°C)	T_{ex}[a] (°C)	dpa	He (appm)	H (appm)	YS (MPa)	UTS (MPa)	UE (%)	TE (%)
–	–	–	–	–	–	0	–	–	1051	1282	7.1	18.0
R1	Q8	108	115	99	129	6.5	500	1950	1112	1438	3.0	13.7
R1	Q7	122	131	111	148	6.5	500	1950	1319	1368	1.5	8.7
R3	Q9	135	149	120	174	7.2	535	2050	1344	1395	1.6	11.3
R1	Q6	150	162	135	185	9.8	795	3250	1281	1451	1.9	9.9
R1	Q5	173	188	155	216	9.8	795	3250	1494	1565	1.4	8.4
R3	Q12	166	185	147	219	10.7	850	3440	1347	1389	2.9	10.1
R1	Q4	210	229	287	266	14.1	1205	4900	1333	1466	1.8	12.0
R3	Q14	236	266	207	321	15.2	1305	5300	1459	1517	3.2	10.6
R1	Q1	326	357	286	419	18.6	1645	6900	1500	1531	1.1	6.9
R3	Q15	361	410	312	500	19.6	1740	7400	1279	1477	3.7	7.0

[a] T_{av} 'average' irradiation temperature defined as $T_{av} = (T_l + T_h)/2$.
T_h upper-bound irradiation temperature.
T_l lower-bound irradiation temperature.
T_{ex} maximum temperature during overfocussing period.

listed in Table 1 represent the total hydrogen quantities produced in the specimens. The retained hydrogen values are not known and it is expected that a significant amount of the produced hydrogen escapes via diffusion. For instance, measurements of hydrogen in ferritic/martensitic specimens irradiated in STIP I have shown that for specimens irradiated at temperatures higher than 250 °C, most of the irradiation-generated hydrogen diffuses out of the steel [8].

Following irradiation, tensile tests were carried out at room temperature with an initial strain rate of $3 \times 10^{-4}\,s^{-1}$. Fracture surfaces of selected tensile specimens were analysed by scanning electron microscopy (SEM).

3. Results

The tensile properties measured at room temperature before and after irradiation (Table 1) are shown in Fig. 1. Irradiation caused an increase in yield stress and ultimate tensile strength. However, and although the data are somewhat scattered, there was little change in the hardening as a function of dose. Of course it must be remembered that in this experiment irradiation temperature and dose in general increase in parallel. The evolution of the ductility reflected that of the strength: the total and uniform elongations were reduced by the irradiation but in the investigated dose range, no clear decrease of the ductility could be observed with increasing dose. Moreover, it must be emphasized that the material retained significant ductility at room temperature, since all irradiated specimens exhibited a uniform elongation over 1% and a total elongation above 8%.

In addition, fracture surface examinations were carried out on the unirradiated reference specimen and on four irradiated speci-

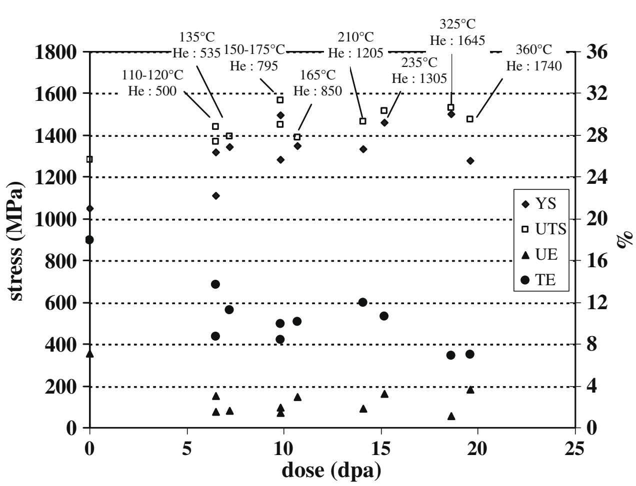

Fig. 1. Tensile properties (YS: 0.2% yield stress; UTE: ultimate tensile strength; UE: uniform elongation; TE: total elongation) as a function of dose measured at room temperature. For each set of data points, Helium content in appm and average irradiation temperature are indicated (in one case, two samples with identical doses and different temperatures were tested).

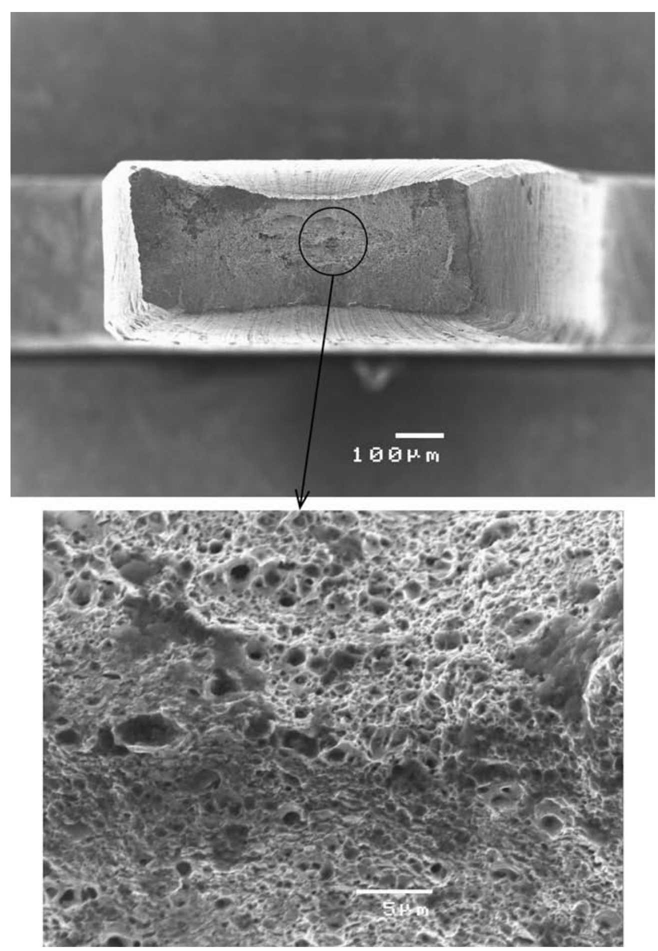

Fig. 2. SEM micrographs showing the fracture surface of the unirradiated reference specimen tested at room temperature.

mens (Q5, Q14, Q1, Q15). As shown in Fig. 2, the unirradiated specimen displayed significant necking at failure and the fracture surface appearance is clearly ductile with a dense array of round dimples. The size distribution of these dimples is large, some of them being well below 1 μm in diameter and have probably been initiated on small oxide particles.

The SEM investigations of the irradiated specimens revealed that their fracture mode was similar and some of the micrographs obtained in the case of specimen Q1 are shown as typical examples. Failure occurred in all cases with some necking, however the reduction of area was less than in the unirradiated case as can be seen in Figs. 2 and 3. The micrographs in Fig. 3 also show numerous cracks which were absent on the fracture surface of the unirradiated specimen. Many of these cracks correspond to 'steps' on the fracture surface. As Fig. 3 demonstrates, the side walls of these steps have a brittle 'fibrous-like' appearance. The main fracture surface perpendicular to the specimen axis has on the whole a ductile appearance. Some typical round dimples are present but the fracture surface also displays a dense array of small features which look like tiny cracks which extend perpendicular to the fracture surface and whose widths range from less than one μm to a few μm.

4. Discussion

The MA957 specimens investigated in the present study were irradiated in the STIPII experiment in order to evaluate the tensile behaviour of this material in a spallation environment, i.e. with simultaneous displacement damage and production of gases, in particular helium, by transmutation reactions. 9Cr tempered martensitic steels (9Cr1Mo EM10 and 9Cr1MoVNb T91 steels) were also irradiated side by side with the MA957 samples and experienced nearly identical irradiation conditions in terms of doses, irradiation temperatures and gas concentrations. These specimens were tensile tested and it was found that those irradiated to high doses (above approximately 16 dpa) displayed a total loss of ductility and broke in a brittle manner [7]. Moreover, the SEM observations revealed significant amounts of intergranular fracture. It was suggested that one of the causes of this drastic embrittlement and intergranular fracture mode was the accumulation of important helium contents in these specimens [7]. As already pointed out, some of the MA957 specimens were subjected to the same amount of displacement damage and gas generation and still retained a ductile behaviour as demonstrated by the tensile properties reported in Table 1 and the SEM investigations which showed that the samples displayed significant necking at failure. If indeed helium is a major cause of the brittle behaviour displayed by the 9Cr martensitic steel specimens irradiated in STIPII, then the present results may provide the first evidence that ODS materials such as MA957 are highly resistant to helium embrittlement, as anticipated in [4]. Of course the irradiated MA957 specimens suffered a reduction of ductility as compared to the unirradiated sample. Moreover there was some modification of the fracture mode: the 'fibrous-like' appearance of the side walls of the 'steps' formed on the fracture surface (Fig. 3) may reveal some kind of 'intergranular' separation. Indeed, following hot-extrusion and cold work, MA957 has small elongated grains approximately 0.5 μm in diameter and with a length/diameter ratio of 10–20 [9], which is consistent with the dimensions of the features seen on the SEM micrographs. Although this phenomenon is obviously promoted by the irradiation and/or helium accumulation since it is absent in the case of the unirradiated specimen, it must be mentioned that

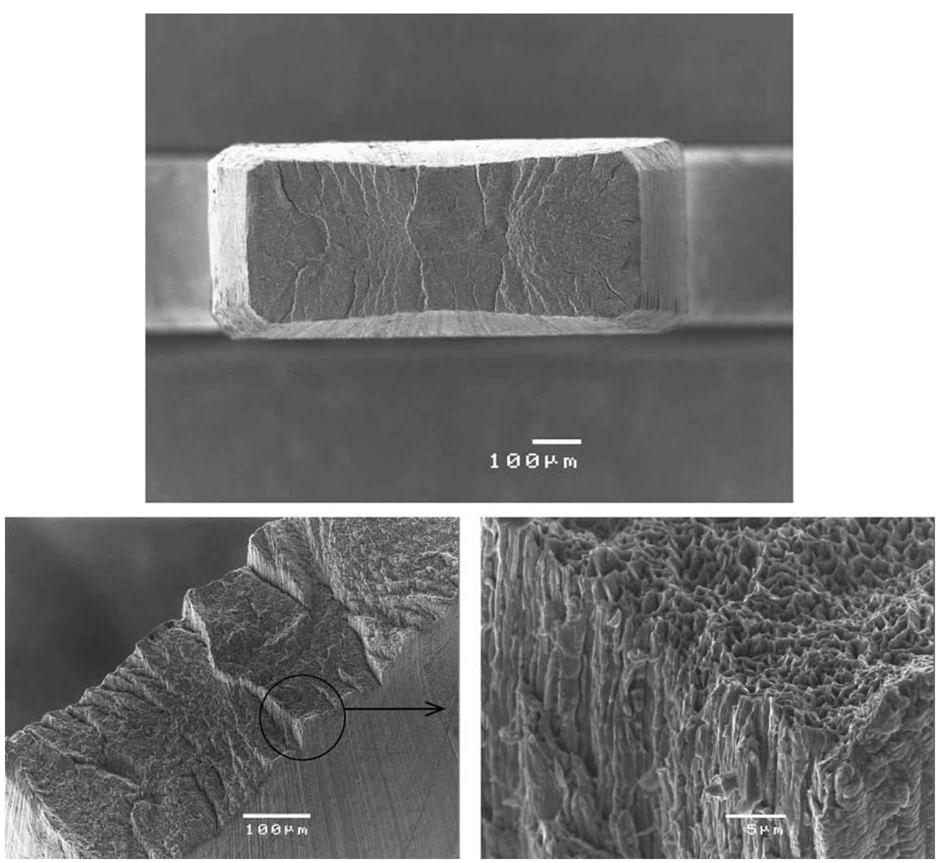

Fig. 3. SEM micrographs showing the fracture surface of specimen Q1 irradiated to 18.6 dpa and tested at room temperature.

a similar fracture appearance has already been observed for unirradiated MA 956 [11].

It is also worth pointing out that MA957 experienced a significantly lower irradiation-induced hardening than that displayed by the 9Cr martensitic steels irradiated in STIPII [7]. The same trend was observed when comparing the tensile properties of MA957 and various Cr martensitic steels irradiated at 300–325 °C [9,10]. This behaviour might be related to the high density of NCs which have been shown to be stable after irradiation [4,12] and which may act as recombination sites for point defects. Of course detailed investigations of the irradiation-induced microstructure would be needed to test this hypothesis and also to check whether helium was trapped by the NCs.

5. Conclusions

MA957 in the hot-extruded and cold-worked metallurgical conditions retained significant ductility after irradiation up to almost 20 dpa in a spallation environment. By contrast, 9Cr martensitic steel specimens irradiated in the same experiment displayed a fully brittle behaviour with an intergranular fracture mode. Although the effects of fission neutron irradiation on the tensile behaviour of several ODS steels has already been investigated [10,13], no results were available in the case where helium is produced together with displacement damage. The present results are the first evidence that ODS steels of MA957 type may retain suitable mechanical properties in fusion conditions and are consistent with recent microstructural data [4] suggesting that this type of material can be used to manage high levels of helium and displacement damage. Additional mechanical tests on specimens irradiated with high helium levels in different temperatures ranges are needed to confirm this point and detailed microstructural examinations should also be carried out to further investigate the role of the NCs as helium trapping sites.

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