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The tensile properties of AISI 316L and OPTIFER in various conditions irradiated in a spallation environment

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Abstract

Tensile specimens, prepared from AISI 316L austenitic stainless steel in three conditions (solution-annealed, cold-worked and electron-beam welded) and from OPTIFER martensitic stainless steel in tempered condition, were irradiated in the Swiss spallation neutron source (SINQ) at 90–400 °C to displacement doses from 3 dpa to 11 dpa. The mechanical properties were measured by tensile testing at room temperature and 250 °C, respectively, and subsequent metallographic analysis was employed. The tensile results indicated that the strength of AISI 316L-SA is quite similar or a little higher than in 316L-EBW but elongation of SA 316L is somewhat larger than EBW for both unirradiated and irradiated samples. The cold-worked specimens revealed much higher strength but almost zero strain-to-necking after irradiation. The results from OPTIFER samples showed that irradiation hardening increases with dose, which is accompanied by a dramatic reduction of uniform elongation beginning at very low dose. The metallographic analysis showed that the samples of AISI 316L-EBW failed in the welded zone.

1. Introduction

Depending on the particular liquid mercury target design, materials used for structural components such as the beam window and the beam entrance window of the mercury container vessel in a spallation neutron source must withstand a variety of severe loads, such as thermal stress, pressure waves and radiation dose, arising from the high power and pulsed proton beam injection [1–3]. In order to avoid boiling of either water or mercury, the temperatures during operation range from 100 °C to 300 °C which is somewhat lower than used in nuclear reactors. Moreover, because of the higher particle energies, the production of transmutation elements, especially hydrogen and helium will be orders of magnitude larger than those in fission and even in fusion reactors. On the other hand, as a complicated structure, the components have to be welded. It is therefore important to characterize not only the response of mechanical properties to the spallation environment for the base materials but also for the welded materials.

To study the radiation effects of structural materials in various conditions in a spallation environment, an international collaboration irradiation experiments (named as STIP) have been performed in the Swiss spallation neutron source (SINQ) at the Paul Scherrer Institut [4]. The main purpose of this materials R&D

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program is to contribute to the database for selecting the optimum materials for resistance against radiation damage. Austenitic stainless steel AISI 316L and modified 9Cr–1Mo martensitic steel T91 have been considered as candidate materials. In order to widen database, two other 9% Cr martensitic steels similar to T91 in chemical composition, such as EM10 developed in French, and OPTIFER, developed in Germany are also included in STIP for comparison. The element of Mo is replaced by W in OPTIFER steel. Some results on mechanical properties from STIP experiment have been published earlier for T91 [5] and EM10 [6].

In this paper we present the comparison of tensile properties of the austenitic stainless steel AISI 316L in different metallurgical conditions, i.e. solution-annealed (SA), 20% cold-worked (CW) and electron-beam welded (EBW) conditions, and the tensile properties of the martensitic stainless steel OPTIFER in the tempered conditions after SINQ irradiation. The results of AISI 316L are discussed and compared to data obtained by fission neutron irradiation. Meanwhile the results of OPTIFER are compared to its analogous steels of T91 and EM10.

2. Experimental

The chemical compositions of the investigated materials are given in Table 1. The solution-annealed AISI 316L [7] and OPTIFER were delivered by ISPRA and by SAARSCHMIEDE GmbH (heat no. 000735), respectively. Some AISI 316L materials were first cut into plates and then were cold-rolled to obtain the CW specimens. To prepare the EBW specimens, two 12 mm thick blocks of AISI 316L were welded together by the electron-beam technique [7].

Miniaturized tensile samples in the present study were cut by spark erosion and then mechanically polished. The sample outline-size was 12 mm long, 2.5 mm wide and 0.4 mm thick, with a gauge volume of $5 \times 1 \times 0.4$ mm^3. The welded specimens were cut in such a way that the welded zone of 2.4 mm width was in the middle of the 5 mm gauge length. A welded specimen was therefore composed of welded zone with base material on each side, as shown in Fig. 1. Fig. 2 shows a metallograph which reveals the different grain structures for the weldment and the base material.

The samples were irradiated in SINQ with a proton current of about 0.85 mA for the first 12 months and about 1.04 mA for the last 2 months, and received a total proton charge of 6.8 Ah. The details of irradiation information can be found in Ref. [4]. Briefly, according to the location, the displacement per atom (dpa) as well as the concentrations of transmutation atoms H and He were calculated for each specimen using the LAHET and MCNP-X codes and cross sections obtained from the accelerator product of tritium program. Furthermore the calculated He concentrations have been corrected based on the gas desorption measurements performed at the Pacific Northwest National Laboratory [8]. The irradiation temperature was also calculated

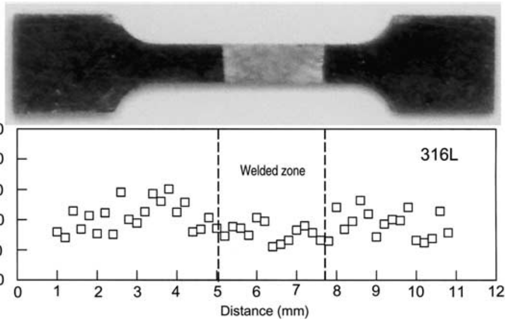

Fig. 1. Hardness distribution before irradiation along the EBW tensile specimen together with a photo showing the position of welded zone and the specimen shape.

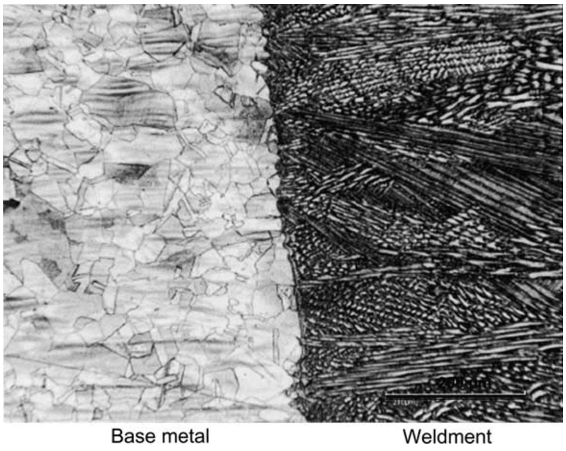

Fig. 2. Metallograph showing grain structure of the base material, welded zone and interface.

Table 1
Chemical compositions of materials (wt%)

	Ni	Cr	Mn	Cu	Mo	Co	W	Ta	C	Si	P	B	S	N	Fe
AISI 316L	12.24	17.17	1.75	0.07	2.31	0.077	–	0.002	0.019	0.35	0.02	0.0009	0.0007	0.073	Bal.
OPTIFER	0.06	9.48	0.55	–	0.002	–	0.985	0.065	0.125	0.04	0.0015	–	0.003	–	Bal.

Table 2
Irradiation conditions

Materials	ID	Rod	$T_{\text{irrad.}}$ (°C)[a]	dpa	He (appm)	H (appm)[b]	T_{test} (°C)
SA316L	A37	10	90/108	3	155	1156	RT
	A16	10	190/220	5.9	322	2368	RT
	A06	10	90/108	3	155	1156	250
	A43	10	190/220	5.9	322	2368	250
CW316L	B19	10	90/108	3	155	1156	RT
	B31	10	158/188	5	268	1978	RT
	B32	10	90/108	3	155	1156	250
	B02	10	190/220	5.9	322	2368	250
316L-EBW	C16	10	88/105	3	155	1156	RT
	C57	10	155/185	5.6	306	2265	RT
	C11	10	88/105	3	155	1156	250
	C18	10	155/185	5	268	1978	250
OPTIFER	M15	10	90/108	3	178	905	RT
	M27	1	215/255	7.6	568	3110	RT
	M08	1	320/380	11.4	1048	4800	RT
	M11	10	90/108	3	178	905	250
	M34	10	186/220	5.9	390	1955	250
	M09	1	278/330	9.8	818	4075	250

[a] The two temperatures for each sample correspond to the temperature in two irradiation periods of different proton beam currents at SINQ target [4].

[b] Calculated values. In fact, the measurements showed that the most of hydrogen diffused out of samples at ⩾250 °C [8].

for each specimen by means of ANSYS code which was also corrected based on the measured values at different positions in the target [4]. A summary of irradiation conditions is given in Table 2.

Tensile tests were conducted in air at a strain rate of about 10^{-3}/s at room temperature and 250 °C, respectively, using a 2 kN MTS tensile machine equipped with a video-extensometer. Subsequently, the metallographs were examined by optical microscopy to identify and characterize their failure regions.

3. Results and discussion

A comparison of typical stress–strain curves of AISI 316L in SA, CW and EBW conditions are shown in Fig. 3 for irradiation to doses of 0, 3 and 6 dpa, respectively. The dose dependence of yield stress and strain-to-necking (STN) is illustrated in Fig. 4. For unirradiated specimens, the EBW had similar strength compared to SA but the elongation was only half of the SA value. However, CW specimens manifested much higher strength and almost complete loss of ability to work-hardening. With increasing dose, SA and EBW specimens showed irradiation hardening accompanied by loss of work-harden. The effect of irradiation hardening was a little less for EBW. However a reasonable strain-to-necking remained for both SA and EBW at the highest dose available. The worst case was for the CW specimen which exhibited unstable plastic deformation at the very beginning of the tensile test after irradiation to the lowest available dose of 3 dpa, the STN dramatically dropping to less than 1%. A striking feature was that the

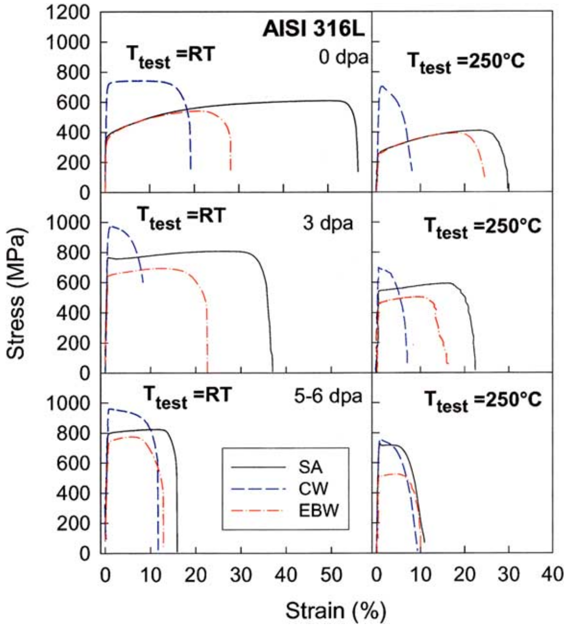

Fig. 3. Stress–strain curves of AISI 316L specimens tested at a strain rate of 10^{-3}/s at 25 °C (R) and 250 °C (L).

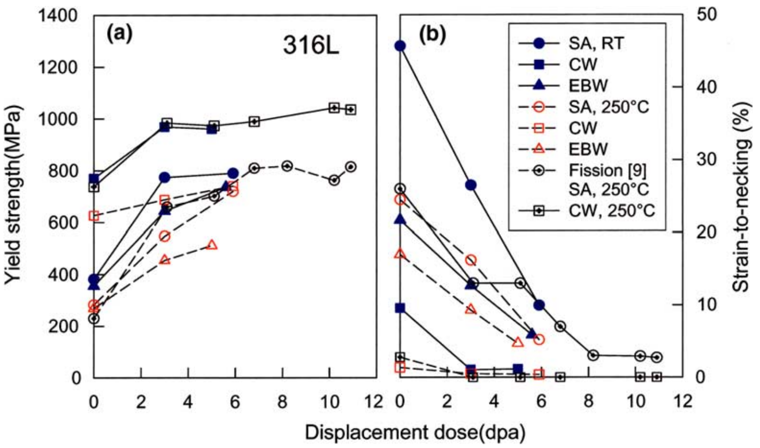

Fig. 4. Yield stresses (0.2% offset) and strain-to-necking are plotted as a function of displacement dose (dpa) for AISI 316L in solution-annealed, 20% cold-worked and electron-beam welded conditions. Filled and empty symbol indicate test temperatures of 25 °C and 250 °C, respectively. For comparison, data obtained by fission reactor irradiation [9] (T_{test} = $T_{irrad.}$ = 250 °C) are included.

difference of UTS between CW and SA specimens was saturated at 180 MPa at T_{test} = 25 °C. This is possibly connected to network dislocations produced during cold-work that contribute to the strength. Generally speaking, the trends of tensile response to irradiation for the test temperature of 250 °C were similar to that at 25 °C. Somewhat smaller values in strengths and elongations were observed for all three metallurgical conditions. Metallographical analysis demonstrated that welded specimens failed in the welded zone during tensile testing, as shown in Fig. 5. This is not surprising because the welded zone is a little softer than the base material, especially after irradiation. It is expected that the measured strain-to-necking of 316L-EBW underestimates the strain in the weld due to contributions of base material in the gauge length. To study the difference in changes of mechanical properties under spallation and fission environments, present results are compared to previous data obtained by fission reactor irradiation [9] as shown in Fig. 4. Besides of the fission data on the yield stress of CW samples tested at 250 °C which surprisingly lie on the room temperature line of our CW data, the other data indicate that the dependence of strength and STN on dose show similar behavior in both environments. Recently such comparison between spallation and fission irradiation for 316L with more complete data was given in Ref. [10]. The general trend is similar as we find hear. The STN value measured in spallation environment are somewhat lower than measured in fission environment at dose region above 5 dpa which may be attributed to a helium effect [11]. It is difficult to make a precise comparison since in individual experiments the temperatures of irradiation and test, which are quite sensitive to tensile properties, were different.

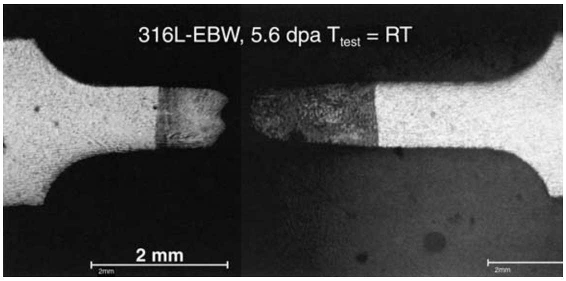

Fig. 5. Micrograph of irradiated AISI 316L (5.6 dpa) showing that the specimen failed in the welded zone.

The tensile properties of OPTIFER in tempered condition before and after irradiation are illustrated in Fig. 6 for test temperatures of 25 °C and 250 °C. The yield strength and uniform elongation as taken from the tensile curves are summarized in Fig. 7. For comparison the data for T91 [5] and EM10 [6] are also included.

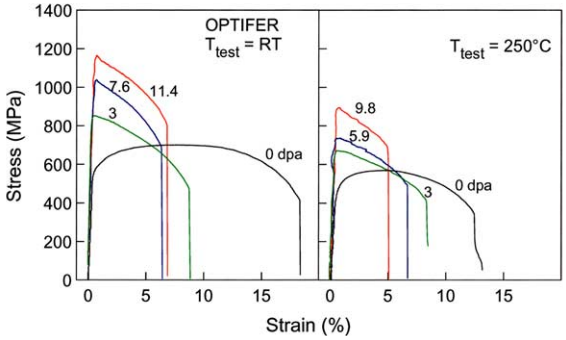

Fig. 6. Stress–strain curves of martensitic stainless steel OPTIFER irradiated to various doses at 25 °C (L) and 250 °C (R). The tensile tests were performed at a strain rate of 10^{-3}/s.

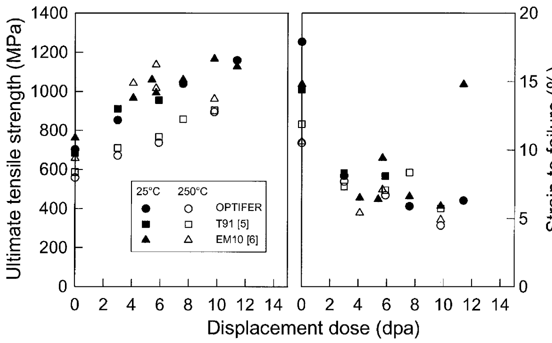

Fig. 7. Ultimate tensile strengths and strain-to-failure are plotted as a function of displacement dose (dpa). For comparison, the data for T91 [5] and EM10 [6] are also included. The circle, square, triangle-up are for OPTIFER, T91 and EM10, respectively. Filled and empty symbols indicate test temperatures of 25 °C and 250 °C, respectively.

In general, the three sets of data from T91, EM10 and this study agree very well. The common trends are a gradual increase of the strength and, following an initial drop, a slow decrease in the strain to failure with increasing dose. The UTS decreases with test temperature whereas the elongation was not sensitive to test temperature. The UTSs of EM10 at $T_{\text{test}} = 250$ °C were astonishingly somewhat higher than of the other two sets of data. The reason for this behavior is unclear.

4. Summary

Tensile tests were performed at 25 °C and 250 °C on AISI 316L in SA, CW and EBW conditions and OPTIFER in tempered condition irradiated up to 11 dpa at SINQ. The results can be summarized as following:

(1) Irradiation induces hardening and embrittlement in AISI 316L for all metallurgical conditions studied. SA and EBW specimens remained reasonably ductile up to 6 dpa, the highest dose available.
(2) The strength of the welded zone is somewhat lower than of the parent material for AISI 316L. This effect is more evident after irradiation.
(3) The measured strain-to-necking of 316L-EBW underestimates the strain of the weld due to contributions of base material in the gauge length.
(4) The 316L-EBW samples failed in the welded zone.
(5) EB-weldments of AISI 316L experience no additional problems arising from irradiation as compared to base materials.
(6) OPTIFER samples show practically the same behavior as T91 and EM10 steels, i.e. irradiation hardening accompanied with a severe reduction of strain-to-necking.

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