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Characterization of Eurofer-97 TIG-welded joints by FIMEC indentation tests

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Abstract

Welded joints of 25-mm thick plates of Eurofer-97, produced by a multi-pass GTAW + filler material method, have been investigated before and after different heat treatments to identify the temperature–time combination that gives mechanical properties as close as possible to those of the base material. After NDE (non-destructive examinations), samples were annealed in the temperature range 730–750 °C for 1 and 2 h, microhardness was measured across the joints and FIMEC (flat-top cylinder indenter for mechanical characterization) tests were carried out in the molten zone (MZ), heat affected zone (HAZ) and in the matrix. The FIMEC method allowed determination on a local scale of the yield stress as obtained in standard tensile tests. Mechanical tests were supplemented by optical microscopy observations. The best results were obtained by the treatment at 750 °C for 2 h: original characteristics are substantially recovered in the HAZ but not completely in the MZ.

1. Introduction

Even though some thought has been given to the use of alternative structural materials (V-, Ti- or Cr-alloys) for fusion reactors [1], it is unlikely that tritium blanket moduli (TBM) of ITER or DEMO could be fabricated of a material other than a reduced activation ferritic–martensitic steel (RAFM). The EU prime candidate for that family of alloys is Eurofer-97, for which at present a good data bank exists, especially aimed for design purposes [1,2].

A TBM is a huge, complicated component, impossible to build as monolithic structure. Joining will be largely used to fabricate that essential part of a magnetic fusion reactor (MFR). The RAFMs weldability is still a major problem. They appear insensitive to cold- and hot-cracking, at least for gas tungsten arc welding (GTAW), but, owing to their feature of self-hardening steels, even air-cooling promotes the martensitic transformation thus the result of a weld process is an unacceptable hard and brittle structure. Therefore, a Post Welding Heat Treatment (PWHT) constitutes a compulsory stage of the joining procedure. However, even if a large part of the material toughness may be recovered by a well-done PWHT, the HAZ remains the weakest region of the joint. Grain coarsening, precipitation and segregation are responsible for this weakness. Deleterious effects of the pre-existing precipitation and segregation are worsened by irradiation, so also these phenomena have to be cured by an 'optimised' PWHT.

The goal of our activities was to identify the temperature–time combination providing mechanical features of Eurofer-97 GTAW-joints as close as possible to those of the base material. In addition to standard techniques, such as microhardness tests and microscopical observation, the present investigation exploited the possibility of FIMEC (flat-top cylinder indenter for mechanical characterization) test to evaluate the yield

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stress on a local scale, i.e. directly measured in different zones of interest across the joints.

2. Material and experimental details

2.1. Characteristics of the base material

The product used for the butt-to-butt welded joints was a 25-mm thick plate of Eurofer-97 (Heat no. E83694). The chemical composition is: Cr 8.87, C 0.10, Si 0.05, Mn 0.45, P 0.005, S 0.004, Mo 0.0027, W 1.15, V 0.20, Ta 0.14, Ti 0.005, N 0.017, Ni 0.028, Cu 0.0035, Co 0.006, Al 0.008, Nb 0.0025, B < 0.001, O 0.0009, As < 0.005, Sn < 0.005, Zr < 0.005, Sb < 0.005, Fe to balance (wt%). The mechanical characteristics are summarised in Table 1 [3].

The steel was normalised at 980 °C (31 min) and tempered at 760 °C (90 min). No δ–ferrite is present and the average grain size is on the order of ASTM no. 9–10. A detailed microstructural characterization of Eurofer-97 is in [4,5].

2.2. Welding procedures and PWHT details

Welding was made by CEA Saclay laboratories using an automated GTAW method with a filler material (a 1-mm diameter Eurofer-97 wire); the work pieces have been 'V-groove' machined and welded along the plate rolling direction. A total of ten or eleven passes were performed per weld. A constant welding voltage of 10 V was used while current ranged from 150 to 250 A. Welding speed varied from 40 to 35 mm/min whereas the wire feed-rate reached a maximum of 1500 mm/min during the later runs. Welded joints have been protected by top and bottom shielding provided by flowing argon gas. Three 500-mm long joints were produced [6].

Soundness of joints was inspected by NDE (non-destructive examinations), X-rays and ultra-sound, and the structure was examined by microscopic observation. From each joint 10 mm-thick slices were cut transversely (Fig. 1), then they were post weld heat treated in argon at 730 and 750 °C, for soaking times of 1 and 2 h. Cooling was in still air.

2.3. Structural and mechanical characterisation

Each sample was mechanically polished, etched using Vilella reagent and observed by optical microscopy. The morphology and the extent of MZ and HAZ were analysed.

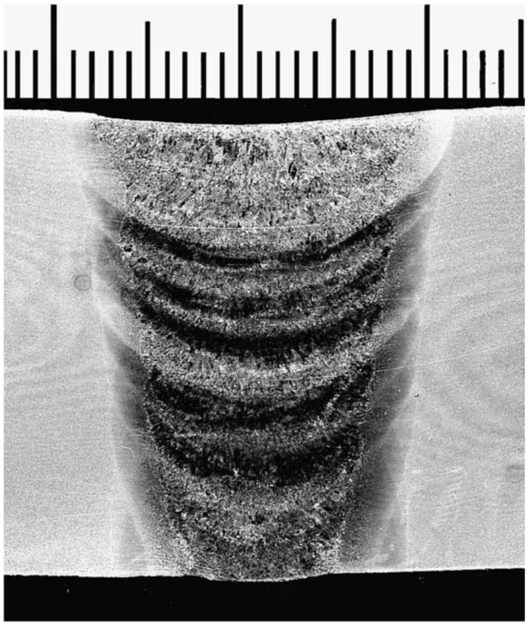

Fig. 1. Cross-section of a weld joint after mechanical polishing.

Before and after PWHT, the properties of all welded joints were studied by Vickers microhardness measurements (load = 0.1 kgf) performed in the same cross-section used for metallographic observations.

GTAW joints were also investigated by FIMEC tests. The FIMEC test, which has been developed by us, is based on the penetration, at constant rate, of a flat WC punch of small size ($\Phi = 1$ mm, $h = 1.5$ mm). During the test the applied load (q) and the penetration depth (δ) are measured, so load vs. penetration (LP) curves can be recorded. From LP curves it is possible to determine stress vs. penetration depth curves by dividing loads by the punch-surface contact area A ($A = \pi(\Phi/2)^2 \cong 0.785$ mm^2). The characteristics of these curves have been described in [7,8]: the limit value q_y is reached after an initial linear stage, which is followed by a stage of work hardening tending to saturation. Under standardised conditions (penetration rate ≅0.1 mm/min and deformation rate in tensile test ≅10^{-3} s^{-1}), $q_y \cong 3\sigma_y$, σ_y being the yield stress (0.2 YS). So, the local value of yield stress has been determined directly in the MZ and HAZ of the joints and in the matrix.

Table 1
Mechanical properties of Eurofer-97 at room temperature (from Ref. [3])

σ_y (MPa)	R_M (MPa)	A (%)	Z (%)	A_V (J)	HV_{30} (kg/mm^2)
550	673	24.8	73.9	223	218

σ_y: yield stress, R_M: ultimate tensile strength, A: total elongation, Z: reduction of area, A_V: impact absorbed energy, HV_{30}: Vickers microhardness.

3. Results and discussion

Joints were analysed by NDE: no flaws were detected by ultrasound but some defects were evident in X-ray examination. The kind of flaw can be identified as lack of penetration (more precisely, lack of filler material deposition) and small solidification cracks. Defects are quantitatively very limited and dispersed, separated from each other and their dimensions much less than those specified in ASME Boiler and Pressure Vessel Code, Section VIII, Division 2 or Section IX. Therefore, the joints can be classified as flawless, sound welds.

Fig. 2 shows the structures of MZ and HAZ in as-welded condition and after the PWHT of 2 h at 750 °C. Weld joints seem free from δ-ferrite islands, the structure is fully martensitic in MZ, although it appears not homogeneous being finer in some zones than in others. Since different portions of the material in the MZ are cooled with different speeds, a finer martensitic structure forms where the higher cooling rates are experienced. After the heat treatment, tempered laths are observed in MZ and a certain degree of non-homogeneity is still observed. The recovery in HAZ seems substantially completed.

In Fig. 3(a) and (b) the microhardness HV values measured across the joint are shown after 1 (a) and 2 (b) hours at 730 and at 750 °C. Data of the material in as-welded condition are also reported for comparison. Before PWHT strong hardness variations can be observed in the MZ: a maximum is reached at a distance of about 5–6 mm from the centre. The structural inhomogeneity is responsible for variations in hardness profile and large data scatter. In any case, in all the parts of the MZ the microhardness is always much higher than that of the matrix. The microhardness values of HAZ show a decreasing trend from a mean value of about 340 MPa near the MZ to the typical value of the matrix.

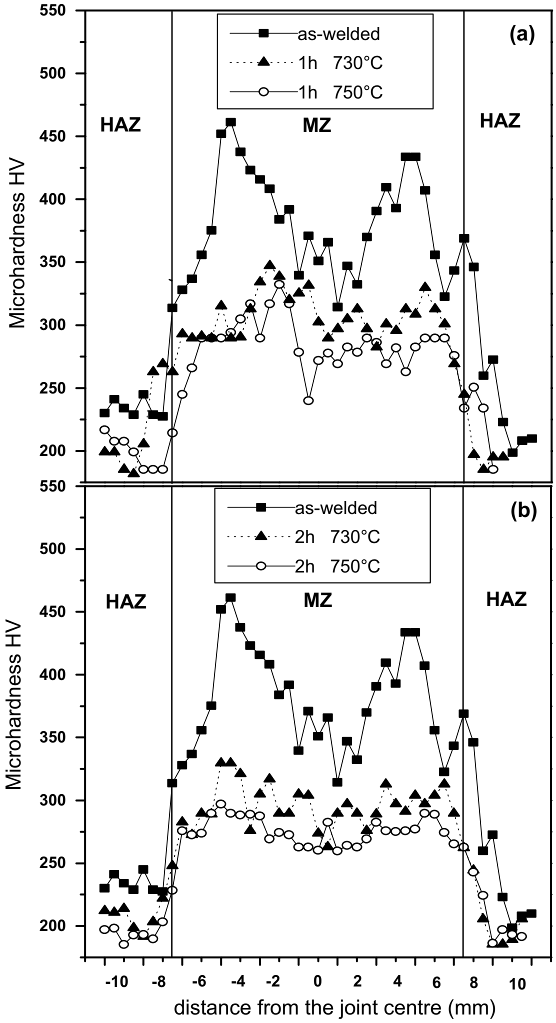

Fig. 3. Microhardness HV across the welded joint after 1 (a) and 2 (b) hours at 730 °C and at 750 °C. Data from the as-welded condition are reported for comparison in (a) and (b).

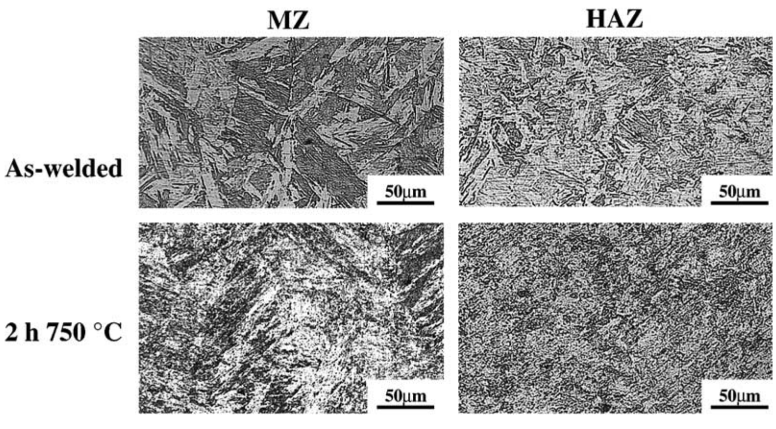

Fig. 2. Optical micrographs taken from the molten and the heat affected zones of samples in as-welded condition and after PWHT of 2 h at 750 °C.

After the 1 h treatment (Fig. 3(a)) a large scatter of data with hardness peaks exceeding 300 HV are still observed in MZ for both treatment temperatures. After 2 h (Fig. 3(b)) the treatment at 750 °C strongly reduces the scattering in MZ even though the mean hardness (~270 HV) remains higher than that of the matrix (~220 HV). In the HAZ the recovery of hardness is complete: its average value (~200 HV) is substantially the same of the base metal in the same sample. The treatment at 730 °C is less efficient: in MZ hardness values are still strongly scattered after 2 h and in some zones values around 330 HV have been measured.

Fig. 4(a) and (b) displays the FIMEC curves obtained in tests carried out in MZ, HAZ (the centres of imprints are at 5 and 10 mm respectively from the joint centre) and matrix of samples in the as-welded condition and after PWHT at 730 °C (a) and at 750 °C (b). Since the curves of the matrix show negligible variations after the different treatments only one is reported.

The $\sigma_y = q_y/3$ values obtained from the curves in Fig. 4 are reported in Table 2.

The yield stress of the matrix obtained from FIMEC (585 MPa) is a little higher than that reported in Table 1 (550 MPa) but the variation is within 7%, a scatter normally observed in performing a series of standard tensile tests [9].

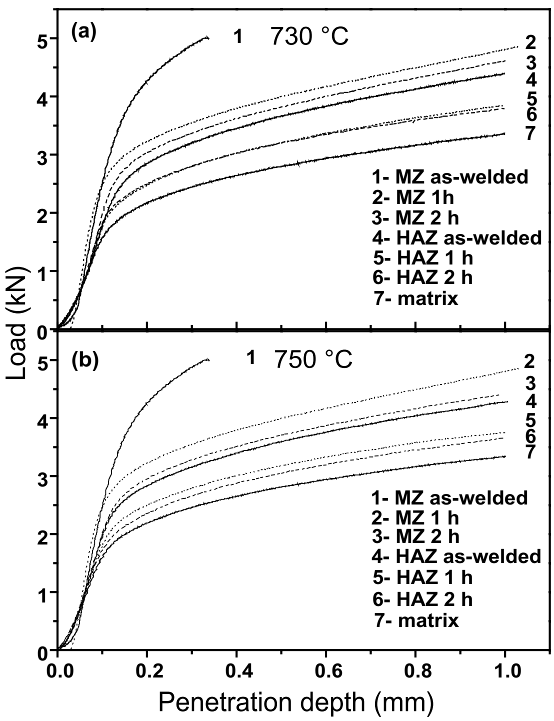

Fig. 4. FIMEC curves from matrix, MZ and HAZ of samples after PWHTs of 1 and 2 h at 730 °C (a) and 750 °C (b). For comparison the curves for the as-welded condition are shown.

Table 2
Yield stress σ_y determined from FIMEC tests (Fig. 4) in the molten zone (MZ), heat affected zone (HAZ) and matrix of the joints in as-welded condition and after the indicated PWHT

Investigated zone	Heat treatments	$\sigma_y = \frac{q_y}{3}$ (MPa)
MZ	As-welded	1240
HAZ	As-welded	812
MZ	1 h 730 °C	962
HAZ	1 h 730 °C	748
MZ	2 h 730 °C	876
HAZ	2 h 730 °C	710
MZ	1 h 750 °C	897
HAZ	1 h 750 °C	705
MZ	2 h 750 °C	855
HAZ	2 h 750 °C	664
Matrix		585

In the as-welded condition, the MZ exhibits a yield stress of 1240 MPa, typical of a fully martensitic structure, while σ_y measured in HAZ is 812 MPa, an intermediate value between those of MZ and matrix. These values are in agreement with microhardness trends (Fig. 3) and microscopical observations (Fig. 2). The different PWHTs reduce σ_y both in MZ and HAZ in different degree depending on treatment temperature and soaking time. As shown by microhardness tests, FIMEC also indicates that the best recovery of original properties is obtained after 2 h at 750 °C.

4. Conclusions

GTAW was employed to produce weld joints in 25 mm-thick plates of Eurofer-97.

The samples have been then submitted to PWHTs of 1 and 2 h at 730 and 750 °C to optimise the time-temperature combination for recovering the original mechanical properties. Before and after the heat treatments, the weld joints have been examined by optical microscopy, microhardness and FIMEC tests, which determined the local yield stress in MZ and HAZ.

Experiments indicate that the treatment of 2 h at 750 °C gives the best results: the original characteristics are substantially recovered in HAZ but only partially restored in MZ.

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