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Diffusion welding parameters and mechanical properties of martensitic chromium steels

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Abstract

To develop and qualify diffusion welding for fabrication of precise high stressed components of fusion reactors, a comprehensive test program was carried out using the martensitic chromium steels OPTIFER-IV and MANET-II. Metallographical and mechanical examinations served to determine the welding quality. The main result is that with a welding pressure of about 50 MPa and a surface roughness of few microns, welding could be produced with a strength and ductility in the range of the base material. Besides, sufficient fracture toughness was achieved when surface machining by wet grinding was replaced by dry milling. © 2000 Elsevier Science B.V. All rights reserved.

1. Introduction

Martensitic chromium steels are a potential structural material for high heat flux components like blankets and divertors of fusion power reactors [1]. The design and layout of these components is determined by geometrical constraints, e.g., the toroidal shape of a tokamak, and functional requirements, in particular high temperatures, static and transient mechanical loads and effects of radiation. These boundary conditions lead to designs which necessitate the application of special manufacturing processes which have to be developed or qualified for the envisaged type of structural material. Joining of parts by diffusion welding is one of the envisaged manufacturing techniques because it allows the manufacturing of complex components with high precision and small effects on material structure and properties. Hot isostatic pressing (HIP) is the mostly applied procedure in diffusion welding [2]. It allows the application of high pressure, which is important to achieve good mechanical properties of the joints. To prevent the access of the pressure gas, sealing and evacuation of the weld area is necessary, either by a thin-walled canister or by seal welding. For components with internal structures of small dimensions like first wall (FW) and cooling plates this is often not possible. In those cases sealing is realized by encapsulating only the outer surface of the component, or by applying the necessary pressure in a mechanical press under vacuum. In both cases the pressure conditions inside the component are no longer isostatic, in a mechanical press the load is even uniaxial. Under these load conditions, deformations are possible by plasticity and creep, and a careful trade-off between the desirable strength of the joint and the tolerable deformations is necessary. This explains the interest in exploring the minimum requirements on the welding pressure.

To develop and qualify the diffusion welding technology for the application described above, a comprehensive test program was carried out using the two martensitic chromium steels OPTIFER-IV and MANET-II developed within the European Fusion Technology Program [3]. The results of this effort, including a comparative discussion of the different mechanical testing methods, are presented in this paper.

2. Mechanical testing methods

To determine the material parameter best suited for the description of the weld quality, the results obtained

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from the tensile, bending and notch impact bending tests of diffusion welded specimens were plotted in force-deformation diagrams. For example, Fig. 1 shows the stress–strain diagrams measured in tensile tests of MANET-II specimens. All force-deformation or stress–strain curves of the welded specimens nearly correspond to those of the base material until they break off. The weld is considered to be of unacceptably poor quality when the yield limit is not reached. This allows total elongation to be used as the only measure of the quality of the weld. The scatter of the curves in Fig. 1 in the stress direction is due to variations in the base material properties, they are not related to the ductility. Parameters analogous to the total elongation in the tensile tests are the bending until cracking in the bending tests and bending until brittle fracture in the notch impact bending test. It was studied how these three parameters correlate and which tests are most suited for the present case. It was found that the characteristic parameters from tensile and bending tests are in good correlation; however, the tensile tests yielded the higher resolution for welding of low quality. Consequently, tensile tests are more suited to judge the strength and ductility of the welding, and bending tests are dispensable. The notch impact bending tests exhibited likewise a satisfactory correlation with the tensile tests, but only for welds with poor fracture toughness. All welding specimens with notch impact bending deformation above 15% of the base material exhibited the tensile properties of the base material. Evidently, the notch impact bending tests

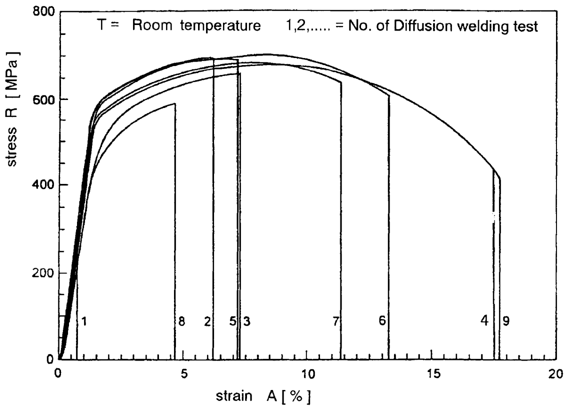

Fig. 1. Stress–strain diagrams from tensile tests of diffusion welded MANET-II specimens.

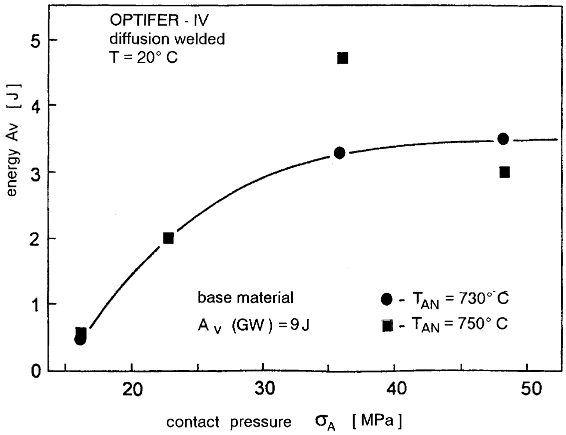

Fig. 2. Notch impact toughness of diffusion welded OPTIFER-IV specimens as a function of the contact pressure (T_{AN} = tempering temperature).

Table 1
Diffusion welding test parameters

Specimen Nos.	Surface preparation		Surface roughness R_t [µm]	HIP parameters		
	Machining	Cleaning		Welding temperature (°C)	Welding pressure (MPa)	Holding time (min)
1	Ground	Etched (KE-Stuttgart-procedure)	$\leqslant 2$	980	50	110
2	Ground	Cleaned with acetone	$\leqslant 2$	980	50	110
3	Ground	Cleaned with acetone	$\leqslant 2$	980	50	180
4	Ground	Cleaned with acetone	$\leqslant 2$	1050	50	180
7[a]	Ground	Cleaned with acetone	$\leqslant 2$	980	150	180
8	Ground	Cleaned with acetone	$\leqslant 2$	980	50	180
9	Dry milled	Cleaned with acetone	$\leqslant 2$	980	50	180
10	Dry milled	Cleaned with acetone	$\leqslant 2$	980	50	180
11	Dry milled	Cleaned with acetone	$\leqslant 12$	980	50	180
12	Dry milled	Cleaned with acetone	$\leqslant 12$	980	150	180
13	Dry milled	Cleaned with acetone	$\leqslant 2$	980	15	180

[a] Specimen Nos. 5 and 6 omitted because of seal welding failure.

simulate another type of loading, and the results must be taken into account when the component under consideration is exposed to related events, e.g., plasma disruptions.

3. Role of welding pressure

As outlined in the introduction, only rather low pressures can be applied when FW or cooling plates with internal cooling channels are diffusion welded under uniaxial pressure conditions. For a typical FW design and a welding temperature of about 1000°C, the welding pressure must be less than 50 MPa. To investigate whether under these conditions a satisfactory quality of the diffusion welded joint can be achieved, a test series was carried out with OPTIFER-IV steel specimens at welding pressures ranging between 16 and 48.5 MPa, welding temperature of 980°C, and holding time of 3 h. The welding surfaces were prepared by grinding to a roughness of 0.7–1.7 μm and subsequent etching. Tensile tests of samples taken from the welding specimens yielded in all cases strength and ductility values corresponding to the base material. Impact energy, however, increased with the welding pressure up to 36 MPa; higher pressures did not result in further improvement (see Fig. 2). The maximum impact energy corresponds to 30–50% of the base material.

4. Isostatic diffusion welding tests

4.1. Test program

To develop and qualify the diffusion welding procedure by HIP, a test program was carried out using the ferritic–martensitic chromium steel MANET-II. The specimens with a diameter of 100 mm and a height of 55 mm had a plane circular welding zone of 80 mm diameter. The vacuum needed in the welding zone was established by EB seal welding in a vacuum chamber. Only specimen No. 8 was evacuated after seal welding via a capillary tube. The seal weld was positioned in such a way that it did not affect the diffusion welding zone.

The test program comprised 11 welding specimens. The parameters are compiled in Table 1. HIP and subsequent heat treatment (750°C, 3 h) was done in the HIP 3000 facility of FZK/IMF. To study the quality of the welding, extensive non-destructive and destructive tests were carried out consisting of ultrasonic (US) inspection, metallographical and microprobe examinations, and mechanical testing. For the latter tests, standard miniaturized samples were cut from the welding specimens by spark erosion.

4.2. Test results

The US scans of all welding specimens indicated flawless bonding over the whole welding area irrespective of the deficiencies observed later on in the mechanical tests and metallographical examinations.

The results of the mechanical tests are compiled in Fig. 3 showing (from top to bottom) the yield and rupture strength, the elongation at rupture measured in the tensile tests, and the impact energy measured in the notch impact toughness tests. The dots indicate mean values from four tensile test samples and eight impact tests, respectively. Scattering of results – if significant – is indicated by vertical lines. The yield and rupture strength are, in all cases, in the range of the base material (typically 617 and 725 MPa, respectively). Like-

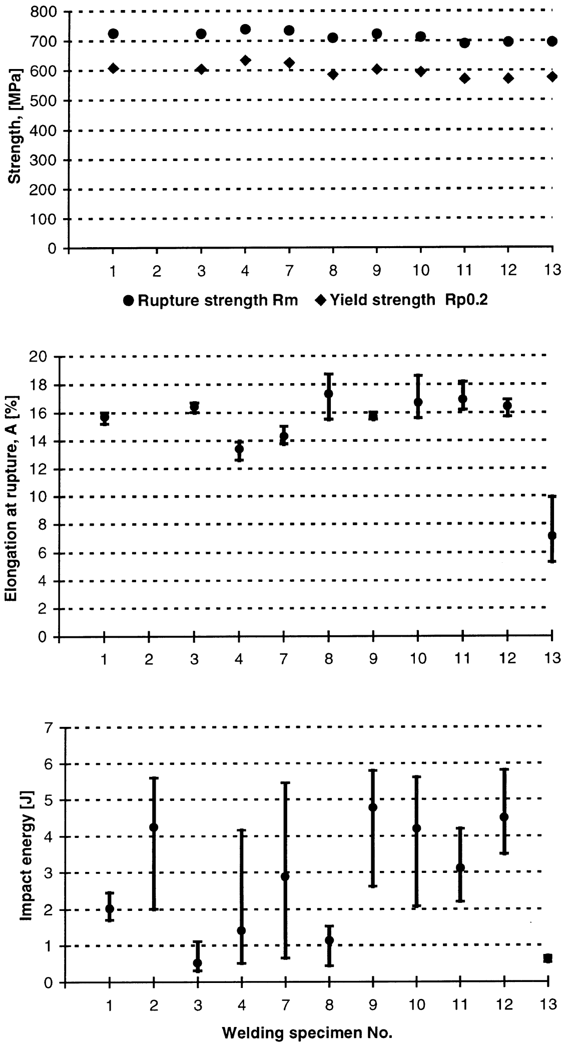

Fig. 3. Mechanical test results of diffusion welded MANET-II specimens.

wise the elongation at rupture corresponds to the base material value of about 15.4% with the exception of the last specimen, see below. In contrast, the energy measured in the notch impact bending tests scatters over a large range. In particular for the specimen Nos. 1–8, the results range between almost 0% and 100% of the base material value of 5.5 J. The average work for these tests is between 10% and 77% of the base material work.

In view of the poor quality and reproducibility of these initial welding tests, a search was started to identify possible causes. The metallographical examinations showed at high magnification (1000×) inclusions or pores in the bonding zone (see top of Fig. 4).

For a more detailed examination, the AUGER scanning electron microscope was applied which allows the identification of such small inclusions. Microsamples with 2×2 mm^2 cross-section were cut by spark erosion and split in the joining zone with a cleavage tool under high vacuum in the recipient of the microscope. The examination revealed different contaminations of the joint area as Si, K, Cl, O and N. With the aid of a Xe ion beam these contaminations could be eroded layer by layer. By this way the contamination thickness was determined to be about 400 nm. Supposedly, these contaminations provoked the diffusion of alloying elements (Cr, Mn, etc.) into the bond region where they affected the quality of the bond in a negative way.

To eliminate possible sources of the contaminations, the surface machining by wet grinding was replaced for the subsequent specimens (No. 9 and following) by dry milling. This modification led to a significant improvement of the fracture toughness. For illustration, the impact work of specimen Nos. 9 and 10 (welded with identical parameters) is plotted in Fig. 5 over the specimen cross-section. The impact work is now between 2.1 and 5.9 J. Eliminating the two lowest values which are located at the border of the bond area and may be affected by the EB seal weld, leads to a range between 3.5 and 5.9 J which corresponds to 65% and 107% of the base material. The results of the mechanical tests are confirmed by the metallographical examination: in the section of one of these two specimens shown in the lower part of Fig. 4 the bond line is practically no longer visible. Meanwhile, the improvement of the quality of

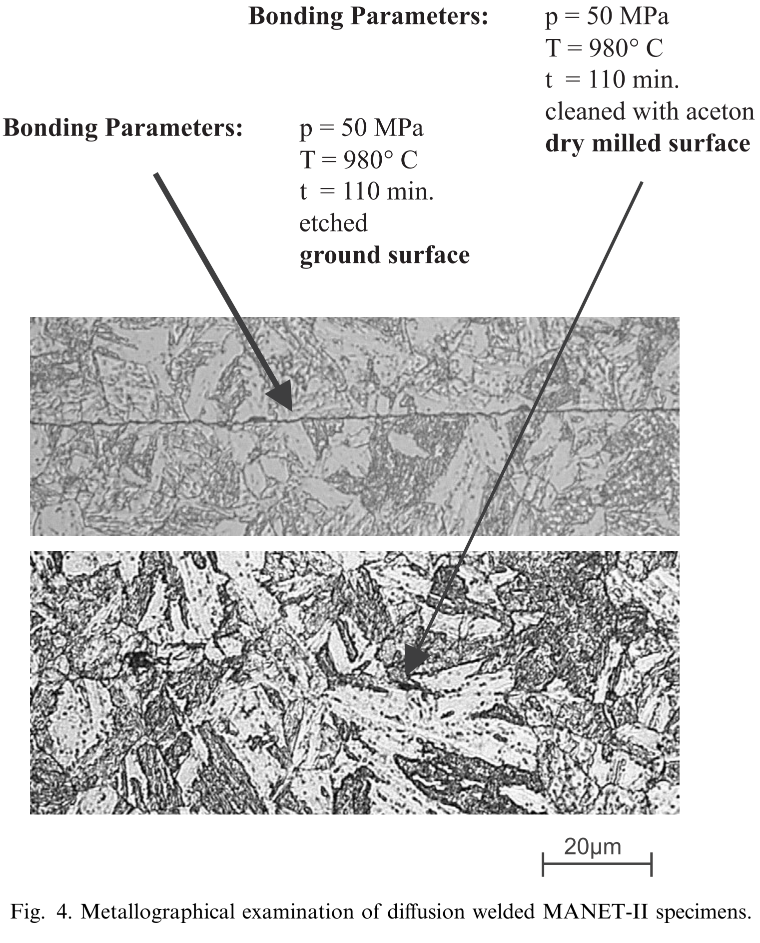

Fig. 4. Metallographical examination of diffusion welded MANET-II specimens.

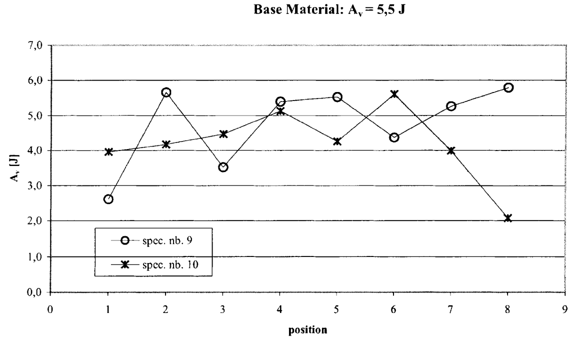

Fig. 5. Notch bar impact energy of diffusion welded specimen Nos. 9 and 10.

diffusion weldings by switching to dry milling was confirmed by investigations carried out at ESPOO/Finland [4].

In the last three tests (specimen Nos. 11, 12 and 13) the welding pressure and the surface roughness were varied with the other parameters being identical to specimen Nos. 9 and 10. Specimen No. 11 with a larger surface roughness shows a loss in impact work as compared to Nos. 9 and 10. This loss can be compensated for by increasing the welding pressure to 150 MPa (specimen 12). The reduction of the welding pressure to 15 MPa (specimen 13) caused a significant reduction of the ductility and a drop of the impact work to 10% of the base material. This effect is in agreement with the result obtained for OPTIFER-IV (see Section 3).

5. Conclusions

The main conclusions from the diffusion welding tests on the martensitic chromium steels OPTIFER-IV and MANET-II can be summarized as follows:

1. Tensile and notch impact toughness bending tests are sufficient to qualify diffusion welding. Bending test reproduce the tensile tests results, however, with lower sensitivity; hence, they are dispensable.
2. Ultrasonic testing is able to detect complete lack of bonding, but is not suited to determine the quality of welding.
3. At a temperature of 980°C and a surface roughness of a few microns, a welding pressure of 50 MPa is sufficient to produce diffusion welding with high strength and ductility, irrespective of the method of surface machining and cleaning. A higher surface roughness can be compensated for by increasing the welding pressure.
4. In addition to good strength and ductility, satisfactory and reproducible fracture toughness results were achieved when the welding surfaces were machined by dry milling.
5. Etching of the surfaces has no advantage against cleaning with acetone.

In total, it has been demonstrated that diffusion welding is an attractive and reliable joining technique, well suited for the manufacturing of fusion reactor components from martensitic chromium steels.

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