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Metallography studies and hardness measurements on ferritic/martensitic steels irradiated in STIP

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Abstract

In this work metallography investigations and microhardness measurements have been performed on 15 ferritic/martensitic (FM) steels and 6 weld metals irradiated in the SINQ Target Irradiation Program (STIP). The results demonstrate that all the steels have quite similar martensite lath structures. However, the sizes of the prior austenite grain (PAG) of these steels are quite different and vary from 10 to 86 μm. The microstructure in the fusion zones (FZ) of electron-beam welds (EBWs) of 5 steels (T91, EM10, MANET-II, F82H and Optifer-IX) is similar in respect to the martensite lath structure and PAG size. The FZ of the inert-gas-tungsten weld (TIGW) of the T91 steel shows a duplex structure of large ferrite gains and martensite laths. The microhardness measurements indicate that the normalized and tempered FM steels have rather close hardness values. The unusual high hardness values of the EBW and TIGW of the T91 steel were detected, which suggests that these materials are without proper tempering or post-welding heat treatment.

1. Introduction

Comparing to austenitic stainless steels, ferritic/martensitic (FM) steels have not only higher strength and better thermal mechanical properties, but also lower swelling and creep rates under neutron irradiation. Therefore, FM steels have been selected as the candidate structural materials for the first wall blankets of the future fusion reactors [1]. For the same reason, FM steels have also been selected as the beam window materials for liquid metal spallation targets such as the MEGAPIE (Mega Watt Pilot Experiment) and prime candidate materials to be used the future ADS (Accelerator Driven Systems) devices. Although the behaviors of FM steels after neutron irradiation have been well studied, little is known in spallation target irradiation conditions due to the fact that the transmutation products, especially gas products such as helium and hydrogen, may play an important role in the irradiation induced effects. As a key irradiation program for providing necessary materials data for developing advanced spallation targets, the SINQ target-irradiation program (STIP) has included a few thousand specimens of various FM steels [2,3]. The FM steels were obtained from various sources. Although these FM steels have been studied in different materials programs, the basic microstructural information of the steels such as grain structure, grain size and precipitate structure is not completely known from the materials' suppliers or the literature. This causes some difficulties for understanding the results of the related post-irradiation examinations, and moreover, for comparing the behaviors of these steels. Therefore, a systematic study has been carried out to obtain such information from more than twenty FM steels, FM-ODS steels, some welds of the FM steels. In the present paper the grain structure and microhardness of fifteen FM steels and six of their weld metals will be reported. Meanwhile, the detailed information of precipitate structure of these steels is described in elsewhere [4].

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2. Experimental

2.1. Materials

2.1.1. Ferritic/martensitic (FM) steels

The FM steels investigated in this study were separated into two groups: one was the conventional FM steels and the other was so-called reduced activation ferritic/martensitic (RAFM) steels. In total fifteen FM steels were studied. The chemical compositions of these steels are given in Table 1. The information of the steel suppliers and the heat treatments of the steels are listed in the following.

(1) T91 (ORNL)
The steel named as 9Cr1MoVNb (mod) was obtained from the Oak Ridge National Laboratory (ORNL). The heat No was 30176. Plates of either 3 mm or 6 mm thickness were normalized at 1040 °C for 1 h and tempered at 760 °C for 1 h. The steel was used in the first STIP irradiation experiment (STIP-I).

(2) T91 (STIP-II)
The steel was obtained from CEA, France. The heat number was 36224. The as-received material was a 15-mm-thick plate which might be cut from a large piece. The heat treatments were performed at PSI: normalized at 1040 °C for 1 h followed by air cooling, and then tempered at 760 °C for 1 h and followed by air cooling. The steel was used in the second STIP irradiation experiment (STIP-II).

(3) T91 (SPIRE)
The steel was supplied by the Ugine (France) company for the EU FP5 SPIRE program. The heat number was 36224. The as-received material was in the form of a 15-mm-thick plate. The steel was normalized at 1040 °C for 1 h followed by air cooling, and then tempered at 760 °C for 1 h and followed by air cooling. Specimens of this steel were irradiated in the second and third STIP irradiation experiments (STIP-II and STIP-III).

(4) EM10 (STIP-II)
The steel was received by CEA, France together with the T91 (STIP-II) steel. The as-received material was also a 15-mm-thick plate and might be cut from a large piece. The heat treatments were performed at PSI: normalized at 990 °C for 1 h followed by air cooling, and then tempered at 750 °C for 1 h followed by air cooling. Specimens of this steel were irradiated in STIP-II.

(5) T92
The steel was obtained from CEA, France. The steel was selected as one of key materials studied in the ADS related materials programs of the EC 6th Framework Program. The as-received material was a 3.5-mm-thick plate. The steel was normalized at 990 °C for 50 min and tempered at 750 °C for 1 h. Specimens of this steel were irradiated in STIP-IV.

Table 1
Chemical compositions of the tempered martensitic steels in wt% (Fe in balance)

Steel	Cr	Ni	Mo	Mn	Ti	V	Nb	W	Ta	Cu	C	Si	P	S	B	N
T91 (ORNL)	8.32	0.09	0.86	0.48	0.001	0.20	0.06	–	–	0.03	0.092	0.15	0.012	0.004	0.007	0.005
T91 (STIP-II)[a]	8.63	0.23	0.95	0.43	0.003	0.21	0.09	–	–	0.046	0.1	0.31	0.02	0.006	–	0.03
T91 (SPIRE)[a]	8.63	0.23	0.95	0.43	0.003	0.21	0.09	–	–	0.046	0.1	0.31	0.02	0.006	–	0.03
EM10 (STIP-II)	8.97	0.07	0.99	0.48	0.007	0.01	< 0.002	–	–	0.05	0.102	0.44	0.013	< 0.003	< 0.001	0.014
T92	8.82	0.12	0.47	0.43	–	0.2	0.06	1.72	–	–	0.1	0.19	0.015	0.002	0.003	0.045
MANET-II	10.3	0.65	0.58	0.85	–	0.19	0.14	–	–	–	0.11	0.18	0.005	0.004	0.030	0.030
DIN1.4313 (FZJ)	13.0	4.0	<0.7	1.0	0.3	–	–	–	–	–	0.05	1.0	0.045	0.03	–	–
DIN1.4922 (FZJ)	11.5	0.5	1.0	0.5	–	0.3	–	–	–	–	0.2	0.3	0.045	0.03	–	–
EP823 (STIP-II)	11.7	0.65	0.73	0.54	–	0.34	0.26	0.63	–	–	0.17	1.1	–	–	–	–
F82H (Mod)	7.87	0.02	0.003	0.1	0.004	0.19	.0002	1.98	0.03	0.01	0.09	0.07	0.003	0.001	0.007	0.007
Optimax A	9.3	<0.01	0.09	0.60	<0.01	0.24	<0.01	0.97	–	<0.01	0.098	0.02	0.01	<.001	0.001	0.001
Optifer-V	9.48	0.06	0.002	0.55	–	0.245	–	0.985	0.065	–	0.125	0.04	.0015	0.003	–	–
Optifer-IX	8.9	–	–	0.325	–	0.21	–	1.238	0.044	–	0.12	0.025	0.003	0.003	–	0.028
Eurofer97 (Rod)	8.93	0.007	<0.001	0.44	0.009	0.19	<0.001	1.10	0.14	0.022	0.12	0.07	<0.005	0.004	<0.001	0.017
Eurofer97 (Plate)	8.93	0.022	0.0015	0.47	0.009	0.20	0.002	1.07	0.14	0.003	0.12	0.060	<0.005	0.004	<0.001	0.018

[a] T91 (STIP-II) was from the same heat of the T91 (SPIRE). Therefore the composition is the same as the T91 (SPIRE) steel.

(6) MANET-II
A piece of 25-mm-thick plate was obtained from the fusion materials community. The steel was normalized at 1075 °C for 30 min followed by air cooling, and then tempered at 750 °C for 2 h followed by air cooling. Specimens of this steel were irradiated in STIP-II.

(7) DIN 1.4313
A piece of cylinder of 40 mm in diameter was obtained from Forschungszentrum Jülich. The steel was normalized at about 1030 °C and tempered at about 610 °C. Some specimens of this steel were irradiated in STIP-IV.

(8) DIN1.4922
A piece of 5 mm thick plate was obtained from Forschungszentrum Jülich. The steel was normalized at about 1050 °C and tempered at about 750 °C. Some specimens of this steel were irradiated in STIP-IV.

(9) EP823 (STIP-II)
The specimens of the EP823 steel were supplied by the Los Alamos National Laboratory (LANL). The steel was originally obtained from the Institute of Physics & Power Engineering (IPPE), Russia. The steel was normalized at 1050 °C for 1 h and followed by air cooling, and then tempered at 730 °C for 2 h and followed by air cooling. Some tensile, bend-bar and TEM specimens of the steel were irradiated in STIP-II.

(10) F82H (mod)
The F82H (mod), IEA Heat 974, was obtained from the fusion materials community in the form of a 15 mm thick plate. The steel was normalized at 1040 °C for 38 min and tempered at 750 °C for 1 h. Specimens fabricated from the plate were irradiated in STIP-I to STIP-V.

(11) Optimax-A
The Optimax-A steel was developed by the Fusion Materials Technology Group of Federal Poly-technique University Lausanne, Switzerland. The steel was in the form of an 8 mm thick plate. It was normalized at 1050 °C and tempered at 750 °C for 2 h.

(12) Optifer-V
The Optifer-V steel was developed at Forschungszentrum Karlsruhe (FZK). The heat number was 735. It was in the form of a forged bar with a cross-section of about 24×24 mm. The steel was normalized at 950 °C for 30 min followed by air cooling, and tempered at 750 °C for 2 h followed by air cooling. Specimens of this steel were irradiated in STIP-I.

(13) Optifer-IX
The Optifer-IX steel was also developed at FZK. The heat No was 803. It was in the form of a forged bar with a cross-section of about 24×24 mm. The steel was normalized at 1075 °C for 30 min followed by air cooling, and tempered at 750 °C for 2 h and followed by air cooling. Specimens of this steel were irradiated in STIP-II.

(14) Eurofer97 (Rod)
The Eurofer97 (Rod) steel in the form of a rod of 100 mm diameter was received from FZK The heat No was E83699. The steel was normalized at 979 °C for 1 h 51 min followed by air cooling, and tempered at 739 °C for 3 h 42 min followed by air cooling. Tensile and TEM specimens of this steel were irradiated in STIP-II.

(15) Eurofer97 (Plate)
The Eurofer97 (Plate) steel (Lot 250) in the form of a 25 mm thick plate was received from FZK. The heat No was E83697. The steel was normalized at 980 °C for 27 min followed by air cooling, and tempered at 760 °C for 1.5 h and followed by air cooling. Specimens of this steel were irradiated in STIP-III to -V.

For a comparison, the normalizing and tempering conditions are listed in Table 2.

Table 2
Heat treatment conditions, mean PAG size and micro-hardness measurement results of the tempered martensitic steels

Materials	Heat treatment conditions		PAG size	Micro-hardness
	Normalization	Tempering	(μm)	(HV0.05)
T91(ORNL)	1040 °C/1 h	760 °C/1 h	23 ± 3	225 ± 5
T91 (STIP-II)	1040 °C/1 h	760 °C/1 h	15 ± 1.5	264 ± 5
T91 (SPIRE)	1040 °C/1 h	760 °C/1 h	29 ± 6	258 ± 14
EM10(STIP-II)	990 °C/1 h	750 °C/1 h	43 ± 15	250 ± 8
T92	1060 °C/20 min	780 °C/1 h	18 ± 1.5	243 ± 5
MANET-II	1075°/30 min	750 °C/2 h	32 ± 2.5	268 ± 5
DIN1.4313	~1030 °C	~610 °C	76 ± 7	243 ± 10
DIN1.4922	~1050 °C	~750 °C	81 ± 5	252 ± 5
EP823 (STIP-II)	1050 °C/1 h	730 °C	25 ± 10	268 ± 12
F82H (mod)	1040 °C/38 min	750 °C/1 h	86 ± 20	220 ± 5
Optimax-A	960 °C/30 min	750 °C/2 h	19 ± 1.5	219 ± 5
Optifer-V	950 °C/30 min	750 °C/2 h	11 ± 0.5	217 ± 5
Optifer-IX	1075 °C/30 min	750 °C/2 h	55 ± 10	266 ± 5
Eurofer97 (Rod)	979 °C/1 h51 min	739 °C/3 h42 min	16 ± 2	227 ± 5
Eurofer97 (Plate)	980 °C/27 min	760 °C/1 h30 min	16 ± 1.5	237 ± 5

2.1.2. Welds of FM steels

In this work electron beam welds (EBWs) of the T91 (ORNL), EM10 (STIP-II), MANET-II, Optifer-IX and F82H steels and the inert gas tungsten arc weld (TIGW) of the T91 (ORNL) steel were investigated. The EBWs were prepared from 3-mm-thick plates and the TIGW was prepared from a 6-mm-thick plate, which was aimed at studying the welding applied to thin components (e.g. liquid metal containers) in spallation targets. The EBW and TIGW of the T91 (ORNL) steel were received from ORNL. The other EBWs were prepared at PSI, which were re-tempered at 730 °C for 2 h after welding.

2.2. The metallographic technique

The grain structures of the steels were mostly investigated on the cross-sections parallel to the rolling or extrusion direction. In some cases, the grain structures on the transverse cross-sections perpendicular to the rolling or extrusion direction were also examined. The specimens were prepared using a standard metallographic technique: fine mechanical polishing plus chemical and/or electrochemical etching. The main etchant used in this study was the solution of 45 ml HCl + 20 ml HNO_3 + 35 ml H_2O. For some steels, in order to get images of a sharp and delineated contrast, another solution of 1.5 g CuCl + 33 ml HCl + 33 ml ethanol + 33 ml H_2O was used after the etching of the first etchant. The observations were performed on an optical microscope. The micrographs of each specimen were taken at magnifications of 100, 200 and 500 times. The grain size was evaluated using the circle line method according to the ASTM E 112-96 Standard Test Methods for Determining Average Grain Size. For each steel 5 or more micrographs were used and 250 or more grains were counted. The variation was mostly below ±15% of the mean values.

2.3. Micro-hardness measurements

The micro-hardness was measured with a Vickers micro-hardness testing machine. The micro-hardness measurement performed according to the ASTM E92-82 Standard Test Method for Vikers Hardness of Metallic Materials. In this work, a load (P) of 0.05 kgf (0.49 N) was applied, and the loading time was 20 s. The Vickers micro-hardness value ($HV_{0.05}$) was evaluated. For each specimen 5-8 measurements were performed, and the average value ($HV_{0.05}$) was calculated. The variation was normally below ±5% of the averaged values.

3. Results and discussion

3.1. FM steels

3.1.1. Grain structure

The features of the grain structure of the 9 conventional steels are shown in Figs. 1–9, and those of the 6 RAFM steels are shown in Figs 10–15. In most cases only the cross-sections parallel to rolling/extrusion direction are illustrated. While for steels T91 (SPIRE), Optifer-V, Eurofer97 (Rod) and Eurofer97 (Plate) the cross-sections of both parallel and perpendicular to rolling/extrusion direction are presented. From the figures one can see that, except for the F82H steel, all other steels show rather similar appearance: clear martensite lath structure inside PAGs. As for the F82H steel, its appearance is somewhat different from the others after the same polishing and etching processing. The martensite laths are not so clear, although the transmission electron microscopy (TEM) reveals that the F82H steel is also fully martensitic [5]. An obvious difference in PAG size can be observed from the figures. Steels such as DIN1.4313, DIN1.4922 and F82H have very large PAGs of 70–80 μm in size. The Optifer-IX and EM10 (STIP-II) steels have also quite large PAGs of about 50 μm. Then followed by steels such as

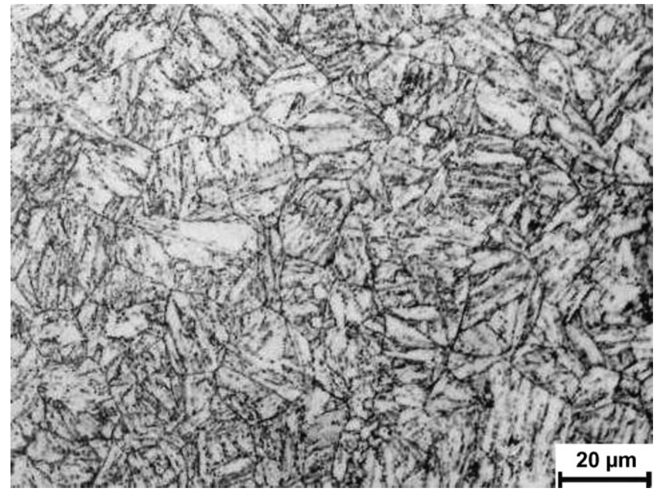

Fig. 1. Optical micrograph showing the grain structure of the T91 (ORNL) steel.

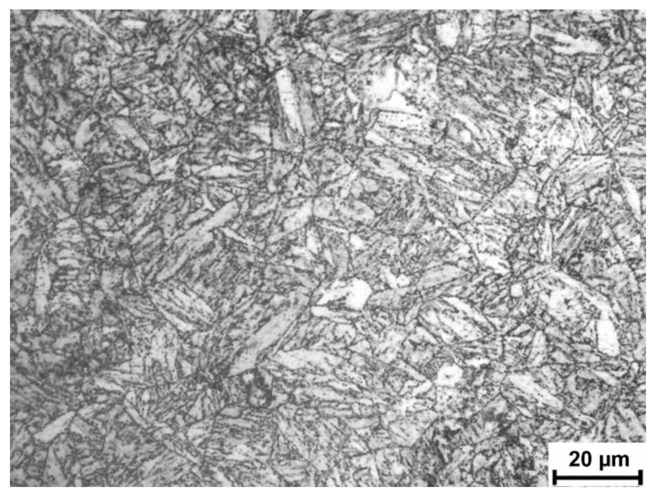

Fig. 2. Optical micrograph showing the grain structure of the T91 (STIP-II) steel.

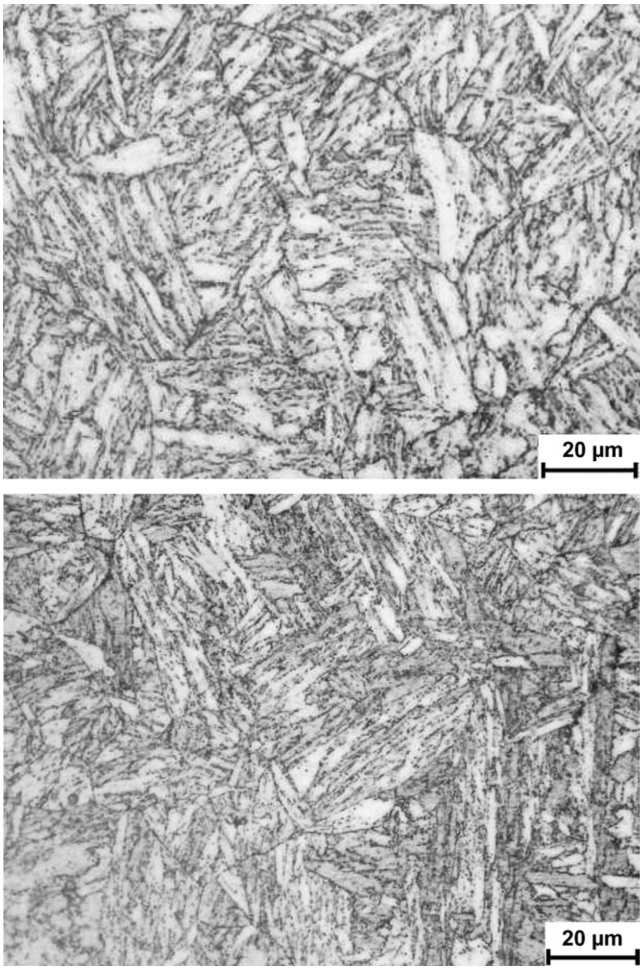

Fig. 3. Optical micrographs showing the grain structure of the T91 (SPIRE) steel on cross-sections parallel (upper) and perpendicular (lower) to the rolling direction.

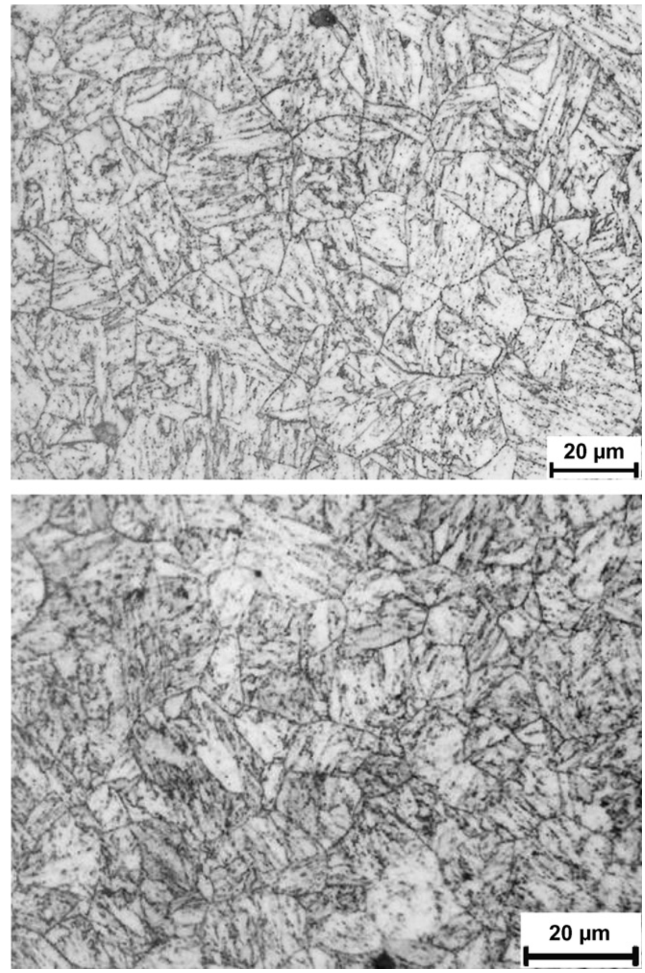

Fig. 5. Optical micrographs showing the grain structure of the T92 steel on cross-sections parallel (upper) and perpendicular (lower) to the rolling direction.

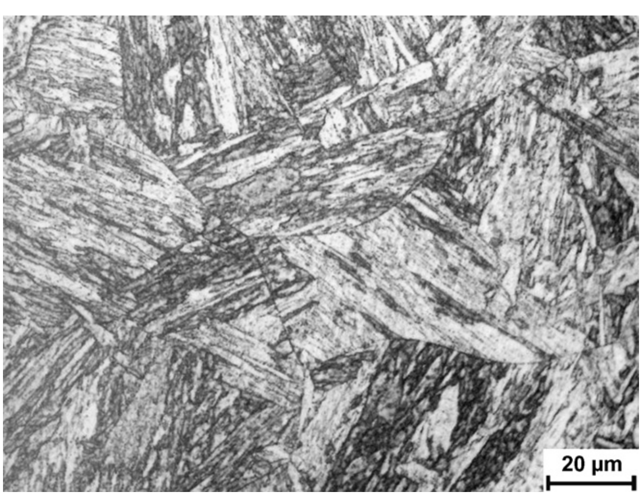

Fig. 4. Optical micrograph showing the grain structure of the EM10 (CEA) steel.

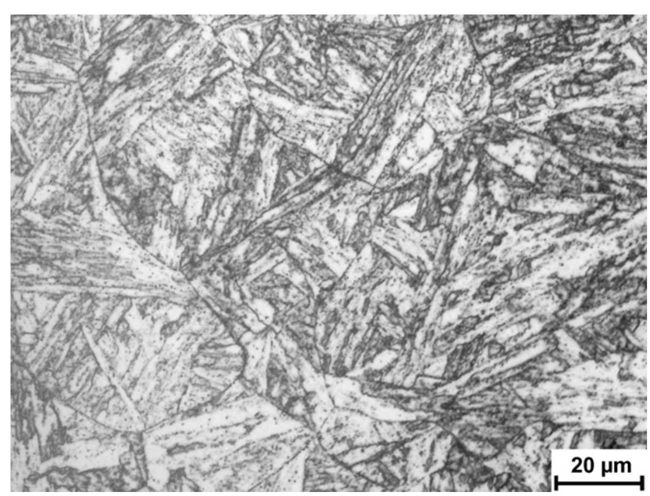

Fig. 6. Optical micrograph showing the grain structure of the MANET-II steel.

MANET-II, T91 (SPIRE) and T91 (ORNL) have 20–30 μm PAGs. The rest are with quite small PAGs of 10–20 μm. The detailed results of the PAG size are given in Table 2 and plotted in Fig. 16. Generally the PAG size

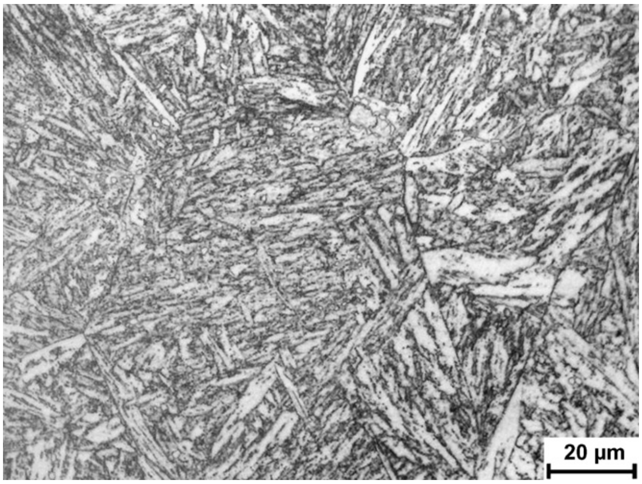

Fig. 7. Optical micrograph showing the grain structure of the DIN 1.4313 steel.

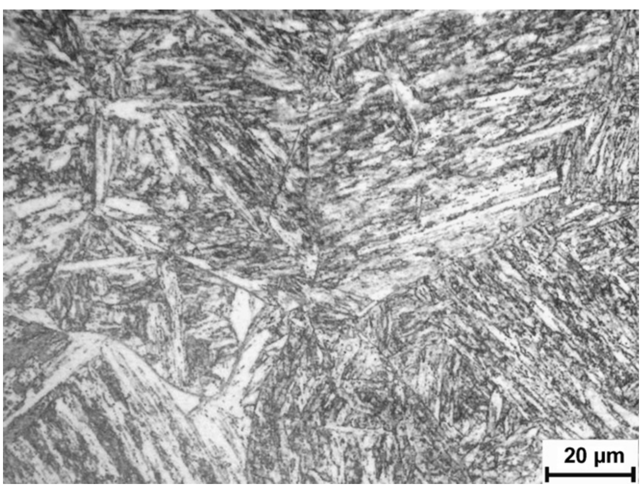

Fig. 8. Optical micrograph showing the grain structure of the DIN1.4922 steel.

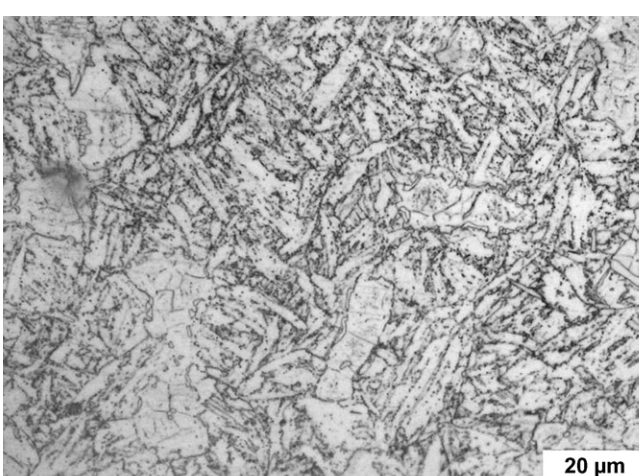

Fig. 9. Optical micrograph showing the grain structure of the EP823 (STIP-II) steel.

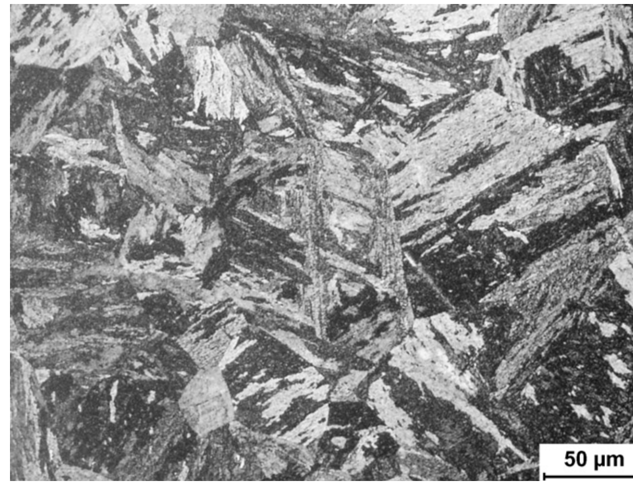

Fig. 10. Optical micrograph showing the grain structure of the F82H (mod) steel.

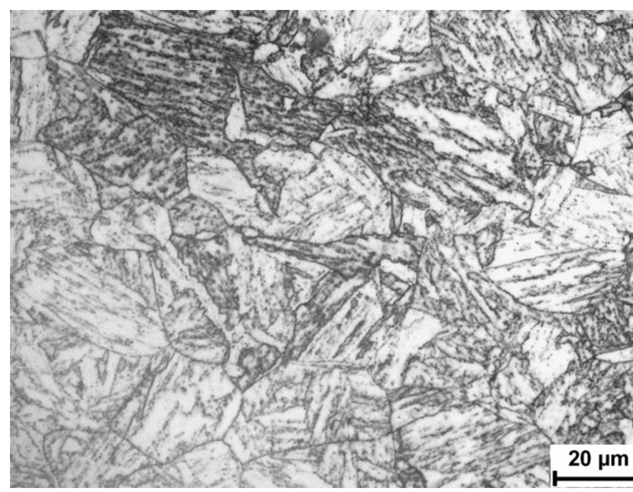

Fig. 11. Optical micrograph showing the grain structure of the Optimax-A steel.

should increase with the normalizing temperature. From Table 2 one can see that, at least for steels normalized at lower temperatures up to 980 °C the PAG size is relatively small, <20 μm. However, for the rest it is difficult to figure out a rule to correlate their PAG sizes with the normalizing conditions. The reasons could be that the different steels are with very different manufacturing history in respect to hot-/cold-rolling and intermediate heat treatments, and with different contents of the austenite-stabilizing elements such as C, N, Ni, Mn, Cu, etc. The PAG sizes obtained in the present work are in agreement with the values given by the steel suppliers or found in the literature, e.g. for Optifer-V [6], Eurofer97 (Plate) [7], Optimax-A [8] and T91 (SPIRE) [9].

The features shown in Figs. 3, 5, 12, 14 and 15 demonstrate that the microstructures illustrated on both cross-

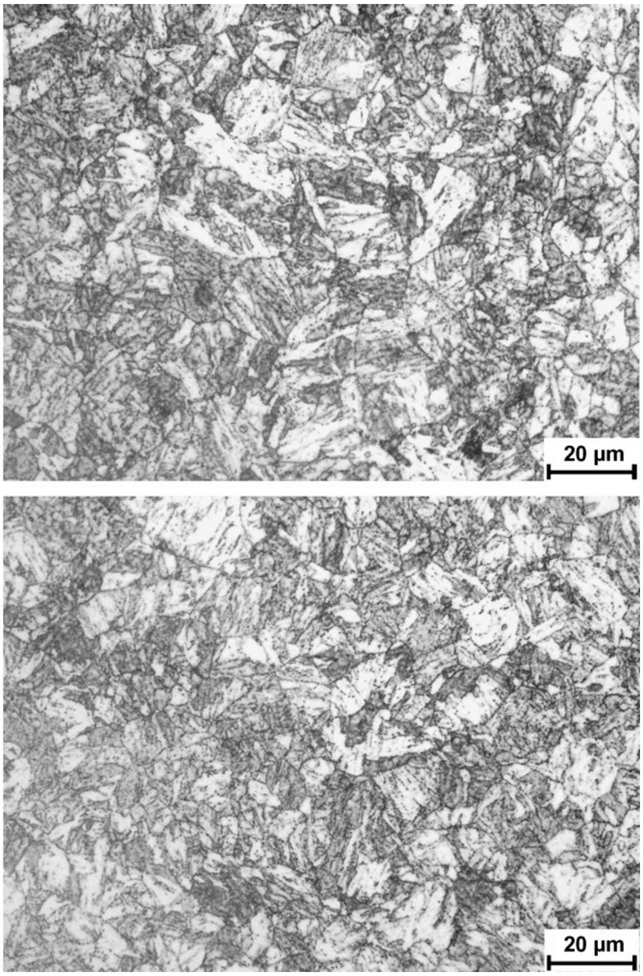

Fig. 12. Optical micrographs showing the grain structure of the Optifer-V steel on cross-sections parallel (upper) and perpendicular (lower) to the extrusion direction.

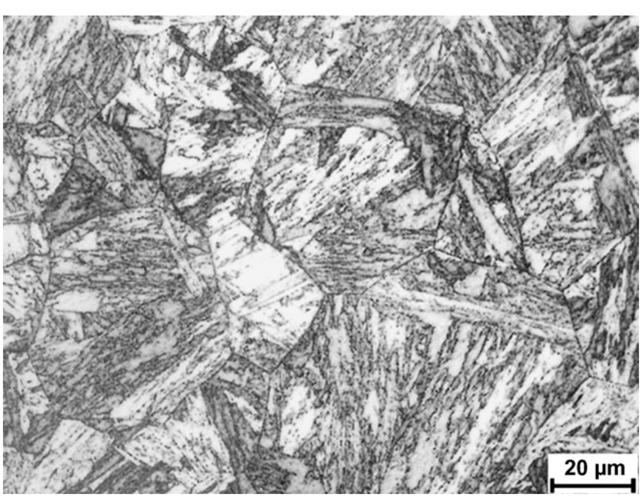

Fig. 13. Optical micrograph showing the grain structure of the Optifer-IX steel.

sections parallel and perpendicular to the rolling/extrusion direction are essentially the same. No obvious texture of

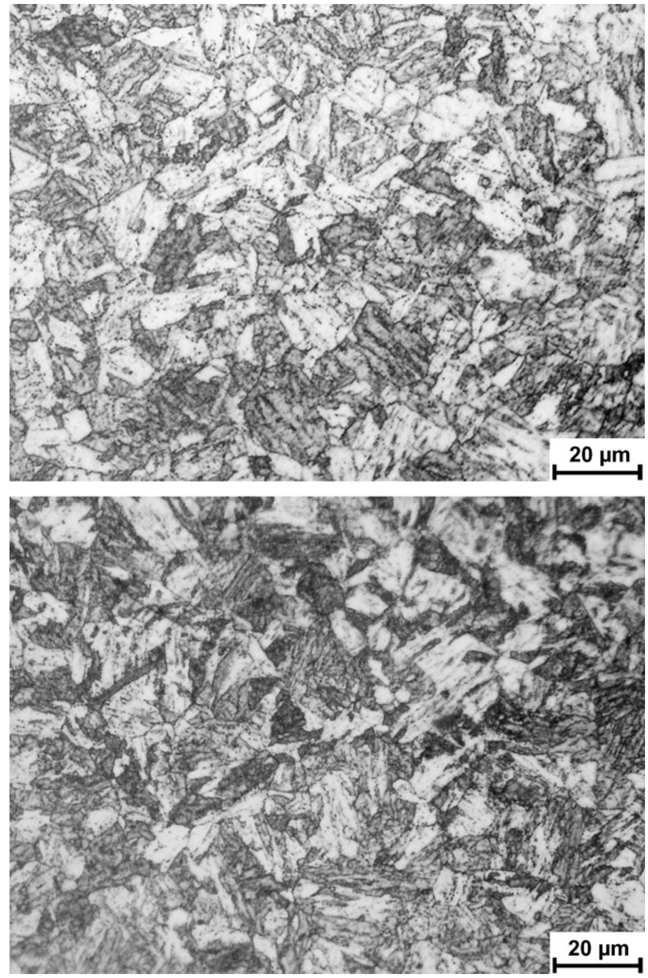

Fig. 14. Optical micrographs showing the grain structure of the Eurofer-97 (Rod) steel on cross-sections parallel (upper) and perpendicular (lower) to the extrusion direction.

PAG in rolling/extrusion direction was detected in these steels.

3.1.2. Micro-hardness

The results of the micro-hardness ($HV_{0.05}$) measurements are shown in Fig. 17 and also summarized in Table 2. The 15 steels show a comparable hardness between about 220 and 270.

Similar to the situation of the grain structure described above, no evident correlation can be drawn between the hardness and the PAG size values and the heat treatment conditions. This suggests that the PAG size should not play an important role in the strength of tempered martensitic steels. This is understandable because the strength of tempered martensitic steels is mainly controlled by the precipitate, dislocation and martensite lath structures. Although the detailed information of the dislocation and lath structures of the steels is not known, a parallel study [4] on the precipitate structure of the steels indicates that steels such as Optifer-IX, MANET-II, EP823 etc. which have

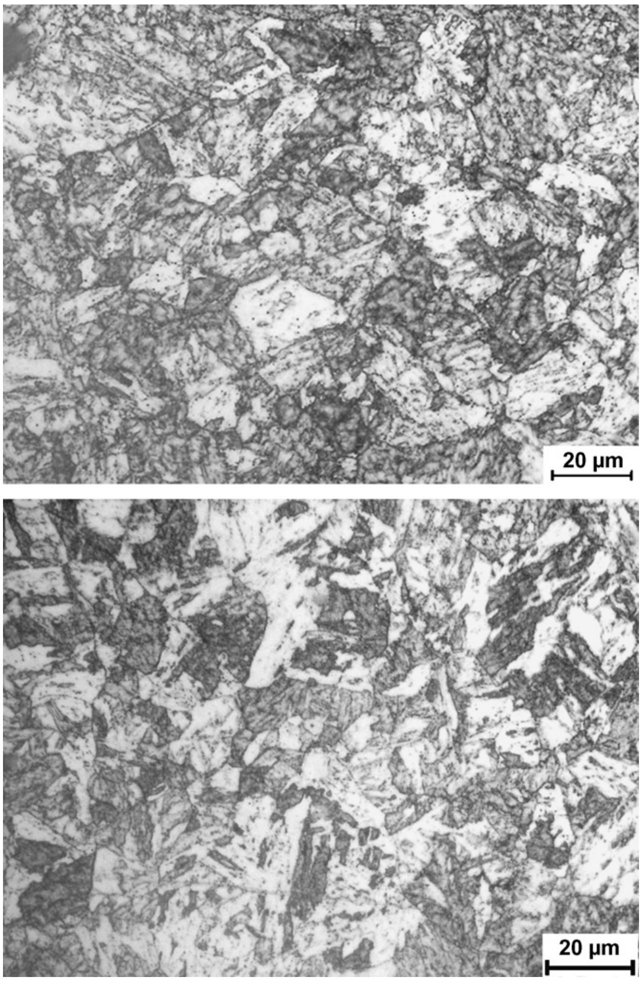

Fig. 15. Optical micrographs showing the grain structure of the Eurofer97 (Plate) steel on cross-sections parallel (upper) and perpendicular (lower) to the rolling direction.

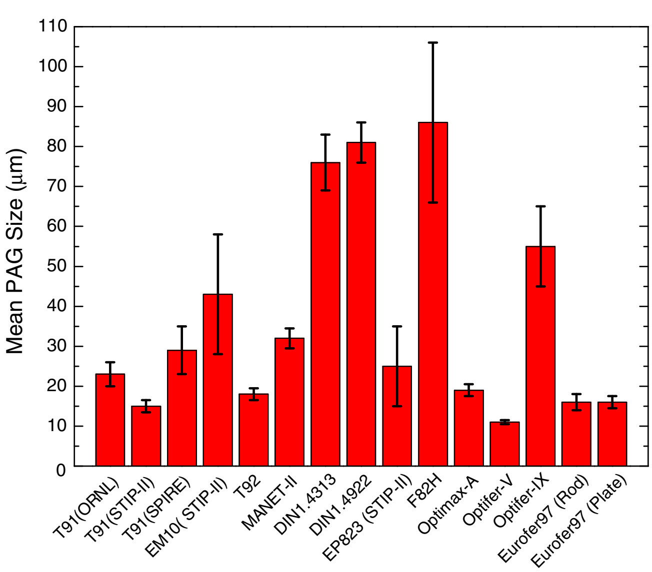

Fig. 16. Mean PAG sizes of the FM steels.

higher hardness values contain high-density small precipitates.

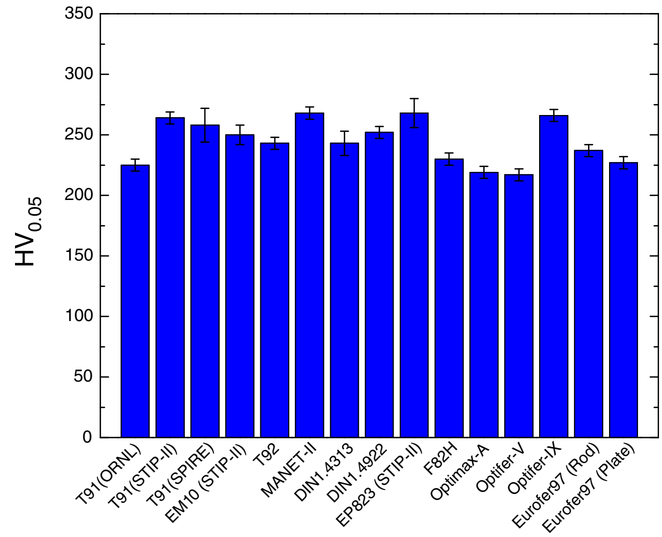

Fig. 17. Micro-hardness ($HV_{0.05}$) of the FM steels.

3.2. Welds of FM steels

The geometries of the EBWs are quite similar, namely the width of the fusion zone (FZ) is about 1mm, and that of the heat affection zone (HAZ) is around 0.5 mm. In all the FZs and HAZs of the five steels, T91 (ORNL), EM10 (STIP-II), MANET-II, F82H and Optifer-IX, almost fully martensite lath structure was observed. The sizes of the PAGs in the HAZs of the different steels are similar to those of the base metals. While the sizes of the PAGs in the FZs are rather close, between 25 and 35 μm, although the PAG sizes of the base metals vary from 18 to 86 μm. As an example, Fig. 18 shows the transverse cross-section (perpendicular to weld-line) of the EBW of the T91 (ORNL) steel and Fig. 19 shows the cross-section (parallel to weld-line) of the EBW of the F82H steel.

In the TIGW of the T91 (ORNL), it was observed that the FZ was of duplex structure of ferrite grains and martensite laths, as shown in Fig. 20, which could be due fast cooling rate after welding.

Micro-hardness was measured along a line perpendicularly to the weld central line cross the areas of the BM, HAZ and FZ. The results of the hardness measurements on the welds are shown in Fig. 21. For the EBWs, an evident increase of hardness is observed in the HAZs and the maximum hardness values are observed in the FZs. For the EBW of the T91(ORNL) steel, the maximum hardness in the FZ is about 430, which implies that no post-welding heat treatment (PWHT) was performed. As for the other EBWs, the hardness values of the FZs are about 20–40% higher than that of the base metals. This suggests that PWHT at slightly higher temperatures may be necessary to reduce the hardening in the FZs.

As mentioned above, the FZ of the TIGW of the T91 (ORNL) steel shows a duplex structure of ferrite and

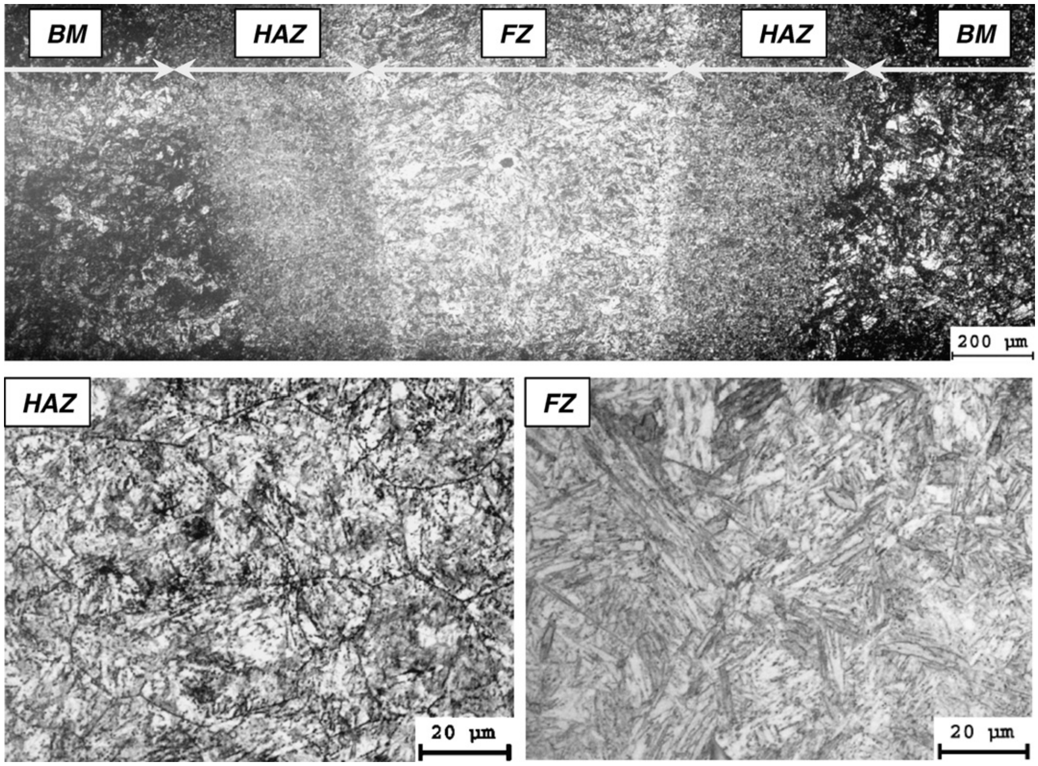

Fig. 18. Micrographs showing the grain structures of the EBW (3 mm thick) of the T91 (ORNL) steel.

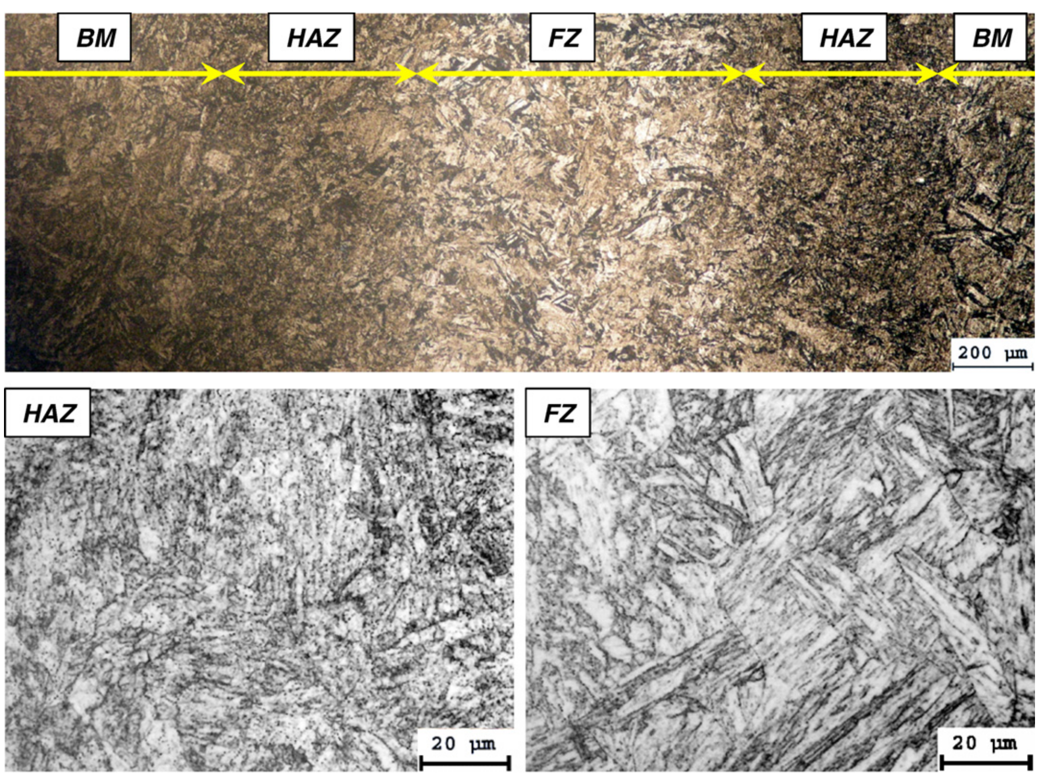

Fig. 19. Micrographs showing the grain structures of the EBW (3 mm thick) of the F82H steel.

martensite. The hardness measurement was carefully performed in both ferrite and martensite areas. In Fig. 21(b), it can be seen that there is a large difference between the hardness values of the two different areas. The high

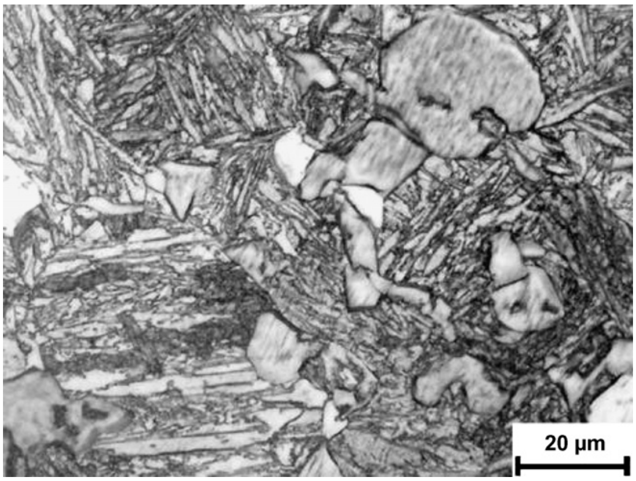

Fig. 20. Optical micrograph showing the grain structure in the FZ of the TIGW (6 mm thick) of the T91 (ORNL) steel.

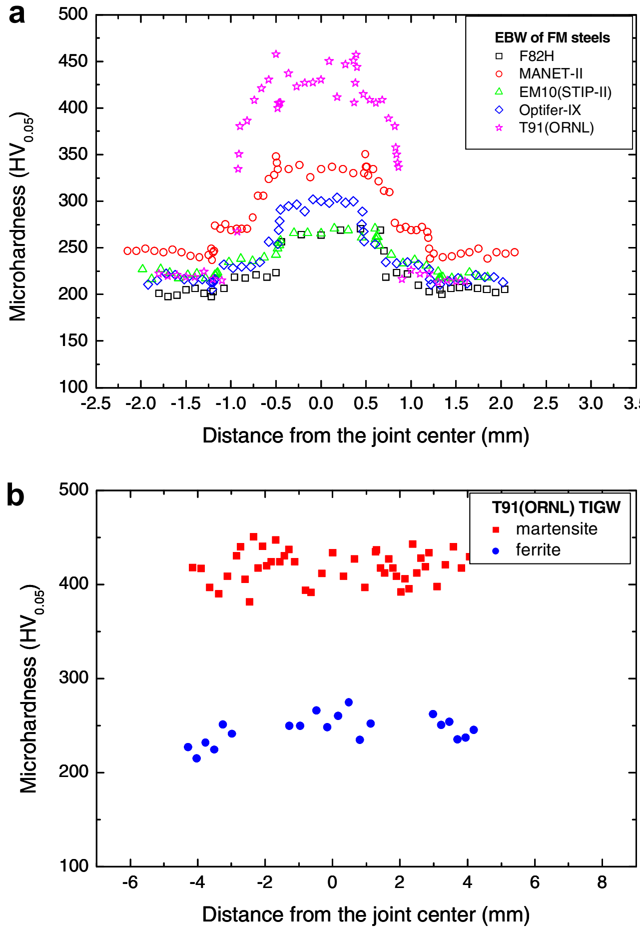

Fig. 21. Microhardness profiles of (a) the EBW of the different FM steels and (b) the TIGW of the T91 (ORNL) steel.

hardness values measured from the martensite areas indicates again that no PWHT was done.

4. Conclusions

Following conclusions can be drawn from the microstructural investigations and hardness measurements of the FM steels and the weld metals:

(1) All the investigated steels showed similar fine and homogeneous tempered martensite lath structure.
(2) The prior austenite grain (PAG) sizes of the 15 FM steels were determined, which showed a quite large variation (from 18 to 86 μm) for different steels.
(3) The $HV_{0.05}$ values of the 15 FM steels varied between about 220 and 270.
(4) The microstructure in the FZs of EBWs of T91 (ORNL), EM10 (STIP-II), MANET-II, F82H and Optifer-IX steels was similar in respect to the martensite lath structure and PAG size. The TIGW of the T91 (ORNL) steel demonstrated a duplex structure of ferrite and martensite.
(5) The microhardness profiles of the 6 welds were determined. For both EBW and TIGW of the T91 (ORNL) steel, the results showed much higher hardness in the FZ and HAZ as compared with that of the base metal. It suggests that the PWHT was not performed on these materials after welding. For the others, the hardness values of the FZs are about 20–40% higher than that of the base metals, which also suggests that PWHT should be performed at slightly higher temperatures.

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