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Letter to the Editors

Simulated coarse-grained heat-affected-zone microstructures in DIN 22NiMoCr37 steel

M.J. Balart *, J.F. Knott

Department of Metallurgy and Materials, School of Engineering, The University of Birmingham,
Edgbaston, Birmingham B15 2TT, UK

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Summary

Samples of DIN 22NiMoCr37 nuclear reactor pressure vessel steel have been heat treated for 1 h in the temperature range 1373–1473 K to obtain different prior austenite grain sizes. Excessive coarsening of prior austenite grains has been observed. This is attributed to the absence in this sample of steel of 'microalloying' elements such as Ti, Nb or V, which can combine with C and/or N and precipitate as carbides, nitrides and/or carbonitrides. These particles are able to pin grain boundaries at high temperatures, and hence prevent excessive grain growth.

1. Introduction

ASTM A508 MnMoNiCr steel and A533B MnMoNi steel, quenched and tempered, are widely used for the construction of nuclear reactor pressure vessels (RPVs). Thick forgings or plates of such steels usually have a tempered bainite microstructure and are fabricated by welding. Tempered martensite microstructures may be generated in the weld heat-affected-zone (HAZ). Prior austenite grain size is a major factor in determining the brittle and ductile properties of MnMoNi alloys, having a tempered martensitic microstructure and similar strength levels [1].

In the present paper, we investigate the effects of austenitizing temperature on prior austenite grain size in a specific sample of DIN 22NiMoCr37 steel, which is said to be equivalent to ASTM A508 Cl.2 [2], forgings of which have been used in the fabrication of nuclear RPVs. The specific sample was from a forged ring section used to generate the Euro data set for fracture toughness (of as-heat-treated forged material). Our aim was to explore how the fracture toughness might be affected by heat-treatments similar to those experienced by the coarse-grained heat-affected-zone (CGHAZ) of a weld. Here, we report observations made on the microstructures developed. The fracture properties of the steel in the simulated CGHAZ microstructures are currently being evaluated and will be presented separately.

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* Corresponding author. Tel.: +44 1214143265; fax: +44 1214145232.

E-mail addresses: M.J.BalartMurria@bham.ac.uk (M.J. Balart), J.F.Knott@bham.ac.uk (J.F. Knott).

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2. Material and heat treatment

The material used was taken from C(T)-4T specimens machined from the steel block SX25.2 used in the Euro data set [2,3]. The heat treatment of the as-received DIN 22NiMoCr37 steel is shown in Fig. 1 [3]. In this heat-treated condition, the steel had a tempered bainitic microstructure, and its fracture properties have been previously determined in the generation of the Euro fracture toughness data set [3]. The chemical composition of the DIN 22NiMoCr37 steel as analysed in previous studies is shown in Table 1. The minimum and maximum values of the elemental analysis determined at 3 different positions (outside, centre and inside, see Fig. 2) of the marked blocks (Pos. 1–4) are given in Table 1. As will be discussed later, we wished to obtain analyses for the microalloying elements Ti, Nb, V and so had the steel reanalysed at the position marked in Fig. 2 and with the results shown in Table 2. Note that in Ref. [3] Cr is given as 0.003 wt%, but, following our analysis and communication with Heerens, it was confirmed that the Cr was indeed 0.38–0.41 wt%, and so this figure is given in Table 1. It may be seen that the contents of V, Nb and, in particular, Ti are very low.

Samples of the steel were austenitised at 1373 K, 1398 K, 1423 K, 1448 K and 1473 K for 1 h, followed by water quenching (Q). They were then tempered at 923 K for 2 h (T) to simulate stress-relief, water quenched (Q) and held isothermally at 793 K for 180 h (E), followed by air cooling, to maximise any susceptibility to intergranular embrittlement. In reporting observations, samples are identified by austenitisation temperature and the heat treatment (QTQE).

Metallographic sections were cut with their normals parallel to the longitudinal (*L*) direction on the 10 mm × 10 mm section. Vickers microhardness measurements were made parallel to both the *T*- and *S*-directions at midthickness (5 mm from the edge) using a load of 0.5 kg (HV 0.5).

3. Results and discussion

Representative macrographs of the samples given different grain coarsening treatments are shown in Fig. 3, and optical micrographs are shown in

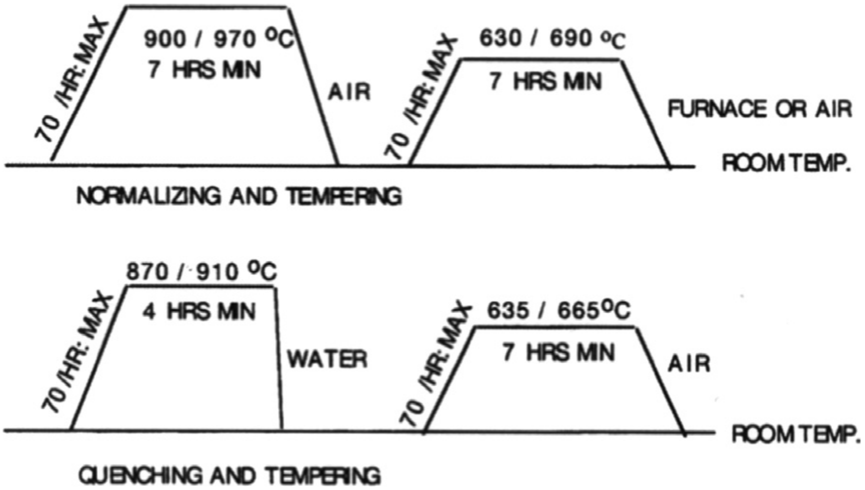

Fig. 1. Heat treatment of the as-received DIN 22NiMoCr37 steel [3].

Table 1
Chemical composition (wt%) of DIN 22NiMoCr37 steel

	C	Si	P	S	Cr	Mn	Ni	Cu	Mo	Positions	References
Min.	0.20	0.23	0.003	0.003	0.38	0.80	0.78	0.049	0.55	[Pos. 1]; [Pos. 2]; [Pos. 3] and [Pos. 4] (see Fig. 2)	[3,4]
Max.	0.21	0.26	0.004	0.004	0.40	0.84	0.80	0.050	0.57		
	0.22	0.23	0.006	0.0047	0.39	0.88	0.84	0.08	0.51	Block 30 taken from the same plate used in the Round-Robin to generate the 'Euro' database	[5]

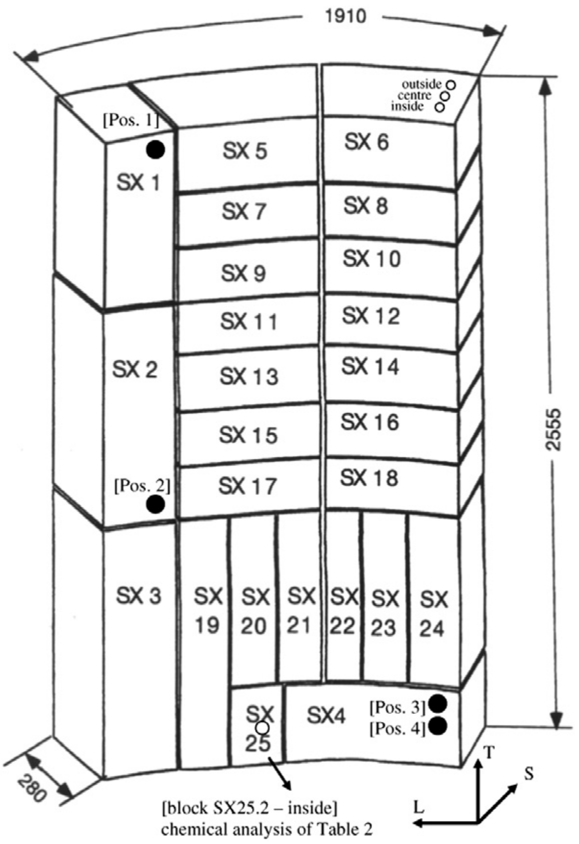

Fig. 2. Large ring segment of the DIN 22NiMoCr37 steel (dimensions in mm) where the chemical analysis given in Tables 1 and 2 was determined [3,4].

Fig. 4. SEM micrographs of the as-received bainitic microstructure and the 1373-QTQE and 1473-QTQE martensitic microstructure are shown in Fig. 5. The as-received sample exhibited a fine-grained bainitic microstructure, Figs. 3(a) and 4(a). The 1373-QTQE showed a prior austenite grain size of approx. 100 μm, Figs. 3(b) and 4(b). The 1398-QTQE and 1423-QTQE showed mixed grain sizes, composed of coarse grained regions (>1 mm diameter) and finer-grained regions (∼100 μm), Fig. 3(c). This is indicative of abnormal grain growth and could result from the presence of a spatially inhomogeneous distribution of grain boundary pinning centres, e.g. carbides, nitrides and/or carbonitrides. The 1448-QTQE and 1473-QTQE exhibited an extremely large, uniform equi-axed prior austenite grain size (diameter ∼1 mm), Figs. 3(d) and 4(c).

The microhardness profiles, determined on the 10 mm × 10 mm cross-sections, are shown in

Table 2
Chemical composition (wt%) of DIN 22NiMoCr37 reanalysed taken from C(T)-4T machined from the steel block [SX25.2 – inside] position (see Fig. 2)

C	Si	P	S	Cr	Mn	Ni	Cu	Mo	Sn	Al	As	B	Sb	Ti	Nb	V	N	O
0.21	0.22	0.005	0.004	0.41	0.87	0.87	0.06	0.52	0.007	0.014	0.006	<0.0005	0.002	0.0004	0.002	0.004	0.008	0.0005

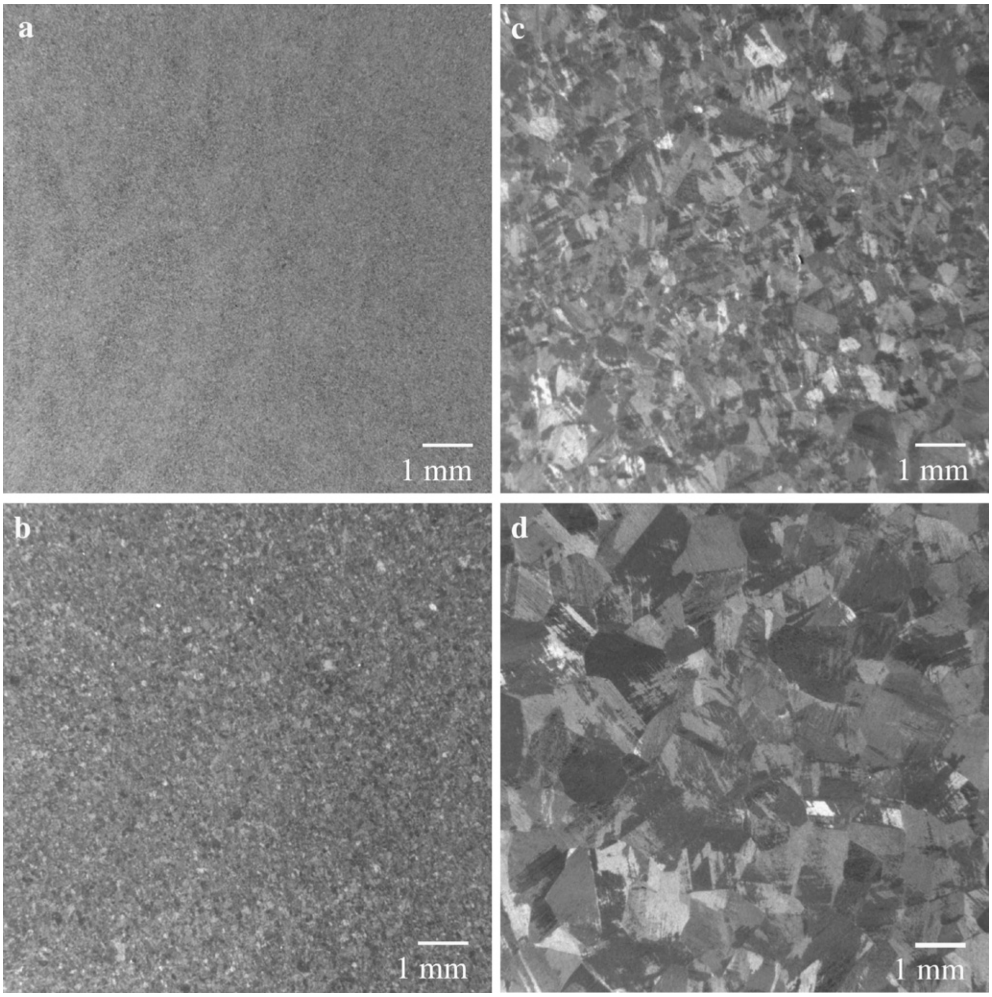

Fig. 3. Macrographs of the DIN 22NiMoCr37 steel (a) as received; and heat treated (b) 1373-QTQE, (c) 1423-QTQE and (d) 1473-QTQE.

Fig. 6. The average and standard deviation (sd) HV 0.5 values are given next to the symbol legend. The values obtained parallel to the *S*-direction are 210 ± 6 for AR, 307 ± 7 for 1373-QTQE, 311 ± 17 for 1398-QTQE, 308 ± 11 for 1423-QTQE, 317 ± 9 for 1448-QTQE and 307 ± 7 for 1473-QTQE. Generally, the values for the CGHAZ are close with small sd and may be compared with the range reported in Ref. [1], 295 ± 25 for tempered martensitic microstructures. The somewhat larger values of sd for 1398-QTQE and 1423-QTQE compared to the other samples may be the result of sampling large and finer grains in mixed grain size microstructures. It is of interest that the microhardness values for the heat treated samples are similar, despite there being over an order of magnitude difference in prior austenite grain size between 1373-QTQE and 1473-QTQE. Equating microhardness to flow stress, it is clear that the resistance to dislocation movement is provided by the fine dispersion of tempered carbides, Fig. 5(b) and (c), rather than by grain boundaries as such. The coarser carbide distribution in the AR bainitic microstructure is associated with a lower value of flow stress/microhardness.

We suspected that the excessive grain growth was due to the absence of Ti, Nb and/or V and so had the steel reanalysed with the results shown in Table 2. The levels of these microalloying elements (in wt%) were, indeed, found to be extremely low: 0.0004Ti, 0.002Nb and 0.004V. A survey was also made of prior austenite grain sizes of A508 and MnMoNi steels reported in the literature. These are summarised in Table 3, together with details of the steel grade and its heat treatment. At an austenitisation temperature of 1473 K for 1 h, a range of prior austenite grain size values, from

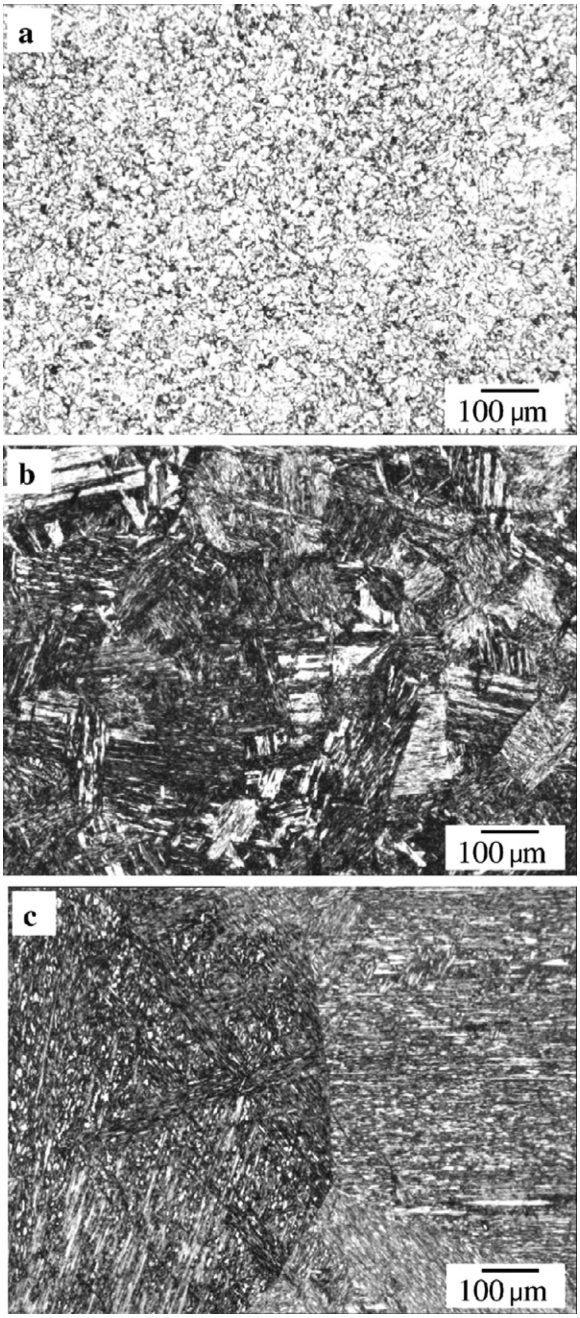

Fig. 4. Optical micrographs of the DIN 22NiMoCr37 steel (a) as received; and heat treated (b) 1373-QTQE and (c) 1473-QTQE.

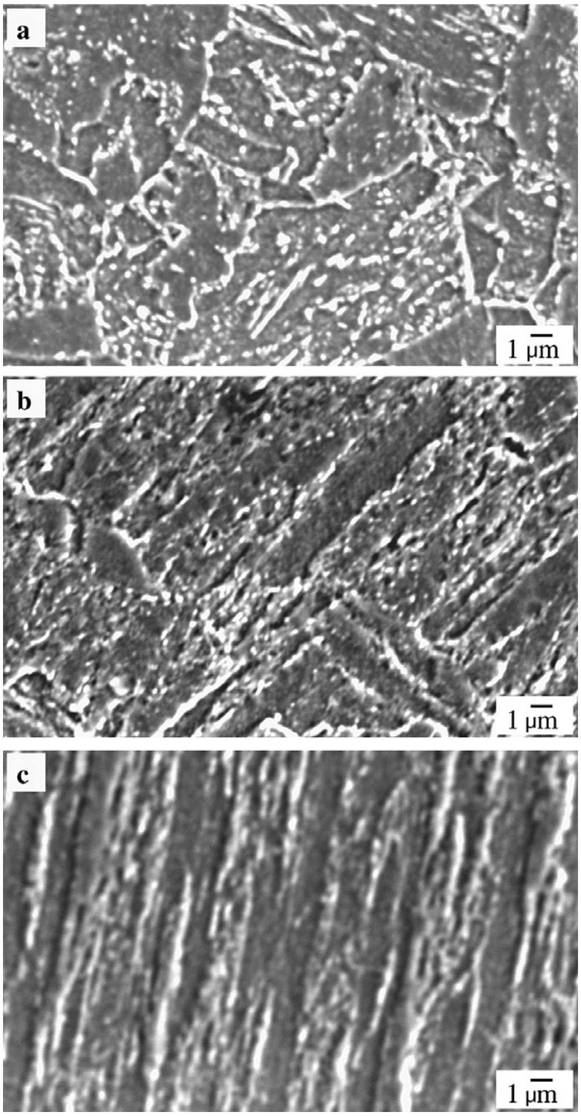

Fig. 5. SEM micrographs of the DIN 22NiMoCr37 steel (a) as received, showing tempered bainite; and heat treated (b) 1373-QTQE and (c) 1473-QTQE, showing tempered martensite.

98 μm to 448 μm is quoted in Table 3, compared to that of ~1 mm in the 1473-QTQE steel. Note that the level of Ti, in particular is not generally quoted. Some ASTM MnMoNi(Cr) and CrMo(V) steels specified as ‘weldable’, implicitly if not explicitly, contain ‘microalloying’ additions of Ti and/or V to control austenite grain size at high austenitisation temperatures, by creating a fine dispersion of carbides, nitrides and/or carbonitride particles stable at high temperatures, Tables 4–7. These particles can pin grain boundaries and prevent grain growth. The critical microalloying element is Ti, to form relatively stable TiN particles which exhibit the highest dissolution temperature [15]. Versions of a steel containing insufficient Ti–N microalloying additions are likely to experience excessive grain growth in the regions of a weld close to the fusion boundaries.

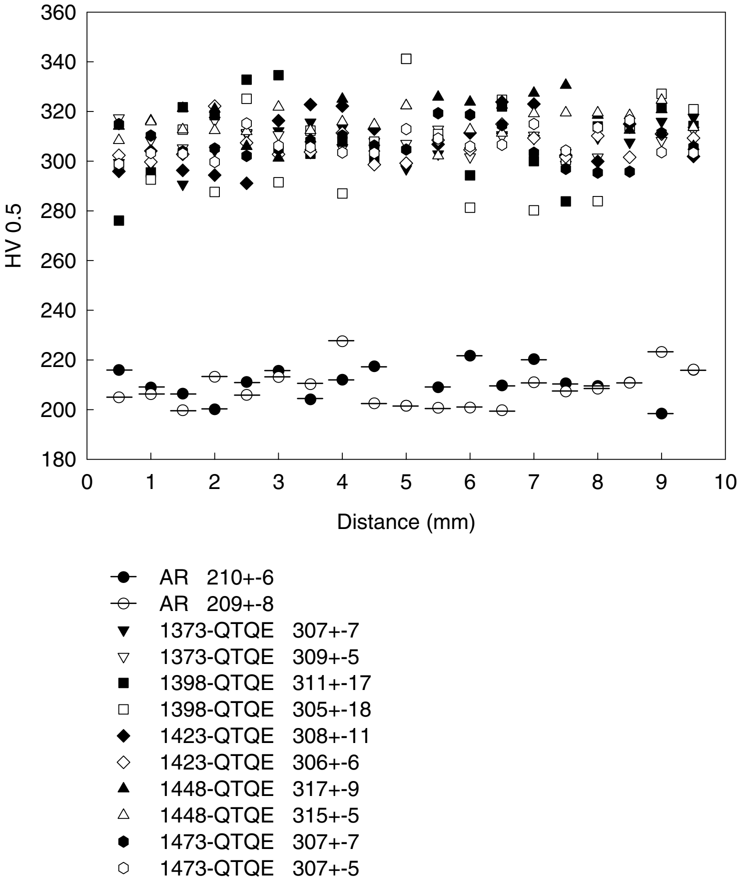

Fig. 6. Microhardness profiles taken on the 10 mm × 10 mm cross-sections, filled symbols – measurements taken parallel to the *S*-direction; and unfilled symbols – measurements taken parallel to the *T*-direction. The HV 0.5 mean and sd values at the measured positions are given next to the figure legends.

The microchemistry and stability of a complex carbide, nitride or carbonitride depend on the steel's alloy composition, steel phases and processing parameters [16–19]. The estimated solubility

Table 3
Prior austenite grain size for some grades of A508 and MnMoNi steels reported in the literature

Materials	Chemical composition (wt%)	Heat treatment	Prior γ grain size; microstructure	References
Experimental melt MnMoNi steel 'pure'	Fe–0.24C–1.37Mn–0.51Mo–0.54Ni–0.26Si–<0.01Cr–0.0027P–0.0047S–0.04Cu–<0.01V–<0.02As–<0.01Sb–0.0017B–<0.01Sn–<0.01W–<0.01Nb–0.0030O–0.0015N–0.0012Al	1123 K 1 h; WQ; 923 K 1 h AC 1273 K 1 h; WQ; 923 K 1 h AC 1373 K 1 h; WQ; 923 K 1 h AC 1473 K 1 h; WQ; 923 K 1 h AC 1573 K 1 h; WQ; 923 K 1 h AC Embrittling treatment: 773 K 1000 h WQ	22 μm 86 μm 99 μm 98 μm 251 μm	[1]
Experimental melt MnMoNi steel 'S doped'	Fe–0.25C–1.47Mn–0.53Mo–0.60Ni–0.27Si–<0.01Cr–0.0025P–0.061S–0.009Cu–<0.01V–<0.02As–<0.01Sb–0.0011B–<0.01Sn–<0.01W–<0.01Nb–0.0048O–0.0048N–0.0087Al	1123 K 1 h; WQ; 923 K 1 h AC 1273 K 1 h; WQ; 923 K 1 h AC 1373 K 1 h; WQ; 923 K 1 h AC 1473 K 1 h; WQ; 923 K 1 h AC 1573 K 1 h; WQ; 923 K 1 h AC Embrittling treatment: 773 K 1000 h WQ	14 μm 64 μm 79 μm 137 μm 185 μm	[1]

(*continued on next page*)

Table 3 (*continued*)

Materials	Chemical composition (wt%)	Heat treatment	Prior γ grain size; microstructure	References
Experimental melt MnMoNi steel ‘Cu doped’	Fe–0.27C–1.39Mn–0.52Mo–0.55Ni–0.28Si–<0.01Cr–0.0086P–0.0053S–0.4Cu–<0.01V–<0.02As–<0.01Sb–0.0011B–<0.01Sn–<0.01 W–<0.01Nb–0.0074O–0.0016N–0.0038Al	1123 K 1 h; WQ; 923 K 1 h AC 1273 K 1 h; WQ; 923 K 1 h AC 1373 K 1 h; WQ; 923 K 1 h AC 1473 K 1 h; WQ; 923 K 1 h AC 1573 K 1 h; WQ; 923 K 1 h AC Embrittling treatment: 773 K 1000 h WQ	17 μm 99 μm 154 μm 448 μm 1064 μm	[1]
Experimental melt MnMoNi steel ‘P doped’	Fe–0.26C–1.44Mn–0.52Mo–0.54Ni–0.29Si–<0.01Cr–0.045P–0.0045S–<0.01Cu–<0.01V–<0.02As–<0.01Sb–0.0014B–<0.01Sn–<0.01W–<0.01Nb–0.0245O–0.0028N–0.0037Al	1123 K 1 h; WQ; 923 K 1 h AC 1273 K 1 h; WQ; 923 K 1 h AC 1373 K 1 h; WQ; 923 K 1 h AC 1473 K 1 h; WQ; 923 K 1 h AC 1573 K 1 h; WQ; 923 K 1 h AC Embrittling treatment: 773 K 1000 h WQ	23 μm 92 μm 104 μm 428 μm 615 μm	[1]
A533B	Fe–0.24C–1.50Mn–0.50Mo–0.66Ni–0.23Si–0.15Cr–0.002S–0.005P–0.11Cu–0.03Al–0.002Ti	1473 K 1 h; WQ; 923 K 2 h; WQ; Embrittling treatment: 793 K 0 h, 30 h, 90 h and 180 h; AC	110 μm	[6]
A508 Cl. 3	Fe–0.16C–1.33Mn–0.49Mo–0.72Ni–0.22Si–0.23Cr–0.0036S–0.010P–0.006Cu–0.012Al–0.005Ti–0.004Nb–0.002V–0.0907N–0.001Pb–0.001Sb–0.007Sn–0.017Co–0.022As–0.005W	1423 K 1 h; OQ 903 K 2 h; AC	∼100 μm; tempered martensite	[7]
A533B Cl. 1	Fe–0.20C–1.43Mn–0.50Mo–0.85Ni–0.26Si–0.12Cr–0.004S–0.019P–0.14Cu–0.012Al–0.003V	1373 K 1 h; OQ; 903 K 2 h; followed by an embrittling treatment: step cooling heat treatment between 873 K and 743 K 160 h	∼200 μm; tempered martensite	[8]
A533B	Fe–0.25C–1.51Mn–0.53Mo–0.63Ni–0.22Si–0.06Cr–0.005P–0.005S–0.04Cu–0.025As–0.001Sb–0.005Sn–0.02Al	(a) 1523 K 1 h; OQ (b) 1523 K 1 h; FC at 293 K/min to 1173 K, holding for 1 h; OQ	(a) 130 μm; auto-tempered lath martensite (b) 102 μm; auto-tempered lath martensite	[9]
A508 Cl.3	Fe–0.18C–1.46Mn–0.51Mo–0.86Ni–0.10Si–0.15Cr–0.006P–0.003S–0.03Cu–0.004V–0.018Al	Simulated HAZ (a) 1173 K peak temperature (b) 1623 K peak temperature	(a) 10 μm; 71% ferrite, 22% martensite, 5% austenite and 2% carbide (b) 410 μm; 4% ferrite, 93% martensite, 2% austenite and 1% carbide	[10]

WQ – water quenching; OQ – oil quenching; AC – air cooling; FC – furnace cooling.

temperatures in austenite are 1323 K for VN, 1373 K for NbC, 1423 K for AlN and >1723 K for TiN [16]. Reynolds [20] reported that, at austenitisation temperatures of 1473 K, 1523 K and 1573 K for 1 h, the average prior austenite grain size in experimental low alloy steels (not of A508/A533B type) was 160, 170 and 200 μm for compositions with <0.005 wt% Ti (residual levels), and 60, 80

Table 4
Chemical composition (wt%) of the ASTM A 508/A 508M - 03 [11]

C	Si	P	S	Cr	Mn	Ni	Cu	Mo	Sn	Al	As	B	Sb	Ti	Nb	V	N	O
Grade 3																		
0.25[a]	0.15–0.40	0.025[a]	0.025[a]	0.25[a]	1.20–1.50	0.40–1.00		0.45–0.60								0.05[a]		
Grade 3V																		
0.10–0.15	0.10[a]	0.020[a]	0.020[a]	2.8–3.3	0.30–0.60			0.90–1.10				0.001–0.003		0.015–0.035		0.20–0.30		

[a] Max.

Table 5
Chemical composition (wt%) of the ASTM A 302/A 302M - 03 grade C product analysis [12]

C	Si	P	S	Cr	Mn	Ni	Cu	Mo	Sn	Al	As	B	Sb	Ti	Nb	V	N	O
0.20–0.25[a]	0.13–0.45	0.035[a]	0.035[a]		1.07–1.62	0.37–0.73		0.41–0.64										

[a] Max.

Table 6
Chemical composition (wt%) of the ASTM A 533/A 533M - 93(2004) type B product analysis [13]

C	Si	P	S	Cr	Mn	Ni	Cu	Mo	Sn	Al	As	B	Sb	Ti	Nb	V	N	O
0.25[a]	0.13–0.45	0.035[a]	0.035[a]		1.07–1.62	0.37–0.73		0.41–0.64										

[a] Max.

Table 7
Chemical composition (wt%) of the ASTM A 542/A 542M - 99(2004) product analysis [14]

C	Si	P	S	Cr	Mn	Ni	Cu	Mo	Sn	Al	As	B	Sb	Ti	Nb	V	N	O
Type B (2.25Cr1Mo)																		
0.09–0.18	0.50[a]	0.015[a]	0.015[a]	1.88–2.62	0.25–0.66	0.28[a]	0.28[a]	0.85–1.15								0.03[a]		
Type C (Cr–Mo–V alloy)																		
0.08–0.18	0.13[a]	0.025[a]	0.025[a]	2.63–3.37	0.25–0.66	0.28[a]	0.28[a]	0.85–1.15						0.005–0.045		0.18–0.33		

[a] Max.

and 100 μm when the steel contained 0.015 wt% Ti. In the complete absence of grain boundary pinning particles, the average prior austenite grain sizes were 152, 660 and 840 μm for austenitisation temperatures of 1473 K, 1523 K and 1573 K, respectively. Optimum additions may be made to weldable steels. Excessive additions can lead to the formation of coarse cubic carbonitrides which can act as initiation sites for cleavage fracture [21].

4. Summary and conclusions

Excessive coarsening of prior austenite grains has been observed in DIN 22NiMoCr37 steel, after austenitizing in the temperature range 1373–1473 K. This is attributed to the absence in the samples of steel of ‘microalloying’ elements such as Ti, Nb, V which are able to form carbides, nitrides and/or carbonitrides, which can pin grain boundaries at high temperatures. The ASTM ‘weldable’ grades include microalloying elements implicitly, rather than explicitly, but the specifications are clearly based on past experience and coarse grain size is thought to be detrimental. The potential dichotomy between a drive for ‘cleanliness’ *per se* and a need to include microalloying elements to prevent excessive grain growth should be exposed as soon as possible.

Acknowledgements

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