Air cooled bainitic steels for strong, seamless pipes Part 2 – properties and microstructure of rolled material

G. Gomez¹, T. Pérez¹ and H. K. D. H. Bhadeshia^{*2}

In the first part of the present study,¹ two steels were designed with the aim of achieving properties consistent with the specifications for the manufacture of seamless pipes. The alloys were intended to develop into mixtures of bainitic ferrite and carbon enriched retained austenite during cooling to ambient temperature from the pipe forming operations, thus avoiding the need for subsequent heat treatment. Part 2 of the study deals with the characterisation and understanding of the mechanical properties of the two alloys. Good combinations of strength and toughness have been achieved.

Keywords: Seamless pipe, Hot rolled, Bainite, Retained austenite

Introduction

In Part 1 of the present study¹ two alloy systems (Table 1) were designed in order eventually to facilitate the production of seamless pipes without the additional quench and tempering heat treatment that is currently a part of the manufacturing process. The alloys were shown to reliably produce the desired microstructure of carbide free bainite under conditions appropriate to industrial parameters. The work reported here deals with the next stage in the development, i.e. the study of the microstructure and properties of the alloys in the hot rolled condition. The experimental techniques have been described in Part 1 and hence are not repeated here.¹

Microstructure in hot rolled condition

The microstructure of alloy 1 in the as rolled condition consists of bainitic ferrite and retained austenite (Fig. 1*a* and *b*); the fraction of austenite was determined using Xray analysis at 0.18 ± 0.01 (Table 1).¹ The hardness of this structure was 382 ± 5 HV20, close to that of the dilatometric sample cooled at 0.2° C s⁻¹.¹ No carbides were observed in the as rolled microstructure, but the size of some regions of austenite was as high as 5 µm. These blocky austenitic regions are considered detrimental to toughness due to their tendency to transform into coarse, untempered brittle martensite under the influence of stress.^{2–4} In contrast alloy 2 has a perceptibly finer structure (Fig. 1*c* and *d*), mostly composed of thin films of bainitic ferrite and retained austenite. There were also some small regions that may be identified as autotempered martensite. The fraction of austenite was determined to be 0.13 ± 0.01 , present mostly as films <1 µm thick between bainite plates. The proportion of blocky austenite appeared far less than in alloy 1. The hardness was much higher than the as rolled alloy 1, at 468 ± 5 HV20, but again, consistent with samples cooled at 0.2° C s⁻¹.¹ It is reasonable therefore to conclude that 0.2° C s⁻¹ is a representative cooling rate for 16 mm plates cooled in air after hot rolling.

The refinement of the microstructure of alloy 2 relative to alloy 1 is expected from its lower transformation temperature at any given cooling rate.^{5–8} Its greater hardness is due both to the structural refinement and its higher carbon concentration.

The tensile and impact properties in the rolled condition are shown in Table 2. The yield to tensile strength ratio is substantially lower in comparison to quenched and tempered steels, $^{9-12}$ although quite consistent with hot rolled ferrite pearlite steels (Fig. 19 of Ref. 13). Low yield ratios are typical in bainitic microstructures containing free dislocations which are introduced due to the displacive mechanism of transformation.^{14–17} An additional factor is the mixed microstructure in which the individual phases have different mechanical properties.^{18,19}

One interesting and counterintuitive observation is that the stronger alloy 2 has better toughness than alloy 1; the ductile to brittle transition temperature (as measured using full size Charpy specimens) for the former is a respectable -20° C whereas this rises to 25° C in alloy 1. The higher carbon alloy therefore outperforms its lower carbon counterpart in both strength and toughness. An

¹Department of Metallurgy, Tenaris Argentina R&D Simini 250, 2804 Campana, Pcia. Buenos Aires, Argentina ²Materials Science and Metallurgy, University of Cambridge Cambridge

CB2 3QZ, UK

^{*}Corresponding author, email hkdb@cam.ac.uk



1 Scanning electron micrographs of hot rolled steel: optical micrographs have been presented in Part 1 (Ref. 1)

increase in carbon content produces microstructural refinement by a reduction in the transformation temperature. This refinement produces an increase in both strength and toughness. There must of course be an upper limit to the amount of carbon that can be added to suppress transformation temperature because the maximum quantity of bainite that can form is then reduced via the incomplete reaction phenomenon.^{20,21}

Heat treatments

Normalising treatments were carried out to study the effects austenitic grain size, at temperatures $20-40^{\circ}$ C above the measured A_{c3} (880 and 840°C for alloys 1 and 2 respectively). Because these are much lower than the finish rolling temperature (1000°C), the austenitic grain size was reduced to about 16–22 µm in both alloys (Table 1).¹ The mechanical property data in Table 3 show that the smaller austenite grain size does not lead to any significant improvement in the tensile or toughness properties, indicating that other factors described below are of greater importance.

Table 1 Compositions (wt-%) of alloys 1 and 2; full details in Ref. 1

	С	Mn	S	Cr	Ni	Ni	
Alloy 1	0·24	0·09	1·27	1∙00	0·23	3∙64	
Alloy 2	0·30	0·10	1·42	1∙03	0·22	3∙48	

The best combination of properties, both in the as rolled and normalised conditions, was obtained with alloy 2. It was suspected that the microstructure may benefit from tempering as a means to optimise properties; tempering treatments at temperatures up to 500° C were therefore performed on as rolled specimens. Figure 2 shows that an important increase in hardness with a maximum at ~ 300° C. The properties measured in the tempered condition are given in Table 4. Consistent with the hardness data, tempering at 300° C led to a large increase in proof strength and a smaller increase in the tensile strength, with their ratio increasing from 0.67 to 0.79; at the same time, the upper shelf energy of the Charpy tests also increased (Table 4). The

Table 2 Mechanical property data for as rolled condition

		Alloy 1	Alloy 2
0.2% proof strength, MPa		816	965
Ultimate tensile strength, MPa		1185	1447
Proof to ultimate strength ratio		0·69	0·67
Elongation, %		15	15
	Test temperature, °C	Charpy energy, J	Shear area, %
Alloy 1	25	24	25
	0	20	<20
	-20	17	<20
Alloy 2	25	69	100
	0	58	100
	20	49	48
	40	42	34



for one hour at temperature indicated

fracture surface, illustrated in Fig. 3, shows 100% ductile fracture at 0°C, with a ductile to brittle transition temperature of about -20° C.

Tempering at 500° C also led to an enhancement of the proof strength, but there was a dramatic deterioration in the toughness, Table 4. The metallurgical mechanisms responsible for the observed changes in mechanical properties were investigated further using microscopy and diffraction.

Figures 4 and 5 show features from the midthickness of the untempered plate, and that tempered at 300°C. The overall bainitic microstructure was essentially unchanged by the low temperature heat treatment. From the microstructural analysis it was uncertain whether there was some kind of precipitation within

Table 3	Mechanical	property	data	for	normalised
	condition				

		Alloy 1	Alloy 2
0.2% proof strength, MPa		838	968
Ultimate tensile strength, MPa		1213	1545
Proof to ultimate strength ratio		0·69	0∙63
Elongation, %		12	14
	Test temperature, °C	Charpy energy, J	Shear area, %
Alloy 1	24	34	38
	0	29	25
	-20	22	15
Alloy 2	24	56	100
	0	48	100
	-20	48	100



3 Surface of Charpy sample broken at room temperature, alloy 2, tempered at 300°C

the bainitic ferrite during tempering at 300°C, or whether the increase in hardness was due to some decomposition of the remaining austenite. The X-ray determined fractions of retained austenite are given in Table 5. The concentrations are derived from the Rietveld refined lattice parameters and assuming the relationship between the parameter and composition as in Ref. 22. The carbon content of ferrite is much higher than consistent with equilibrium or paraequilibrium, but its value remained almost unchanged during tempering. This is consistent with previous studies,^{22,23} which indicated that the excess of carbon in ferrite is segregated to dislocations and hence is stabilised against precipitation.²⁴ This has been confirmed by direct imaging.²⁵ The retention of excess carbon is valid for temperatures up to $\sim 500^{\circ}$ C, where recovery processes begin to anneal the dislocation structure.²² Thus, the precipitation of carbides from the supersaturated ferrite during tempering at 300°C was ruled out as an explanation of the observed hardening; indeed, the carbides could not be observed with certainty using transmission electron microscopy (Fig. 5).

Another possible hardening mechanism is an additional decomposition of austenite into fine bainite during isothermal holding at 300°C.^{26–28} Considering that the maximum volume fraction of bainite corresponding to the isothermal transformation at 300°C was probably not achieved during continuous cooling, the

Table 4 Mechanical property data for alloy 2 following tempering

		As rolled	Tempered 300°C	Tempered 500°C
0.2% proof strength, MPa		965	1232	1040
Ultimate tensile strength, MPa		1447	1563	1409
Proof to ultimate strength ratio		0.67	0.79	0.74
Elongation, %	-	15	14	15
	Test temperature, °C	Charpy energy, J	Shear area, %	
As rolled	0	58	100	
	-20	49	48	
	-40	42	34	
Tempered 300°C	25	75	100	
	0	68	100	
	-20	52	49	
Tempered 500°C	25	18	23	
	0	15	11	
	-20	15	8	



a as rolled condition; *b* after tempering at 300°C Scanning electron micrographs of alloy 2

4

retained austenite may continue to transform during tempering. This possibility is consistent with:

- (i) a reduction in the retained austenite fraction from 0.13 ± 0.01 in the as rolled material to 0.10 ± 0.01 in the sample tempered at 300°C
- (ii) an increase in the austenite carbon content
- (iii) a dilatometric expansion during tempering (Fig. 6).

Nevertheless, the measured decrease in V_{γ} seems too small to account for the large increase in the proof stress. It is likely that the tempering at 300°C helps reduced the density of mobile dislocations.

As pointed out earlier, tempering at 500° C leads to a dramatic decrease in toughness, as is evident also from the mode of fracture (Fig. 7). The tempering temperature is high enough to cause the retained austenite to decompose into a mixture of ferrite and carbides, a



5 Transmission electron micrograph of alloy 2 after tempering at 300°C



6 Dilatometer strain recorded when sample of as rolled alloy 2 is tempered at 300°C: expansion occurs indicating transformation of austenite

phenomenon which is well established to lead to a decrease in toughness.^{2,3} This was supported by the X-ray diffraction analysis, which showed that the retained austenite in the tempered sample had decreased to almost zero (V_{γ} =0.03±0.02).

Summary

Mixed microstructures of bainitic ferrite and retained austenite have been studied. The importance of maintaining a low transformation temperature consistent with microstructural refinement is evident from the observation that toughness can be greater for a steel which is stronger and contains a higher carbon concentration.

The steels studied have a low proof strength to tensile strength ratio but it is found that tempering at a low temperature can significantly enhance the proof strength, presumably by allowing the free dislocations to be pinned by carbon. This is a useful tool given that some requirements for pipes used in the oil and gas

Table 5X-ray diffraction data: V_{γ} is volume fraction of
retained austenite;
 x_{γ} and x_{α} are carbon
concentrations of austenite and ferrite
respectivelySteelVxwt-%

Sleel	ν _γ	<i>x</i> _γ , wι-70	\mathbf{X}_{α} , WL-70
Alloy 2, as rolled	0.13 ± 0.01	1.2 ± 0.1	0.14 ± 0.03
Alloy 2, tempered at 300°C	0.10 ± 0.01	1.4 ± 0.1	0.15 ± 0.03



a scanning electron micrograph; b surface of broken
Charpy specimen tested at room temperature
7 Alloy 2 tempered at 500°C

industries specify a minimum proof strength and a maximum hardness; to fulfill both conditions a high proof to tensile strength ratio is needed. However, tempering at a high temperature leads to the loss of retained austenite and a large decrease in toughness.

Promising results were obtained with alloy 2 both in the as rolled and tempered at low temperature conditions. However for many high strength applications the impact properties would have to be improved. The next stage in the development of these alloys will involve some chemical modifications and the analysis of other processing routes. The results obtained will be used to assess the feasibility of a full scale industrial trial.

Acknowledgement

The authors are grateful to Tenaris Argentina (Research and Development) for funding this work.

References

- G. Gomez, T. Pérez and H. K. D. H. Bhadeshia: *Mater. Sci. Technol.*, DOI: 10.1179/174328408X388130.
- 2. H. K. D. H. Bhadeshia and D. V. Edmonds: Met. Sci., 1983, 17, 411–419.
- H. K. D. H. Bhadeshia and D. V. Edmonds: *Met. Sci.*, 1983, 17, 420–425.
- S. Chatterjee and H. K. D. H. Bhadeshia: *Mater. Sci. Technol.*, 2006, 22, 645–649.
- K. J. Irvine and F. B. Pickering: in 'Physical properties of martensite and bainite', Special report 93, 110–125; 1965, London, Iron and Steel Institute.
- F. B. Pickering: 'Physical metallurgy and the design of steels'; 1978, Essex, Applied Science Publishers.
- F. B. Pickering: 'Constitution and properties of steels', 339–399; 1992, New York, VCH Publishers.
- S. B. Singh and H. K. D. H. Bhadeshia: *Mater. Sci. Eng. A*, 1998, A245, 72–79.
- 9. G. E. Pellissier: Eng. Fract. Mech., 1968, 1, 55-75.
- A. N. Kumar and R. K. Pandey: *Mater. Sci. Eng.*, 1984, 63, 121– 136.
- W. S. Lee and T. T. Su: J. Mater. Process. Technol., 1999, 87, 198– 206.
- J. Henry, X. Averty, Y. Dai, P. Lamagnere, J. P. Pizzanelli, J. J. Espinas and P. Wident: J. Nucl. Mater., 2003, 318, 215–227.
- S. B. Singh, H. K. D. H. Bhadeshia, D. J. C. MacKay, H. Carey and I. Martin: *Ironmaking and Steelmaking*, 1998, 25, 355–365.
- H. K. D. H. Bhadeshia: 'Bainite in steels', 2nd edn; 2001, London, Institute of Materials.
- 15. M. Sudo and I. Kokubo: Scand. J. of Metall., 1984, 13, 329-342.
- K. Sugimoto, T. Sakaki, T. Kurihashi and O. Miyagawa: *Tetsu-to-Hagane*, 1986, **72**, 2101–2108.
- E. Swallow and H. K. D. H. Bhadeshia: *Mater. Sci. Technol.*, 1996, 12, 121–125.
- Y. Tomota, K. Kuroki, T. Mori and I. Tamura: *Mater. Sci.Eng.*, 1976, 24, 85–94.
- H. K. D. H. Bhadeshia and D. V. Edmonds: *Met. Sci.*, 1980, 14, 41–49.
- H. K. D. H. Bhadeshia and D. V. Edmonds: Acta Metall., 1980, 28, 1265–1273.
- P. G. Self, H. K. D. H. Bhadeshia and W. M. Stobbs: Ultramicroscopy, 198, 16, 29–40.
- M. Peet, C. Garcia-Mateo, F. G. Caballero and H. K. D. H. Bhadeshia: *Mater. Sci. Technol.*, 2004, 20, 814–818.
- 23. H. K. D. H. Bhadeshia and A. R. Waugh: Acta Metall., 1982, 30, 775–784.
- 24. D. Kalish, M. Cohen: Mater. Sci. Eng., 1970, 6, 156-166.
- F. G. Caballero, M. K. Miller, S. S. Babu and C. Garcia-Mateo: Acta Mater., 2007, 55, 381–390.
- F. G. Caballero, H. K. D. H. Bhadeshia, K. J. A. Mawella, D. G. Jones and P. Brown: *Mater. Sci. Technol.*, 2002, 18, 279–284.
- F. G. Caballero and H. K. D. H. Bhadeshia: Curr. Opin. Solid State Mater. Sci., 2005, 8, 186–193.
- 28. H. K. D. H. Bhadeshia: Mater. Sci. Technol., 2005, 21, 1293-1302.