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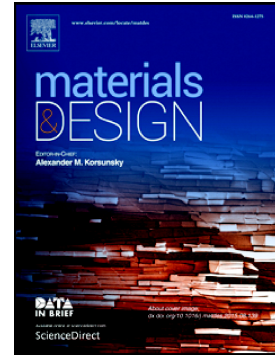
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## Journal Pre-proof

The influence of Widmanstätten ferrite, martensite and grain boundary carbides on the strength and impact behaviour of high Al (0.2%) and Nb containing hot rolled steels



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# The influence of Widmanstätten ferrite, martensite and grain boundary carbides on the strength and impact behaviour of high Al (0.2%) and Nb containing hot rolled steels

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## ABSTRACT

The influence of Al and Nb on the strength and impact behaviour of hot rolled 0.06%C, 1.4%Mn steels has been determined after hot rolling to 15 and 30mm thick plate. When 0.16%Al was added to the plain C-Mn steel, the impact behaviour significantly improved even though Widmanstätten ferrite (WF) was present. This improvement was due to refinement of the grain boundary carbides and removing the N from solution as AlN. The hot rolled steels all contained WF but when Nb was added more WF formed as well as MA giving poor impact behaviour. Reducing the hardenability from that shown in previous work by decreasing C from 0.1 to 0.06%, Nb from 0.03 to 0.02%, and cooling rate from 33 to 17K/min had no effect in improving the impact performance of hot rolled Nb steels. To ensure optimum properties not only is it necessary to reduce the hardenability, but WF formation must be discouraged by having a high Ar<sub>3</sub>. This can only be presently achieved by refining the austenite grain size via control rolling the Nb containing steels; the benefit of adding Al can then, readily be seen. Suggestions are made as to how this might be achieved for hot rolling.

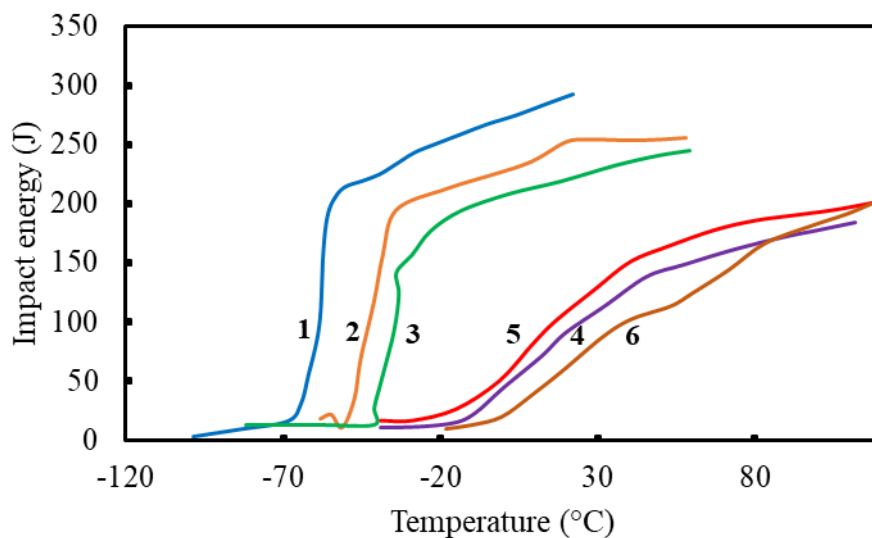
**Keywords:** Hot rolling, high Al additions, Nb, steel, MA, Widmanstätten ferrite, grain boundary carbides, impact and strength

## 1. Introduction

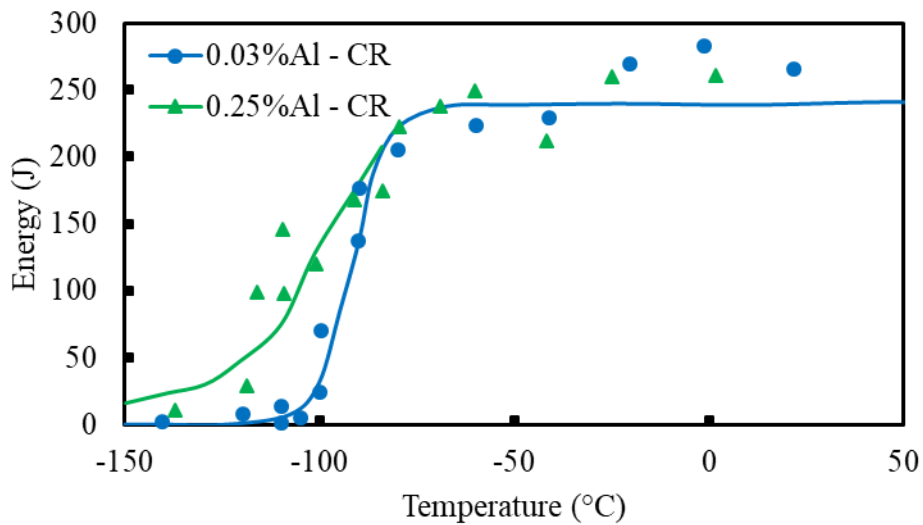
Control rolled steels are characterised by their good mechanical properties and are employed in many exacting engineering applications. However, the cost factor and the unavailability of the control rolling facilities in many of the smaller steel plants make it necessary for research workers to explore alternative options. Hot rolling is cheaper than control rolling but the mechanical properties are poorer mainly because of the coarser grain size, giving inferior impact resistance and lower strengths.

Interest over recent years has focused on using Al additions to improve the impact behaviour of hot rolled low C steels, as previous work [1,2,3] had shown that adding 0.2% Al to a plain C-Mn steel (0.1%C, 1.4%Mn) is beneficial to impact behaviour, the ITT (impact transition temperature) being decreased by 30-40°C. The 0.2% Al addition resulted in only a small fall in strength (~10MPa) but an excellent Impact transition temperature (ITT) of ~ -95°C was obtained. However, the yield strength (LYS) of ~300MPa was too low to replace many of the control rolling specifications. Nevertheless, it should be possible to increase the strength level and still end up with adequate impact performance to meet some of these control rolling specifications at the lower end of the strength spectrum. This can be inferred, from the several examinations that have been carried out to find the effect of strengthening by precipitation hardening on the ITT of ferrite/pearlite low C steels were values of 0.37, 0.35, 0.42, 0.55°C/MPa have been quoted [Refs 4,5,6,7, respectively]. If an average multiplying factor of 0.45°C/MPa is used, then one would expect that a strength increase from 300 to 400MPa would result in an increase of the 27J ITT by ~ 50°C, which is still an acceptable strength/ITT combination for many of the control rolled steels.

A niobium addition was therefore chosen as the means of providing this strengthening, as on hot rolling it gives both precipitation hardening and grain refinement. In the last paper by the authors [3], the Al level in a plain C-Mn steel (0.1%C, 1.4%Mn) was increased from 0.02% to 0.3% resulting in the formation of Widmanstätten ferrite (WF) and bainitic type structures giving poor impact behaviour, worse than given by a normal addition of ~0.02% Al. Adding Nb (0.03%) introduced more WF and martensite/retained austenite (MA) also formed making the impact performance even worse. The impact transition curves for the hot rolled steels from this previous examination [3] are summarised in **Fig. 1** and show a continuous deterioration in the ITT when either Al is increased from 0.02 to 0.30% or Nb is added.



**Fig. 1.** Influence of 0.3% Al and 0.03%Nb additions on the impact transition curves of hot rolled 15mm thick, 0.1%C, 1.4%Mn plate [3]. Curve 1, 0.02%Al, curve 2, 0.3%Al, curve 3, 0.02%Al and Nb, curves 4, 5 and 6, 0.3%Al and Nb.



**Fig. 2.** ITT curves of control rolled Nb containing steel at two Al levels, 0.03 and 0.25%, (30mm thick plate) [3].

In contrast, the finer grained control rolled Nb containing steel, which was free of WF and lower transformation products (LTPs), showed excellent impact properties and the benefit from adding Al was clearly shown, **Fig. 2**.

As a result of these earlier examinations [1-3] the Al level in this work has been reduced from 0.3% to 0.16%, the C level from 0.1% to 0.06% and the Nb from 0.03% to 0.02% in order to reduce the hardenability and hopefully avoid on hot rolling the formation of LTPs.

The extent to which Al and Nb can be used to improve the properties of hot rolled steels has formed the major objective in this paper, the ultimate aim being to obtain mechanical properties similar to those given by the more expensive, control rolled or normalised route, for example API 5L PSL2 X56M line pipe which requires a minimum yield strength of 390MPa and minimum impact energy of 41J at 0°C. In this context the role of Al, the grain boundary carbides, WF and martensite in influencing the mechanical properties has also been studied.

## 2. Experimental

The base composition of the steels was ~0.06%C, 1.4%Mn, 0.5%Si, 0.005%S, 0.005%P, 0.02%Al and 0.008%N to which either ~0.2%Al or ~0.2%Al together with ~0.02%Nb had been added (denoted in many of the tables as plain C-Mn for the base steel with 0.02%Al, and the other two being denoted as Al and Al/Nb, respectively). Two final plate thickness levels were studied for the hot rolled state, 15 and 30mm

gauge, the air cooling rates through the transformation (800-500°C) being 33 and 17 K/min, respectively. The steels were cast as 22kg laboratory vacuum melts when finish rolling at 15mm thick plate or cast as 60kg vacuum melts when rolled to 30mm to ensure that sufficient deformation was available to break down the as cast structure.

For comparison purposes, the steels were also examined after control rolling but in this case, the steels were only rolled to 15mm gauge.

The compositions of the casts, (wt.per.cent.) and the plate thickness are given in **Table 1**.

**Table 1**

Chemical compositions of steels, (wt.per.cent.).

Steel	Type	C	Mn	Si	S	P	Nb	Al	N	Thickness (mm)
H1	HR*	0.051	1.40	0.46	0.004	0.005	-	0.02	0.009	15
H2	HR	0.060	1.40	0.47	0.005	0.005	-	0.16	0.007	15
H3	HR	0.056	1.39	0.46	0.005	0.005	0.018	0.16	0.006	15
C2	CR <sup>+</sup>	0.060	1.39	0.47	0.005	0.005	-	0.16	0.007	15
C3	CR	0.056	1.40	0.46	0.005	0.005	0.020	0.16	0.006	15
H4	HR	0.061	1.39	0.49	0.005	0.004	-	0.02	0.007	30
H5	HR	0.062	1.38	0.49	0.004	0.004	0.018	0.18	0.006	30

HR\* hot rolled      CR<sup>+</sup> control rolled

All the casts were soaked at 1200°C. Steels H1, (0.02%Al), H2, (0.16%Al) and H3, (0.16%Al, 0.02%Nb) were hot rolled to a thickness of 15mm, finish rolling at ~950°C. The plates were then air cooled from 950°C to room temperature; the cooling rate being 33K/min.

Similar steels to H2 (Al) and H3 (Al/Nb) with nominally the same compositions, C2 and C3 were control rolled. For the control rolling process, after rolling the steel to 45 mm thick plate, it was held at 950°C followed by further rolling to 15mm, the finish rolling temperature (FRT) being ~900°C. The steel plates were then left to air-cool.

Because of the poor impact behaviour that was found in these hot rolled 15mm thick plates, two steels, H4 plain C-Mn (~0.02%Al) and H5 (~0.2%Al, 0.02%Nb) in **Table 1** were hot rolled to thicker plate, 30mm and air cooled. This aimed to improve the impact performance by reducing the hardenability further by slowing down the cooling rate through the transformation from 33 to 17K/min, (15 and 30mm thick plate, respectively).

Duplicate tensile specimens were machined from all the plates in the transverse direction and strained to failure using a cross head speed of 0.025cm/min. Standard Charpy V notch impact samples were machined from the hot rolled and control rolled plates in the rolling direction and the ITT curves were established. The volume

fraction of the phases present was measured by point counting and the grain size by the linear intercept method. Samples were prepared for SEM (scanning electron microscope) examination and the grain boundary carbide thickness ( $t$ ) was measured by the method developed by Mintz et al. [4] and the grain boundary carbide density ( $de$ ) by the number of grain boundary carbides intersected in a 20 mm linear traverse. The larger particles  $\geq 0.5\mu\text{m}$  were analysed on the SEM using EDAX analysis (Energy-dispersive X-ray spectroscopy).

### 3. Results

The tensile and impact behaviour are summarised in **Table 2** and the metallographic measurements are given in **Table 3**.

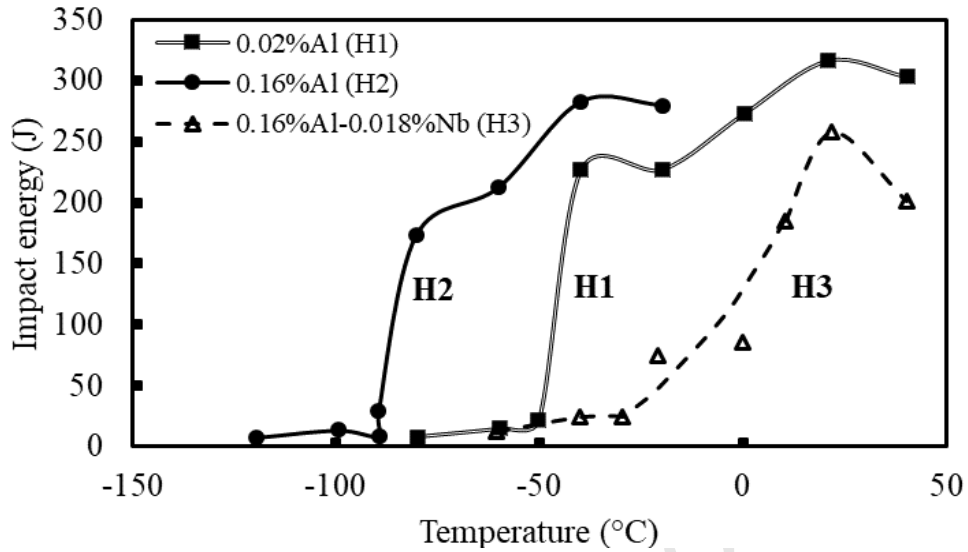
**Table 2**

Summary of the impact and tensile results for hot rolled steels, H1 to H5 and control rolled steels, C2 and C3.

Steel	Type	Type of Rolling	Thickness (mm)	Al (%)	Nb (%)	LYS (MPa)	UTS (MPa)	Elongation (%)	ITT at 54J ( $^{\circ}\text{C}$ )	ITT at 27J ( $^{\circ}\text{C}$ )
H1	<b>C-Mn</b>	HR*	15mm	0.02	-	305	451	38	-48	-50
H2	<b>Al</b>	HR	15mm	0.16	-	293	448	34	-88	-90
H3	<b>Al/Nb</b>	HR	15mm	0.16	0.018	385	539	27	-23	-30
C2	<b>Al</b>	CR <sup>+</sup>	15mm	0.16	-	288	442	38	-90	-90
C3	<b>Al/Nb</b>	CR	15mm	0.16	0.018	389	534	22	-60	-65
H4	<b>C-Mn</b>	HR*	30mm	0.022	-	273	437	42	-58	-58
H5	<b>Al/Nb</b>	HR	30mm	0.17	0.018	343	499	35	-25	-35

HR\* hot rolled      CR<sup>+</sup> control rolled

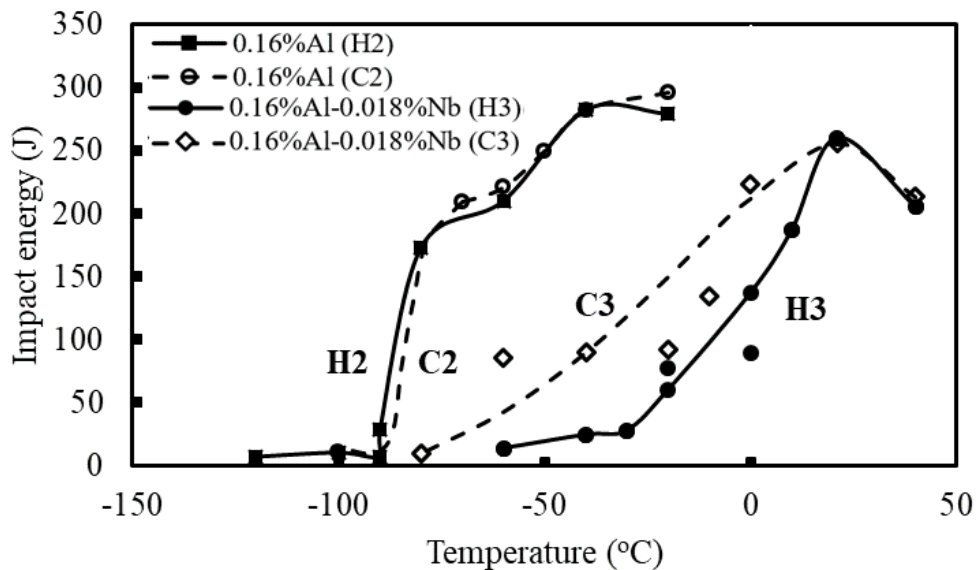
The impact transition curves for the hot rolled 15 mm thick plates, H1, H2 and H3 are shown in **Fig. 3**.



**Fig. 3.** ITT curves for steels, H1, H2 and H3, containing respectively, 0.02%Al, 0.16%Al and combined 0.16%Al and 0.018%Nb.

The addition of 0.16%Al, plate H2, to the base plain C-Mn steel H1, slightly reduced the strength but gave a significant decrease of 40°C to the 27J ITT, **Fig. 3** and **Table 2**, as had been found in previous examinations [1,2].

The ITT curves for the 15mm thick control rolled plates for the high Al steel, C2 and the high Al, Nb containing steel, C3 together with the ITT curves of the hot rolled steel plates of similar composition, H2 and H3, are shown in **Fig. 4**. The plates H3 and C3 having a Nb addition gave the worst impact behaviour of all the steels examined and resulted in a very wide transition temperature range, and a lower ductile shelf energy compared to the Nb free steels, H2 and C2, **Fig. 4**.



**Fig. 4.** Impact transition curves of hot rolled plates H2 and H3 and control rolled plates with similar compositions C2 and C3.

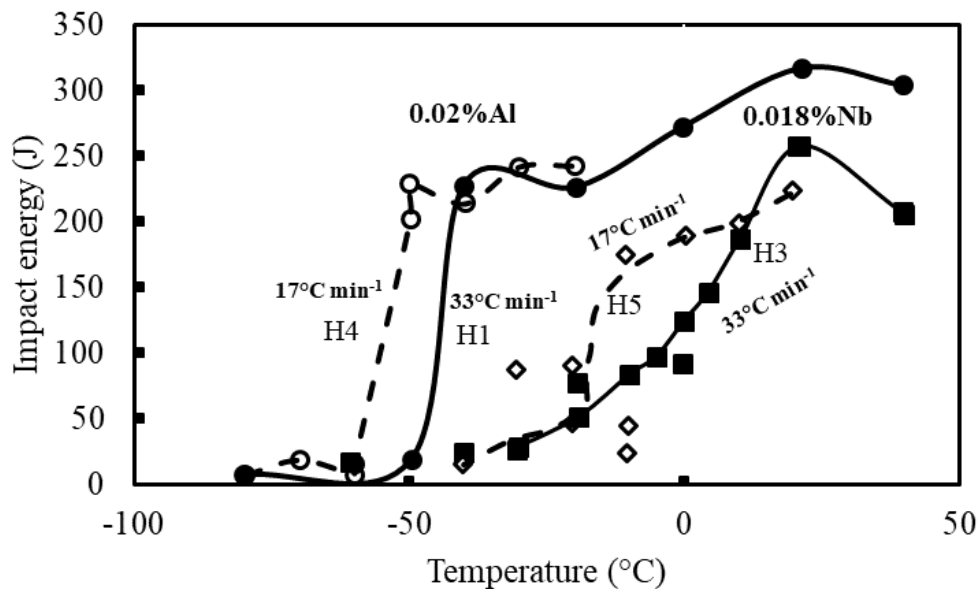


The Nb free high Al steels, H2 and C2 showed little difference in either strength level or impact behaviour between the control rolled and hot rolled plate, **Fig. 4** and **Table 2**. This is to be expected as the grain size was similar, **Table 3**, ( $6.4d^{-1/2}\text{mm}^{-1/2}$  ( $24\mu\text{m}$ ) and  $6.7d^{-1/2}\text{mm}^{-1/2}$  ( $22\mu\text{m}$ ) for H2 and C2, respectively). The similarity in properties between H2 and C2 also suggests that the 0.16% Al addition was likely to be as beneficial to the impact behaviour of the control rolled steel as it had been to the hot rolled steel.

The addition of Nb, to the hot rolled plate H3, brought the strength closer to the desired yield strength of 400MPa, equivalent to the lower end of the control rolled strength spectrum, but the impact behaviour was poor, the 27J ITT being  $-30^\circ\text{C}$ , **Fig. 3** and **Table 2**. In contrast to the high Al steel, grain refinement did take place on control rolling the Nb containing steel, C3, **Table 3**, resulting in a yield strength of 389 MPa and a 27J ITT of  $-75^\circ\text{C}$ .

In the case of the thicker 30mm plates, H4 and H5, the slower air cooling resulted in little change in the ITT curves, the ITT curve being about  $5^\circ\text{C}$  higher than for the slower cooling rate, (15mm thick plates H1 and H3), **Fig. 5**.

The yield strength of the slower cooled plates, H4 and H5 were 30-40MPa, lower than the faster cooled plates, H2 and H3, **Table 2**, most likely due to a coarsening of the Nb(CN) precipitation and the coarser grain size.



**Fig. 5.** Influence of cooling rate on ITT curves. The curves are for the hot rolled low Al (0.02%) steels, H1 and H4, and the Al/Nb steels ( $\sim 0.2\%$  Al and 0.02% Nb), H3 and H5, at the two cooling rates, 17 and 33K/min.

**Table 3**

Microstructural measurements.

Steel	Thick- ness (mm)	Cond- ition	Al (%)	Nb (%)	Cooling rate (K/min)	Grain size ( $\mu\text{m}$ )	Grain size ( $\text{mm}^{-1/2}$ )	Pearlite volume fraction (%)	WF (%)	Grain boundary carbide thickness <b>t</b> ( $\mu\text{m}$ )	Grain boundary carbide density, <b>de</b> ( $\text{N mm}^{-1}$ )
H1	15	HR*	0.02	-	33	24.4	6.4	4.8	0.75	0.25	16.2
H2	15	HR	0.16	-	33	23.7	6.5	7.9	0.30	0.21	14.1
H3	15	HR	0.16	0.02	33	14.9	8.2	4.1	1.7	0.30	18.9
C2	15	CR <sup>+</sup>	0.16	-	33	22.3	6.7	7.7	0.25	0.20	14.3
C3	15	CR	0.16	0.02	33	12.6	8.9	5.0	1.3	0.225	21.8
H4	30	HR	0.02	-	17	26.9	6.1	4.6	0.6	0.27	15.4
H5	30	HR	0.16	0.02	17	18.3	7.4	4.0	1.4	0.32	17.1

\* Pearlite includes WF    HR\* hot rolled    CR<sup>+</sup> control rolled

Relative errors of measurements: grain size 2%, Pearlite volume fraction 2.0-2.7%, grain boundary carbide thickness 7% and grain boundary carbide density 7%, WF 10% [8].

The hot rolled steel plates, H1 to H5, all contained some WF the greatest amount being when Al and Nb were both present together, see H3 and H5, 1.7% and 1.4% WF, respectively, **Table 3**.

The grain size was coarse for all the hot rolled plates, H1 to H5, ( $\sim 6\text{-}8\text{mm}^{-1/2}$ ) but adding Nb did lead to some grain refinement, by  $\sim 1.3\text{mm}^{-1/2}$ , compare H3 and H5 with all the other Nb free steels, H1, H2 and H4 in **Table 3**.

Grain boundary carbide thickness varied over a relatively narrow range from 0.2 to  $0.32\mu\text{m}$ , **Table 3**. The addition of 0.16%Al to the hot rolled steel, H1 and H2, refined the carbide thickness and reduced the carbide density both by a small amount. In contrast adding Nb coarsened the carbides and increased the carbide density, (compare H1 with H3, **Table 3**).

Control rolling led to a finer grain size for the Nb containing steel, (compare C3,  $12.6\mu\text{m}$  with H3,  $14.9\mu\text{m}$  in **Table 3**) but not the refinement one would normally expect, (typical grain size on control rolling is in range  $5\text{-}7\mu\text{m}$ ). However, no grain refinement occurred on increasing the Al level from 0.02 to 0.16%, either on hot or control rolling (compare C2 with H2, **Table 3**).

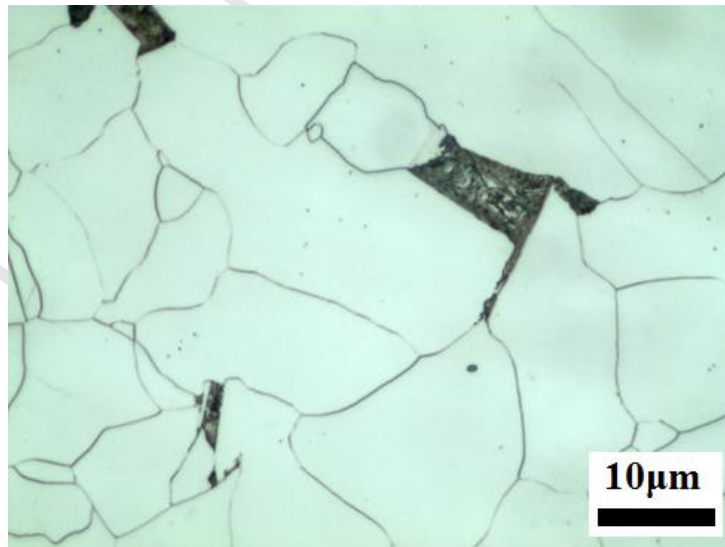
### Micrographs

A selection of micrographs for the plates, after hot and control rolling, are shown in **Figs. 6-10** taken using the optical microscope (OM) and scanning electron microscope (SEM).

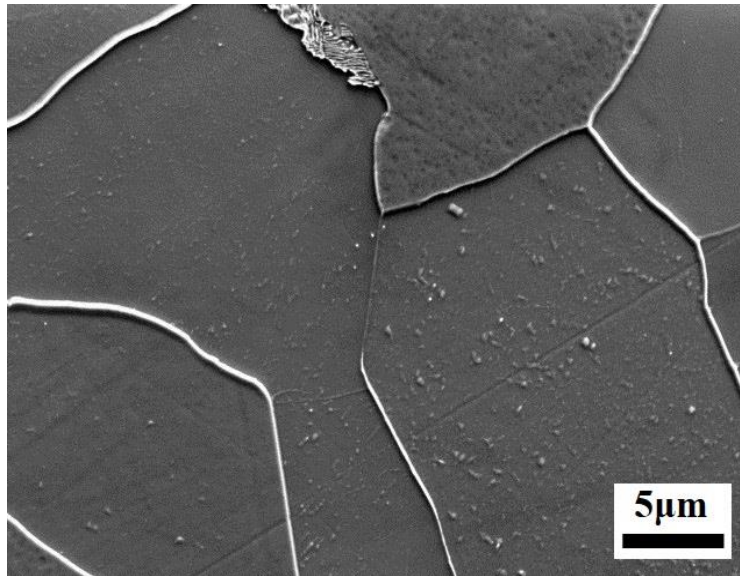
The microstructures of the hot and control rolled plates were all ostensibly ferrite/pearlite (>95%). The coarse particles in the micrographs  $\geq 0.5\mu\text{m}$  in size, shown in for example **Figs. 6b** and **7b** were identified as MnS inclusions with AlN precipitation often at the peripheries of the inclusions.

Adding Nb resulted in grain refinement and a mixed grain structure.

The micrographs selected fitted into two groups. The first group H1, H2, C2 and H4 where for Nb free plates with either 0.02 or ~0.2%Al and were characterised by having small amounts of WF (<1% **Table 3**) but no MA, **Figs. 6-8**. The other group H3, H5 and C3, all Nb containing steels contained both WF (>1%, **Table 3**) and MA, **Figs. 9** and **10**. The control rolled 15mm thick plate, C3 was not immune to the presence of either WF or MA, **Figs. 10a** and **b** but did have smaller amounts than the hot rolled plates, **Figs. 9a, b** and **c**. MA was found to be present in the slower cooled hot rolled plate H5, **Fig. 9c** but a much smaller amount was present than in the faster cooled plate H3, **Fig. 9a**. This made it more difficult to detect but because of its presence the impact behaviour remained poor, the ITT for both plates, H3 and H5, being -30 to -35°C, **Fig. 5** and **Table 2**.

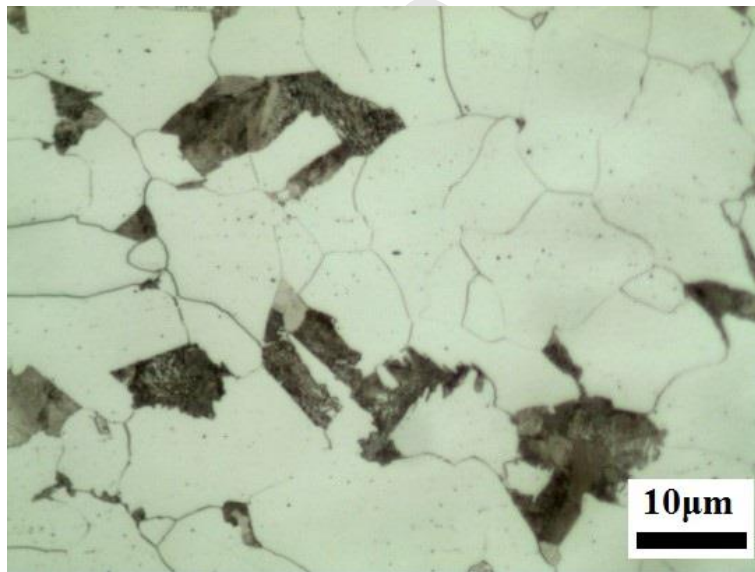


(a)

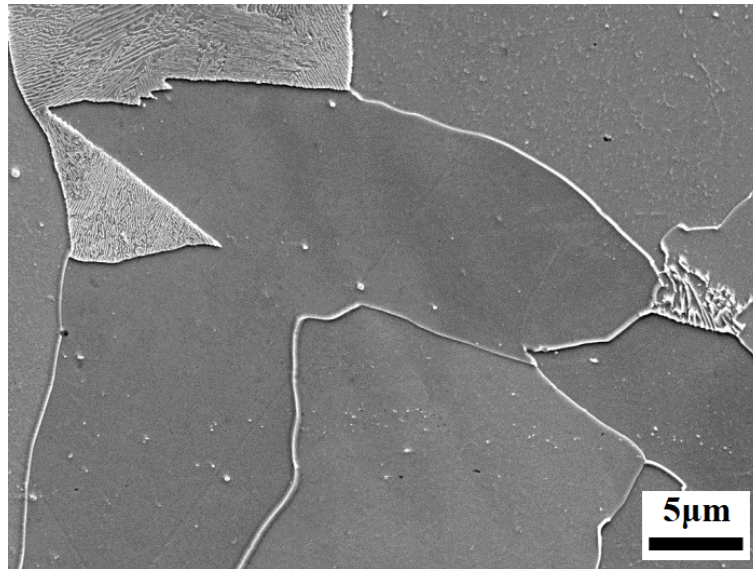


(b)

**Fig. 6.** Hot rolled microstructure of plate H1, the base plain C-Mn steel, (0.02% Al) showing evidence of a small amount of WF (0.75% WF) but no MA. (a) OM (b) SEM.

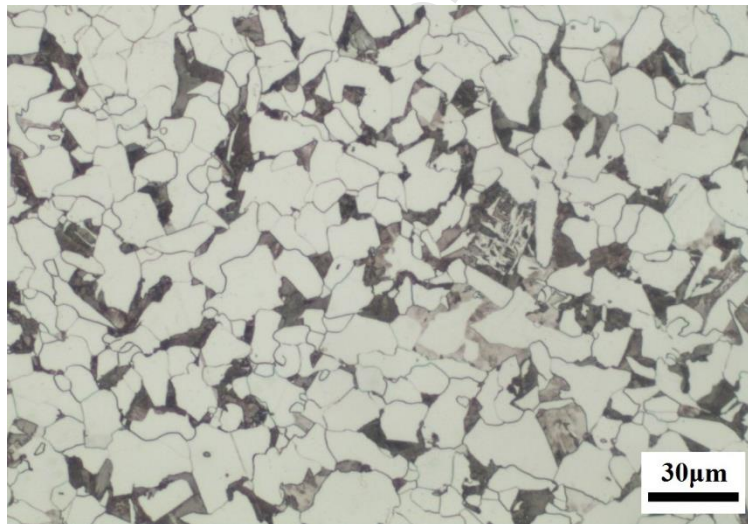


(a)

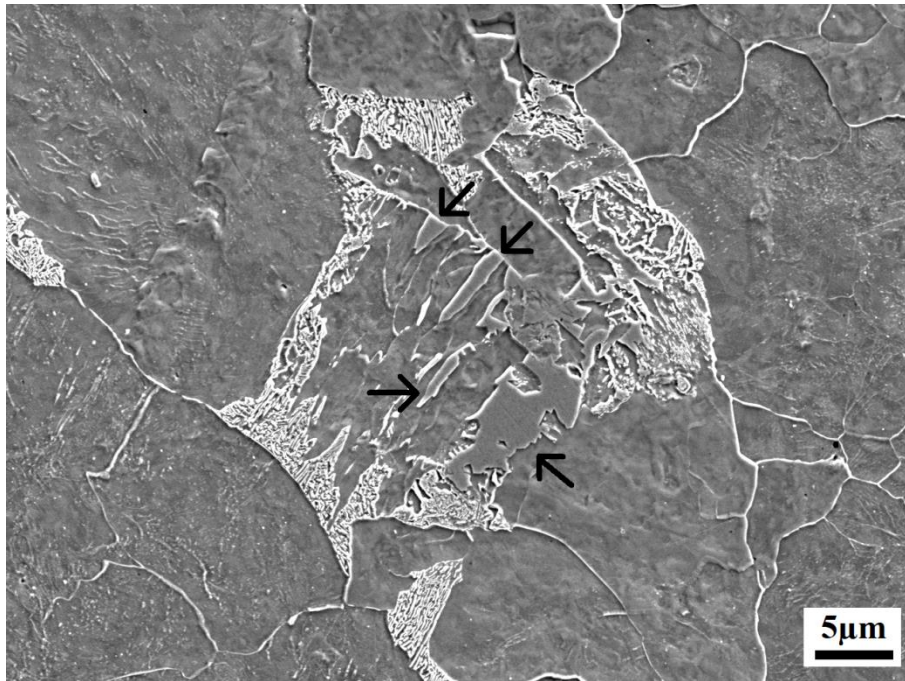


(b)

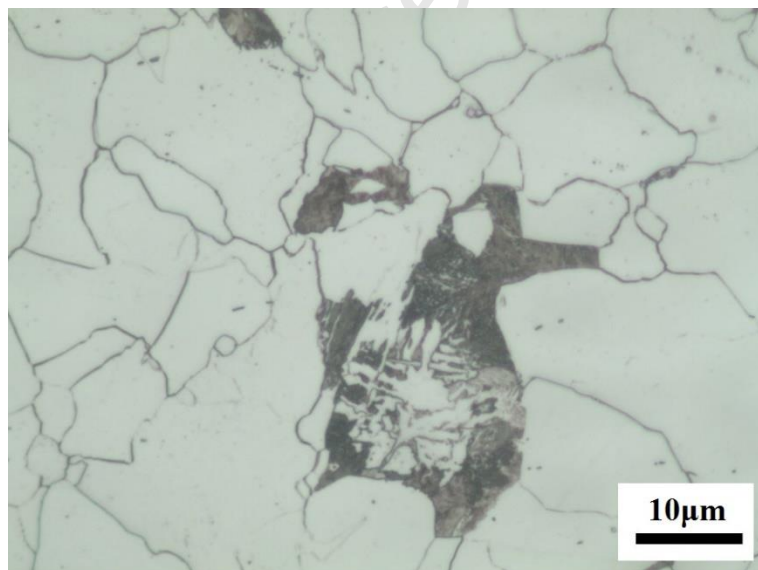
**Fig. 7.** Control rolled plate C2 having 0.16% Al. Essentially ferrite-pearlite but saw cuts are noticeable. The amount of WF was 0.25%. (a) OM (b) SEM.



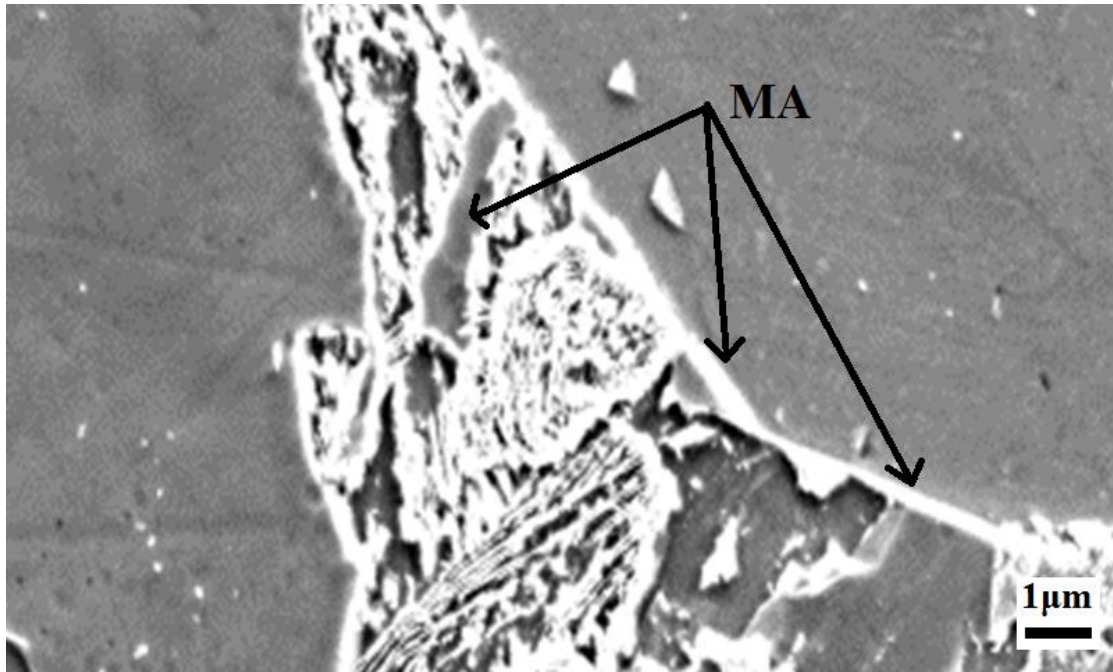
**Fig. 8.** WF is still present in the more slowly cooled low Al (0.02%) thicker 30mm plate, H4, (0.6% WF) but MA was not observed.



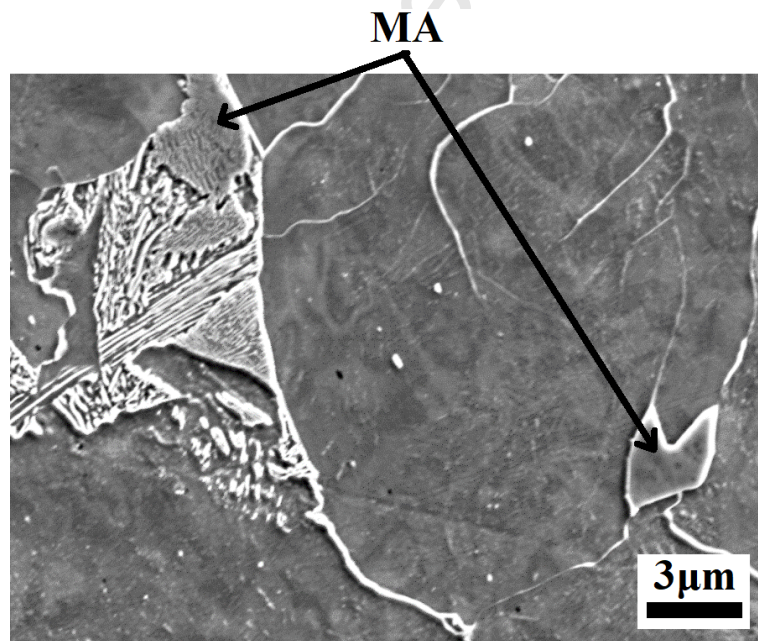
**Fig. 9a.** Presence of Nb leads to more WF (1.7%) and islands of MA. Plate H3. Arrows mark the regions where MA is present.



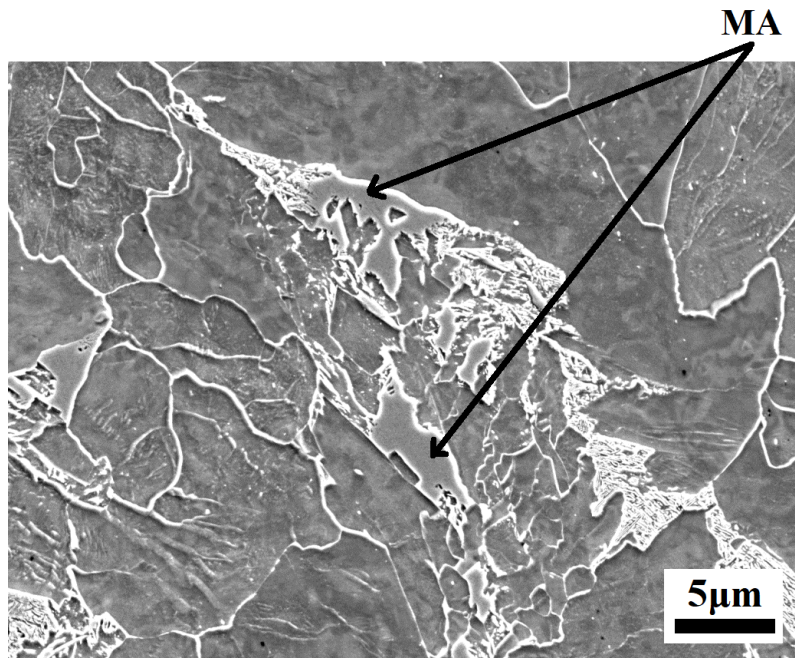
**Fig. 9b.** Nb containing plate, H5 showing WF (OM). The presence of WF is more obvious here (1.4%) but a very small amount of MA was also detected in the structure of this slower cooled plate, Fig.9c.



**Fig. 9c.** Very small islands of MA detected in plate H5 compared to the larger more numerous islands found in the faster cooled plate H3.



(a)

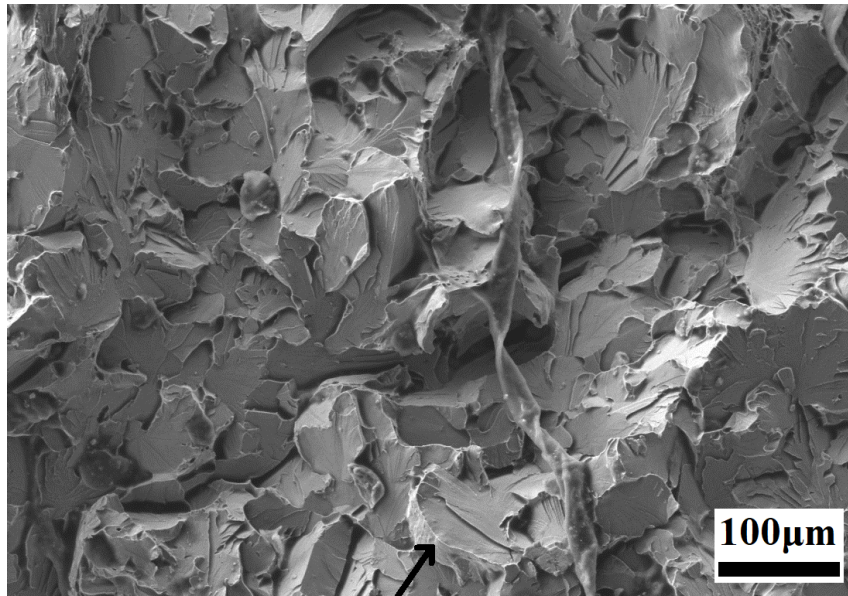


(b)

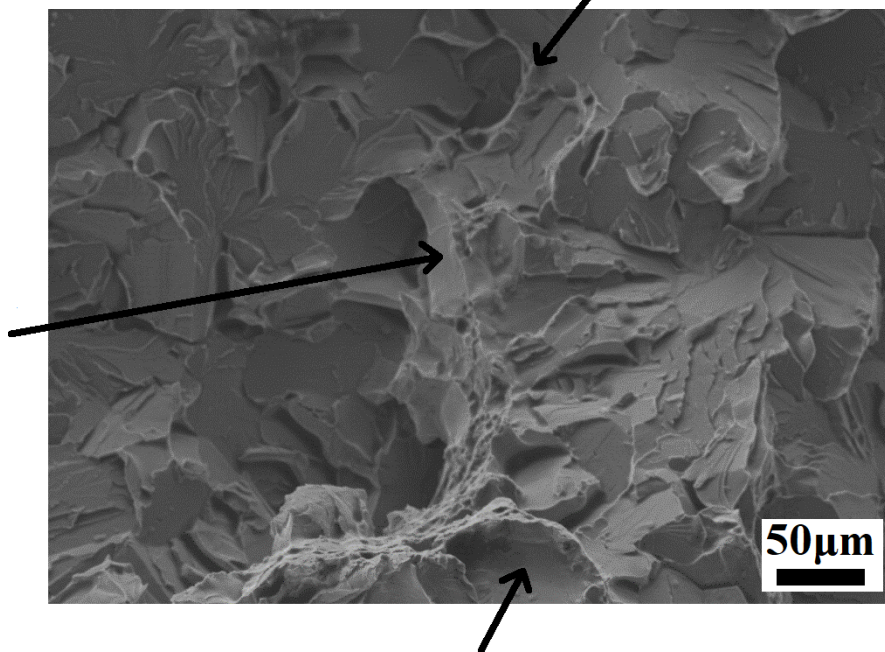
**Fig. 10a. and b.** Control rolled high Al/Nb containing steel, C3 showing examples of regions with WF (1.3%) and MA. The plate thickness is 15mm.

The fracture surfaces of the Charpy samples for the two 30mm thick plates, H4 and H5 tested at temperatures on their brittle shelves at  $-70^{\circ}\text{C}$  and  $-40^{\circ}\text{C}$ , respectively, are shown in **Figs. 11a** and **b**. They were examined under the SEM to see whether they were normal cleavage or whether there was evidence of any intergranular failure which might be caused by coarse carbides or MA at the grain boundaries. The fractures, **Fig. 11a** and **b** were mainly normal brittle cleavage failures but "circular" regions on the fracture surface did possibly indicate some intergranular passage of the cracks (arrowed in figures).





(a)

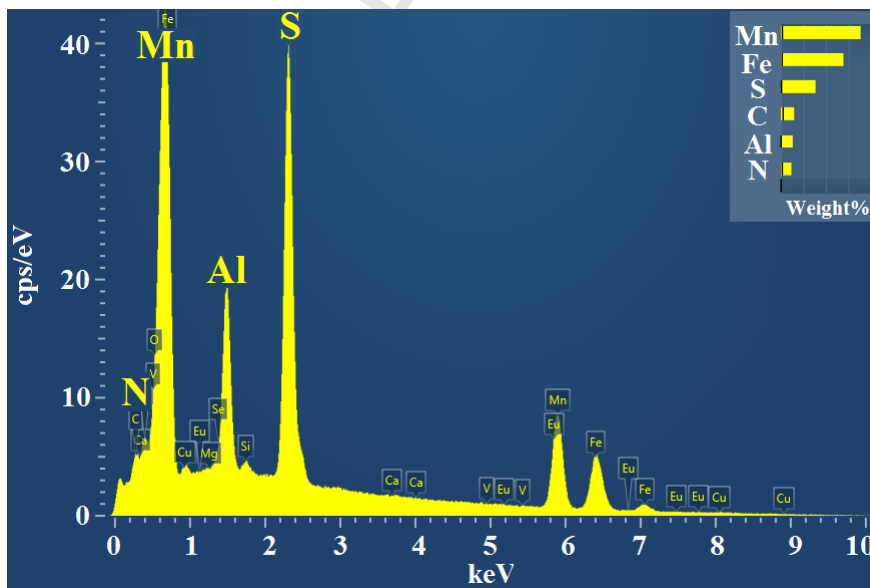
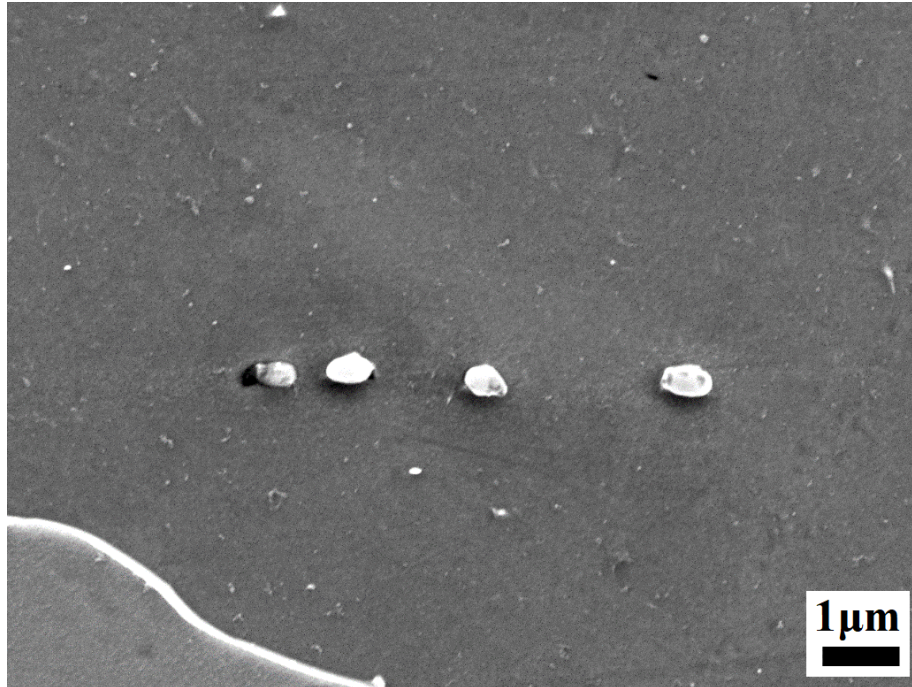


(b)

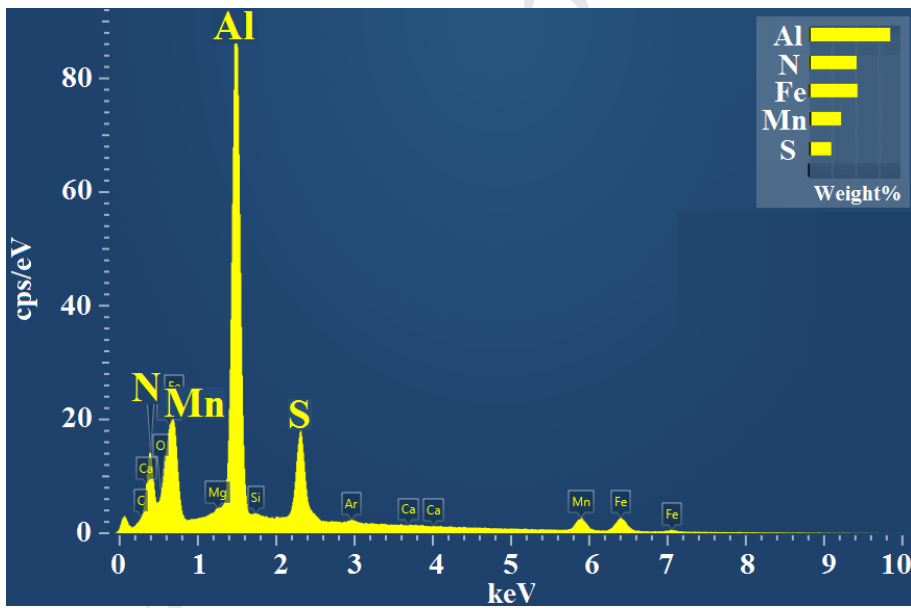
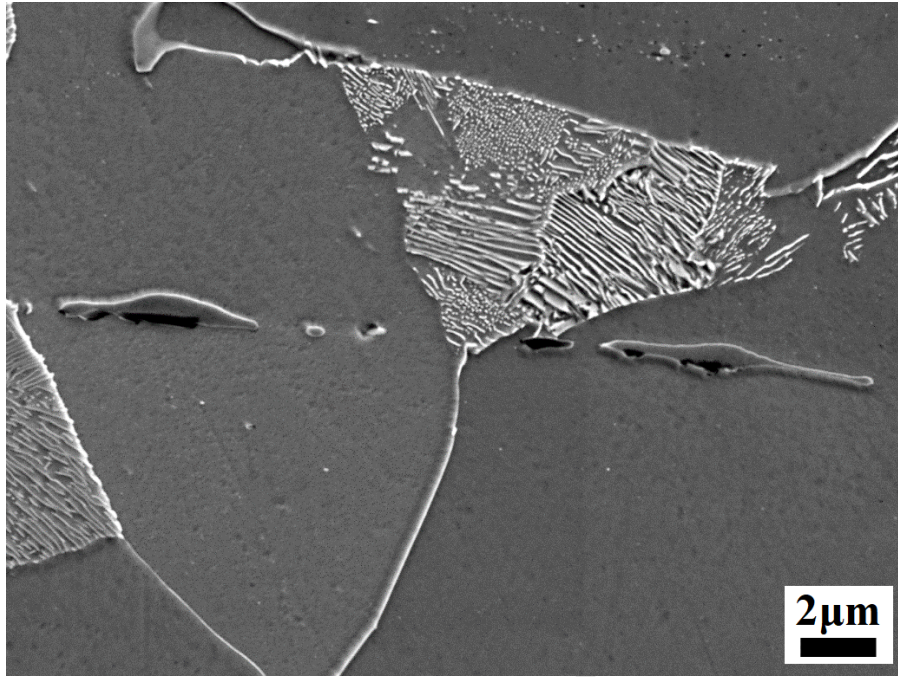
**Fig. 11.** Fracture surface of the hot rolled 30mm thick plate (a) Nb/Al, H5 tested at  $-40^{\circ}\text{C}$ , and (b) low Al, H4 tested at  $-70^{\circ}\text{C}$ .

The coarse particles in the matrix were identified as MnS and AlN. Quite often the particles were combined AlN and MnS particles, **Fig. 12a** and **12b**. The black areas on the micrographs are AlN and the grey areas are the MnS inclusions. It has been shown that AlN has difficulty in precipitating out from the austenite and MnS inclusions offer themselves as suitable nucleation sites [9, 10]. The AlN precipitates

were usually at the periphery of the MnS inclusions and their more hexagonal nature does not become noticeable until they have grown outside the inclusion when they are more developed as is found in the higher Al, as-cast (1.5%Al) TWIP steels [10], **Fig. 13.**



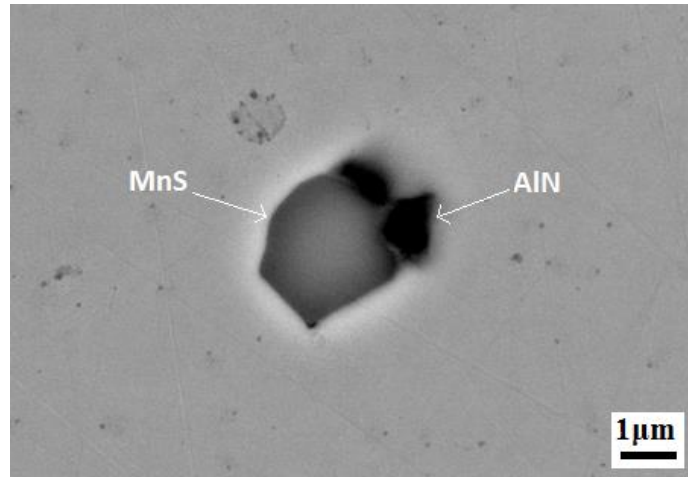
Typical Spectrum analysis	N Weight %	Al Weight%	S Weight %	Mn Weight %
MnS/AlN	4.66	5	15	35.0
MnS/AlN	5.48	11	8.0	19.0



(b)

Typical Spectrum Analysis	N Weight %	Al Weight %	S Weight %	Mn Weight %
MnS/AlN	24	37	12	16.0
AlN	27	42	-	-

**Fig. 12.** Typical particles and their analysis (a) Particles were manganese sulphides and AlN, and (b) Elongated MnS inclusions combined with AlN. The micrographs (a) and (b) were taken from plate C2.



**Fig. 13.** AlN precipitating on a MnS inclusion in an as cast TWIP steel [10].

#### 4. Discussion

##### Analysis of results

In examining the strength and impact results it is normal to analyse them using empirical regression equations that have been developed for plain C-Mn and HSLA (high strength low alloy) steels. Any significant difference in the actual measured results with those predicted by these equations then warrants further investigation to establish the cause. The following two equations [4] have been chosen because they were developed specifically for hot rolled steels:

$$\text{LYS (MPa) for plain C-Mn steels} = 105 + 43.1\% \text{Mn} + 83\% \text{Si} + 1540N_{\text{free}} + 15.4d^{-1/2}$$

**(Equation 1)**

$$27J \text{ ITT } ^\circ\text{C} = 173t^{1/2} - 8.3d^{-1/2} + 0.37\Delta p - C$$

**(Equation 2)**

Where  $N_{\text{free}}$  is the N in solution,  $d$  the grain diameter in  $\mu\text{m}$ ,  $t$  is the grain boundary carbide thickness in  $\mu\text{m}$  and  $\Delta p$  is the precipitation hardening contribution in MPa and  $C$  is a constant.

The constant in equation (2) was originally  $-42^\circ\text{C}$  (Ref.4, 1979) but as steelmaking practice has improved over the years particularly with reducing the S level [3] the constant has decreased to  $-85^\circ\text{C}$ . The precipitation hardening component  $\Delta p$  is taken as the Actual LYS - Predicted LYS for plain C-Mn steels from equation 1.

Using Leslie et al's solubility equation [11] for AlN the amount of free N at  $1200^\circ\text{C}$  can be calculated and is given in **Table 4**.

**Table 4**

The amount of free N and N combined as AlN at 1200°C.

Steel	Al (%)	N <sub>total</sub> (%)	N combined as AlN (%)	Free N at 1200°C (%)
H1*	0.02	0.009	0.0022	0.0068
H2	0.16	0.007	0.0052	0.0018
H3	0.16	0.006	0.0042	0.0018
C2 <sup>+</sup>	0.16	0.007	0.0052	0.0018
C3	0.16	0.006	0.0042	0.0018
H4	0.022	0.007	0.0028	0.0042
H5	0.17	0.007	0.0053	0.0017

\*H1-H5 hot rolled    <sup>+</sup>C2-C3 control rolled.

Leslie et.al solubility equation [11] predicts there will be for the plain C-Mn steel plates, H1 and H4, ~0.007 and 0.004%N in solution, respectively at 1200°C. It is generally believed that at these normally added low Al additions (0.02-0.04%Al), because the precipitation of AlN is very sluggish [9], the N in solution at 1200°C will not be precipitated out as AlN on hot rolling and cooling to room temperature. The values calculated in **Table 4**, therefore, have been used for the N in solution at room temperature in all calculations.

For the other plates with the higher Al content, ~0.16%Al, the driving force for precipitation may be enough to precipitate out the remaining N in solution on cooling to room temperature. In addition the presence of Nb(CN) in the Nb containing steels is also likely to take a very small amount of N out of solution. Nevertheless, as one cannot confirm this, for the purpose of all calculations the free N has again been taken as that calculated at 1200°C, i.e. 0.0018%. (The N in solution is difficult to analyse unless sophisticated techniques such as internal friction are used).

Using these equations (1) and (2), the following **Table 5** for the predicted LYS and 27J ITT can then be calculated and compared with the actual experimental values.

**Table 5**

Predicted and Actual LYS and 27J ITT°C.

Steel	Al (%)	N (%)	Nb (%)	t <sup>+</sup> (μm)	Δp* (MPa)	Predicted LYS (MPa)	Actual LYS (MPa)	Predicted 27J, ITT (°C)	Actual 27J, ITT (°C)
H1	0.02	0.009	-	0.25	-8	312.6	305	-55	-50
H2	0.16	0.007	-	0.21	-14	307.2	293	-65	-90
H3	0.16	0.006	0.018	0.30	53	332.1	385	-36	-30
C2	0.16	0.007	-	0.20	-22	310.3	288	-71	-90
C3	0.16	0.006	0.018	0.23	46	342.9	389	-64	-65
H4	0.022	0.007	-	0.27	-33	305.9	273	-57	-60
H5	0.17	0.007	0.018	0.32	21	321.7	343	-41	-35

P\* precipitation hardening t<sup>+</sup> carbide thickness Δ

The measured LYS of the Nb free, 15mm thick plates, H1, H2, C2 and H4 is ~300MPa whilst the Nb containing steels, H3 and C3 were considerably higher ~385MPa. The higher LYSs of the Nb containing 15mm thick plates are due to both precipitation hardening and grain refinement as shown in **Tables 2** and **5**.

The 0.16%Al addition to the plain C-Mn steel, plates H2 and C2 can be seen to reduce Δp by ~15MPa, **Table 5**, most likely due to N removal (discussed later in the paper).

For the Nb containing steels, the precipitation hardening contribution to strength from Nb(CN) for the 15 mm air-cooled plates, H3 and C3 is similar on hot rolling as to when control rolling, ~50MPa, **Table 5**. This is reduced at the slower cooling rate to ~20MPa, plate H5, **Table 5**. The grain refinement benefit to strength for these Nb containing 15mm thick plates {~1.3d<sup>-1/2</sup>mm<sup>-1/2</sup> x 15.4 from equation (1)} contributes a further ~20MPa to the yield strength. Nb due to its powerful grain refining ability in the control rolled containing steel has a lower ITT than the hot rolled steel of similar composition, ~20°C lower, **Tables 2** and **3**. The only other microstructural factors that are different between the Nb free and Nb containing steels are the amounts of WF and LTPs. Whereas the Nb free steels have very small amounts of WF and have no MA, adding Nb encourages more WF and in addition MA forms, suggesting that the impact behaviour would be better if a solely ferrite/pearlite structure could be achieved.

Again, the plates can be divided into those that have less than 1% WF, plates H1, H2, H4 and C2 and those that have ≥1% WF, H3, H5 and C3, all of which have Nb as an addition and have MA, **Table 3**. The predicted ITT (P) and experimental ITT (E) for the Nb free and Nb containing steels are given in **Table 6** and **7**, respectively. For the plain C-Mn steels H1 and H4, **Table 6** the predicted ITT (P) is close to experimental

(E), (5 and  $-3^{\circ}\text{C}$  difference) but the other two plates show the beneficial influence of Al giving  $\sim 20^{\circ}\text{C}$  lower ITT, (E-P of  $-25$  and  $-19^{\circ}\text{C}$ ) and this is without materially influencing the strength, **Table 6**.

**Table 6**

Predicted and Experimental 27J ITT $^{\circ}\text{C}$  for Nb free steels with Al

Steel	Type	LYS MPa	Predicted(P) ITT $^{\circ}\text{C}$	Experimental(E) ITT $^{\circ}\text{C}$	Difference E-P $^{\circ}\text{C}$	WF %
H1	C-Mn	305	-55	-50	5	0.75
H4	C-Mn	273	-57	-60	-3	0.60
H2	Al	293	-65	-90	-25	0.30
C2	Al	288	-71	-90	-19	0.25

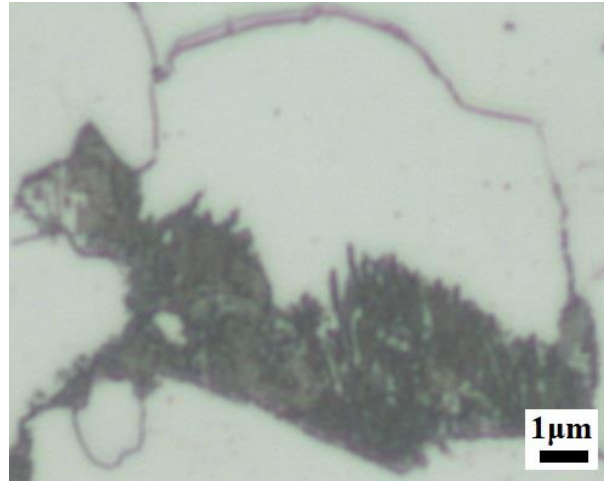
The predicted and experimental ITTs are given together with the volume fraction of WF in **Table 7** for the Nb containing plates, H3, H5 and C3. On adding Nb, the WF percentage volume fraction increases to  $\geq 1\%$  and there is always MA, so any benefit from the Al addition is masked (E-P is  $-5$  to  $6^{\circ}\text{C}$ , **Table 7**).

**Table 7**

Predicted and Experimental 27J ITT $^{\circ}\text{C}$  for Nb containing steels.

Plate	Type	Predicted (P) ITT $^{\circ}\text{C}$	Experimental (E) ITT $^{\circ}\text{C}$	E-P $^{\circ}\text{C}$	WF %
H3	Al/Nb	-36	-30	6	1.7
H5	Al/Nb	-41	-35	6	1.4
C3	Al/Nb	-60	-65	-5	1.3

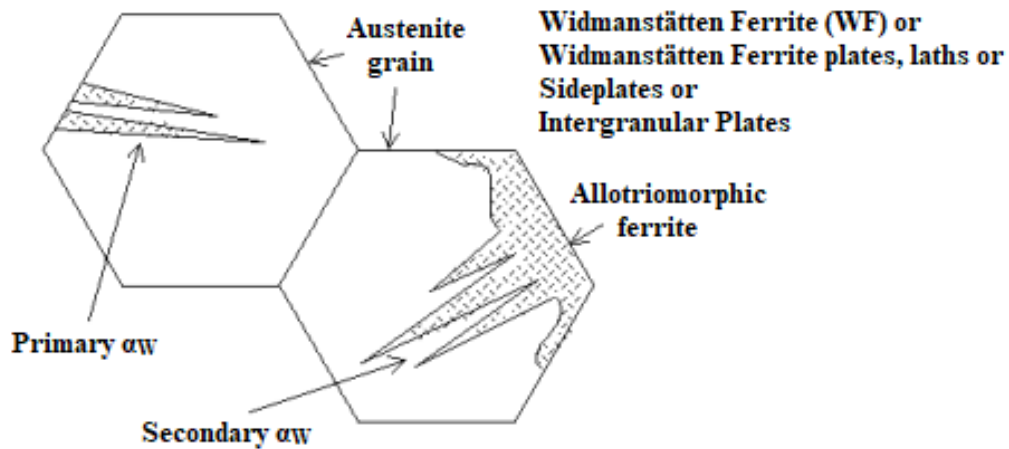
It is clear from this analysis, that WF and the LTPs are associated with and may be responsible for the poor impact behaviour shown by the Nb containing hot rolled steels and the role of these phases in influencing the properties needs to be explored. In the present instance, "upper" bainite is also present in some of the steels. **Fig. 14** and this too is detrimental to impact behaviour because of the presence of coarse carbides, so that WF, MA and "upper" bainite may all in part be responsible for the poorer than expected impact behaviour of the hot rolled Nb containing steels.



**Fig. 14.** Bainitic carbides protruding into the ferrite interior in plate H5.

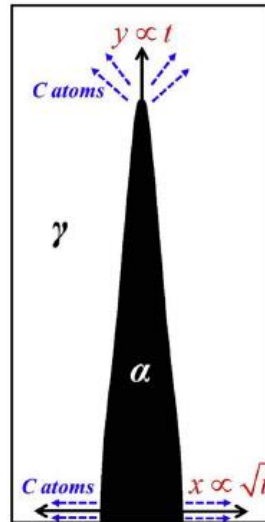
#### Importance of Widmanstätten ferrite

Depending on the cooling rate, the first phase precipitated out from the austenite on cooling, is either granular allotriomorphic ferrite or WF. When the austenite grain size is sufficiently coarse and the cooling rate is high enough, primary WF forms,  $\alpha_W$ , and grows directly from the austenite grain surfaces [12]. Secondary WF develops from any allotriomorphic ferrite, that may be present in the microstructure, **Fig. 15**.



**Fig. 15.** Various forms of WF [12].



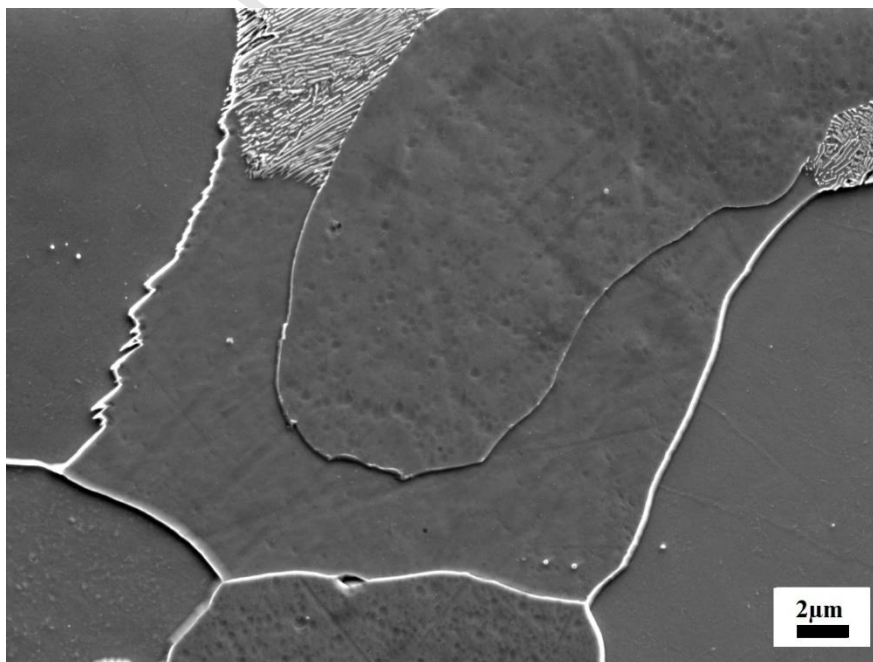


**Fig. 16.** The growth features of the acicular WF precipitate at the X-axis and Y-axis, where  $t$  is the time [13].

WF can form at temperatures close to the  $Ae_3$  temperature and hence can occur at very low driving forces; the undercooling needed amounts to a free energy change of only  $50\text{ J mol}^{-1}$ . This is much less than required to sustain a diffusionless transformation [14].

Two models have been proposed to account for its formation [12,15].

Bhadeshia [14] has suggested that it is formed by a shear transformation as with martensite but is more complex requiring a cooperative shear mode. This may account for the jerkiness noted in the boundaries in the present work when it is not able to develop easily, **Fig. 17**.



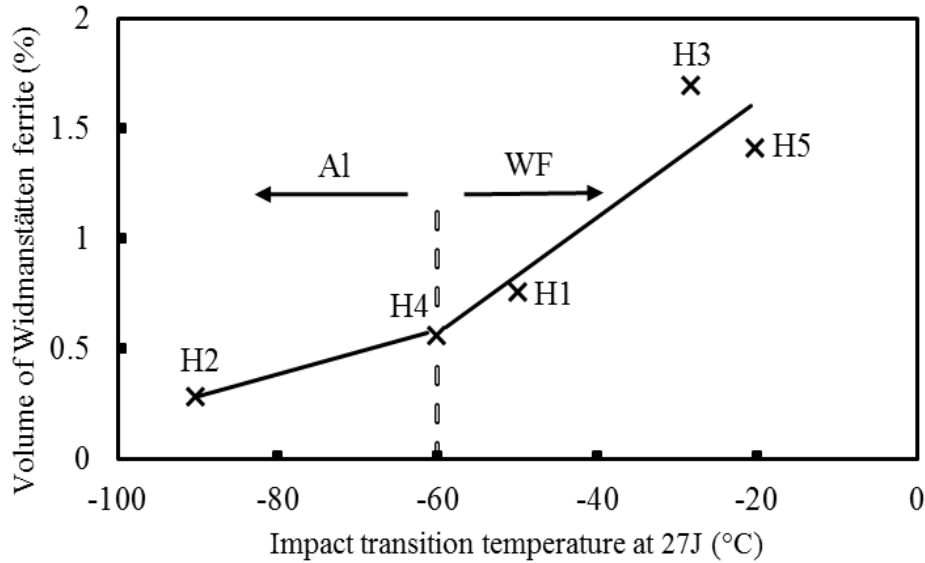
**Fig. 17.** Zig zag boundary possibly indicating the onset of WF formation.

Although martensite which is definitely formed by a shear transformation (displacive) and WF which may also be, the cooling rate needed to form them from a fixed composition and coarse grain size is different. WF although probably displacive in itself, needs carbon to diffuse out of the ferrite [14] before it can take place and is therefore slower than the martensite transformation, even though the transformation takes place at higher temperatures.

In the second model (reconstructive) Aaronson [15] suggests that WF is formed by a diffusional mechanism with the migration of ledges on the broad face of the  $\alpha/\gamma$  interface and this is supported by the experimental work of Phelan and Dippenaar [16]. Ohmori has reviewed all the phases that can form from the austenite when the carbon is precipitated out [17] and concludes that either theory using a displacive or reconstructive model can be used to explain most of the experimental results.

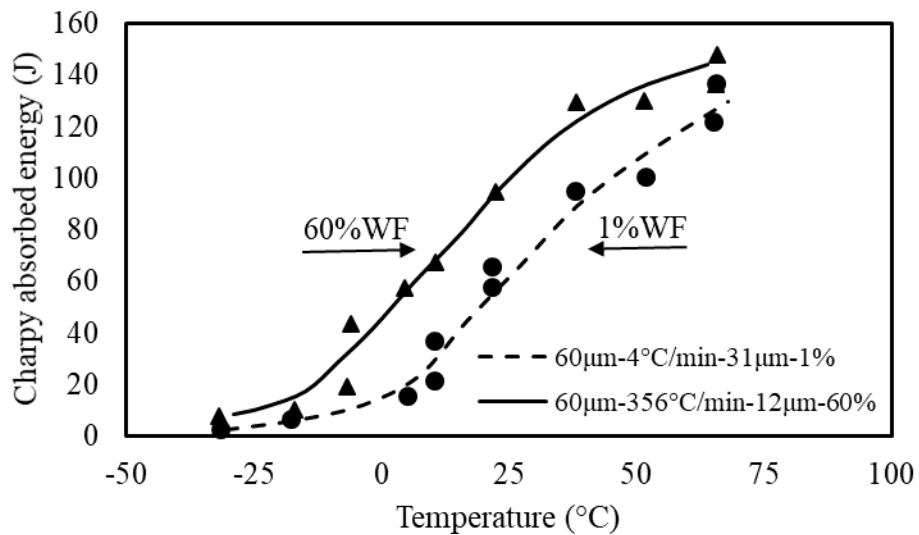
In the present work, WF was observed to be present without MA in the hot rolled plates, H2 and H4. This suggests that the cooling rate required to form WF is slower than that needed to form MA, as might be expected. Bodnar and Hansen [18] have shown that it is possible to obtain up to 60% WF without MA forming and the impact behaviour is hardly affected. Only, when the hardenability is increased by the addition of Nb or there is a higher Al content as shown in previous work [1] MA can form. Todorov and Khristov [19] note that the presence and dispersion of WF depends on the C content, size of the austenite grain size and the cooling rate. It is suggested that there is a critical austenite grain size below which it will not form [19]. Similarly for C, the amount formed depends on the cooling rate and austenite grain size. For an austenite grain size of 80-90 $\mu$ m slow furnace cooling resulted in the normal ferrite/pearlite microstructure but with air cooling (80-100K/min) WF formed and the amount increased as the C level increased from zero, reached a maximum of 60% at 0.1%C and decreased to zero as the C level reached 0.5%C [19].

However, in the present work the impact behaviour in contrast to that shown by Bodnar and Hansen [18] appears to deteriorate markedly as the amount of WF colonies increases **Fig. 18**.

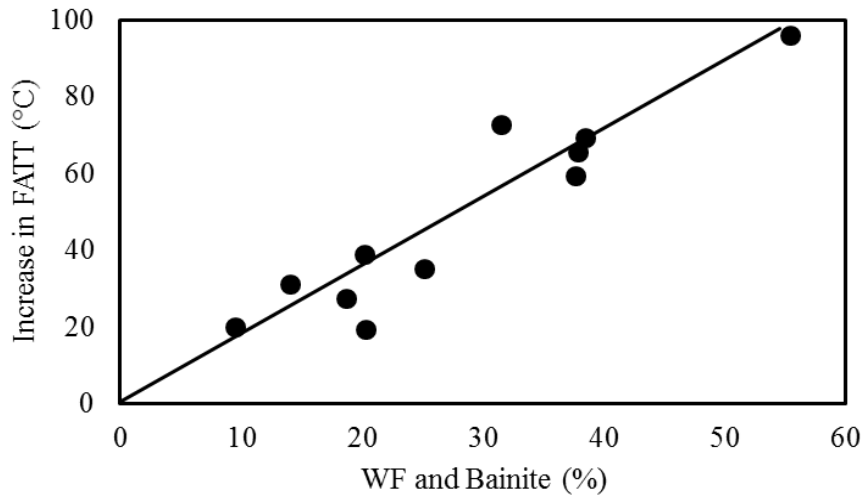


**Fig. 18.** Apparent influence of Widmanstätten ferrite on the impact behaviour of the hot rolled steels examined.

Unfortunately, the effect of WF on impact behavior is unclear from the literature [18,20]. Bodnar and Hansen [18] as mentioned found 60% WF hardly influenced the impact behavior of plain C-Mn steels, **Fig. 19a** whilst Morrison and Preston [20] found it detrimental when produced in Nb containing steels, **Fig. 19b**.



**Fig. 19a.** ITT curves for an 0.2%C, 0.7%Mn steel having the same  $\gamma$  grain size of  $60\mu\text{m}$  cooled at two different cooling rates, 4 and  $\sim 360^\circ\text{C}/\text{min}$  showing little change in ITTs although WF changes from 1 to 60% [18].



**Fig. 19b.** Influence of Widmanstätten ferrite or bainite on the ITT of a Nb containing steel. 1%WF increases ITT by 2°C [20].

Morrison and Prestons' work [20] show that at a coarse ferrite grain size of 14µm in a 0.15%C 1.4%Mn 0.028%Nb steel, increasing the WF from 32 to 53% substantially reduced the toughness. Their multiplying factor for WFs influence on the ITT was 1%WF increases the ITT by ~2°C, **Fig. 19b**. This is similar to the multiplying factor for pearlite of 2.2% [21] and is much too low for the present work where 1% WF appears to have an order of magnitude worse effect on the ITT, **Fig. 18**. Hence the poor impact behavior of the hot rolled steels cannot be ascribed to WF alone. In the present exercise although it would appear that WF is detrimental to impact behavior it is probably MA which is the major culprit as Bodnar and Hansen [18] have suggested to account for Morrison and Prestons' results [20]. The cause of the poor impact behavior may not be WF in itself but that when WF forms there is this build-up of carbon at the peripheries of the colonies, **Fig. 16**, favouring coarse grain boundary carbides or MA. Thus the more WF there is, the more likely a region with martensite or thicker colony carbide boundaries can form and the statistically probability of crack formation are increased. Nb encourages both WF and martensite. Al in contrast reduces the volume fraction of WF but because it prevents the precipitation of Fe<sub>3</sub>C in bainitic ferrite it also increases the C content of the austenite favouring the presence of MA [22]. This prevention of the precipitation of Fe<sub>3</sub>C probably accounts for Al being able to refine the grain boundary carbides.

It is interesting to note that Bodnar and Hansen have shown examining the fracture path that the cracks often go along the allotriomorphic grain boundaries of the WF [18] where coarse grain boundary carbides or MA can form. Just examining the Charpy specimens fracture surfaces as in the present work, **Figs. 11a** and **b** was not sufficient to confirm this but their examination of the mode of fracture was more detailed than the present one.

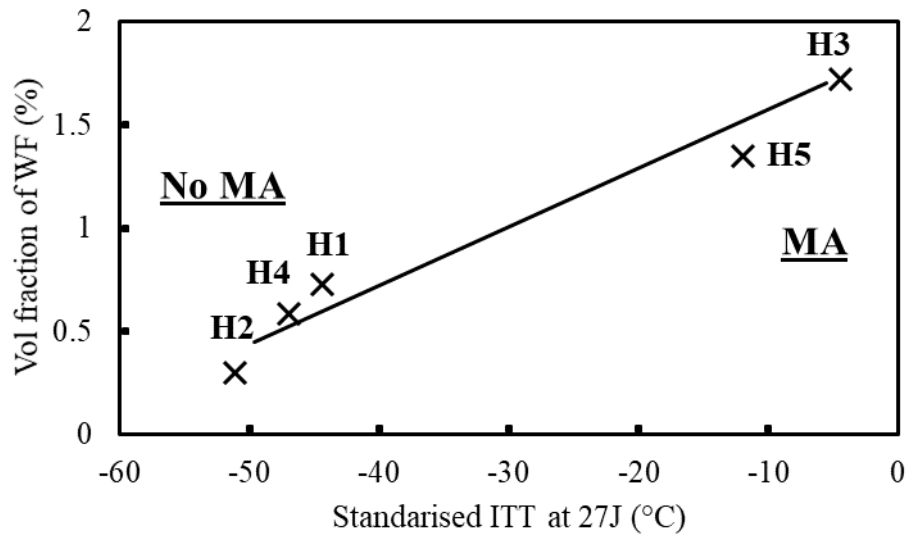
In the present work, the impact behaviour seems to change radically when the volume fraction of WF changes from 1 to 2%, **Fig. 18**. This figure can indeed be modified to isolate the influence of only WF on the impact behavior by normalizing all the hot rolled data to a grain size of  $d^{-12} = 6\text{mm}^{-1/2}$  and zero precipitation hardening using equation (2) and removing the beneficial effect of Al by subtracting 30°C (the average improvement from this work and the previous examinations [1-3]) for all steels containing 0.16%Al. This indicates that although there is an apparent relationship of the impact behaviour with the volume fraction of WF, given the normal scatter in measurement, there may be only two points on the curve, a point for steels without MA and a point for steels with MA (the Nb containing steels). It is most likely that it is the MA rather than the WF that causes the impact behavior to deteriorate. It also appears that only a very small amount of MA is needed to seriously impair the impact behaviour, **Fig. 9c** and once this is achieved further amounts make little material difference. It is suggested that the WF alone will not influence impact behavior more than pearlite does but if the hardenability is increased by adding Nb or increasing the Al to ~0.5%, MA can form and then impact behaviour rapidly deteriorates [23]. The reason as to why increasing the Al level to 0.3%Al caused the impact behaviour to deteriorate was not explored in that examination [3] and the boundaries were not examined for evidence of very small martensite colonies.

Even though the higher Al additions ( $\geq 0.5\%$  Al) have led to martensite formation on hot rolling [23], the lower addition ~0.2% to the plain C-Mn steel, plate H2, did not result in MA forming and in consequence gave better impact behaviour. Although after hot rolling, a small amount of WF formed, **Fig. 18**, this had no influence on the impact behaviour, **Fig. 3** and the benefit from adding Al was observed. A niobium addition, in contrast, in these hot rolled steels, always resulted in having more WF but importantly MA constituents form and it is the latter which may be responsible for the poor impact behaviour rather than the WF, **Fig. 3**. Whereas Al raises the transformation temperatures, the  $A_{r3}$  and  $M_s$ , encouraging polygonal ferrite and making it harder for martensite to form [24,25], Nb, lowers them, encouraging both WF and martensite formation [25,26,27].

The various non equilibrium structures, WF, bainite and martensite all have a major influence on the tensile and impact performance. Martensite in particular can easily cause brittle failure and will often cause pre-yielding of the ferrite removing the yield point. However, there is so little MA present in these steels that pre-yielding was not observed.

Probably what is more serious is that when WF forms, the austenite surrounding the acicular WF becomes rich in carbon, **Fig. 16** and depending on the cooling rate, poor impact behaviour occurs. This arises because there is a build-up of carbon at the tips of the ferrite needles as well as at the allotriomorphic ferrite boundaries producing MA, **Fig. 16** [13].

Evidence of the early stages of WF formation is shown by the unusual, zig zag boundaries, **Fig. 17** and the saw tooth appearance shown in **Figs. 7a** and **b**.



**Fig. 20.** WF, (volume fraction %) vs 27J ITT°C, standardized to  $d^{-1/2} = 6\text{mm}^{-1/2}$ ,  $\Delta p = 0$  and the absence of any benefit from Al for the hot rolled plates.

The finding of MA constituents even in the control rolled Nb containing steel, **Figs. 10a** and **b** also suggests that the control rolled steel is not reaching its full potential, with an ITT of  $-65^{\circ}\text{C}$  and strength level of 389MPa, and the benefit from the addition of Al is being masked by the MA.

In the present exercise it is possible to have WF without martensite giving good properties, H2 but as soon as MA constituents appear the impact behavior deteriorates probably even in a control rolled steel, C3, **Figs. 10a** and **b**. This may be because the austenite grain size is more critical for WF formation than it is for martensite; the more grain boundaries the easier it is to form granular polygonal ferrite rather than WF.

Bodnar [28] has also shown using a dilatometry simulation of hot rolling that the volume fraction of WF increases with cooling rate and austenite grain size.

The influence of WF on strength is also not clear. WF has been shown to have after hot rolling [18] a low density of dislocations similar to that found in polygonal ferrite. In itself, the LYS is then unlikely to be that different from when polygonal ferrite is present particularly at these coarse grain sizes. A finer austenite grain size leads to a finer polygonal ferrite grain size as well as a finer WF grain size. Thus, the strength will increase as the grain size refines and the LYS will be essentially controlled by the polygonal ferrite grain size [18]. The presence of martensite can cause pre-yielding but the small amount less than 0.25% present in these steels is so low that no pre-yielding was noted.

Previous work [3] has shown that a ~0.2%Al addition is beneficial to impact behaviour for both hot rolled plain C-Mn steels as well as control rolled Nb containing steels, **Fig. 2**. The hot rolled Nb containing steels may be indeed, showing the benefit of adding Al but its influence is being masked by the presence of MA.

#### Role of MA and grain boundary carbides

MA is a two phase structure of martensite and retained austenite and is regarded as the most deleterious combination as the martensite is the very brittle high C twinned martensite. During cooling from the austenite, the austenite transforms to ferrite and as with WF this causes a build-up of carbon in the remaining austenite and depending on the grain size, cooling rate and composition MA can form. Pipeline steels can be particularly prone for this to happen and centre line segregation of Mn during casting must be avoided. Welding can also be a problem as the coarse grained HAZ, (heat affected zone) is susceptible to the formation of MA [30-33].

Nb containing steels are particularly prone to its formation [32] and the form it is in is important, whether it is granular or lenticular, the latter being particularly detrimental presumably as it is possible to crack more easily [27].

With grain boundary carbides, the range of thickness that has been experimentally observed is ~0.2 $\mu\text{m}$  to ~0.8 $\mu\text{m}$  [4] and this can be seen from equation (2) to give an increase in the ITT of 75°C. In Nb containing steels in the present work, the acicular MA regions are also about 1 $\mu\text{m}$  in thickness, **Fig. 9** and the ITT has again increased by a similar amount compared to the ITT of the Nb free steels (compare in **Fig. 3**, the ITT curve of H2 with that of H3). It is often difficult to tell the difference between coarse carbides or MA at the boundaries using the SEM but as far as their influence on the impact behaviour is concerned it has been shown to make little influence [29]. The carbides also set off the pearlite reaction which when WF can form at higher temperatures leads to coarser carbides. In many cases, the pearlite reaction seems to start and end prematurely as it runs out of carbon, **Fig. 17**.

#### Importance of Ar<sub>3</sub> temperature

Reducing the C, Nb levels and the cooling rate from that of previous work [3] made little difference to the impact behaviour suggesting that hardenability is perhaps not the only concern for improving the properties, rather it may be as to whether WF can form and help trigger off the formation of MA. The austenite grain size seems very important as to whether these phases form and the cooling rate and grain size needed are likely to be different; MA requiring a faster cooling rate to freeze in the C in solution. With WF, the C has to diffuse away from the advancing acicular ferrite plate, **Fig. 16**. This becomes easier the more grain boundaries there are and the more favoured will be polygonal ferrite rather than WF and the more grain boundaries will reduce the carbon gradient. Yang and Bhadeshia [34] have also shown that in a 0.12%C, 2.3%Mn, 5%Ni steel the M<sub>s</sub> temperature decreases as the grain size is refined. This is the opposite behaviour to the Ar<sub>3</sub> which increases as the grain size is

refined. It seems likely from the evidence that increasing the  $A_{r3}$  temperature is as important as the hardenability for avoiding WF and MA. The higher the  $A_{r3}$  the more likely polygonal ferrite will form rather than WF.

Mintz et al. [24] have obtained a regression equation for steels with similar composition to those under examination for the  $A_{r3}$  (un-deformed) temperature as follows:

$$A_{r3} (^{\circ}\text{C}) = 868 - 181\%C - 75.8\%Mn + 1086\%S - 1767\%Nb - 0.0933CRt - 3799\%N_{\text{free}}$$

Where CRt is the cooling rate in K/min. From this equation, it can be seen that Nb has a very big influence in lowering the  $A_{r3}$ . A decrease in Nb content by 0.010% would from this equation raise the  $A_{r3}$  temperature by 17-18 $^{\circ}\text{C}$ . The change in C level from 0.1% to 0.06% would raise the  $A_{r3}$  by a smaller amount, 9 $^{\circ}\text{C}$  and this might be the reason as to why reducing the carbon from that of previous examinations [1-3] did not have such a big influence in improving properties. Also reducing the cooling rate from 33 to 17K/min will from this equation have only a very small influence on the  $A_{r3}$ . All this fits in well with the experimental observations. Al was not included in this analysis but a more limited linear regression analysis on a smaller sample size specifically including many TRIP steels with Al additions up to 2% gave a multiplying factor of +18.1 for Al for its influence on the  $A_{r3}$  [20]. This would mean that, reducing the Al from 0.3 to 0.16% will decrease the  $A_{r3}$  by 3 $^{\circ}\text{C}$  but if it takes the N out of solution this could restore the balance, for example if there is 0.008%N in solution, removing that amount would raise the  $A_{r3}$  by ~25 $^{\circ}\text{C}$ .

Again using a smaller sample size, the influence of austenite grain size on the  $A_{r3}$  was also established:

$$A_{r3} (^{\circ}\text{C}) = 833.6 - 190.6\%C - 67.4\%Mn + 1522\%S - 2296N_{\text{free}} - 1532\%Nb + 7.91 d^{1/2} - 0.117CRt. [20].$$

A change of austenite grain size from 100 $\mu\text{m}$  to 20 $\mu\text{m}$ , the range normally covered in hot rolling (higher end of range) and control rolling (lower end of range) processes, would from this equation increase the  $A_{r3}$  substantially by 32 $^{\circ}\text{C}$ . Thus, the benefit of the finer grain size from control rolling is very apparent.

It seems likely that both hardenability and the  $A_{r3}$  temperature may be important in preventing MA from forming in these steels. If the  $A_{r3}$  is low WF can form more easily. If the hardenability is not sufficiently high, only WF forms and this has only a relatively small influence on the impact and strength. If WF forms and the hardenability is high then MA forms at the peripheries of the WF.

Although there has been little success in improving the impact behaviour of hot rolled Nb containing steels, adding ~0.2%Al to the finer grained Nb control rolled steels in which the structure was entirely ferrite/pearlite did result in the expected improvement in ITT, **Fig. 2**.



Although ensuring that WF and MA are avoided to attain the optimum properties, the benefit of adding Al in improving impact performance is the major concern in the paper and refinement of the grain boundary carbides and N removal seems to be the most likely cause for any improvement [3].

Importance of grain boundary carbide thickness (**t**) and carbide density (**de**) on the impact behaviour.

The two microstructural features, grain boundary carbide thickness and density have been found to be important in influencing brittle failure [4, 35, 36]. Thicker carbides provide a wider crack length, and the density defines the statistical chance of finding a grain boundary carbide which is thick enough to crack and propagate into the ferrite and cause brittle failure. An increase in the number of grain boundary carbides intersected per mm in a linear traverse by 20 has been shown to raise the ITT by  $\sim 26^{\circ}\text{C}$  [3]. Of the two carbide thickness and density, the thickness has been found to be more important. For the ranges covered in the present examination, **Table 3**, a change from 0.2 to  $0.32\mu\text{m}$ , increases the ITT by  $21^{\circ}\text{C}$ , (equation 2) and for the carbide density, a change from 14 to 22/mm would result in an increase of  $\sim 10^{\circ}\text{C}$ . The last two columns in **Table 8** give the calculated benefit for both **t** and **de**.

**Table 8**

Influence of Al on the carbide thickness (**t**) and density (**de**) and on the ITT.

Plate	Rolling	Type	Al (%)	Cooling Rate (K/min)	Grain Size ( $\mu\text{m}$ )	Grain Size ( $\text{mm}^{-1/2}$ )	<b>t</b> ( $\mu\text{m}$ )	<b>de</b> (N/mm)	Change in ITT due to <b>t</b> ( $^{\circ}\text{C}$ )	Change in ITT due to <b>de</b> ( $^{\circ}\text{C}$ )
H1	HR	C-Mn	0.02	33	24.4	6.4	0.25	16.2	-	-
H2	HR	Al	0.16	33	23.7	6.5	0.21	14.1	-7	-3

It can be seen from **Table 8** that adding 0.16%Al, H2 to a plain C-Mn steel, refines the carbide thickness, **t** from 0.25 to  $0.21\mu\text{m}$  and decreases the carbide density, **de** from 16.2 to 14.1/mm. This would result in a total decrease in the ITT of  $\sim 10^{\circ}\text{C}$ .

Influence of Nb on the carbide thickness (**t**) and density (**de**) on the ITT

In **Table 9**, as has been found in previous work [36], Nb additions cause both an increase in the thickness as well as density (compare H3 with H2). The total change in ITT on adding Nb, due solely to its influence on carbide formation, is to increase the ITT by 10 to  $20^{\circ}\text{C}$  as shown in **Table 9**.

**Table 9**

Influence of Nb on carbide thickness (**t**) and density (**de**) and their influence on ITT.

Plate	Rolling	Type	Grain Size ( $\mu\text{m}$ )	Grain Size ( $\text{mm}^{-1/2}$ )	<b>t</b> ( $\mu\text{m}$ )	<b>de</b> (N/mm)	Change in ITT due to <b>t</b> ( $^{\circ}\text{C}$ )	Change in ITT due to <b>de</b> ( $^{\circ}\text{C}$ )	Total Change in ITT due to carbides ( $^{\circ}\text{C}$ )
H2	HR	Al	23.7	6.5	0.21	14.1	-	-	-
H3	HR	Al/Nb	14.9	8.2	0.30	18.9	16	5	21
H4	HR	C-Mn	26.9	6.1	0.27	15.4	-	-	-
H5	HR	Al/Nb	18.3	7.4	0.32	17.1	8	2	10
C2	CR	Al	22.3	6.7	0.20	14.3	-	-	-
C3	CR	Al/Nb	12.6	8.9	0.23	21.8	6	10	13

Control rolled plates because of their finer grain size will have both a higher carbide density and finer grain boundary carbides, **Table 10** [35]. The similar grain size for H2 and C2 result in no significant change in the impact behaviour due to the grain boundary carbides. When grain refinement does occur, the finer grain size of C3 compared to H3 results in finer carbides and in consequence, better impact behaviour. However, the differences are small, the total change in the ITT due to the finer carbides being  $-8^{\circ}\text{C}$ , **Table 10**.

**Table 10**

Influence of control rolling on carbide thickness (**t**) and density (**de**) on the impact behaviour.

Plate	Rolling	Type	Carbide Thickness <b>t</b> ( $\mu\text{m}$ )	Carbide Density <b>de</b> (N/mm)	Grain Size ( $\mu\text{m}$ )	Grain Size ( $\text{mm}^{-1/2}$ )	Cooling Rate (K/min)	Change in ITT due to <b>t</b> ( $^{\circ}\text{C}$ )	Change in ITT due to <b>de</b> ( $^{\circ}\text{C}$ )
H2	HR	Al	0.21	14.1	23.7	6.5	33	-	-
C2	CR	Al	0.20	14.3	22.3	6.7	33	-2	-
H3	HR	Al/Nb	0.30	18.9	14.9	8.2	33	-	-
C3	CR	Al/Nb	0.23	21.8	12.6	8.9	33	-12	4

Finally, for the influence of cooling rate, the slower cooled plates H4 and H5, gave coarser carbides and fewer of them, **Table 11**.

However, the change in ITT due to carbide thickness and density on altering the cooling rate can be seen from **Table 11**, to be too small ( $2^{\circ}\text{C}$ ) to have a significant influence on the impact behaviour.

**Table 11**

Influence of cooling rate on the carbide thickness (**t**) and density (**de**) and impact behaviour.

Plate	Carbide Thickness <b>t</b> ( $\mu\text{m}$ )	Carbide Density <b>de</b> (N/mm)	$\Delta p$ (MPa)	Grain Size ( $\mu\text{m}$ )	Grain size $d^{-1/2}$ ( $\text{mm}^{-1/2}$ )	Cooling Rate (K/min)	Change in ITT due to <b>t</b> ( $^{\circ}\text{C}$ )	Change in ITT due to <b>de</b> ( $^{\circ}\text{C}$ )
H1 C-Mn	0.25	16.2	-8	24.4	6.4	33	-	-
H4 C-Mn	0.27	15.4	-33	26.9	6.1	17	3.5	-1
H3 Al/Nb	0.30	18.9	53	14.9	8.2	33	-	-
H5 Al/Nb	0.32	17.1	21	18.3	7.4	17	3.0	-2

### Importance of N

Although an Al addition, improves the impact behaviour of C-Mn and C-Mn-Nb steels by refining the grain boundary carbides, the ITT decreasing by  $\sim 10^{\circ}\text{C}$ , (compare H1 with H2) **Table 8**, this is unlikely to be the main cause of the  $\sim 30^{\circ}\text{C}$  improvement ( $40^{\circ}\text{C}$  if not accounting for the fall in yield strength). The possibility of Al behaving like Si and reducing the  $k_y$  value in the Hall-Petch yield strength relationship, so making it easier for dislocations to be generated from grain boundaries preventing their build up has also been shown not to occur [36, 37]. Therefore, removing the N from solution as AlN is the most likely reason for the remaining improvement in the impact behaviour. The removal of 0.001%N from solution by Al has been shown to reduce the ITT by  $2.75^{\circ}\text{C}$  [37] and thus removal of 0.008%N by Al in the current steels would reduce the ITT by  $\sim 22^{\circ}\text{C}$ , while the refinement of grain boundary carbide thickness and reduction in their numbers would also give rise to a further fall in the ITT of  $10^{\circ}\text{C}$ , making a total fall in ITT of  $32^{\circ}\text{C}$ .

The small influence of adding Al in reducing the strength is less easy to explain. Al at the level examined does not grain refine in these hot rolled steels but Al can take the N out of solution.

Morrison et al [38] have reviewed all the research work that has been carried out on the effect of free N on the lower yield strength and concluded that an addition of 0.001% free N to steels increases the yield strength by  $\sim 5\text{MPa}$ . Hence, for the high Al containing steel (steel H2) the removal of N (0.007% at  $1200^{\circ}\text{C}$ ) to form AlN would [38] reduce the yield strength by  $\sim 35\text{MPa}$ . However, the solid solution hardening effect of Al will increase the yield strength by a small amount, 12MPa (1%Al has been shown to increase the yield strength by 70MPa, [23]) so that the strength loss

should not be greater than 23MPa. This fits in reasonably well with the average value of  $\Delta P$  of -18MPa that is shown in **Table 5** for H2 and C2.

### Role of Al and Nb

The hot rolled Al/Nb containing plate, H3, achieved the highest strength level among all tested steels but the increase in strength by 100MPa resulted in the impact behaviour markedly deteriorating as shown in **Fig. 3**. The considerable improvement in the strength, **Table 2** has been shown to be due to a combination of precipitation hardening and grain refinement, **Table 3**.

In the Nb free steels, on increasing the Al content from 0.02% to 0.16 %, the benefit of adding Al is clearly shown, the ITT decreasing by about 40°C. Grain refinement during hot deformation is observed in the hot rolled Al/Nb containing steel plates H3 and H5, **Table 3**, and this improves both strength and toughness but the formation of WF and MA impairs the impact behaviour.

This improvement on adding Al is also noticeable in Al/Nb containing steels when control rolling is used provided the austenite grain size is fine enough to prevent both WF and MA from forming. In contrast, for the coarser grained hot rolled plates, Nb causes the impact behaviour to deteriorate sharply, **Fig. 3**, despite the great advantage of Nb to strength. It is known that both high Al additions and micro-alloying additions of Nb can encourage the formation of martensite. Bhadeshia [32] has shown that Nb containing steels are particularly exposed to martensite formation leading to the creation of local brittle zones and thus influencing overall toughness.

Crowther [39] has also studied the influence of Al and Nb and the rolling schedule on the mechanical properties of control and hot rolled steels. In his work, a lower FRT of 800°C was used so that the ferrite grain size at room temperature was finer, 7 $\mu$ m rather than the ~12 $\mu$ m as in this work and more typical of the fine grain size normally associated with control rolled steels. The plate thickness in that study was 30mm and the C level higher at 0.11%. The finer grained control rolled steel, (7 $\mu$ m), gave no WF and MA resulting in excellent properties whereas for the present paper, the hot rolled steel H3, which had WF and MA in the microstructure and a coarse ferrite grain size of ~12 $\mu$ m gave rise to poor impact behaviour.

Two steels, H7 and C4 taken from his work [39], with similar compositions are compared with the current steels containing Nb, H3 and C3 in **Table 12**.

**Table 12**

Comparison of the mechanical properties between hot rolled and control rolled plates when the control rolled steel has MA present and when it is absent.

Steel	Rolling schedule	C (%)	Al (%)	Nb (%)	LYS (MPa)	Elongation (%)	27J, ITT (°C)	$d^{-1/2}$ (mm <sup>-1/2</sup> )
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H3	Hot rolling	0.06	0.16	0.018	385	27	-30	8.2
C3	Control rolling	0.06	0.16	0.018	389	22	-65	8.9
H7	Hot rolling	0.11	0.17	0.023	371	29	-45	9.5
C4	Control rolling 1	0.11	0.18	0.024	404	32	-120	11.8

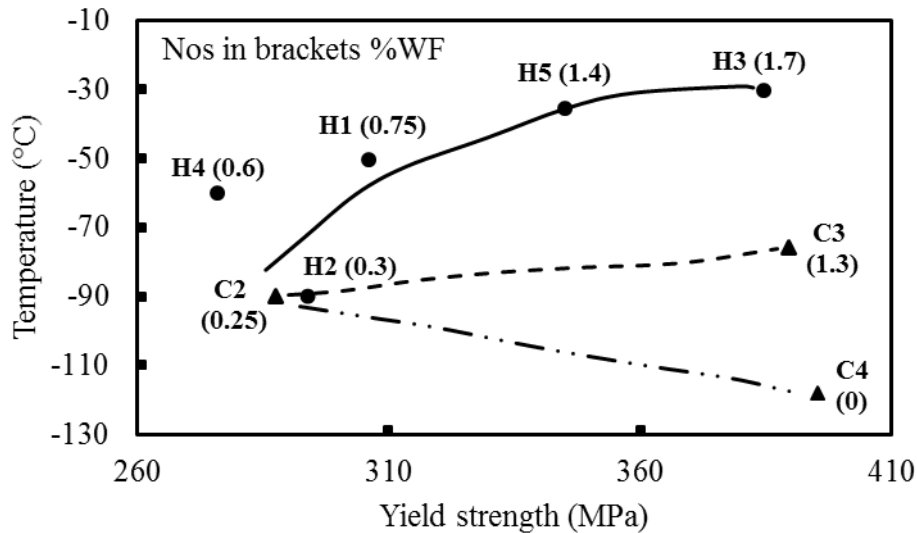
H7 was hot rolled FRT at 970°C and C4 was control rolled FRT 800°C.

C4 had no WF and no MA so the true benefit from Al could be obtained, resulting in a strength level of 400MPa with a 27J ITT of -120°C, **Fig. 21**.

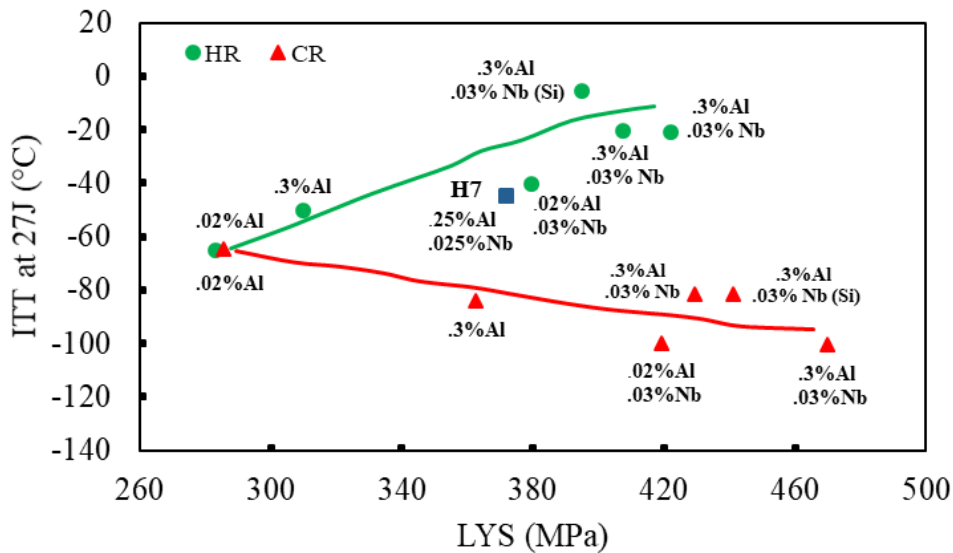
#### Combined effect of ITT and LYS

In order to assess the results it is always necessary to examine the combined effect of strength and toughness.

The impact transition temperatures are plotted against the LYS in **Fig. 21** for the presently examined steels. To clarify the trends, the ITT versus LYS curves are also shown from the previously reported work, both for the hot and control rolled steels, **Fig. 22** [3]. It is clear from these plots, that for the hot rolled steels except for the plate with ~0.2%Al, H2, **Fig. 21**, the plates have poor impact behaviour and the impact behaviour is worst when the Nb addition is combined with the high Al addition, **Figs. 21** and **22**. The data in **Figs. 21** and **22** can be separated into two curves, one for the hot rolled and the other for control rolled. Whereas the hot rolled Nb containing steels showed the worse combinations of properties, the control rolled Nb containing steels gave the best. Similar behaviour is shown with the presently examined hot rolled Nb containing steels, **Fig. 21**. The control rolled Nb containing steel does give much better properties than the Nb hot rolled steels but the presence of MA does appear to prevent it from reaching its true potential. The true potential is shown in C4 which although containing more pearlite due to its higher carbon content, gives both a higher yield strength (15MPa higher) and better impact properties than C3, (55°C lower ITT) **Fig. 21**.



**Fig. 21.** Impact/strength relationship for presently examined 0.06% C steels. The figures in the brackets are the percentage volume fraction of WF.



**Fig. 22.** The impact/strength relationship for the previously examined 15mm thick plates having 0.1% C, in which an Al addition of 0.3% had been made [3].

The behaviour can be seen to be similar in **Figs. 21** and **22** for the two carbon levels, except the properties of the control rolled steel plate C3 are not as good as might have been expected (compare C3 with C4 the latter being free of MA, **Fig. 21**).

## 5. Summary

The evidence suggests that it is the presence of martensite at the allotriomorphic grain boundaries or the tips of the WF colonies which is responsible for the worse than expected impact behaviour encountered when Nb is added to the hot rolled steels. It is unlikely that WF by itself can be responsible for the poor impact behaviour but it does seem to be a necessary requisite for MA formation in these steels. WF can form

without materially influencing impact behaviour but if the steel is sufficiently hardenable the regions which are high in C as a result of the WF transformation will transform to MA on air cooling of 15-30mm hot rolled plate causing the impact behaviour to markedly deteriorate. To avoid this, a high  $A_{r3}$  is required to encourage polygonal ferrite formation rather than WF and the steel should have its hardenability reduced so that MA cannot form even when WF is present. Nb unfortunately, lowers both the  $A_{r3}$  and makes the steel more hardenable and also results in coarser and more numerous grain boundary carbides. Refining the austenite grain size is one way of avoiding all these undesirable phases and accounts for the excellent properties that are present in Nb control rolled steels. However, without the grain refinement Nb is too hardenable to prevent MA from forming. In contrast, Al in solution although able to form martensite on air cooling is not as hardenable as Nb. It is also interesting to note that in a global model perspective, only two elements, Al and Co, increase the  $M_s$  when they are added to the alloy; all other elements lower the  $M_s$  [40].

Because Al raises the  $A_{r3}$  it discourages WF from forming making granular polygonal ferrite more likely. It also has the benefit of refining the grain boundary carbides so improving impact behaviour. The reason as to why an Al addition decreases the ITT has now been more clearly established. Part of the benefit comes from refinement of the grain boundary carbides ( $\sim 10^\circ\text{C}$ ) and part from removing the N from solution ( $\sim 20^\circ\text{C}$ ).

Unless the austenite grain size can be refined sufficiently to prevent MA and WF formation, Nb from this work is not an addition to add to conventionally hot rolled, 15-30mm thick air cooled plate if the required benefit to properties from adding Al is to be obtained. Unfortunately, without Nb austenite grain refinement is limited when it comes to conventional hot rolling. V as it is a weaker carbide and nitride former than Nb may be a possibility at the 0.05% level [41] but the high solubility of vanadium nitride in the austenite compared to NbCN ensures that it will give relatively little grain refinement with only moderate precipitation hardening for the relatively slow cooling rate of these air-cooled plates [41]. A V/Nb combination in the presence of  $\sim 0.2\%$  Al, maybe a better possibility as the presence of V and Nb together slows down the diffusion of carbon [42] and this may result in finer carbides [43]. The presence of a lower Nb content, around 0.01%, will also reduce the hardenability.

Ti might also be seen as a suitable addition, although previous work [43] on hot rolled C-Mn steel plates has shown that of the microalloying additions B, Zr, Ti and Al, an addition of 0.2% Al was found to give the best combination of strength and impact behaviour. This may be because Ti both coarsens as well as appreciably increases the number of grain boundary carbides as does Nb [43]. Al thus, has rather a unique position in that because it is a non-carbide former it is able to refine the carbides and remove N from solution and discourage the formation of WF. However, if the grain size is sufficiently fine to avoid WF, as when control rolling, then the addition of Nb and 0.2% Al are a very good combination.

## 6. Conclusions

- (1). Adding 0.16% Al to a hot rolled plain 0.06%C-Mn steel reduces the ITT by about 40°C without influencing strength.
- (2). This improvement has also been obtained in a higher C-Mn steel at the 0.1%C level on adding 0.2% [1] but when this was increased to 0.3% Al [3], the impact behaviour was impaired due to the presence of WF and LTPs.
- (3). Adding Nb to the hot rolled high Al steel leads to poor impact behaviour due to the WF and MA. The amount of MA required to cause problems is very small but is enough to obscure the beneficial effect of Al.
- (4). In contrast, control rolling because of its finer grain size can allow Al to benefit the impact properties. Although in the present work after control rolling the ferrite grain refinement from Nb was finer than in the Nb free steel, at ~12µm, MA was still present so that the full potential improvement in impact behaviour was not achieved. A bigger improvement occurs when the ferrite grain size after control rolling is in the region of 7µm, as in addition to the normal benefits associated with a finer ferrite grain size, the corresponding austenite finer grain size does not allow WF or MA to form. An Al addition of ~0.2% can then be seen to give its benefit of further reducing the ITT by ~40°C.
- (5). Future work on hot rolled steels must therefore concentrate on adding a precipitation hardener that has only a small influence on the hardenability and the Ar<sub>3</sub>.

### Data Availability statement

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

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**Declaration of interests**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

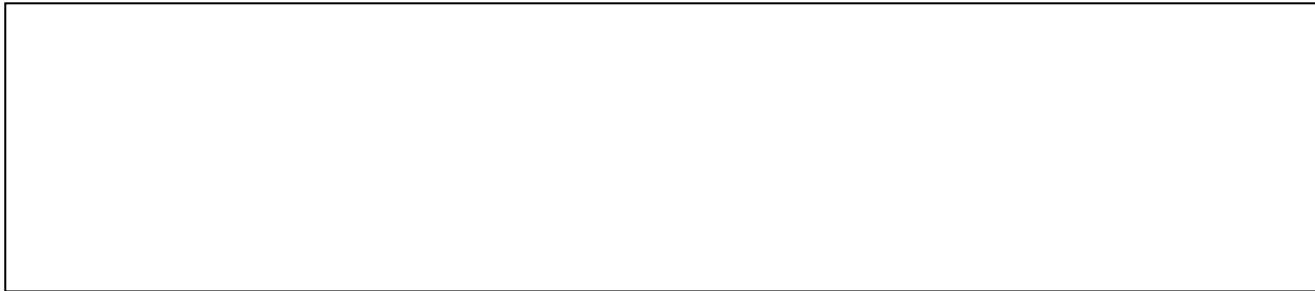
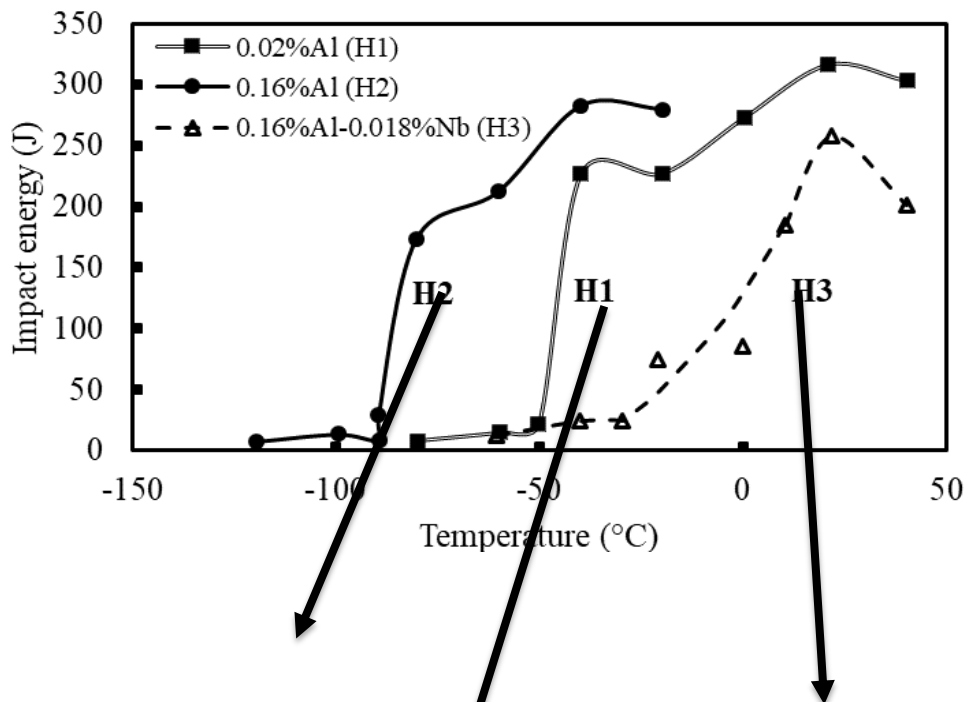
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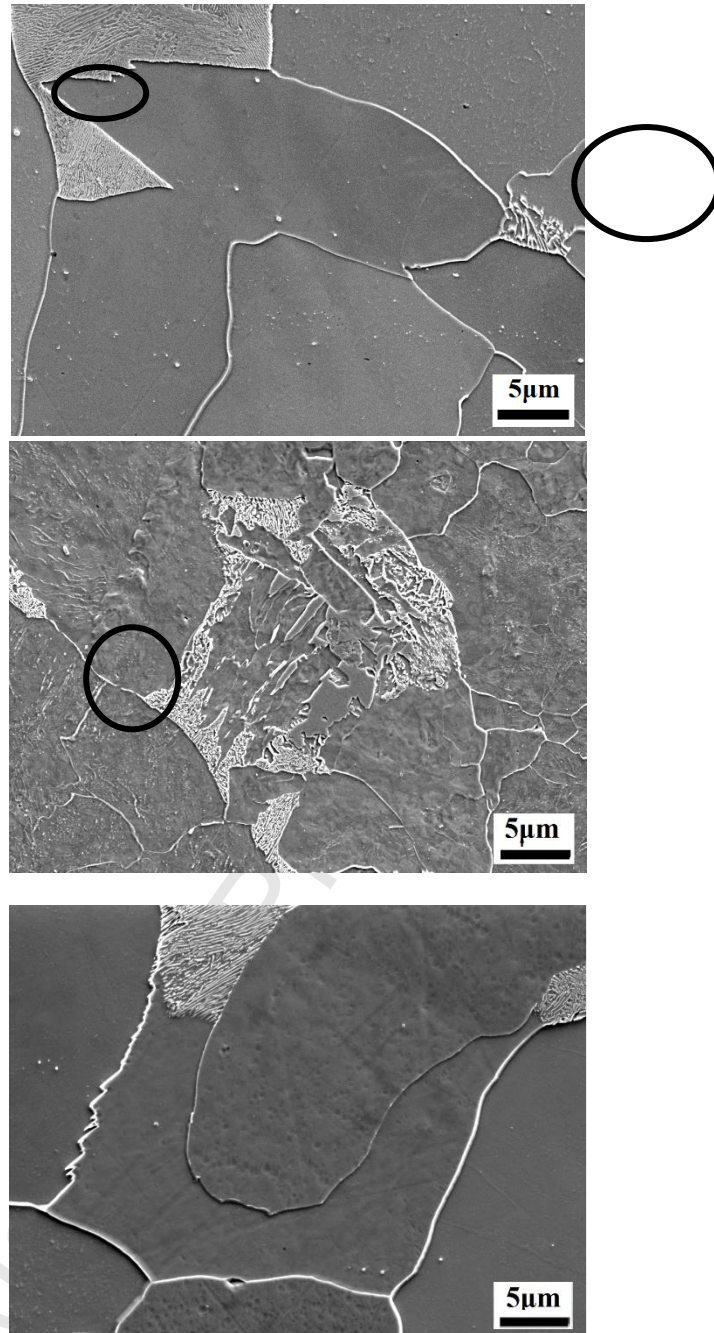
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**Graphical abstract**



### Highlights

- \* Adding 0.2% Al improves impact behaviour of hot rolled C-Mn steels whereas Nb does not.
- \* Widmanstätten ferrite causes small deterioration in toughness but can lead to brittle martensite formation.
- \* Phases which impair toughness can be removed by reducing hardenability and raising  $A_{r3}$  temperature (the temperature at which austenite begins to transform to ferrite during cooling).

- \* Al is unique, raising Ar<sub>3</sub> temperature and Ms temperature (the temperature at which the transformation of austenite to martensite is complete) as well as refining carbides and removing nitrogen.
- \* On hot rolling, Nb lowers Ar<sub>3</sub> and Ms, favouring martensite.
- \* Grain refinement raises Ar<sub>3</sub> so that a 0.2%Al addition improves toughness in control rolled Nb steels giving high strength.

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