Influence of V Precipitates on Acicular Ferrite Transformation

Part 2: Transformation Kinetics

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A combination of thermodynamic models as well as some physical metallurgical principles has been used to analyse the influence that V(C, N) precipitates have on the transformation kinetics of acicular ferrite by isothermal decomposition of austenite. Those precipitates were found absolutely necessary for the nucleation of acicular ferrite as reported in Part 1 of this work, now in Part 2, through the proper design of a heat treatment route we have studied the effect that those precipitates have on the kinetics of acicular ferrite transformation.

KEY WORDS: acicular ferrite; bainite; V(C, N); dilatometry; temperature time transformation diagrams; transformation kinetics.

1. Introduction

In part 1¹⁾ of this two part study it was proven how N through its combination with V enhances the formation of V(C, N) precipitates within austenite that will act as nucleation sites for acicular ferrite (AF) as long as the austenite grain boundaries are, previously to the transformation, decorated with proeutectoid ferrite. In this part two heat treatment routes were designed on the basis of some thermodynamics and equilibrium criteria, so in one of the two routes precipitation is enhanced while in the other one is not, and a comparative study of the kinetics of AF transformation in both routes allow us to analyse the role that the aforementioned precipitates are playing.

2. Materials and Experimental Procedure

The chemical composition of the alloy used is listed in **Table 1**, denoted as 5B steel in part 1.¹⁾

An Adamel Lhomargy DT1000 high resolution dilatometer has been used to determine the TTT diagrams of the steels, and to track the evolution of AF transformation.

Cylindrical dilatometric specimens of 2 mm diameter and 12 mm length were austenitised and then isothermally transformed at different temperatures until transformation was completed followed by gas quenching to room temper-

ature. A simple lever rule based method allow us estimating the evolution of the normalized volume fraction of AF V_{AF}/V_{AF}^{max} with time from the recording of the change in length of the specimen during transformation, a method that has been previously validated in Ref. 2).

$$\frac{V_{AF}}{V_{AF}^{max}} = \frac{L(t) - L_0}{L_f - L_0} \dots\dots\dots(1)$$

where V_{AF}^{max} is the maximum volume fraction of AF formed at the end of the isothermal reaction, $L(t)$, L_0 and L_f are the lengths of the specimen at t , just before the beginning and right at the end of transformation respectively.

Specimens were polished and etched in the usual way for further metallographic examination by means of optical and field emission gun scanning electron microscopy. The previous austenite grain boundary was revealed by means of the thermal etching technique.³⁾

A set of theoretical calculations concerning temperature evolution of different phases present, as well as bainite start temperature (B_s) were performed with the help of a commercial package for thermodynamic calculations in equilibrium in combination with the SG SOL-SGTE Solution database 3.0.⁴⁾

Thermoelectric power measurements (TEP) were performed to experimentally estimate the level of V(C, N) precipitation. TEP measurements are based on Seebeck effect, thus, a difference in the temperature at both extremes of the sample creates a potential difference ΔV , the relative TEP is given by $\Delta S = \Delta V / \Delta T$. Restoration, recrystallisation and precipitation processes increase ΔS values, while cold working (increase of dislocation density) and dissolution processes decrease ΔS . For these experiments, $30 \times 2 \times$

Table 1. Chemical composition of the experimental grade, in wt%.

	C	Mn	V	N	Al	Si	S	Cr	Ni
5B	0.08	1.46	0.25	0.018	0.016	0.007	0.001	0.01	0.009

0.8 mm³ samples were machined longitudinally to the rolling direction. A schematic representation of the TEP apparatus and further detail on the theory lying beneath this technique is given elsewhere.⁵⁾

The martensite start temperature (M_s) of the steel was determined by dilatometry, where standard cylindrical specimens were austenitised and then rapidly cooled. Martensite formation during cooling was detected by monitoring the fractional change in dilatation with temperature, to ensure reliability of the result the test was performed at least twice.

3. Results and Discussion

Figure 1 shows the two routes designed and adopted in this work to analyze the influence of V(C,N) precipitates on AF transformation kinetics. Route 2 was designed to have a higher fraction of potent nucleation sites for AF formation compared to Route 1, as it will become clear in the following paragraphs.

Several are the reasons for the selection of 1125°C for 120 s as the austenitising conditions, first is that according to **Fig. 2**, at this temperature the microstructure is austenite, avoiding the interference of other phases in the study. The second is that according also to **Fig. 2**, there is a small percentage of V precipitates present in the microstructure, those together with the small fraction expected to precipitate within austenite during quenching, 500°C/s, to the isothermal transformation temperature leaves an austenite with a scarce level of potent nucleation sites for AF formation, case of Route 1. It is also very important, the fact that at this temperature the measured austenite grain size is

Fig. 1. Heat treatments routes adopted to study the influence of V(C,N) on AF transformation kinetics.

Fig. 2. Calculated equilibrium mass fractions of austenite (γ), ferrite (α), cementite (θ) and V(C,N) precipitates.

60 μm , *i.e.* such a big grain size is expected to enhance AF formation in detriment of bainite by increasing the ratio between intragranular and grain boundary nucleation sites.^{6,7)}

In the case of the Route 1 the necessary thermodynamic calculations to estimate the B_s temperature according to Bhadeshia's theory⁸⁾ were performed by means of Ref. 4), and the obtained result was 590°C. The experimental M_s temperature was obtained by dilatometry giving an average value of 480°C.

In the case of Route 2 a more profuse V(C,N) precipitation in austenite is intended, therefore, following austenitisation, samples were quenched at 500°C/s down to 900°C and held for 2500 s. The dilatometric curve $\Delta l/l_0$ vs. t revealed an Avrami type shape that reached a plateau, at about 2200–2400 s, meaning that at 2500 s the equilibrium conditions described in **Fig. 2** has been reached, *i.e.* about 0.1 % of V(C,N) precipitates. Therefore the level of precipitation in austenite is representatively higher than that obtained solely by austenitisation at 1125°C, Route 1. To further assess this asseveration a set of TEP measurements were performed on quenched samples that were held at 900°C for different periods of time, 5 to 2500 s, after applying the selected austenitisation conditions. **Figure 3** shows the relative TEP values measured, where the reference condition for the relative values was a sample that after austenitisation was quenched to room temperature, *i.e.* corresponding to the level of precipitation attained in Route 1. Giving the fact that the only metallurgical difference between the reference microstructure and that after holding at 900°C is the level of precipitation, it is reasonable to assure that any increment in the relative TEP measurements, as the holding time at 900°C is increased, can only be justified in terms of an increasing precipitation level. Also **Fig. 3** shows that based on EDX analysis the precipitates can be identified as N rich V(C,N) after following Route 2 but quench-

Fig. 3. (a) Evolution of thermoelectric power at 900°C as function of holding time at 900°C. And (b) identification of precipitates at 2500 s as V(C,N).

ing to room temperature after the isothermal treatment at 900°C–2 500 s.

As it was mentioned, given that 2 500 s at 900°C allow to reach the equilibrium conditions, new calculations to estimate the B_s of impoverished austenite in C, V and N, once precipitation has been considered as in Fig. 2, were performed as previously described. The results showed a negligible variation of B_s as compare to that of Route 1. But in the case of the M_s temperature, much more sensitive to austenite C content,⁹⁾ a slight increase is expected. This point was confirmed by means of dilatometry measurements by quenching to room temperature after 2 500 s at 900°C, providing with a M_s average value of 520°C.

Therefore the selected temperatures for the isothermal heat treatments in both routes were in the range between 650 and 450°C, at intervals of 25°C. All the tests were carried out until the dilatometry signal showed that the tracked transformation had ceased, then gas quench to room temperature was applied.

For Routes 1 and 2 martensite is identified at temperatures equal and lower than 475 and 500°C, respectively, so the M_s temperature for Route 1 is expected between 475–500°C and for Route 2 between 500–525°C, very similar values to those obtained in the previous dilatometric measurements, 480 and 520°C respectively. **Figures 4(a)** and **4(f)** show examples of the microstructure obtained below M_s for both routes, the profuse precipitation that can be observed is due to a self-tempering process since samples were held more time than strictly necessary to complete the martensitic transformation.

On the other hand AF, were detected for Route 1 between 500–575°C, see *e.g.* Figs. 4(b) to 4(d), and for Route 2 between 525–575°C, Figs. 4(g) to 4(i). In Fig. 4(g) it is also possible to observe a lot of small white round features, see *e.g.* those within the dotted white circle, that were identified as V(C,N),^{1,10)} when comparing with *e.g.* Fig. 4(c), so it is possible to confirm that effectively Route 2 has promoted V rich precipitates. Also in Fig. 4(g) it has been pointed the occurrence of cementite precipitation, which is part of the austenite to AF/bainite transformation process when the steel composition does not contain Si in enough quantities.⁸⁾ Figures 4(c) and 4(h) show examples of what it seems to be a set of AF plates growing in opposite directions, a clear example of the absolutely random distribution of AF plates in opposition to the more parallel exhibited by bainite. In both cases it was impossible to identify the precipitate where they nucleated, but it is necessary to remember that this is a planar section of a 3D microstructure. A more general view of chaotic distribution of AF plates is shown in Fig. 4(d). It is also obvious from the micrographs presented that the AF plates tend to be thinner as the transformation temperature decreases, this is due to the fact that both austenite strength and driving force for the transformation increase by lowering the transformation temperature, therefore there is a larger resistance to interface motion and an increased nucleation rate, leading to smaller plate thickness.¹¹⁾

A remarkable feature in both routes is the total absence of grain boundary ferrite in order to obtain AF, this proeutectoid ferrite was proved to be absolutely necessary to form AF under the anisothermal heat treatments described

Fig. 4. Micrographs showing some examples of microstructures achieved after different isothermal heat treatments. Thus, for Route 1 (a) 450°C, (b) 525°C, (c) 550°C, (d) 575°C and (e) 675°C, for Route 2 (f) 500°C, (g) 525°C, (h) 550°C, (i) 575°C and (j) 650°C.

in the first part of this work,¹⁾ which shows the high effectiveness of the V(C,N) particles inside the austenite matrix as intragranular nucleation sites for AF.

Finally in the temperature range between 600–650°C, for both routes, the observed microstructure is mainly proeutectoid ferrite with some pearlite as the micrographs in Figs. 4(e) and 4(j) reveal. Therefore from the above results it is possible to set the B_s temperature for both routes between 575–600°C which is in good agreement with the calculated value of 580°C.

With the help of those results in conjunction with the dilatometric analysis it is possible to outline the TTT diagrams for both routes and the results thus obtained are presented in **Fig. 5**. It is interesting to note that in the region of interest for this study, AF region, no big differences in

Fig. 5. TTT diagrams for both routes, the error is ± 1 s which is the dilatometer resolution. F=ferrite, P=pearlite, M=martensite, AF=acicular ferrite.

Fig. 6. Experimental results for the overall transformation kinetics of AF. Beware of different time scales.

terms of transformation kinetics are found, for both routes the beginning of AF transformation takes place in just few seconds. It is necessary a more detailed analysis of the dilatometric curves to appreciate the differences introduced by an increasing presence of V(C,N) precipitates. Thus, making use of Eq. (1) it is possible to sketch the overall transformation kinetics of AF for Route 1 and 2 as the time evolution of estimated normalized fraction, V_{AF}/V_{AF}^{max} , see Fig. 6. As it has been just mentioned the beginning of transformation for both routes is very similar, about 1–2 s, but this is not the case for the end of it. While for Route 2 the end of transformation is detected in between 5 and 15 s for Route 1 is in the range of 30 to 60 s. Therefore the results allows us to indicate that, indeed the more profuse presence of V(C,N) precipitates in Route 2 leads to an AF transformation kinetics that is much faster than that detected for conditions in Route 1, where the precipitation of such particles is scarcer.

4. Conclusions

In this work two different routes were design so the effect of V(C,N) precipitates on acicular ferrite formation could be analyzed. To avoid the formation of bainite instead of acicular ferrite, the selected previous austenite grain size was $60 \mu\text{m}$, big enough to ensure the reduction of bainite nucleation sites and, at the same time increasing the probability of trapping more of those precipitates within. Then two heat treatment routes were designed, in one of them precipitation of V(C,N) is enhanced by applying after austenitisation, a precipitation isothermal treatment at 900°C (austenite field) during 2 500 s. Given the fact that the only difference between both routes is the amount of precipitates in austenite, the observed acceleration of acicular ferrite transformation kinetics, in the route where pro-

fuse precipitation took place as compared with the other one, can only be explained in terms that V(C,N) precipitates effectively enhance acicular ferrite formation by strongly increasing the ratio between intragranular and grain boundary nucleation sites.

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