# On the Formation Potential of Acicular Ferrite Microstructure in Different Steel Grades Focusing on the Influence of Carbon Content

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## INTRODUCTION

Since the 1970s comprehensive effort was spent on the explanation of the acicular ferrite formation. Acicular ferrite (AF) nucleates intragranularly on non-metallic inclusions. The needle or lenticular shaped plates radiate in various directions and create a chaotic, interlocking microstructure. The growth of acicular ferrite grains is diffusionless, but excess carbon is rejected to the remaining austenite shortly after transition. The carbon enriched austenite transforms during the ongoing cooling process to perlite, bainite or martensite, or remains as retained austenite in the final structure. The created multiphase microstructure provides excellent mechanical properties, most notably toughness, so that acicular ferrite steels are of increasing interest for steel producers. In literature four main nucleation mechanisms are described: destruction of the crystal structure, creation of dislocation arrays, reduction of lattice mismatch and chemical changes in the local matrix. Literature suggests that a combination of at least two effects is responsible for the nucleation of acicular ferrite, but the exact impact of the mechanisms is not completely understood yet.<sup>1–8</sup>

Acicular ferrite formation is mainly influenced by the steel composition, cooling rate, non-metallic inclusions and austenite grain size. In the last decades extensive work was conducted to evaluate the effects of these parameters, but there are still controversial opinions. This may be a result of their strong interaction. Thus, a reasonable study of their impact is only possible with a systematic methodology.<sup>4-7</sup>

In literature the microstructure formed after austenitization is directly related to the cooling rate between 800 and 500 °C. It is commonly accepted that the cooling rate for acicular ferrite is situated between those of bainite and coarse-grained ferrite. However, there is less agreement about the exact value; cooling rates between 6 and 1200 °C/min are described as appropriate in literature. This wide range of cooling rates demonstrates very clearly the complexity of the acicular ferrite formation. The evaluation of the cooling rate's influence is therefore only possible if interactions with the other impact factors, especially the steel composition, are considered. The steel composition itself is also seen as a major influencing parameter for the austenite-ferrite-transition. Alloying elements affect the formation of acicular ferrite as solute element in the matrix or because they form active non-metallic inclusions. Carbon is described as one of the dominating elements concerning microstructure development. Until today several publications about the interaction of carbon and acicular ferrite emerged, but they describe divergent effects. Evans<sup>15,16</sup> found a steady increase in the acicular ferrite amount with increasing

carbon content from 0.05 to 0.15 wt.-%. Mu et al.<sup>17</sup> investigated the influence of carbon between 0.01 and 0.40 wt.-%; they calculated the driving force for ferrite nucleation and showed a steady decrease with rising carbon content. Furthermore, they demonstrated that the nucleation capability of inclusions is reduced by higher carbon contents; subsequently, inclusion's minimum size to act as nuclei for acicular ferrite is significantly increased. Düren<sup>18</sup> observed that there is an optimum carbon content for acicular ferrite at 0.07 wt.-%. Below acicular ferrite is substituted by coarser ferrite veins, above by martensite.

To clarify the effect of carbon on the acicular ferrite formation the present work investigates four steel grades with varying carbon contents from 0.04 to 0.65 wt.-%. Special attention is paid to the evaluation of the interaction between steel composition and cooling rate. Therefor a systematic four-step methodology is used, including thermodynamic calculations, melting experiments, heat treatment experiments by a high-temperature laser scanning confocal microscope (HT-LSCM) and computerized metallographical analyses. Many previous studies, e.g. Ref. 5,17,19–23, added synthetic oxide powders to the steel to create the desired inclusion types which act as nucleation sites for acicular ferrite. In contrast, this study only uses deoxidation and desulphurization products, directly generated by reactions in the melt, to provide the desired nucleation sites. The advantage of this approach is that the inclusion content in the final product is not additionally increased by the addition of artificial particles.

## EXPERIMENTAL PROCEDURE

The current work investigates four steel grades with varying carbon contents from 0.04 to 0.65 wt.-%; their chemical compositions are given in Table 1. To link up with literature a Ti-alloyed HSLA (high strength low alloyed) steel, which is already well described, is included in this study. The carbon content of this steel grade is with 0.23 wt.-% in medium range. Less publications are available about acicular ferritic pipeline steels, although acicular ferrite structures show a low sensitivity to hydrogen induced cracking and sulphide stress cracking.<sup>24</sup> Due to this excellent resistance against H<sub>2</sub>S, acicular ferrite would be a highly beneficial constituent in pipeline steels.<sup>24-26</sup> Hence, a pipeline steel with 0.04 wt.-% carbon is also analysed in this work. Acicular ferrite's remarkable combination of toughness and strength would also point out rail steels as possible application field. But until yet, no research work regarding the capability of rail steels for acicular ferrite has been published. For that reason, a bainitic rail steel with a medium carbon content of 0.24 wt.-% and a perlitic rail steel with a high carbon content of 0.65 wt.-% are also investigated in the present paper.

	C	Si	Mn	Al	Ti	Nb	S	0	N	Fe
Steel H	0.230	-	1.500	0.005	0.050	-	0.007	0.007	0.005	98.196
Steel P	0.040	0.150	1.900	0.007	0.020	0.050	0.006	0.001	0.002	97.824
Steel B	0.240	1.800	1.900	0.004	0.030	-	0.006	0.002	0.004	96.014
Steel R	0.650	0.500	1.000	0.006	0.050	-	0.006	0.001	0.002	97.785

Table 1: Steel compositions [wt.-%].

A four-step methodology is applied to evaluate the impact of the carbon content and cooling rate systematically:

- (1) In the first step, the methodology uses thermodynamic calculations to predict inclusion formation and modification and to set the necessary experimental parameters. For that purpose FactSage 6.4 with the databases FactPS, FToxid and FSstel is used. Inclusion diagrams, which can be used to set the appropriate steel composition to form the defined inclusions types, are created by the module PhaseDiagram: two elements are varied (x-/y-axes) and the stable inclusion phases at a defined temperature are plotted. The determined steel composition is used as input for a process model using the module Equilib. This model simulates changes in the inclusion landscape during the melting experiments and the subsequent cooling. A detailed discussion of the calculation results as well as a comparison between calculated and experimental results are presented elsewhere.<sup>27</sup>
- (2) Second, melting experiments on laboratory scale are performed in a Tammann type furnace (Ruhrstrat HRTK 32 Sond.). Nucleation sites for acicular ferrite, in the form of non-metallic inclusion, are only created by deoxidation and desulphurization. The Tammann type furnace is an electric resistance furnace that can be heated to 1700 °C. A description of the Tammann type furnace and the detailed experimental procedure is to be found in Ref. 27–29.
- (3) Third, heat treatment experiments by HT-LSCM are conducted. By HT-LSCM the nucleation and growth of acicular ferrite plates can be observed in situ. The HT-LSCM consists of a VL2000DX laser scanning confocal microscope, produced by Lasertec, an attached SVF17-SP high-temperature furnace and the associated hardware and software from Yonekura. The function and advantages of this system are discussed in Ref. <sup>30–33</sup>.
- (4) Finally, the samples are analysed metallographically with respect to the austenite grain size, acicular ferrite amount and inclusion landscape, utilizing modern, computer-based analysing techniques. The determination of the prior austenite grain size and the amount of acicular ferrite in the final microstructure are done by computerized routines using the image analysis software Clemex Vision 7.0. The routines calculate the austenite grain size distribution,

respectively the fraction of acicular ferrite based on images taken by an optical Polyvar Pol microscope combined with a digital camera Clemex 4 megapixel. Detailed information on the evaluation routines and the necessary sample preparation was published previously by the authors.<sup>32,34</sup> Inclusion characterization is performed by manual and automated SEM/EDS measurements using an FEI Quanta 200 MK2 scanning electron microscope (SEM), equipped with an energy dispersive X-ray spectrometer (EDS) system of Oxford Instruments. The procedure of the manual and automated SEM/EDS analysis are explained in detail elsewhere.<sup>35–37</sup>

#### **RESULTS & DISCUSSION**

To evaluate the influence of carbon on the formation of acicular ferrite a low carbon pipeline steel, a medium carbon HSLA steel, a medium carbon rail steel and a high carbon rail steel are studied within this paper. Using HT-LSCM the formation of acicular ferrite can be observed clearly, as illustrated in Figure 1 for the HSLA steel. Non-metallic inclusions act as nucleation sites during austenite-ferrite-transition. With ongoing cooling process the formed acicular ferrite plates grow until they are stopped by impingement with other phases.

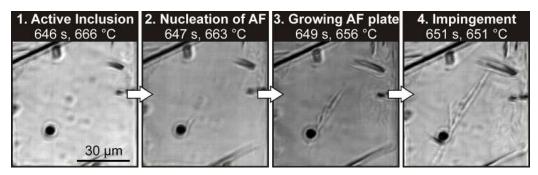


Figure 1. In situ observation of acicular ferrite formation in sample H-1.

Only special types of non-metallic inclusions act as nuclei for acicular ferrite. Within the present work especially  $(Ti,Mn,Al)O_xS_y$  inclusions turned out as highly potent.  $(Ti,Mn,Al)O_xS_y$  particles are found in the HSLA, pipeline and bainitic rail steel as essential active inclusion type. A typical  $(Ti,Mn,Al)O_xS_y$  particle, acting as nucleation site for four acicular ferrite plates, is shown in Figure 2. Due to the small particle size compared to the interacting volume of the SEM, the phase boundaries in the EDS mapping are slightly blurred, but it is obvious the particle is heterogeneous, consisting of  $TiO_x$ ,  $Al_2O_3$  and shells of MnS.

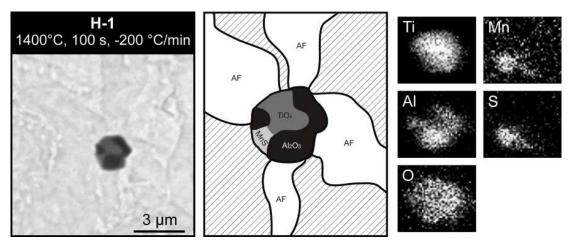


Figure 2. Active (Ti,Mn,Al)O<sub>x</sub>S<sub>y</sub> particle in sample H-1.

The influence of the cooling rate on the formation of acicular ferrite in the medium carbon HSLA steel was already examined previously by the authors. Figure 3 shows two heat treatment regimes, which differ in austenitization temperature. Specimen H-1 was austenized at  $1400\,^{\circ}$ C, specimen H-2 at  $1300\,^{\circ}$ C. In both samples the microstructure mainly consists of acicular ferrite with perlite, grain boundary ferrite and Widmannstätten ferrite. The mean austenite grain size in H-1 is with  $212\,\mu$ m considerably larger than in H-2 with  $157\,\mu$ m. Generally larger austenite grains are seen as more favorable for

acicular ferrite.<sup>2,5–7,14,38,39</sup> However, in the present case the acicular ferrite amount does not significantly change with austenite grain size. Similar observations were made previously by the authors, so that they suggested a critical austenite grain size. Above this value, the austenite grain size is not a limiting factor any more, the amount of acicular ferrite only depends on the steel composition, cooling rate and inclusion landscape. A further increase in grain size without any changes of the other parameters will not increase the acicular ferrite fraction. In this study the critical austenite grain size is obviously reached in both cases. Nevertheless, the medium carbon HSLA steel shows the highest capability for acicular ferrite, what matches well to observations in literature.

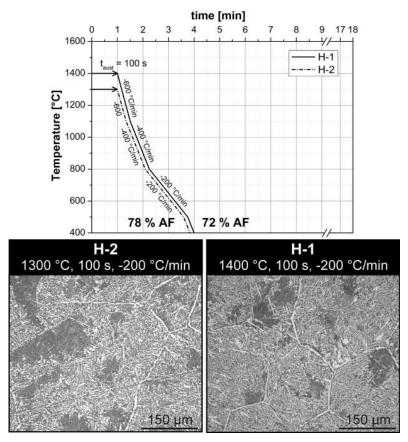


Figure 3. Heat treatment regime and resulting microstructures of the HSLA steel with 0.23 wt.-% C.

Figure 4 shows the tested cooling regimes for the bainitic rail steel with medium carbon content. It can be seen that the optimum cooling rate depends strongly on the steel grade. While for the HSLA and pipeline steel the highest acicular ferrite amount are achieved with a cooling rate of -200 °C/min between 800 and 500 °C, the bainitic rail steel reaches the maximum value if cooled with -100 °C/min in this temperature range. Above and below the cooling rates are the same. At high cooling rates the acicular ferrite and bainite are the main constituents, containing smaller amount of Widmannstätten ferrite, perlite and grain boundary ferrite. With decreasing cooling rate the amount of bainite is reduced in favor of perlite and the fraction of grain boundary raises strongly reducing the area for intragranular acicular ferrite formation. Furthermore, the pattern of acicular ferrite is getting coarser as can be clearly seen in Figure 4. Bainitic rail steels have not been linked to acicular ferrite so far; but the current results prove that also Ti-alloyed bainitic rail steels provide a high potential for acicular ferrite. This information is of particular importance, because it opens a new, promising application field for acicular ferrite.

However, classical high carbon rail steels tend in accordance with the binary Fe-Fe<sub>3</sub>C-phase diagram, which is shown in Figure 5, to transform to perlite with only very small fraction of ferrite. To evaluate the practical capability for acicular ferrite of high carbon rail steels, sample R with 0.65 wt.-% is studied. The perlitic rail steel R is cooled with four different cooling rates between 800 and 500 °C. But, as shown in Figure 6, in this high carbon steel no acicular ferrite can be formed, regardless of austenite grain size and cooling rate. The high carbon content generates a large fraction of cementite precipitations, which cause the formation of perlitic structures. Only very less grain boundary ferrite can be produced.

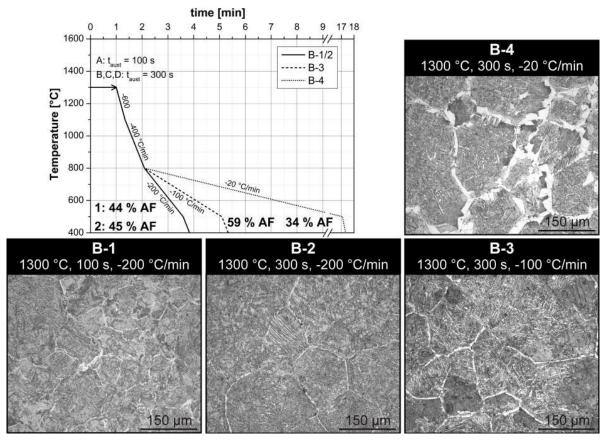


Figure 4. Tested cooling rates and resulting microstructures of the bainitic rail steel with 0.24 wt.-% C.

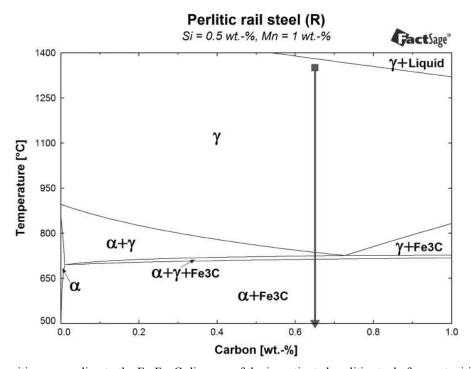


Figure 5. Phase transitions according to the Fe-Fe<sub>3</sub>C-diagram of the investigated perlitic steel after austenitization at 1350 °C.

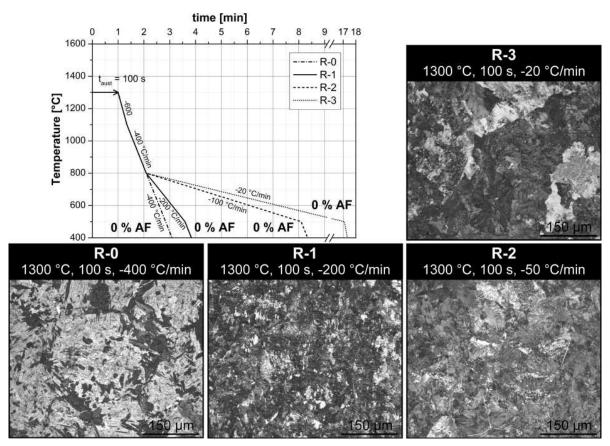


Figure 6. Heat treatment regime and resulting microstructure for the perlitic rail steel with 0.65 wt.-% C.

Figure 7 displays the tested cooling rates for the pipeline steel. It can be seen that the pipeline steel provides a low potential for acicular ferrite. For all tested cooling rates the steel showed a bainitic structure with less acicular ferrite. Changes in the cooling rate do not significantly enhance the capability, what indicates that even though an appropriate cooling rate is necessary to form acicular ferrite, a steel's potential for acicular ferrite cannot be raised by this parameter. An increase in a steel's capability is only possible by variations in the other influencing factors. If the critical austenite grain size is already reached, an optimization can only be done by steel composition and non-metallic inclusions.

In literature controversial opinions about the effect of carbon can be found. Evans <sup>15,16</sup> suggested a steady increase in acicular ferrite if the carbon content is raised from 0.05 to 0.15 wt.-%. A similar effect is observed within this paper, where the acicular ferrite amount increases steeply if the carbon is changed from 0.04 to 0.23 wt.-%. In contrast, Mu et al. <sup>17</sup> assumed a steady decrease in the nucleation probability of acicular ferrite if the carbon content is raised from 0.01 to 0.4 wt.-%. This observation cannot be supported by the findings of this study. Düren <sup>18</sup> indicated that the effect of carbon follows a C-curve, with a maximum acicular ferrite amount at 0.07 wt.-% carbon. The current investigations also show a C-curve dependence of carbon and acicular ferrite, although the peak value is significantly higher than suggested by Düren. The present results indicate an optimum carbon content of > 0.2 wt.-%; due to the limited investigated carbon levels, no exact peak value can be determined.

In addition to the amount of acicular ferrite, carbon also has a strong impact on the pattern of acicular ferrite microstructures:

- Morphology of acicular ferrite plates: Several authors 9,10,13,25,40 reported that the typical lenticular shape of acicular ferrite is often not fully developed in low carbon pipeline steels. This observation is also made in the present study, as shown in Figure 8.
- Structure of intergranular regions between acicular ferrite grains: In literature 41,42 silicon and nickel are described to influence the structure of the intergranular regions between acicular ferrite grains. Within this work it is observed that also carbon has a significant impact on the intergranular regions. As illustrated in Figure 8, the area of intergranular regions is increased with raising carbon content. This results from the diffusionless growth of acicular ferrite. During the growth of acicular ferrite, the remaining austenite is enriched in carbon; if a critical carbon content is reached, the growth of acicular ferrite is stopped. It can be assumed, that this critical carbon value is reached earlier if the initial carbon content in the steel is higher. Hence, at higher carbon contents acicular ferrite has less time to grow, so that more remaining austenite will transform to intergranular phases. It should be noted, that

this effect hinders the classification of acicular ferrite in low carbon steels, because the grains are not surrounded by clearly visible intergranular regions, e.g. dark perlite between bright ferrite grains.

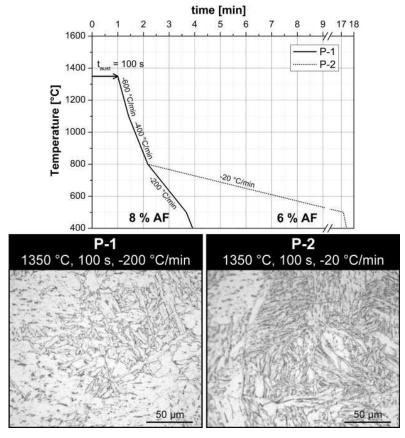


Figure 7. Tested cooling rates and resulting microstructures for the pipeline steel with 0.04 wt.-% C.

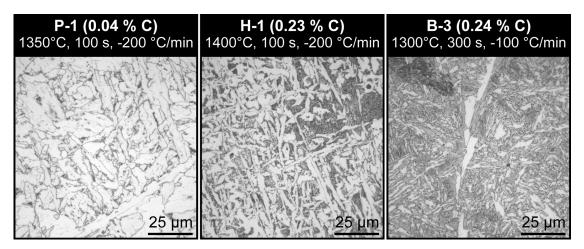


Figure 8: Varying fraction of intergranular regions between acicular ferrite grains with raising carbon content.

## CONCLUSIONS

Due to its excellent mechanical properties acicular ferrite is in the focus of welding engineers and steel producers since many years. The formation of acicular ferrite formation is influenced mainly by the parameters steel composition, cooling rate, non-metallic inclusions and austenite grain size. On the effect these parameters controversial opinions exist. Especially acicular

ferrite's formation in steel grades with different carbon contents is less described in literature. Therefore, the present work studies extensively the influence of carbon content and cooling rate on acicular ferrite. Using a systematic four-step methodology the following knowledge is gained:

- (1) The acicular ferrite potential of steels with varying carbon content is studied. The carbon content is identified as crucial parameter:
  - a. HSLA and bainitic rail steels with medium carbon contents are most efficient. The formation of acicular ferrite in rail steels have not been observed before; hence, the findings of the present thesis open a new, interesting application field.
  - b. The low carbon pipeline steel shows less efficiency. In the high carbon rail steel the formation of acicular ferrite is completely suppressed, which is in accordance with the Fe-Fe<sub>3</sub>C-diagram.
  - c. Furthermore, the carbon content is found to influence the morphology of acicular ferrite plates and the structure of intergranular regions.
- (2) The cooling rate's influence is analysed with respect to the chemical composition. A significant impact of the cooling rate is found:
  - a. The optimum cooling rate depends strongly on the steel composition. While the HSLA and pipeline steels achieve the highest acicular ferrite fraction with a cooling rate of -200 °C/min between 800 and 500 °C, the bainitic rail steel produces the maximum acicular ferrite amount if cooled with -100 °C/min in this temperature range.
  - b. Additionally, the cooling rate affects the grain size; with increasing cooling rate acicular ferrite grains become finer.
  - c. Nevertheless, it is demonstrated by the current work that a steel's potential for acicular ferrite cannot be enhanced by variation of the cooling rate. An appropriate cooling rate is required to produce acicular ferrite, but the steel's capability is only affected by alloying elements, inclusions and austenite grain size.

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