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In situ observation of acicular ferrite formation using HT-LSCM: Possibilities, challenges and influencing factors

Abstract

By using a Laser Scanning Confocal Microscope combined with a High Temperature Furnace (HT-LSCM) for the in situ investigation of acicular ferrite (AF) formation in HSLA steels, new information about the mechanism of formation of this high toughness phase can be gained. Due to the utilization of an inert furnace atmosphere, the ability to accurately adjustment the austenitizing temperature and the well-controllable cooling conditions, the interactions between steel composition, austenite grain size, cooling rate and the fraction of AF formed have been analyzed in detail. The present work focuses on necessary adaptations and appropriate settings of the HT-LSCM for the investigation of the formation of AF. An intensive study of setting parameters for the visualization of the AF formation is done. Special attention is paid to the techniques by which austenite grain size is determined and the effect of austenite grain size on the fraction of AF formed. In addition, the complexity of studying manganese alloyed steels in the HT-LSCM has been elucidated.

Keywords

influencing, challenges, possibilities, lscm, factors, ht, observation, formation, ferrite, acicular, situ

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***IN-SITU* OBSERVATION OF ACICULAR FERRITE FORMATION USING HT-LSCM: POSSIBILITIES, CHALLENGES AND INFLUENCING FACTORS**

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Abstract

By using a Laser Scanning Confocal Microscope combined with a High Temperature Infrared Furnace (HT-LSCM) for the *in situ* investigation of acicular ferrite formation in HSLA steels, new information about the mechanism of formation of this high toughness phase can be gained. Due to the utilization of an inert furnace atmosphere, the ability to accurately adjust the austenitizing temperature and the well-controllable cooling conditions, the interactions between steel composition, austenite grain size, cooling rate and the fraction of acicular ferrite formed have been analyzed in detail. The present work focuses on new developments and improvements in high-temperature laser-scanning confocal microscopy that enabled a detailed investigation of the formation of acicular ferrite. Special attention was paid to the techniques by which austenite grain size is determined and the effect of austenite grain size on the fraction of acicular ferrite formed. In addition, the complexity of studying manganese alloyed steels in the laser-scanning microscope has been elucidated.

Keywords: Acicular Ferrite, HT-LSCM, Microstructure, Phase Transformation

Introduction

In the 1990s two new scientific fields emerged: On the one hand, the specific control of steel microstructure by means of appropriate tailoring of non-metallic inclusions, called “Oxide Metallurgy” [1-2]. On the other hand, the first studies concerning the use of laser scanning confocal microscopy combined with high-temperature furnaces for the observation of metallurgical reactions [3-6]. Knowledge about the role of non-metallic inclusions in controlling the grain size of steels was originally gained by studies on weldments in the fusion zone [7-9]. In addition, a considerable effort has been expended in recent years in finding a method of grain refinement by nucleating ferrite within austenite grains by means of thermo-mechanical treatment in the course of steel production. Up to now some essential and fundamental work has been done through determining the relationship between the presence of non-metallic inclusions and the development of acicular ferrite (AF) structures, mainly focusing on HSLA (High Strength Low Alloyed) steels [10-11]. Various authors reported that an increase in the AF content in the microstructure leads to significant improvements in mechanical properties. They attributed these improvements to different propagation paths of cleavage cracks in the presence of acicular ferrite [6-7]. It is now well established that the nucleation of AF is mainly affected by steel composition, cooling rate, austenite grain size and the presence of non-metallic inclusions [10-11]. By the use of a high-temperature laser-scanning confocal microscopy (HT-LSCM) various metallurgical reactions in the solid and liquid states can be observed *in-situ*. Phase transformations such as the $\alpha \rightarrow \gamma$ transition in steel; peritectic transformations; the growth of austenite grains; inclusion nucleation, growth and agglomeration in steel; inclusion dissolution in slag and reactions at the steel refractory interface can be observed. Through the use of a laser as light source even luminous materials

can be observed up to 1700 °C [12]. The use of laser-scanning confocal microscopy combined with an infra-red furnace originated in Japan and only a few high-temperature confocal microscopes are located in Europe, Australia and the USA [12].

In the last decade, the fields of inclusion engineering and high-temperature metallography have been integrated and exciting new information of AF formation has been gained by the use of *in-situ* observations in high-temperature laser-scanning microscopes. [12-19]. Komizo *et al* [13] were one of the first groups to visualize the formation of acicular ferrite formation by using high temperature laser-scanning confocal-microscopy. Conventional methods for the analysis of AF, most notable optical microscopy, manual and automated SEM/EDS and TEM, only allow the analysis of the final microstructure and the inclusion landscape at room temperature. By contrast, HT-LSCM enables *in-situ* observation of AF formation during cooling. This new method offers interesting opportunities to examine the interaction of acicular ferrite and non-metallic inclusions. Due to the use of an inert furnace atmosphere, the ability to accurately adjust the austenitizing temperature and the well-controllable cooling rate, the interrelationship between steel composition, austenite grain size, cooling rate and the amount of acicular ferrite can be studied in detail. Zhang *et al* [14,15,17,18] conducted a detailed study on the formation of acicular ferrite in low alloyed steels by *in-situ* observations in a LSCM. Starting with very basic examination of phase transformations in general [14], they proceeded to conduct a very intensive study of the interactions between non-metallic inclusions and acicular ferrite [15]. Bin and Bo [16] used the LSCM to investigate the influence of rare earth elements – especially cerium – on the formation of acicular ferrite. Zhang *et al* [15,17,18] confirmed that the austenite grain size increases with increasing holding time at a given austenitizing temperature, but argued that the final grain size depends mostly on the steel composition (e.g. boron segregation- constrained grain growth) and on pinning by inter-metallic compounds and inclusions. [? – is my statement true?] Zhang *et al* [14,15,17] also observed that there is a significant increase in the fraction of acicular ferrite formed with an increase in austenite grain size. They found that although the transformation temperature for grain boundary transformations such as Bainite and Widmannstätten ferrite is influenced by the austenite grain size, the transformation temperature for intragranular transformations such as acicular ferrite, is not.

The present work focuses on new developments and improvements in high-temperature laser-scanning confocal microscopy at the Chair of Ferrous Metallurgy aimed at quantifying and optimizing the formation of acicular ferrite. More specifically, the effect of austenitization temperature and time; cooling rate and the microstructure of the as-received steel have been examined. Special attention was paid to the techniques used to determine the austenite grain size and the effect of austenite grain size on the formation of acicular ferrite. In addition, the complexity of studying manganese-alloyed steels in the HT-LSCM has been evaluated.

Experimental Procedure

Steel Composition

All investigations are done with so-called dipping samples. A steel melt was produced in a vacuum induction furnace, and samples were taken by dipping a submerged body into the melt onto which the steel solidified. The detailed procedure of the dipping tests has previously been described in detail and there is no need to repeat these details [20]. The chemical composition of the melt used in the present study is given in **Table 1**.

Table I. Chemical Composition of the investigated steel [wt.-%]

C	Si	Mn	P	S	Al	Ti
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Characterization Techniques

HT-LSCM, optical microscopy and SEM/EDS analysis have been used to investigate the formation of AF and the parameters that influence the formation thereof. The system for the *in-situ* observation of phase transformations consists of the laser-scanning confocal microscope type VL2000DX, produced by Lasertec, the attached high temperature furnace type SVF17-SP and the associated hard- and software of Yonekura. A detailed description of the system as well as its function and advantages are to be found elsewhere [12,21,22,23]. In order to attain an oxygen free furnace atmosphere, the furnace chamber is repeatedly evacuated and flushed with argon 6.0 [ultra-high purity?] for at least two hours before the experiment starts. Additionally, the argon stream is purified in a gas-train before entering the chamber in order to remove any residual oxygen. The austenite grain size is determined by means of optical microscopy, which is carried out with a Polyvar Pol microscope combined with the image analysis software Clemex Vision 4.0 and a digital camera Clemex 3 Mpixel. Because austenite grain boundaries are revealed by thermal etching in the course of the HT-LSCM treatment, generally no additional chemical etching is necessary. It is important to note that the thermally-etched austenite grain boundaries remain as thermal grooves following the subsequent transformation to ferrite and hence, the resulting ferrite structure can be directly related to the pre-existing austenite grains [Ref. X] In order to compare the microstructure of the sample surface observed in the high-temperature microscope with the bulk microstructure, several specimens were cut and etched with picric acid to visualize the austenite grain boundaries. The evaluation of grain size was done manually in the first step (determination of grain boundaries) and computer based in the second step (calculation of grain size) using the image analysis software Clemex. Figure 1 illustrates the procedure of austenite grain size analysis. For SEM/EDS analyses a Scanning Electron Microscope (SEM) manufactured by Fei (Quanta 200 MK2) and equipped with an energy dispersive X-ray spectrometer (EDS) system of Oxford Instruments (INCA) was used.

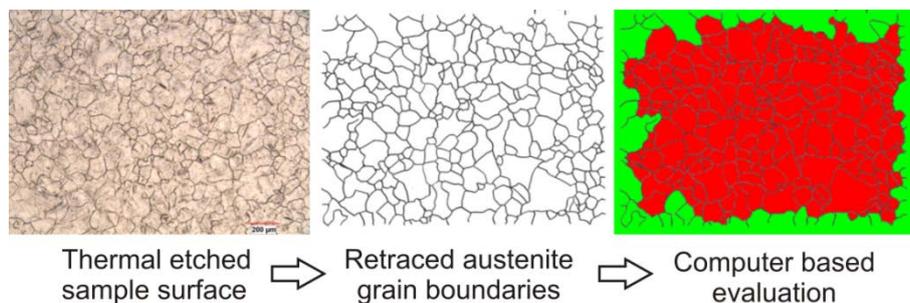


Figure 1. Procedure of austenite grain size analysis.

Results and Discussion

Austenitization Temperature

The austenite grain size was determined at various austenitization temperatures between 1200 and 1400 °C following an isothermal hold for 300 seconds. Specimens were then cooled at pre-determined rates and the fraction acicular ferrite was determined in each case.. Figure 2 shows that below 1400 °C, the austenite grain size increases exponentially with increasing austenitizing temperature. It has been reported in literature, and also observed in previous studies at the Chair of Ferrous Metallurgy [25], that the amount of AF increases with an increase in austenite grain size. Notwithstanding these findings, the amount of AF detected in this study does not change significantly as a function of austenite grain size. This apparent

discrepancy can be explained by considering the fact that the precise steel composition as well as a raft of processing parameters can influence the amount of acicular ferrite that forms. It is therefore suggested that there is a critical austenite grain size at which the maximum AF-content is obtained in any given steel at optimized processing conditions. A further increase in grain size without a change in any of the other parameter, does not lead to an increase in the AF-amount. In the present study, the critical austenite grain size for the given system may well have been reached at the lowest austenitizing temperature used.

Austenitization Time

In this study, the movement of austenite grain boundaries, more specifically the velocity of austenite grain growth v_{GB} , has been determined by analysing successive HT-LSCM images. Using reference points such as non-metallic inclusions or porosities, the distance l travelled by an austenite grain boundary with respect to this reference point is measured and the measurement repeated for several other grain boundaries. After following the grain boundary movement over time t , the grain boundary velocity is calculated as:

$$v_{GB} = \frac{l_{t2} - l_{t1}}{t2 - t1} \quad (1)$$

Figure 3 shows the increase of austenite grain growth with increasing temperature and time and it is evident that the velocity of an austenite grain boundary changes over the austenitizing time. Starting with very fast grain boundary movement, the velocity decreases exponentially which is in accordance with the equation proposed by Andersen and Grong [24]. They described the grain growth velocity as an exponential function of austenitization time and temperature. For a temperature of 1400 °C, an austenitization time of more than 100 seconds is not appropriate because the velocity decreases below 0.25 $\mu\text{m/s}$ (15 $\mu\text{m/min}$) within this time span, which is indicated as no significant movement of grain boundaries.

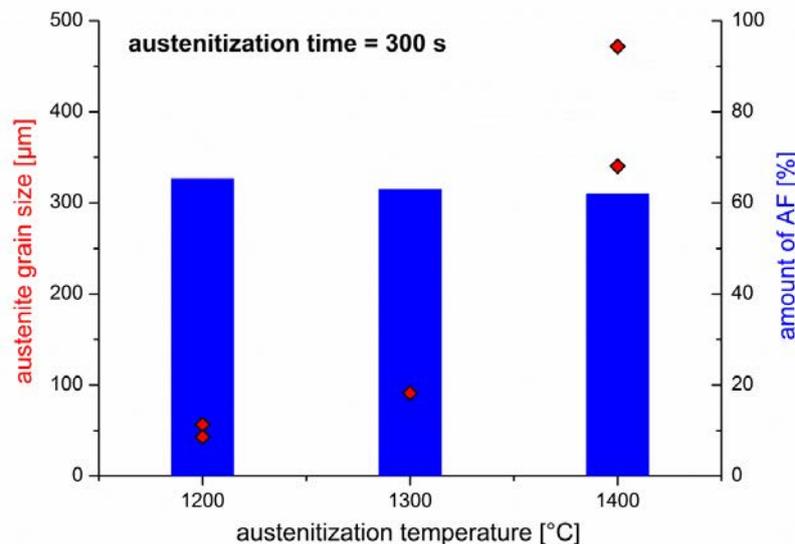


Figure 2. Influence of austenitizing temperature on the austenite grain size and the AF-amount in microstructure.

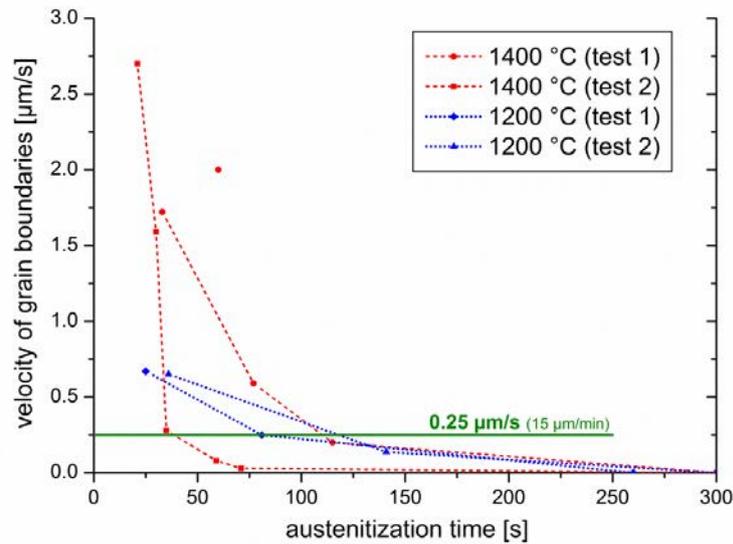


Figure 3. Influence of austenitizing time on the velocity of moving austenite grain boundaries.

[Denise, we need to be careful here. The velocity we measured by this technique only applies to grain boundaries that are located perpendicular to the plane of observation. Grain boundaries that are inclined to the surface will move ostensibly at a lower velocity simply because we measure only the component of the velocity at the surface.]

Cooling Rate

Five different cooling regimes have been tested and the final amount of AF in each case determined by optical microscopy after etching. Figure 4 shows that the largest amount of acicular ferrite (70 %) is obtained with cooling regime C4. It is interesting to note that specimens that were cooled by regimes C2 and C5, at the same rate between 800 and 500 °C as those of C4, contained less acicular ferrite, 59 % in the case of C2 and 52 % in the case of C5. Specimens cooled by regimes C2 and C5 were cooled at different rates between 1400 °C and 800 °C and it therefore seems that the cooling rate between 1400 °C and 800 °C has an influence on the amount of acicular ferrite formed, notwithstanding the fact that AF formation does not occur before a temperature in the range 800 to 700 °C is reached.

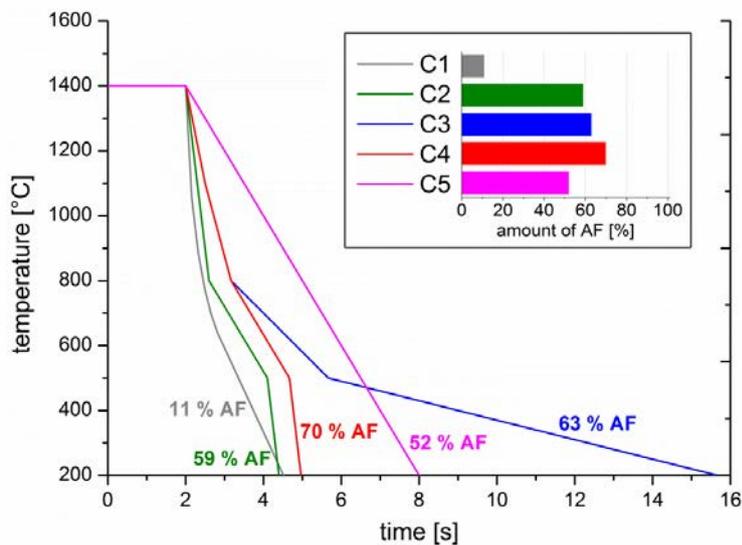


Figure 4. Influence of various cooling regimes on the AF-amount in the final microstructure.

In Figure 5 an image sequence of the austenite – ferrite phase transformation during HT-LSCM treatment C4. After the formation of pearlite, the nucleation of AF on non-metallic inclusions as well as the growth of AF grains are clearly observed.

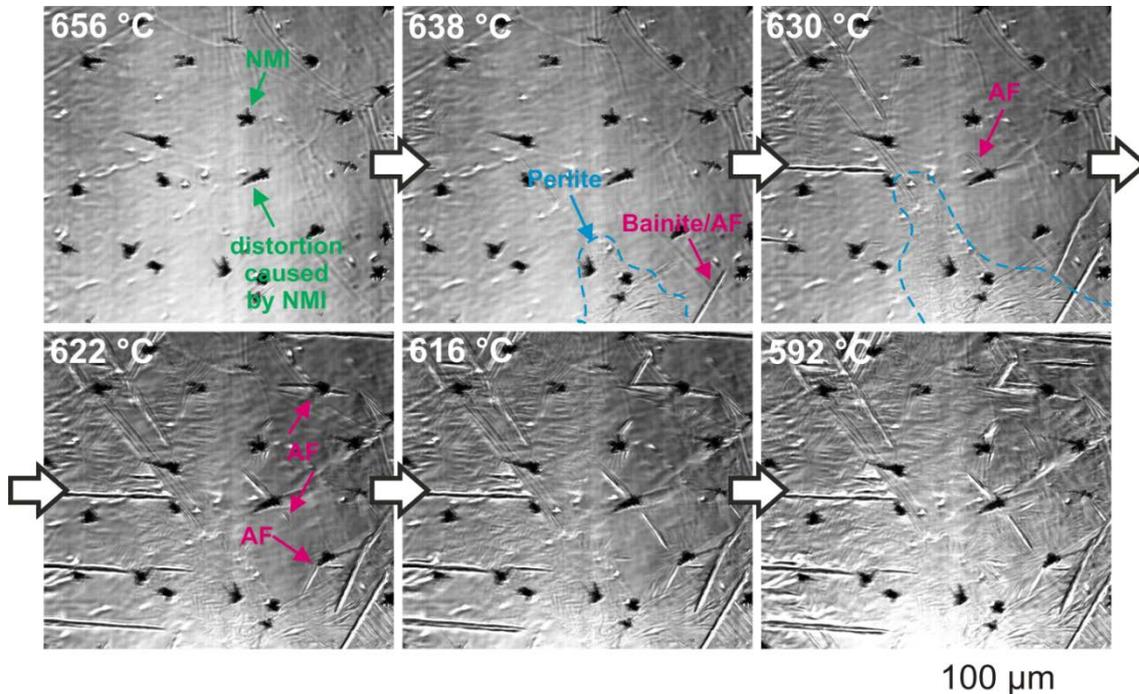


Figure 5. Observation of the phase transformations during HT-LSCM treatment C4.

Microstructure of the as-received steel

The dipping samples used for the HT-LSCM investigations show an acicular ferritic microstructure. In order to determine if the microstructure of dipping sample influences the final microstructure following HT-LSCM treatment, a dipping sample was water quenched instead of air cooled. This sample is fully martensitic before heat treating by cooling regime C4 and no differences between the quenched and air-cooled samples could be detected. It therefore seems that the microstructure of the dipping sample does not have a significant influence on the final amount of AF microstructure following heat treatment C4.

Investigation of Mn-alloyed Steels in the HT-LSCM

Several investigations of Mn-alloyed steels by the HT-LSCM at the Chair of Ferrous Metallurgy including the present study showed that manganese evaporates and/or reacts with oxygen on the sample surface. This results in a manganese-depleted zone to a distance of about 80 μm below the sample surface and complicates the interpretation of observations on the surface of manganese containing steel, such as in the present study. An attempt was made to eliminate, or at the very least, reduce the reaction of manganese with oxygen by positioning a zirconium ring on the crucible, which acts as an additional oxygen getter during the experiment. The extent of manganese depletion was significantly reduced, but not eliminated. The manganese-depleted zone was reduced to a depth of about 20 μm. Hence, the oxygen-getter suppresses the reaction of manganese with oxygen, but does not lead to a satisfying solution to the problem and further investigations are necessary.

[Denise, you mention manganese enrichment, but do not give information about that. Is there really manganese enrichment below the surface? if so, where and to what extent. Unless you expand and explain the enrichment, I think it best to not say anything about it.]

Summary and Conclusion

Within the present work an intensive study of the necessary adaptations and appropriate settings of the HT-LSCM at the Chair of Ferrous Metallurgy was done. The effect of austenitization temperature, austenitization time, cooling rate and microstructure of the dipping samples were examined. Finally, the complexity of investigating Mn-alloyed steels in the HT-LSCM is described. HT-LSCM, optical microscopy and SEM/EDS analysis have been used to examine the formation of AF and the parameters that influencing the formation thereof. The evaluation of grain size was done in three steps: thermal etching, manual retracing of grain boundaries and computer based evaluation of grain sizes.

The austenitizing temperature has a direct influence on the austenite grain size, although it appears that there is a critical austenite grain size, above which no significant influence can be detected of grain size on the amount of AF formed.

The velocity of austenite grain growth changes over austenitizing time, after about 100 s austenitization at 1400 °C no notable movement of grain boundaries has been detected.

Five different cooling regimes have been tested, which showed that the cooling rate above 800 °C has an effect on the amount of AF formed, notwithstanding the fact that the formation of AF phase does not start before a temperature of 800 °C is reached on cooling.

The microstructure of the dipping samples has no significant influence on the amount of AF formed.

During the investigation of Mn-alloyed steels in the HT-LSCM, a reaction of Mn near the sample surface can occur, leading to a Mn-depleted zone. A zirconium oxygen getter can alleviate but not solve the problem and further investigation is required to eliminate manganese depletion near the sample surface.

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