CHARACTERIZATION OF ACICULAR FERRITE MICROSTRUCTURES USING ETCHING METHODS, OPTICAL MICROSCOPY AND HT-LSCM

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ABSTRACT

Acicular ferrite is a needle shaped modification of ferrite, which nucleates intergranularly at non-metallic inclusions. Due to the fine grained structure of acicular ferrite, it offers excellent toughness. By increasing the amount of this component within the microstructure, the properties of HSLA steels can be optimized significantly. The formation of acicular ferrite is influenced by four main parameters: Steel composition, cooling rate, austenite grain size and non-metallic inclusions. These parameters are interacting strongly, making a systematic study essential. By using a Laser Scanning Confocal Microscope combined with a High Temperature furnace (HT-LSCM) for the in situ observation of acicular ferrite formation in HSLA steels, fundamental information about the formation mechanism can be gained. Due to the inert furnace atmosphere, the accurate adjustment of austenitizing temperature and the well controllable cooling conditions, the interactions between steel composition, austenite grain size, cooling rate and the acicular ferrite amount can be analyzed in detail. Up to now no automated quantification of the acicular ferrite amount has been described in literature. The present study focuses on the characterization of acicular ferrite microstructures by a combination of metallographical methods. Conventional sample preparation is combined with the in situ observation of the acicular ferrite formation in a HT-LSCM. Special attention is paid to the determination of austenite grain size by optical microscopy and its influence on the acicular ferrite amount. Finally, various etching methods for the illustration of acicular ferrite are presented, focusing on their applicability for an automated quantification of the acicular ferrite amount.

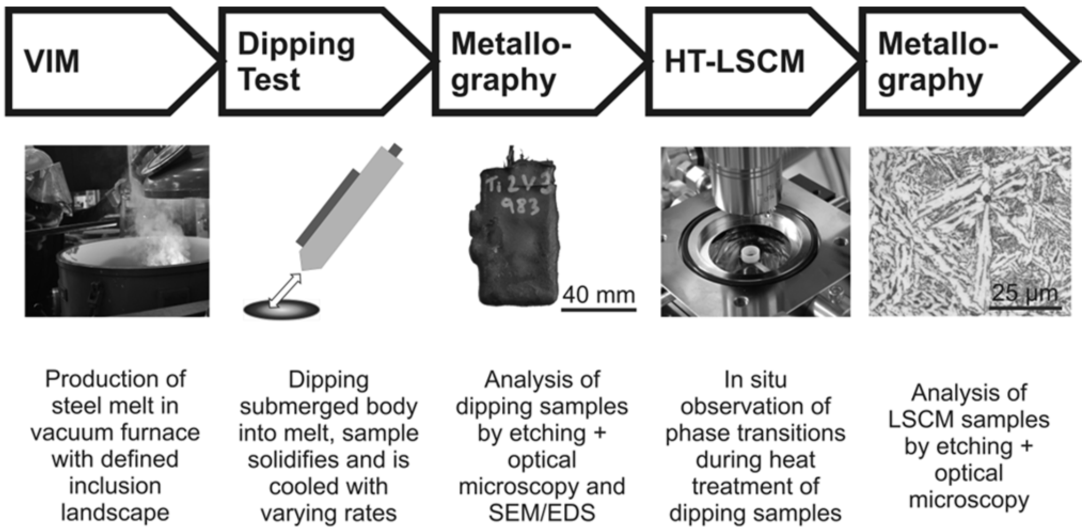
1. INTRODUCTION

In the 1990s a new scientific field called “Oxide Metallurgy” developed which aims on the specific control of steel microstructure by an appropriate tailoring of non-metallic inclusions [1-2]. Up to now there have been considerable efforts to develop a method for grain refinement during steel production by means of oxide metallurgy. Acting as heterogeneous nucleation sites during the $\gamma \rightarrow \alpha$ transition, non-metallic inclusions can refine the microstructure by promoting the formation of acicular ferrite (AF) within the austenite grain. Beside non-metallic inclusions the nucleation of AF is mainly affected by steel composition, cooling rate and austenite grain size [3-4]. Conventional methods for the analysis of AF, most notable optical microscopy, manual and automated SEM/EDS and TEM, only allow the analysis of the microstructure and the inclusion landscape after heat treatment. Hence, during the last decade a new analytical method for the investigation of AF microstructures emerged. Using a Laser Scanning Confocal Microscope attached to a

High-Temperature furnace (HT-LSCM), the formation of AF can be observed in situ [5-11]. Using a laser as a light source, even luminous materials such as steel can be observed up to 1700 °C [5]. Due to the inert furnace atmosphere, the accurate adjustment of austenitizing temperature and the well controllable cooling conditions, the interactions between steel composition, austenite grain size, cooling rate, non-metallic inclusions and the AF amount can be analysed in detail. The present work focuses on the characterization of AF microstructures in HSLA steels using a combination of conventional metallographic methods and modern high temperature microscopy. Samples are heat treated in the high-temperature furnace attached to a LSCM. The determination of the final microstructure is done by etching and optical microscopy. Special attention is paid to the analysis of austenite grain size, which is one of the major influencing parameters on the formation of AF. The advantages of HT-LSCM and the essential benefits of the combination of conventional and modern metallographic methods shall be pointed out.

2. EXPERIMENTAL PROCEDURE

The present study focuses on the characterization of acicular ferrite structures in Ti-alloyed HSLA steels with 0.23 % C, 1.5 % Mn and 0.05-0.4 % Ti. All investigations are done with so-called dipping samples. Therefore a steel melt was produced in a vacuum induction furnace, wherefrom samples were taken by dipping a submerged body into the melt on which the steel solidified. The detailed procedure of the dipping tests was already described in a previous work [12]. The dipping samples are heat treated in the HT-LSCM. [5,13-14]. Afterwards the samples are examined concerning AF amount and austenite grain size by etching and optical microscopy. Fig. 1 gives an overview about the experimental setup for the systematic analysis of the AF formation done within the project. The present work focuses on the metallographic analysis of this setup.



	VIM	Dipping Test	Metallography	HT-LSCM	Metallography
Steel composition	x				
Cooling rate		x		x	x
Austenite grain size		x	x	x	x
Non-metallic inclusions	x		x		

Fig. 1: Experimental setup and considered parameters of AF formation

3. RESULTS

3.1 SETTING A DEFINED INITIAL MICROSTRUCTURE BY AUSTENITIZATION

The austenite grain size is one of the major influencing parameters for the formation of AF. Therefore the setting of a defined austenite grain size before cooling the sample, in which the formation of AF occurs, and the determination of the austenite grain size are essential for a systematic investigation of the AF nucleation.
A standardized method for the determination of the austenite grain size [16] exists, but within this study a computerized method is used in order to achieve optimized results with less time effort. Due to the small observed area in the HT-LSCM the analysis of austenite grain size is done best by optical microscopy after HT-LSCM treatment. For the grain size determination on the sample surface no chemical etching is necessary because of the thermal etching during the HT-LSCM treatment. The determination of austenite grain size inside the sample can be done after etching with picric acid. Fig. 2 illustrates the procedure of austenite grain size analysis, which is a combination of manual and computer based evaluation. Based on this computer aided method grain size distribution can be determined, in contrast to the standardized methods [16] which just provide the amount of grains per length or area unit.

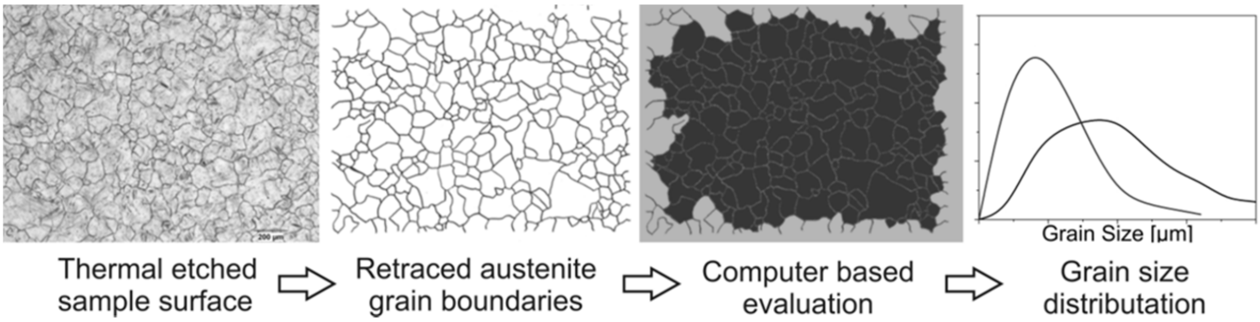


Fig. 2: Procedure of austenite grain size analysis

In Fig. 3 the influence of austenitizing temperature on the austenite grain size is illustrated. It can be seen that the mean grain size increases with increasing treatment temperature.

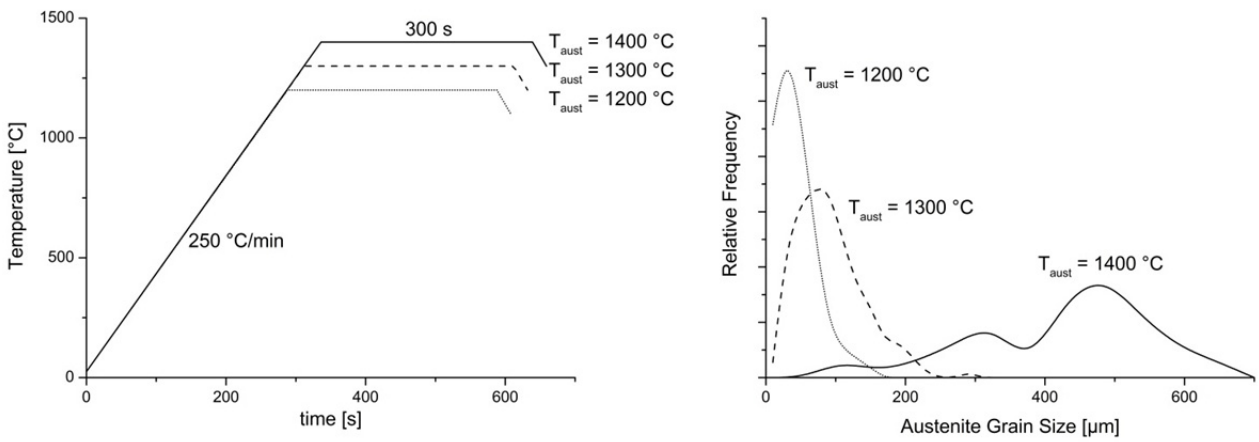


Fig. 3: Tested austenitizing temperatures and the corresponding austenite grain size distributions

3.2 IN SITU OBSERVATION OF THE ACICULAR FERRITE FORMATION DURING COOLING WITH DEFINED RATES

After setting of the defined initial austenite grain size the sample is cooled with defined rates. Fig. 4 shows an image sequence of the in situ observation of phase transformations during heat treatment by HT-LSCM. The formation of AF inside the austenite grain is clearly observable. It is apparent that AF nucleates on non-metallic inclusions, contrary to perlite and bainite which nucleate at grain boundaries. Due to the well controllable temperature cycles the effect of austenitizing temperature, holding time and cooling rate can be investigated in situ in a systematic way.

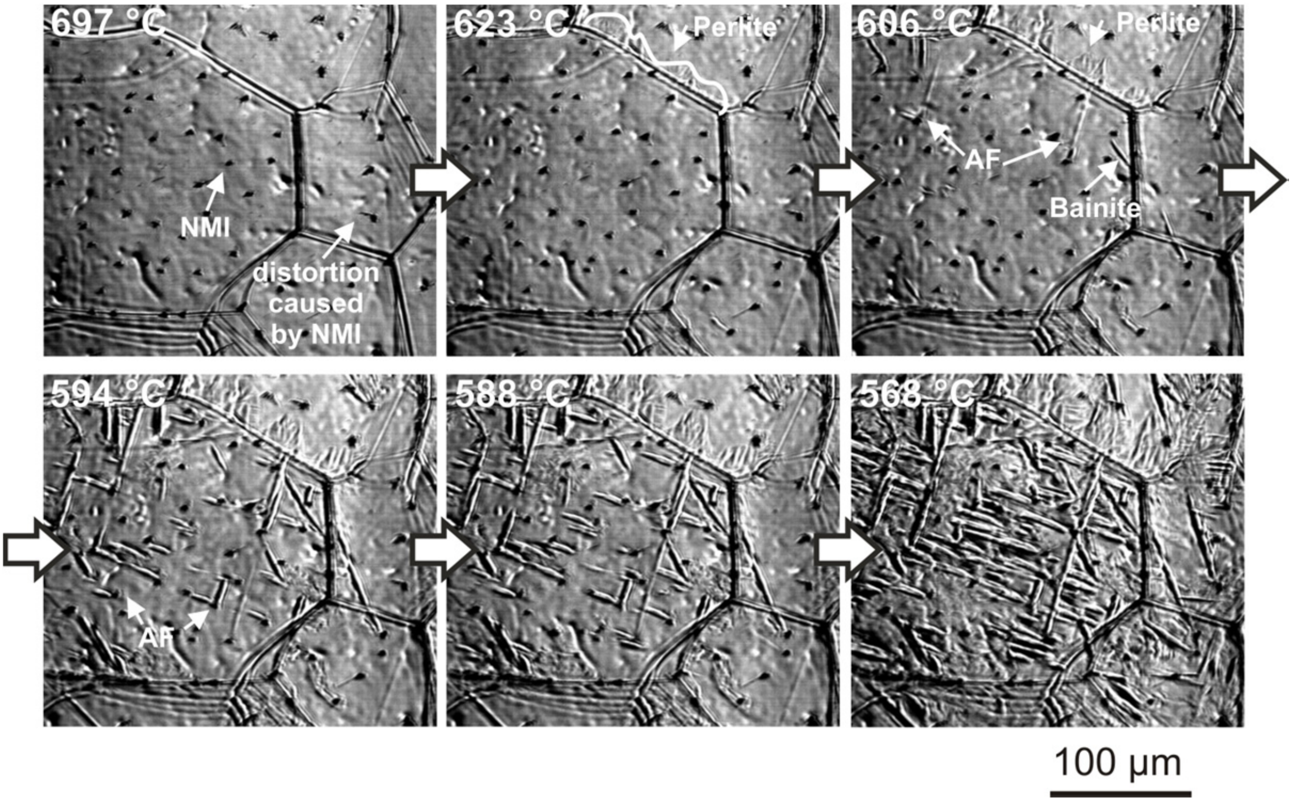


Fig. 4: In situ observation of AF-formation by HT-LSCM (sample austenitized for 100 s at 1400 °C and cooled with about -7 °C/s between 800 and 600 °C)

3.2 ANALYSIS OF ACICULAR FERRITE AMOUNT

A standardized method for the determination of the AF amount [15] exists. Although these method offer a valuable basis for manual analysis, within this study a computerized method is developed and used in order reduce time effort. The amount of acicular ferrite in microstructure is determined computer based using an evaluation routine of the image analysis software Clemex: selection of ferrite by color thresholds → sorting out polygonal ferrite and Widmannstätten ferrite by grain size → correction of grain boundary etching effects → calculation of selected acicular ferrite area. Fig. 5 illustrates this process. The comparison of the computerized method using Nital etching with the standardized method [15] shows a good agreement.

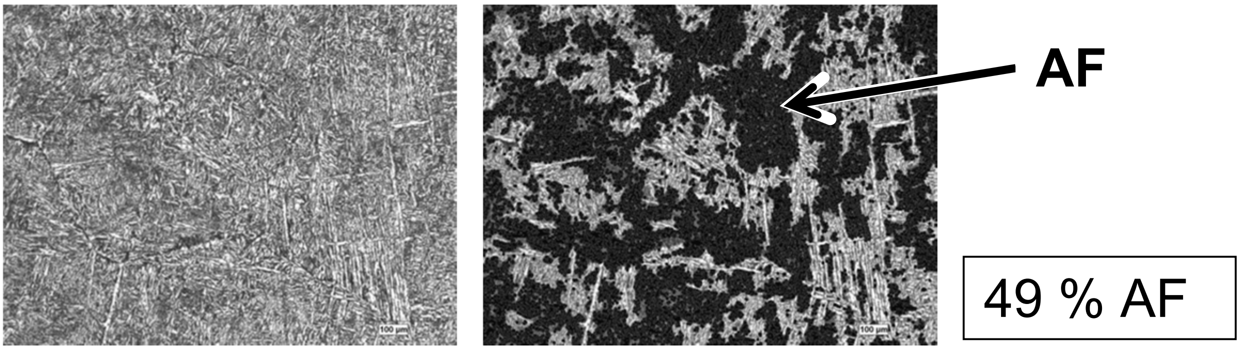


Fig. 5: Computer based evaluation of acicular ferrite amount in microstructure

The sample shown in Fig. 4 is subsequently etched with Nital and analysed by the computer based evaluation to an AF amount of 11 % on the total sample area. The pretended higher AF amount in the LSCM view field is maybe caused by the large austenite grain on which the LSCM focused. This demonstrates the benefit of a combination of HT-LSCM and optical microscopy. HT-LSCM can be done with high magnification to illustrate the phase transformation and optical microscopy is performed at lower magnification to investigate larger areas to obtain statistical results.

3. SUMMARY

The present study focuses on the characterization of AF microstructures in HSLA steels by a combination of metallographical methods. Heat treatment is done in the high-temperature furnace attached to a LSCM. HT-LSCM provides an accurate adjustment of temperature, well controllable cooling conditions and the opportunity of an in situ observation of phase transformations at high magnification. The austenite grain size is one of the major influencing factors for the nucleation of AF. The study of austenite grain size by HT-LSCM is difficult due to the high magnification and the low amount of grains visible in the microscope view field. The computerized investigation by optical microscopy enables the determination of grain size distribution on the total sample area. The influence of austenitizing temperature, holding time and cooling rate can be analysed systematically. The amount of AF after heat treatment can be determined by conventional metallography including etching, optical microscopy and a computerized data evaluation. The lower magnification of optical microscopy and the computer based evaluation makes it possible to analyse larger areas compared to the HT-LSCM to get statistically reliable results. Summing up, the combination of HT-LSCM and conventional metallography allows a systematic study of the influence of cooling conditions and austenite grain size on the amount of AF in microstructure for different steel compositions.

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