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Enhanced Homogenization Strategy by Electroslag Remelting of High-Manganese TRIP and TWIP Steels**

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In the production of high-manganese TRIP and TWIP steels, significant micro-segregation effects are observed after solidification. Homogenization can be achieved by heat treatment followed by deformation at a significant time and effort (forging, annealing, hot rolling). In an attempt to achieve the best homogeneous microstructure and reduced thermomechanical processing, the cast alloy is treated by electroslag remelting (ESR). After a simple hot rolling procedure with a thickness reduction of 90% and without further heat treatment, micro-segregation of manganese can be reduced to about 5 wt%. There is a potential to achieve even lower values with further optimization of the ESR process and an adapted thermomechanical processing.

High-manganese (≈ 15–30 wt% Mn) austenitic steels (HMS) offer an interesting combination of high strength and high plastic formability thus enabling to face the new challenges in the automotive industry, to improve the fuel efficiency and to achieve a better environment preservation. Depending on the Stacking Fault Energy (SFE) TRansformation Induced Plasticity (TRIP), and TWinning Induced Plasticity (TWIP) processes dominate the forming behavior.[1–4]

In the collaborative research centre (SFB761) ''Stahl—ab initio'' of the Deutsche For schungsgemeinschaft (DFG) numerical steel design based on atomistic models is combined with state of the art steel design and characterization.[5] Several work packages in this collaboration also focus on the technical challenges of the common process chain for the production of samples and potential semi-products of high-manganese steels. Typically, the obtained raw materials are characterized for impurities, weighed, and alloyed by vacuum induction melting (VIM). The aim of this first process step is to achieve fast melting under vacuum in order to reduce losses of oxidation-prone alloying elements (especially with regard to Mn) and obtain a homogeneous melt that is subsequently cast and solidified. Upon solidification Scheil-cooling effects can be observed and result in significant micro- and macro-seggregation.[5,6] It was reported that an HMS Fe-Mn22-C0.3 cast block with a weight of ~100 kg exhibits micro-segregations of Mn of more than 8 wt%. Therefore, these blocks were homogenized in a process route consisting of forging at 1 150 °C with about 60% thickness reduction and annealing at 1 150 °C for 5 h. The segregations could be reduced to ~4 wt% manganese. Additional hot rolling and a total reduction in thickness of 99% leads to manganese segregations of less than 2 wt%.

Figure 1 illustrates the general approach of the work presented in this paper. In order to achieve the same final level of micro-segregation, with less thermo-mechanical processing steps or reduced effort (time and energy), cast electrodes were refined by electroslag remelting (ESR). This process is widely applied in the production of specialty steels and superalloys nowadays, in order to obtain ingots with superior cleanliness, minimized macro-segregation, and a more homogeneous microstructure. As a basic principle the tip of a consumable electrode, made from the alloy to be re-fined, is submerged into a molten slag which is contained in a water-cooled copper crucible (see Fig. 2). Electrical current (most commonly AC) is applied between the electrode and the slag resulting in joule heating up to the melting point of the metal. A liquid metal film forms on the downside of the electrode and hence metal droplets are released which sink through the slag bath due to the difference in density. The slag bath floats on a liquid metal pool which collects the droplets. Through cooling at the mold surfaces, solidification occurs and the process continuously builds up a refined ingot with a controlled microstructure and smooth outer surface.

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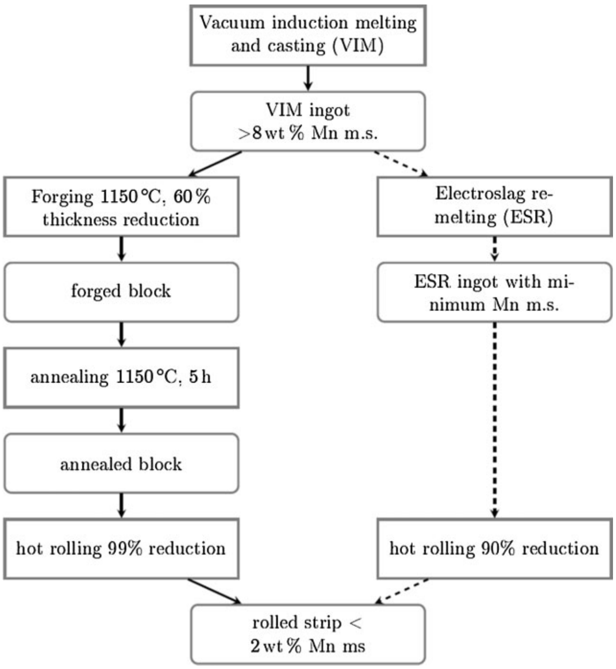

Fig. 1. Proposed process chain for reduced micro-segregation in HMS.

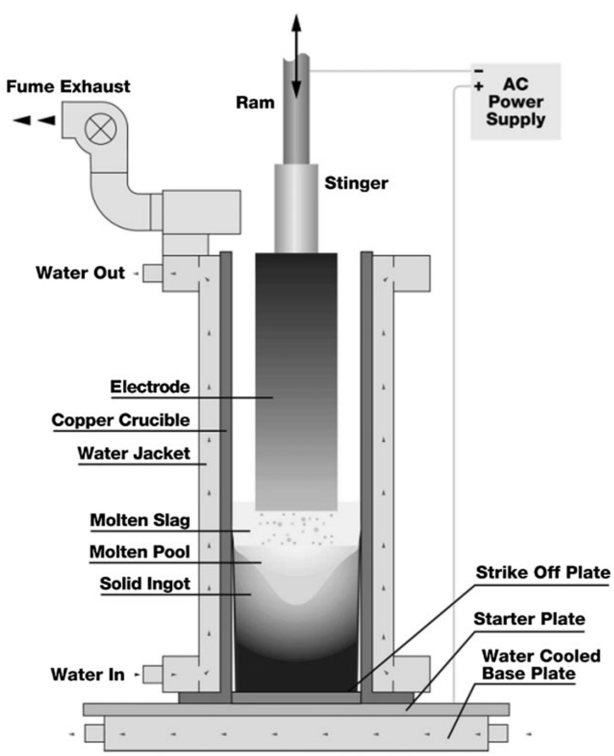

Fig. 2. Principle of ESR.[8]

Materials and Methods

Electrode Preparation

The raw material was melt and cast by IEHK, department of ferrous metallurgy of the RWTH Aachen University, into squared blocks of 45 kg, with a $140 \times 140\ mm^2$ cross-section.

Fig. 3. Electrode prepared for ESR by forging and welded to power connection (stub).

From this block an electrode for ESR was prepared at the Institute of Metal Forming (IBF) by forging the square cross-section into a polygonal shape with 110 mm outer diameter using a 6.3 MN hydraulic forging press. The block was handled with a hydraulic 6-axis heavy-duty robot. The forging temperature was 1 150 °C and the block was reheated between forgings. The forging was processed by picking the block consecutively from both ends in order to obtain the maximum electrode length and to close solidification cavities as best as possible at one end of the electrode. Forging losses were minimal and led to an electrode mass of 43.6 kg. The obtained electrode is presented in Figure 3 and exhibits a length of 610 mm.

Electroslag Remelting

In order to prevent manganese oxidation and allow for better composition-control the melt in this work was carried out under Argon atmosphere in a pilot-scale PESR furnace originally produced by Leybold-Heraeus (now ALD Vacuum Technologies, Hanau). The equipment is capable of operating at inert gas overpressures up to 50 bar and is powered with a 5 kA/66 V supply. It features latest data-logging, monitoring, and melt-control systems. Melting took place in a tapered copper-crucible (diameter, 159–178 mm; length, 880 mm) using the most common industrially available ESR-slag for steels with an approximate composition of 31 wt% CaF_2, 30 wt% CaO, 33 wt% Al_2O_3 and minor content of other oxides.

Start-up of the process was realized by a specially invented procedure using a starter-box made from iron sheet and filled with scrap metal and turnings for arc ignition in the slag. In order to minimize micro-segregation the target melt-rate in kg h^{-1} was determined by an industrially proven rule-of-thumb for ESR or steels or superalloys (1).

$$\mathrm{MR} = F \cdot d_c \tag{1}$$

with MR is the melt rate (kg h^{-1}), F the empirical factor (kg mm^{-1} h), and d_c is the crucible diameter (mm).

The factor F typically ranges from 0.4 for segregation prone materials (superalloys, high Mn-steels) to 0.8 for less segregation sensitive steels with lower contents of alloying elements. For the present melt a factor of 0.45 was chosen giving a target melt rate of 1.2 kg min^{-1}, which for the present set-up can be achieved at a power supply of $\approx$ 110 kW.

The melting process was conducted according to industrial standards by melt-rate-control for uniform melting speed at slightly decreasing power supply and swing-control for control of optimal immersion depth of the electrode in the slag. After the melting process, the ingot was allowed to

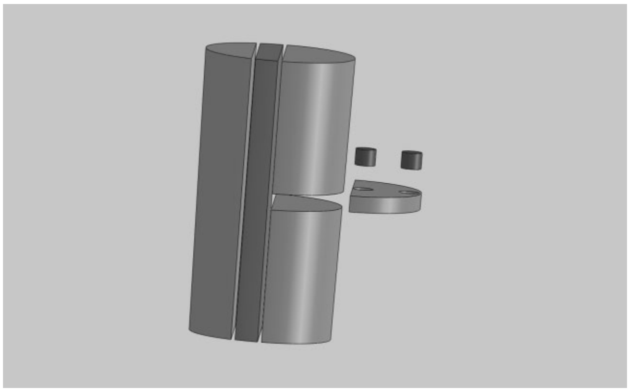

Fig. 4. Scheme of sampling and extraction of rolling strip from the ESR ingot.

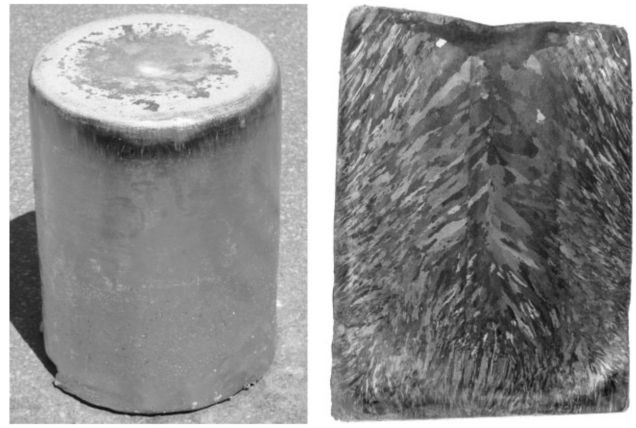

Fig. 5. Mn-steel ingot after removal of slag skin and longitudinal section which shows typical semi-directional solidification structure after ESR.

cool down for 2 h and then carefully removed from the crucible.

Thermo-Mechanical Processing

A longitudinal section was obtained from the ESR ingot by sawing for further rolling as illustrated in Figure 4. At about half the ingot length where stationary melting and cooling conditions are achieved, two samples were extracted on the outer radius and from the core of the ingot in order to compare micro-segregation at different radii and thus at different local solidification times.

In contrast to the processing by forging and annealing presented in ref.[7] no heat-treatment was conducted. The strip of the material obtained from the ESR ingot was simply pre-heated to 1 150 °C and directly hot-rolled thereafter. The hot rolling of the 28 mm thick slab was performed within 6 passes using a two high mill stand with 300 mm roll diameter and 1 200 kN maximum rolling force. Before the first pass the slab was heated through for 1 h at initial rolling temperature of 1 150 °C, after each pass the slab was reheated to this temperature for 5 min. The occurring scale was removed with a high pressure water jet directly before each pass. The final thickness of $\approx$ 2.8 mm equals to a total thickness reduction of only 90% compared to a reduction in thickness of 99% in the previous works.[7] The sheet was cooled down at free air.

Investigation of Microstructure

Samples were obtained from the ESR ingot as indicated in Figure 4 from the outer radius of the block and from the central section in order to investigate the influence of local solidification time on the obtained micro-structure. Electron microprobe measurements were performed by the Central Facility for Electron Microscopy of the RWTH Aachen University (GFE) for local determination of Fe, Mn, C, and Cr concentration. The Electron Probe Micro Analyser (CAMEBAX SX 50) is equipped with four wavelength-dispersive (WD) spectrometers. A focused electron beam of 15 keV with a current of 80 nA was used. The characteristic X-ray intensities, i.e., Mn Kα, Cr Kα, Fe Kα, and C Kα, were calibrated by using the following standards: Mn, Cr, Fe, and Fe_3C. The calibrated intensities (*k*-ratios) were transferred into elemental concentrations with the help of a matrix correction procedure.

A macro-etching and a Baumann sulfur print of the longitudinal and perpendicular cross-section of the ingot were performed at IEHK.

Results

A good utilization of the electrode (almost 99%) was achieved during ESR resulting in an ingot with a weight of 41.4 kg and a height of 23.5 cm. Through computer control, constant melt rates of 1.2 kg min^{-1} could be achieved and a thin slag skin of $\approx$ 1 mm was observed that could easily be removed after melting. Figure 5 displays the obtained ingot and a macroetching of the longitudinal section, showing the classical as-produced structure of ESR ingots with a columnar solidification pattern at the mold walls and increasing grain-sizes toward an equiaxed solidification zone in the center-line of the ingot. The cooling effect of the bottom plate on the direction of heat transfer during the start-up phase and thus on solidification is clearly visible in the lower third of the etching.

The cross-section macro-etching and the BSE micrographs presented in Figure 6 clearly exhibit the well known effect of increasing local solidification times from the mold walls toward the center of the ingot. Grain-structure in the central part is pronouncedly more coarse and segregation effects of Mn on micron-level are qualitatively visible through more intense b/w contrast in the BSE imaging.

On the obtained samples micro-segregation of Fe, Mn, Cr, and C can quantified as the difference in wt% between the minimum and maximum values analyzed on the whole scanning distance in a first and simple approach. As can be observed in Figures 7 and 8, the line scans offer a scanning resolution of 1 μm, which is sufficient for dendrite arm spacings in the range of 50 μm as observed in the casting structure after ESR (see Figure 5).

After hot rolling, significant refining of the structure can be observed with regard to local Fe and Mn concentrations, as

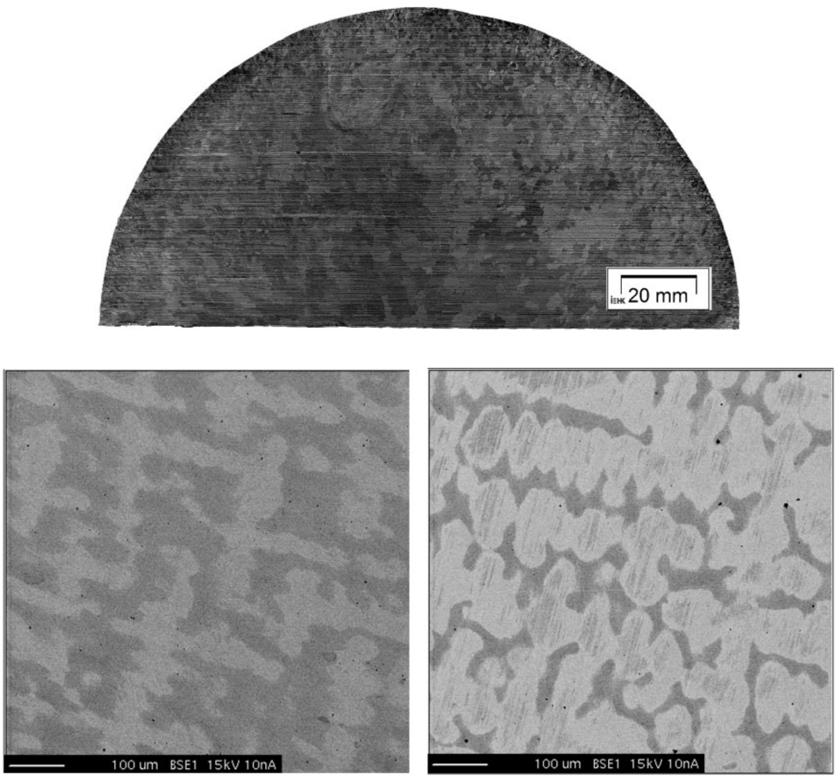

Fig. 6. Macro-etching of cross-section (above) and SEM/BSE exhibits ESR casting-type microstructure. With increasing local solidification times from radius (left) to core (right) Mn micro-segregation becomes more pronounced.

can be seen in Figure 7. A drop in Mn concentration at a position of 150 μm in one of the samples can be recognized that cannot be explained and is most likely due to a measurement failure or the back-scatter of a non-metallic inclusion. This requires a slight modification with regard to the quantification of micro-segregation: For all four samples we determined micro-segregation as the span (maximum–mininum) on a 3 μm moving median of the line scan which gives good compensation of exceptional values and still offers sufficient resolution (compare Table 1).

The sulfur print obtained from the ESR material did not show any exceptional concentrations of sulfur on the strip and thus a certain desulfurization can be observed as expected from a CaO-containing slag. However, this effect is not in the scope of the present paper and was therefore not examined in detail on the material.

Assessment

As laid out above, the measurement and quantification of micro-segregation from a line-scan is relatively straightforward and segregation can be calculated as the span of the observed values. However, to compensate for measuring errors a reproducible method has to be found for fair comparison of the results. For the present case, we proposed to smoothen the line-scan signal by a running median over three positions, i.e., a resolution window of 3 μm which still gives valid results at the length scale observed, but removes exceptional values in a comparable way.

Regarding preparation of the electrode by intensive hot forging, it has to be noted that this procedure has no effect on the microstructure of the obtained ESR-ingot. During ESR the metal is completely molten and it is generally agreed that the super-heat of the metal pool is in the range of 100 K. As a result the solidification structure and the effect of simplified forming in microstructure observed in this paper are merely a result of the solidification conditions during ESR. However, macro-segregation effects in the cast block on the

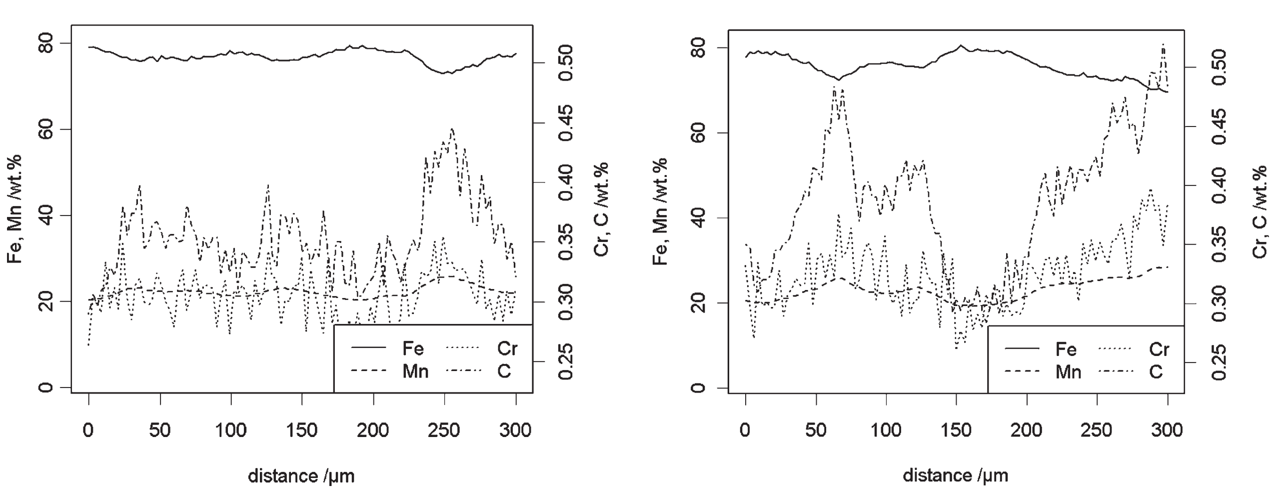

Fig. 7. Mn micro-segregation quantified by line-scans increases with increasing local solidification time from 5.49 wt% on the outer radius (left) to 8.94 wt% in the core (right) of the ESR ingot.

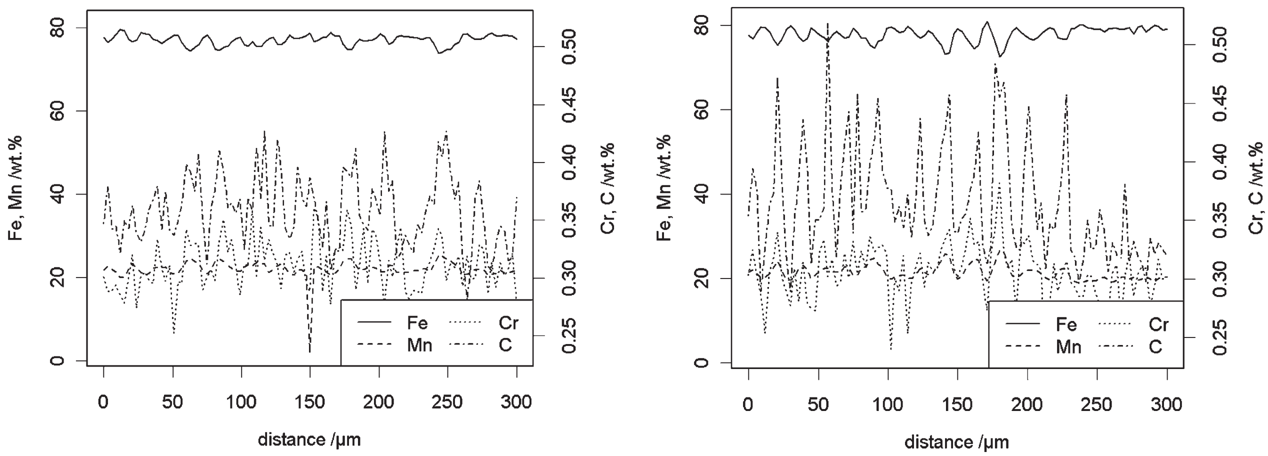

Fig. 8. After rolling Mn micro-segregation could be reduced to 4.8 wt% (left) and 6.88 wt% (right), respectively.

Table 1. Quantification of Mn micro-segregation (MS) in the obtained samples.

	MS (wt% Mn) no smoothing	MS (wt% Mn) with 3 µm smoothing
ESR radius	5.64	5.49
ESR core	9.13	8.94
Rolled 1	23.50	4.80
Rolled 2	7.68	6.88

scale of the block length may be observed in the ESR-ingot even if the process homogenizes these effects to a certain extent. With regard to a simplified process chain (Fig. 1) we have to add that the preparation of a cylindrical electrode by forging was only necessary for the present experimental conditions and can be avoided by either conducting the ESR process in a slab mold or by casting the electrodes in a cylindrical mold directly.

Conclusions

Reduction of micro-segregation is a prominent topic in the processing of high-manganese steels, which can be tackled by advanced melting and thermo-mechanical processing techniques. In the presented works a cast electrode of the composition Fe-21Mn-0.3 C was processed by ESR and subsequent hot rolling. A reduction of micro-segregation from 9 wt% to a final level of 4.8–6.8 wt% could be achieved as opposed to a final level of 2 wt% obtained after intensive forging, annealing, and hot rolling in a previous work.

It could be shown that ESR offers an interesting potential for simplification of processing of these materials with respect to the reduction of micro-segregation prior to thermo-mechanical treatment when correctly controlled. In this context a close and fundamental investigation of slag properties (e.g., melt interval) and process parameters (e.g., melt rate) is necessary to establish a segregation-optimized ESR procedure which can reduce effort and time in mechanical processing.

Apart from micro-segregation also the removal of sulfur and non-metallic inclusions by ESR and the investigation of reduced macro-segregation in ESR ingots offer an interesting perspective for further research and will be examined in separate publications.

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