An alternative approach for the experimental verification of microsegregation models using an in-situ hot tensile test during solidification of steel

Michael BERNHARD[1], Christian BERNHARD[1], Peter PRESOLY[1] and Dali YOU[1]

[1]Chair of Ferrous Metallurgy, Montanuniversitaet Leoben, Franz-Josef-Strasse 18, Leoben, Austria

michael.bernhard@unileoben.ac.at

Keywords: steel; solidification; hot tearing; microsegregation; continuous casting;

Abstract. The presented paper deals with the development of an alternative approach for the experimental verification of microsegregation models with the potential extension to advanced steel grades using an in-situ hot tensile test. Based on experimental results for more than 100 plain carbon steels, the presented method is generally evaluated using a 1D-FV solidification algorithm and a modified microsegregation model as suggested by Ohnaka [1,2]. The study shows that slight modifications of the input parameters for the microsegregation model show significant influence on the predicted results. The high sensibility makes it possible, to clearly identify the influence of alloying elements on the final part of solidification.

Introduction

In continuous casting of steel the exact calculation of non-equilibrium solidification and solute enrichments on microscopic scale is an essential part of a successful process control. Particularly the implementation of concepts with respect to industry 4.0, adjustment of soft reduction and optimized cooling strategies requires an accurate prediction of the shell formation and the final solidification point. In the past decades various analytical and numerical microsegregation models have been published in literature [2-8]. Coupling the calculations with high precise thermodynamic databases is a powerful way to investigate solidification phenomena also for advanced steel grades.

The experimental verification of microsegregation models with respect to steel can be generally divided into two methods: *Directly* the composition along the dendritic microstructure subsequently to a controlled solidification process can be measured using Electron Microprobe Analysis [6,8]. Since carbon and other elements show quite high diffusivity in iron, results for steels are conditionally usable. Within the *indirect way* the common hot tensile test [9,10] is applied: After complete remelting, the test sample is cooled down to a given temperature and the tensile strength and hot ductility are measured. Based on the temperature dependent results, the Zero Strength Temperature (ZST) and Zero Ductility Temperature (ZDT) of the investigated steel grade can be determined assuming correlated fractions of solid. However, it is quite challenging to guarantee defined experimental conditions and literature information is limited to just a few plain carbon steels.

The objective of this work is to develop and critically evaluate an alternative approach for the experimental verification of microsegregation models with a possible application also to advanced steel grades. Several advantages of the present in-situ method are:

- Controlled heat transfer and temperature evolution,
- Defined columnar dendritic growth,
- The possibility of an accurate numerical calculation of the solidification progress and
- Conditions close to continuous casting of steel.

Experimental and numerical approach

In the past decades the Submerged-Split-Chill-Tensile (SSCT) equipment, originally developed at EPF Lausanne [11] has been successfully applied for the characterization of hot tear sensitivity of various steel grades at the Chair of Ferrous Metallurgy at Leoben [12]. As a “co-product” it is possible to correlate the position of the formed hot tears during the experiment with simulation results for the solidus isotherm in order to validate microsegregation models on laboratory scale. The experimental procedure should be described here only briefly, detailed information can be found elsewhere [13].

In-situ hot tensile test (SSCT-test). The cylindrical test body is made of construction steel and consists of an upper and a lower part. Two thermocouples of type k are placed 2 mm beneath the surface of the lower part to measure the temperature and to determine the heatflux during the experiment. In general the SSCT-test procedure can be divided into four stages, Fig. 1 [13]:

- The test body is placed above the liquid steel at *stage I*. The melt is prepared in an induction furnace with a capacity of 25 kg.
- During *stage II* the test body is submerged into the liquid steel with a velocity of 1.2 m/min and shell growth proceeds for a specified period of time.
- At *stage III* the actual tensile test starts: The lower part moves down with a given velocity (corresponds to a given strain rate) and a predefined total strain is induced into the solidifying shell. If the strain exceeds critical values, hot tear segregations (HTS) will be formed during the experiment.
- After the tensile test has finished the test body is lifted out of the melt during *stage IV*, whereby the velocity is identical to submerging. After the specimen is cooled down to room temperature the solidified shell is cut into 16 samples for the metallographic investigations.

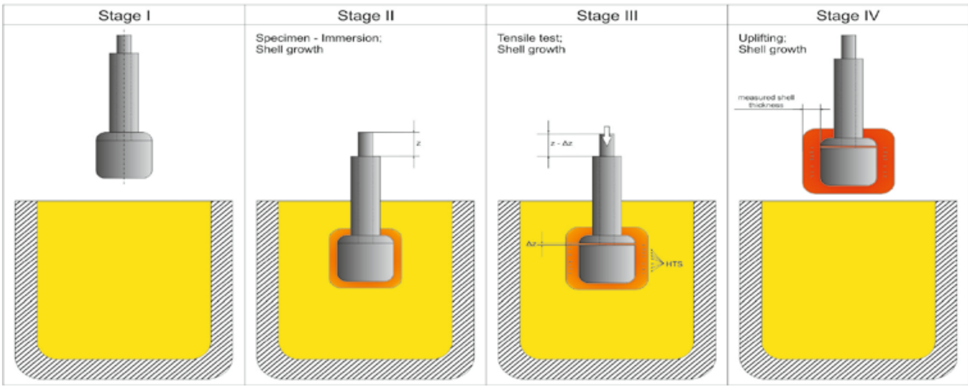

Fig. 1: Different experimental stages of the SSCT-test [13]

Correlation of hot tear statistics and numerical simulation. The principle of correlating the metallographic results with the numerical simulation of the experiment is shown in Fig. 2. A typical Low Carbon (LC) steel grade is taken as an example.

Metallographic analysis and hot tear statistics: The samples are etched with Bechet-Beaujard solution [14] to visualize present HTS. Within the microscopic analysis the position of HTS with respect to the test body surface as well as the shell thickness is measured Fig. 2 (a). The statistical analysis of the HTS position gives the frequency maxima of start and end of HTS in the sample. In case of the LC steel most of the HTS start to form at a distance of 6.18 mm from surface and end at 7.01 mm Fig. 2 (b). Since HTS only occur if the solidifying shell is mechanically loaded, the maxima correspond to the start and end of the tensile test, respectively. Under the assumption that HTS form within a preferred temperature range very close to solidus temperature (f_s = 0.96 - 1) [15] two points for the solidus isotherm (6.18mm/18s and 7.01mm/23s) are obtained. The measured shell thickness after the SSCT-test is 10.62 ± 0.37 mm and generally corresponds to a calculated fraction of solid f_s = 0.2 (10.62mm/25s). Hence, three points are available to accurately reconstruct the shell growth during the SSCT-experiment and to evaluate the calculated non-equilibrium solidus isotherm as illustrated in Fig. 2 (c). Regarding microsegregation models for advanced steel grades (e.g. coupling with thermodynamic databases) it is perhaps necessary to perform the tensile test under different experimental conditions (solidification time or testing time), subsequently one gets even more points for a fixed chemical composition and reproducibility is guaranteed.

Solidification simulation and microsegregation evaluation: An in-house 1D-FV solidification model is used to calculate the shell growth during the SSCT-test, Fig. 2 (c). Input parameters for the simulation are the superheat of the melt (typically 20-30°C) and the determined heatflux based on the measured temperature. For the general evaluation of the present approach the semi-integrated form of Ohnaka's equation according to You et al. [1] is implemented to predict the microsegregation during solidification. Note that the used microsegregation model [16] is a "stand-alone" version and therefore not as high sophisticated as the model of You et al. [1]. The advantage of the semi-integrated form is the possibility to use local partition coefficients dependent on temperature or solute enrichment. An additional tool of the software enables the plot of various temperature profiles to determine the "measured" solidus isotherm. In case of the LC steel the results of solidus temperatures from the SSCT experiment are 1492 °C and 1489 °C. Finally, the "measured" temperature is 1490.5±2°C which is in very good agreement with the calculated one using the semi-integrated Ohnaka equation [1] (1491.7 °C).

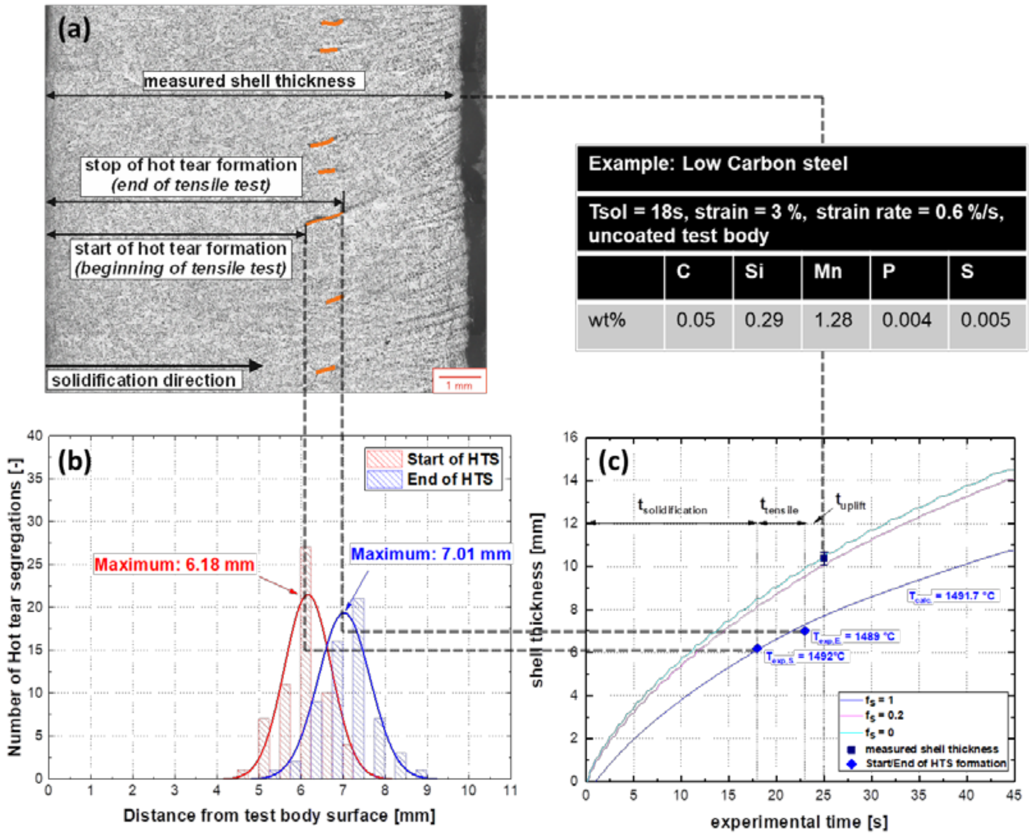

Fig. 2: Principle of determining the solidus temperature from SSCT-test: (a) metallographic examination, (b) hot tear statistics and (c) correlating metallographic results with the solidification simulation

Results and discussion

The previous explained procedure for the LC steel grade was applied for more than 100 plain carbon steels. Composition ranges of the investigated steel grades are listed in Table 1.

Table 1: Composition ranges of investigated steel grades

C [wt%]	Si [wt%]	Mn [wt%]	P [ppm]	S [ppm]
0.02-1.05	0.01-0.36	0.08-2.01	20-260	20-230

Within the first analysis the partition coefficients k_i as proposed by Ueshima et al. [6] were used to perform the microsegregation calculations. As can be seen in Fig. 3 the predicted solidus temperature of LC and Medium Carbon (MC) steels is generally too high (10-15°C) compared to the “measured” temperature. In order to improve the microsegregation model the partition coefficients were slightly modified by extracting the values for k_i from the FSStel2015 database using the thermodynamic software FactSage [18]. In austenite the concept of a local partition coefficient for carbon is applied to define the value dependent on the carbon enrichment $c_l(C)$ itself (Table 2). Although this procedure is not the conservative way to couple the microsegregation model with a thermodynamic database as suggested by You et al. [1], it is a simple and fast possibility to adjust the model and also to visualize the benefit of using newly assessed databases.

Table 2: Equilibrium partition coefficients k_i used for the microsegregation calculations

		C	Si	Mn	P	S
δ	(FSStel2015) [-]	0.17	0.70	0.70	0.32	0.04
	(Ueshima et al.)	0.19	0.77	0.76	0.23	0.05
γ	(FSStel2015)	$0.295+0.057c_l(C)$ $-0.005c_l(C)^2$	0.83	0.70	0.16	0.02
	(Ueshima et al.)	0.34	0.52	0.78	0.13	0.035

By repeating the microsegregation calculations with the coefficients from FSStel2015 [17] significantly better results can be obtained due to the optimized segregation description of the alloying elements. In case of LC steel grades the deviations are smaller than 5 °C. Obviously the segregation of carbon was underestimated using the coefficients from Ueshima et al. [6]. Since the concentration Phosphorus, Sulphur and Manganese of the investigated MC alloys is in a wide range, the improvement is not as clear as for the LC steel grades. The extension of local partition coefficients to all alloying elements would definitely enhance the results. Although, a better agreement between measurements and calculations can be found particularly at lower solidus temperatures the accurate description and further development of thermodynamic data for strong segregating elements (e.g. Phosphorus) is of high importance. This topic is part of recent research at the Chair of Ferrous Metallurgy at Leoben.

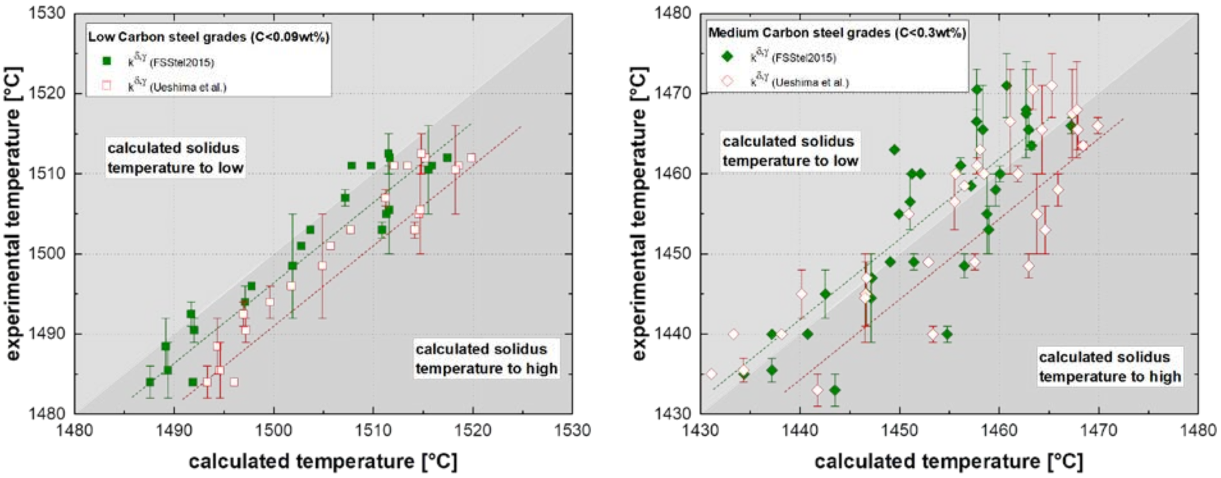

Fig. 3: Evaluation of calculated solidus temperature for Low Carbon and Medium Carbon steels

The investigation of High Carbon (HC) and Ultra High Carbon (UHC) steel grades gives promising results and confirms the present approach to adjust microsegregation models based on the SSCT-test: High Carbon (HC) and Ultra High Carbon (UHC) steels with carbon concentrations between 0.5-1 wt% solidify in pure austenitic microstructure and carbon is the dominating alloying element. Due to the high initial concentration and the reduced diffusivity at lower temperatures, typical carbon enrichment can exceed 3wt% in the interdendritic liquid. As a consequence the accuracy of the microsegregation calculation is mainly defined by the carbon segregation description. Inserting the mentioned concentration into the equation for k_C (Table 2) gives a local partition coefficient of 0.42 close to the end of solidification. Fig. 4 shows the comparison of the predicted and "measured" solidus temperatures. The prediction of higher carbon segregation tendency based on the fixed carbon coefficient (k_C=0.34) leads to a deviation of nearly 80 °C (!) whereas the prediction with adjusted microsegregation model shows only small differences to the experimental solidus temperature. This observation further indicates that already the calculation of low alloyed UHC steels requires a (partial) consideration of precise thermodynamic data (e.g. partition coefficients).

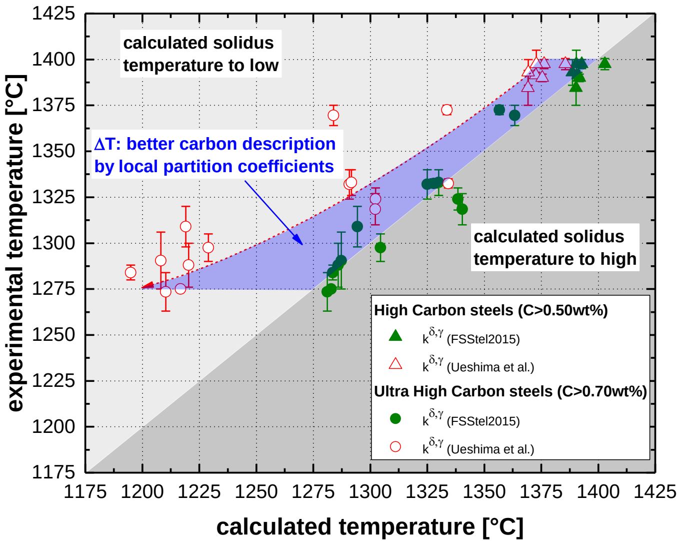

Fig. 4: Evaluation of calculated solidus temperature for High Carbon and Ultra High Carbon steels

Summary and outlook

First evaluations of a microsegregation model using the Submerged-Split-Chil-Tensile (SSCT)-test showed quite promising results. Within the analysis of more than 100 plain carbon steels, it was observed, that already slight modifications of input parameters can improve the predicted solidus temperature. As an example the partition coefficients of the alloying elements were adjusted according to the FSStel2015 [17] database. Particularly in case of High Carbon and Ultra High Carbon steel, the concept of an optimized local partition coefficient for carbon leads to a significant better agreement with the experimental results.

Future research on this topic aims to extend the present method to advanced steel grades and couple the microsegregation models with high precise thermodynamic databases. The development and investigation of thermodynamic databases for microsegregation calculations is part of a further work package within the project (*Presoly, P.:"High concentrations at the final solidification of advanced steels: Thermodynamic evaluation of replicated "segregation-samples" by means of DTA/DSC-measurements"*).

Acknowledgement

Financial support by the Austrian Federal Government (in particular from Bundesministerium für Verkehr, Innovation und Technologie and Bundesministerium für Wirtschaft, Familie und Jugend) represented by Österreichische Forschungsförderungsgesellschaft mbH and the Styrian and the Tyrolean Provincial Government, represented by Steirische Wirtschaftsförderungsgesellschaft mbH and Standortagentur Tirol, within the framework of the COMET Funding Programme is gratefully acknowledged. This work is part of the ongoing K2-MPPE project P3.1 "SmartCast".

References

[1] You, D., C. Bernhard, G. Wieser and S. Michelic, Microsegregation Model with Local Equilibrium Partition Coefficients During Solidification of Steels, Steel Research International 87 (2016), 7, 840–849.

[2] Ohnaka, I., Mathematical analysis of solute redistribution during solidification with diffusion in solid phase, Transactions ISIJ 26 (1986), 12, 1045–1051.

[3] Scheil, E., Bemerkungen zur Schichtkristallbildung, Zeitschrift für Metallkunde 34 (1942), 3, 70–72.

[4] Brody, H.D. and M.C. Flemings, Solute redistribution in dendritic solidification, Transactions of the Metallurgical Society of AIME 236 (1966), 5, 615–624.

[5] Clyne, T.W. and W. Kurz, Solute redistribution during solidification with rapid solid-state diffusion, Metallurgical and Materials Transactions A 12 (1981), 6, 965–971.

[6] Ueshima, Y., S. Mizoguchi, T. Matsumiya and H. Kajioka, Analysis of Solute Distribution in Dendrites of Carbon Steel with delta / gamma Transformation During Solidification, Metallurgical Transactions B 17 (1986), 4, 845–859.

[7] Kobayashi, S., A Mathematical Model for Solute Redistribution During Dendritic Solidification, Transactions ISIJ 28 (1988), 7, 535–542.

[8] Matsumiya, T., H. Kajioka, S. Mizoguchi, Y. Ueshima and H. Esaka, Mathematical analysis of segregations in continuously-cast slabs, Transactions ISIJ 24 (1984), 11, 873–882.

[9] Schmidtmann, E. and F. Rakoski, Influence of the carbon content of 0.015 to 1% and the structure on the high-temperature strength and toughness behaviour of structural steels after solidification from the melt, Archiv fuer das Eisenhuettenwesen 54 (1983), 9, 357–362.

[10] Seol, D.J., Y.M. Won, T. Yeo, K.H. Oh, J.K. Park and C.H. Yim, High temperature deformation behavior of carbon steel in the austenite and delta - ferrite regions, ISIJ International 39 (1999), 1, 91–98.

[11] Ackermann, P., Kurz, W., and Heinemann, W., In situ testing of solidifying Aluminium and Al-Mg shells, Materials Science and Engineering 75 (1985), 1-2, 79-86.

[12] Pierer, R. and Bernhard, C., The nature of internal defects in continuously cast steel and their impact on final product quality, AIST Proceedings, Pittsburgh, USA, 2010, 193-203.

[13] Arth, G., S. Ilie, R. Pierer und C. Bernhard, Experimental and Numerical Investigations on Hot Tearing during Continuous Casting of Steel, BHM 160 (2015), 3, 103–108.

[14] Bechet, S. and Beaujard, L., Nouveau reactif pour la mise en évidence micrographique du grain austénitique des aciers trempésou trempés-revenus, Revue de Metallurgie, 92 (1995), no.10, 923–929

[15] Bernhard, C. and Pierer, R., A new hot tearing criterion for the continuous casting process, Proceedings of the 5th Decennial International Conference on Solification Processing, Sheffield, UK, 2007, 525–530

[16] Bernhard, M., Presoly, P., Bernhard, C., Six, J. and Ilie, S., On the relevance of microsegregation models for process control in continuous casting of steel, Proceedings of the 26th International Conference on Metallurgy and Materials, Brno, CZE, 2017, 38-44.

[17] Bale, C.W. et al., FactSage thermochemical software and databases, 2010–2016, Calphad 54 (2016), 35–53.