THE NEWLY DEVELOPED IN-SITU MATERIAL CHARACTERIZATION (IMC-) TEST: INSTALLATION, START-UP AND FIRST RESULTS

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ABSTRACT

The experimental characterization of the mechanical properties of steel at high temperatures, regarding strength, ductility and crack formation under continuous casting conditions, is of vital interest. The commonly employed method for determining the hot ductility of steel is the determination of the reduction of area from ruptured hot tensile test samples, c.f. [1]. However, in order to simulate continuous casting conditions, on a laboratory scale, and to characterize the mechanical properties, especially, in the temperature range of the second ductility trough, the newly installed In-situ Material Characterization (IMC) Test has been developed. With this new method (patented by Siemens VAI [2]), interdendritic crack formation could be identified, even within the lower temperature range associated to the second ductility trough. Interdendritic crack growth is typically related to the first ductility trough, i.e. susceptibility close to the solidification temperature, as investigated by Bernhard et al. [3]. However, crack growth mechanisms have been found changing from interdendritic to intercrystalline typically expected for the second ductility trough, c.f. [4, 5]. The IMC test therefore offers a new and different approach to the investigation of mechanical properties for in-situ solidified steel samples, especially crack mechanisms. This paper describes the IMC test equipment and procedures and the first results are presented.

KEYWORDS

Second ductility trough, in-situ material characterization, crack formation, hot tensile test

INTRODUCTION

A wide variety of hot tensile test procedures exists, based on either the homogenization or the re-melting of the sample (in-situ test) before further controlled cooling and holding at testing temperature. Subsequently, the sample is ruptured under tensile loading. More recently, these tests are, not only performed isothermally, but also consider thermal gradients over the cross section of the specimen e.g. [6] or after thermal cycling before testing. The intention of these developments was to closely simulate casting conditions.

Already in the 1970s, Wilber et al. [7] and Palmaers [8] were the first to demonstrate the strong dependency of the measured ductility on the thermal cycle before the test. Both authors performed tests with and without prior partial melting of the specimen ("in-situ testing"), with the result of comparably lower strength and ductility values in the latter case. Therefore, in the 1980s, in-situ tensile testing became the most common method for the investigations into hot crack formation [9 – 12].

In the experimental simulation of transverse crack formation, the situation is more diverse: Revaux et al. [13] see the in-situ testing as a necessity for the validity of the results to the transverse cracking problem. Mintz [14] confirms this necessity, especially for the determination of the influence of segregating elements, like S, and elements that tend to form interdendritic precipitates, like Ti. For the determination of the influence of other micro-alloying elements, like Nb, V and Al, a prior solution treatment should be adequate. At least in the case of Nb, the situation might be more complex, as an interdendritic precipitation of (Nb,Ti)(C,N) is possible for micro-alloyed steels [15].

Besides segregation and precipitation, the microstructure has an important influence on the results of tensile tests: The thermal history in the continuous casting process results in the formation of coarse austenite grains near the strand surface [16]. This, together with the marginal deformation of the shell during the straightening by only a few percent [6], prevents the strand shell from dynamic recrystallisation and the associated improvement of ductility [14]. The high temperature end of the ductility trough, estimated from hot tensile tests, might therefore be shifted towards higher temperatures for the continuous casting process. The adjustment of the austenite grain size to the continuous casting process and the limitation of the applied strain, should therefore improve the relevance of experimental data for the problem of transverse crack formation.

A further insight into the role of microstructure in the hot tensile test resulted from early work of Fujii et al. [17]: performing hot tensile tests on specimens taken from different locations inside a slab indicated that the highest crack susceptibility is found with a coarse columnar structure, tested perpendicular to the orientation of columnar growth. In comparison, fine columnar structures and equiaxed structures show a better ductility at elevated temperature. The relevance of a columnar structure for simulating transverse crack formation was shown later by Mintz et al. [18].

A further limitation in the conversion of the results of hot tensile tests to the continuous casting process is the prediction of critical strain values from the measured reduction of area. Existing formulae contain often correction factors, fitted to the respective testing conditions or data from literature, and are therefore not universally valid [6]. A controlled straining of the specimen, limited by the onset of crack formation, and the prediction of the critical strain from this experiment, could be helpful in order to overcome these problems.

This all leads to the question of what are the necessary requirements of an experimental procedure to accurately simulate the formation of surface cracks during continuous casting.

From this brief survey, the following indicators for possible improvements of laboratory hot tensile tests, with relevance for the problem of transverse crack formation in continuous casting can be found:

- in-situ testing is a necessity to take account of the influence of segregating elements, like S, and elements which tend to form interdendritic precipitates, like Ti;
- in-situ testing might be helpful to understand the influence of elements like Nb, with a tendency to segregate and precipitate, depending on steel composition, during or immediately after solidification;
- the adjustment of the initial cooling conditions to the continuous casting mold, and thus the adjustment of microstructure with the main parameters being microsegregation, grain size and grain structure reflecting the continuous casting process;
- the direct measurement of the critical strain would be much more reliable, compared with the prediction of the critical strain from the measured reduction of area;
- the prevention of dynamic recrystallisation by a limitation of the total strain and the generation of a coarse columnar structure would give a more realistic view of crack susceptibility at temperatures between the first and second ductility trough.

This list of arguments makes clear that there still exists a motivation to contribute to the discussion about the experimental simulation of high temperature mechanical properties with respect to the continuous casting process. A feasibility study was conducted in order to answer the question for the potential and the limitations of a modified SSCT (Submerged Split-Chill Tensile) – test with respect to the arguments as listed above. In the following, the development of this "In-situ Material Characterization" (IMC) – test method and some first results will be discussed.

Siemens-VAI Metals Technologies and the University of Leoben performed a feasibility study on alternative testing methods. This project resulted in the development of the patent registered In-situ Materials Characterization (IMC)-test. In 2007, the worldwide first IMC-test stand was installed at the University of Leoben and has since been put into operation. The test method is based on the principle of the Submerged Split-Chill Tensile (SSCT)–test, [3, 16]. A cylindrical chill submerges into a steel bath inside an induction furnace. After the controlled solidification of a thin shell and the subsequent cooling of the shell under inert gas protection, the shell is elongated by pressing apart the two parts of the chill. The applied total strain is limited to only a few percent, sufficient to initiate crack formation and growth in the as-cast material.

In **Fig. 1** the new developed test stand as installed at the University of Leoben and additionally the induction furnace and the hydraulic equipment are shown in this figure. In detail the sample carrier with upper and lower part and the Pt-Pt-Rh thermocouples are visible.

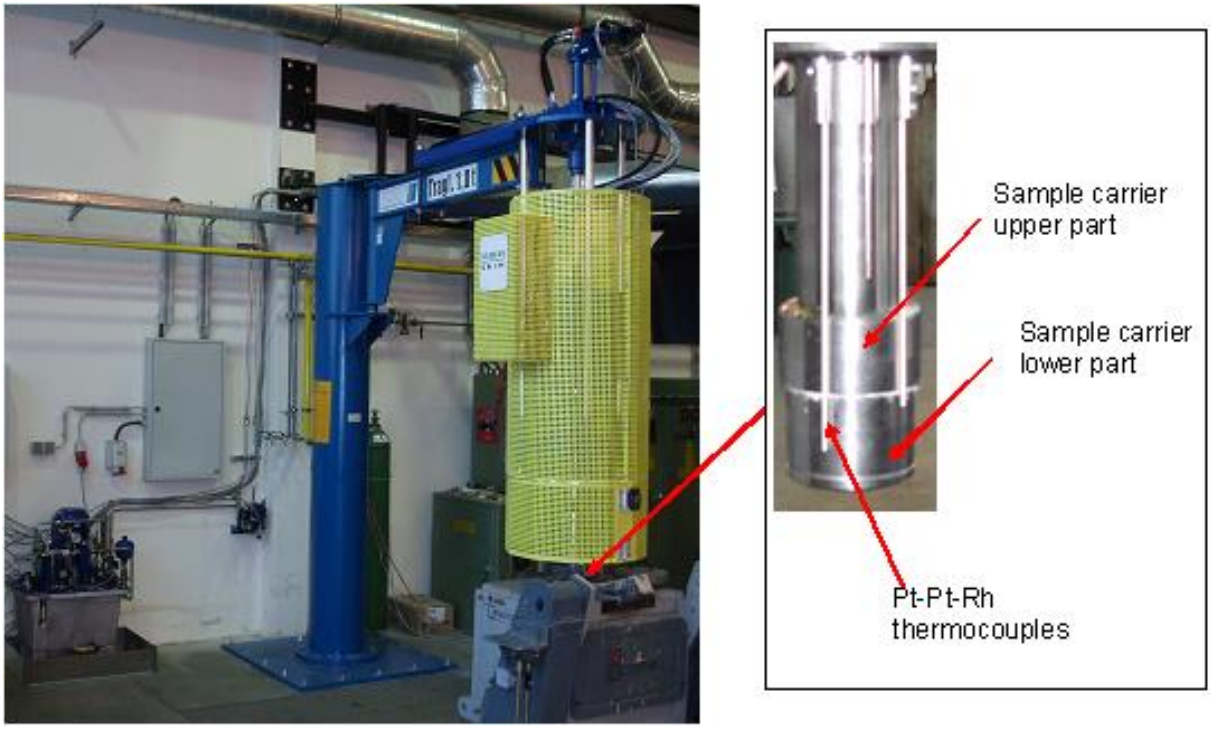


Fig. 1: The IMC – test stand at the University of Leoben.

The method offers the following specific characteristics:

- The possibility to adjust the initial cooling conditions, and thus the coarse columnar grain growth comparable to the continuous casting process;
- The limitation of the total strain and the generation of a coarse columnar structure is preventing dynamic recrystallisation during loading, resulting in quantitative results of crack

susceptibility at temperatures between the first and the second ductility trough; closer to experience from the continuous casting process.

- The direct measurement of critical limits to prevent defect formation (critical strain) is possible;

EXPERIMENTAL PROCEDURE

Approx. 20 kg of steel with a given composition are prepared in an induction furnace. The initial temperature of the melt is set to 25-30 °C above liquidus, similar to continuous casting conditions. During the steel preparation, the IMC - testing device is placed in the assembling position, adjacent to the induction furnace. The testing sequence starts with the submergence of the sample carrier. During lowering into the bath, solidification and cooling, the software controlled hydraulic system keeps the shell free from contraction forces in the axial direction - which is the main loading direction for the test later - by moving the two parts of the sample carrier into each other. The lower part of the sample carrier is connected to a hydraulic cylinder with position transducers and force measuring system. The upper part is fixed to a plate. The sample carrier is equipped with 4 Ni-Cr-Ni thermocouples, positioned 2 mm below the surface, as shown in **Fig. 2**. The sample carrier is covered with a layer of Zirconium-Oxide with a thickness sufficient to create the same heat flux as in the mold of a continuous casting machine. To measure the temperature of the solidifying shell, Pt-Pt-Rh thermocouples are placed near the interface of sample carrier. All testing parameters, as listed in **Tab. 1**, are pre-defined in the software system and the testing program proceeds in fully automatic mode. The reported steel grades are listed in **Tab. 1**. All these 3 steel grades resulted in the same crack formation behaviour as described below.

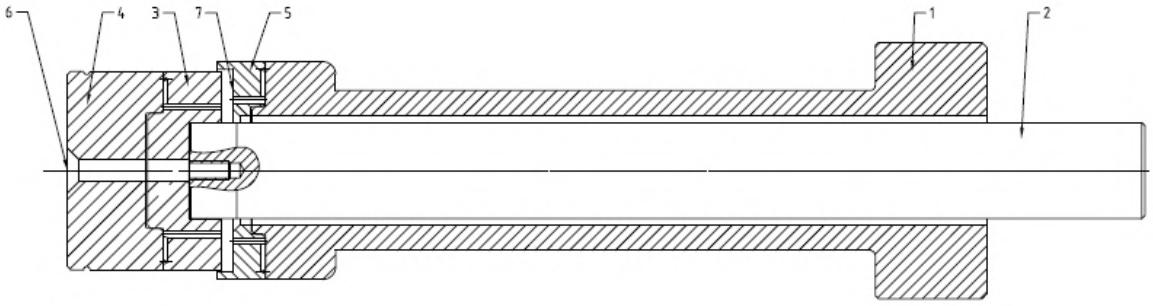


Fig. 2: Detail of the complete sample carrier where the upper and the lower parts are visible.

The numbers in **Fig. 2** indicate the following components:

1	upper part of the sample carrier
2	compression bar for the lower part of the sample carrier
3	fixing plate for the thermocouples at the lower part
4	lower part of the sample carrier
5	fixing plate for the thermocouples at the upper part
6	fixing screw
7	indicates the space between upper and lower part where the lower part can move to during solidification and cooling to the test temperature

Tab. 1: Basic pre-defined data for the IMC – Test:

Chemical analysis [mass %]		C	Si [%]	Mn [%]	Nb [%]	N [%]	Al [%]	Fe [%]
	Steel 1	0.170	0.40	1.50	0.000	0,0025	0.040	rest
	Steel 2	0.170	0.40	1.50	0.022	0.0040	0.040	rest
	Steel 3	0.140	0.40	1.40	0.028	0.0060	0.040	rest
Immersion speed	50 mm/sec							
Removal speed	80 mm/sec							
Solidification time	8 seconds							
Thickness of the zirconium oxide layer	0.4 mm							
Inert gas	Argon							
Expansion ratio	0.2 mm/sec							
Expansion time	25 sec							
Test temperature	900 °C (shell temperature)							

At the start of the testing procedure, the testing device is positioned central above the induction furnace and then immersed with a pre-defined speed. After a short pre-defined solidification time in the steel bath the sample carrier is taken out of the steel bath and is allowed to cool in an inert gas atmosphere to prevent scaling. The thickness of the solidified sample is dependent on the residence time in the steel bath. The pre-defined solidification time of 8 seconds results in a shell thickness of approximately 5 mm. After cooling the sample to the test temperature, which is in the second ductility trough, the lower part of the sample carrier is moved downwards by the pre-defined controlled velocity and time exceeding the ultimate strain. During the test, all data such as temperatures, cylinder position and force are recorded.

After removing the sample from the sample carrier, the solidified shell and the possibly created cracks are metallographically examined.

RESULTS

One important issue of the IMC – development was to simulate the coarse columnar grain structure at the surface of a cast strand. **Fig. 3** and **Fig. 4** show micrographs of a slab sample, compared to an IMC – test sample, both etched with picric acid. The anisotropy of the grain structure is in both cases clearly visible. To investigate the grain structure in detail, an IMC – test sample was compared to a continuous casting slab sample with respect to the grain size located at 1 mm and 2 mm below the surface. **Fig. 5** and **Fig. 6** show the grain size (intercepts) perpendicular to the columnar growth direction for the two samples. The maximum of the grain size distribution for the

IMC – test sample is about 30% smaller than the slab sample. In **Table 2** the average values of the grain size evaluation are listed.

Fig. 3: A sample of a slab edge area. The austenite grain boundaries are highlighted by a yellow line.

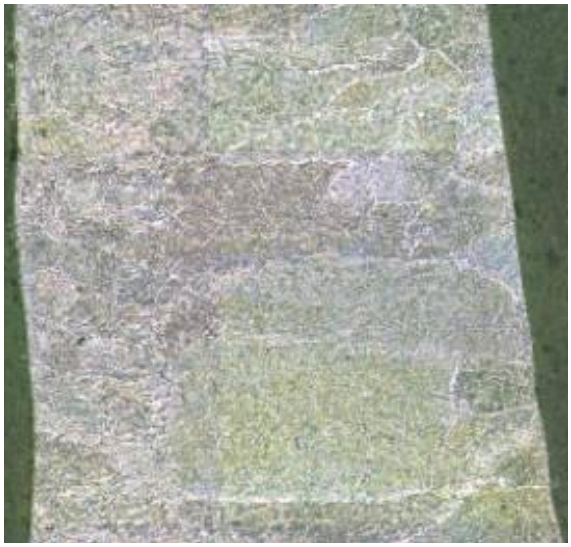


Fig.4: A sample of an IMC – test specimen

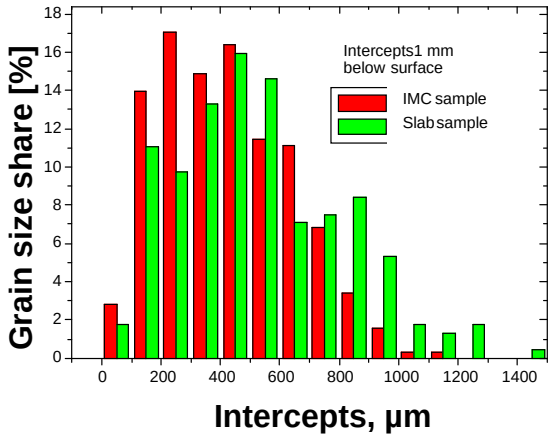


Fig. 5: Measured grain size parallel to the surface of a slab and an IMC – test sample 1 mm below the surface.

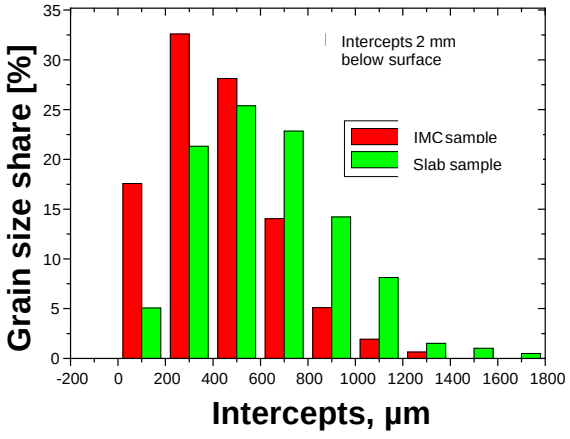


Fig. 6: Measured grain size parallel to the surface of a slab and an IMC – test sample 2 mm below the surface.

Tab. 2: The average measured values of the grain size evaluation:

Average measured values:	1.0 mm blow the surface	2.0 mm below the surface
Slab:	623 µm	784 µm
IMC –test:	433 µm	525 µm

NUMERICAL SIMULATION

In order to understand the stress, strain and the temperature distribution within the test specimen during the testing procedure, a thermo-mechanical model of the IMC – test has been developed based on the Finite Element method in the commercial code (ABAQUS). **Fig. 7** shows the axisymmetric model of the solid steel test body and the chosen Finite Element mesh of the 5 domains; the upper and the lower part of the sample carrier, the zirconium oxide layer, the air gap between the two sample carrier parts and the steel melt. **Fig. 8a - c** illustrates the axisymmetric mesh and the calculated solidified strand shell after 8 seconds solidification time.

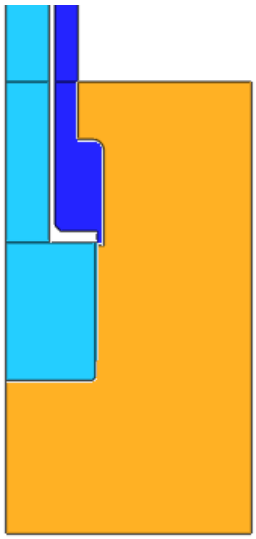


Fig. 7: Domains of the axisymmetric 2D-FE-model

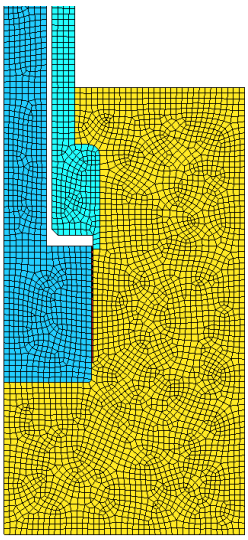


Fig. 8a: Finite Element Mesh for the Heat Transfer Analysis

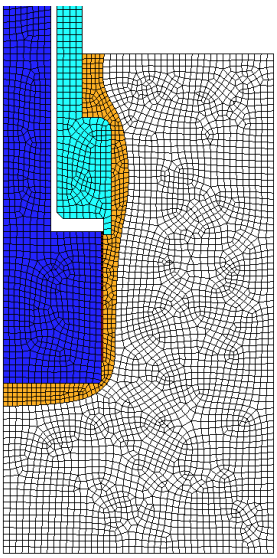


Fig. 8b: Solidified strand shell after 8 seconds of solidification with domains and mesh

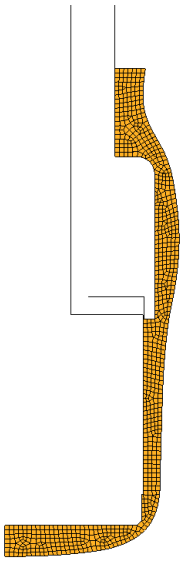


Fig.8c: Solidified shell domain with axisymmetric mesh used for the Stress/Displacement Analysis. The test body is represented by rigid surfaces

The whole analysis is divided into three parts:

1. Determination of solidified steel portion (shell formation) by a transient Heat Transfer Analysis. The mesh, which is displayed in **Fig. 8a**, is used in order to calculate the solidified area of the liquid pool. The solidification time period is assumed to be 8 seconds.
2. As mentioned above, after 8 seconds of solidification time a new domain is created (solidified shell). The complete model is remeshed taking into account that there is a new formed solidified area. After removal of the sample from the liquid pool a further cooling period of 12 seconds at free air is calculated by a further transient Heat Transfer Analysis. The resultant final temperature field is used for the subsequent Stress/Displacement Analysis. **Fig. 8b** shows the remeshed domains.
3. Stress/Displacement Analysis for the solidified shell. The structure of the test-body is now represented by an axisymmetric rigid surface. The solidified shell is built from 4-node axisymmetric solid elements (model see **Fig. 8c**). A temperature dependent elastic-plastic material model is used for the solidified shell. All other deformable domains are switched off during the Analysis. The inner part of the test body (rigid surface) is moved downwards and applies the deformation onto the solidified steel shell. The moving distance amounts to 5 mm with a moving speed of 1mm/s. Contact conditions between the rigid surface and the shell are established.

Fig. 9 shows the Axial Plastic Strain (PE22) field for a tensile test after 8 seconds solidification time and subsequent 12 seconds cooling time to approximately 900°C. The inner-test-body movement was 5 mm downwards. For these testing conditions, the local maximum Axial Strain component PE22, in the range of 50-110%, arises in the shell between the upper and the lower part of the sample carrier. The examination of the test specimens indicate that crack initiation is much earlier in the testing process, since for most of the samples no plastic localization was visible as discussed in the next chapter.

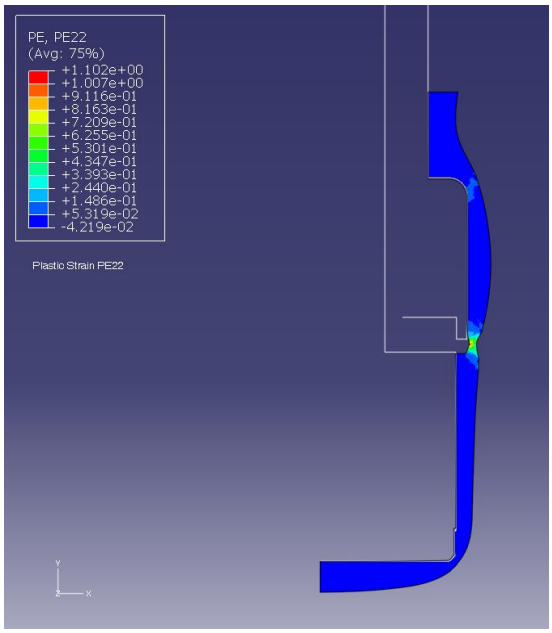


Fig. 9: Axial Plastic Strain Field (PE22) Distribution during IMC – test at the solidified shell for 5mm inner body movement

EVALUATION OF MEASURED DATA

Fig. 10 gives the results of a test series to characterize material properties in the range of the second ductility trough. Detailed test data are given in **Tab. 3**. The tensile tests were performed at temperatures between 700°C and 900°C, in steps of 50°C. It can be seen in **Fig. 10** that the maximum tensile force decreases with decreasing test temperature. The achievement of the maximum tensile force coincides with the onset of massive crack initiation and growth in the sample. The critical elongation for the onset of crack formation – a measure for the critical strain - decreases thus with decreasing temperature. **Fig. 10** also shows the measured temperature inside the test body and inside the solidified shell.

Tab. 3: Parameters for the tests in Fig. 10:

	VA1	VA2	VA3	VA4	VA5
Strain rate	0.2 mm/s	0.2 mm/s	0.2 mm/s	0.2 mm/s	0.2 mm/s
Total elongation	10 mm	10 mm	10 mm	10 mm	10 mm
Elongation Time	50 sec.	50 sec.	50 sec.	50 sec.	50 sec.
Solidification time	8 sec.	8 sec.	8 sec.	8 sec.	8 sec.
Start temperature	900 °C	850 °C	800 °C	750 °C	700 °C
Steel melt temperature	1550°C	1548°C	1553°C	1547°C	1552°C

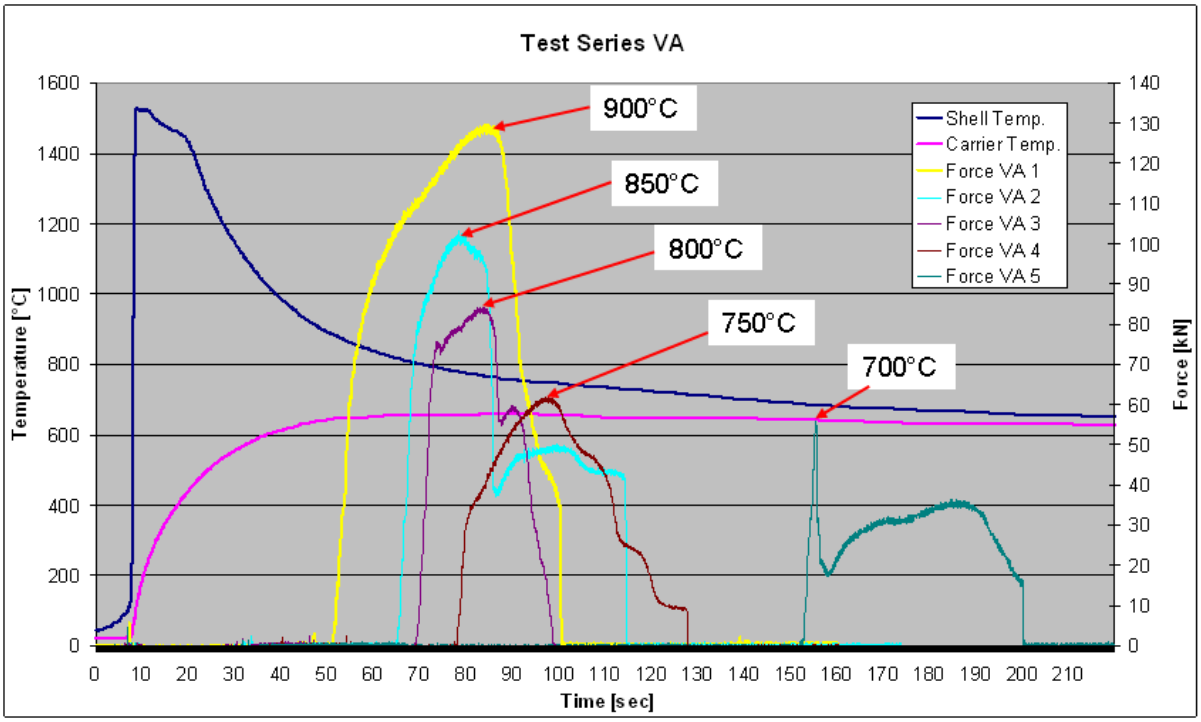


Fig. 10: Measured values of force and temperatures in the solidified shell and the test body

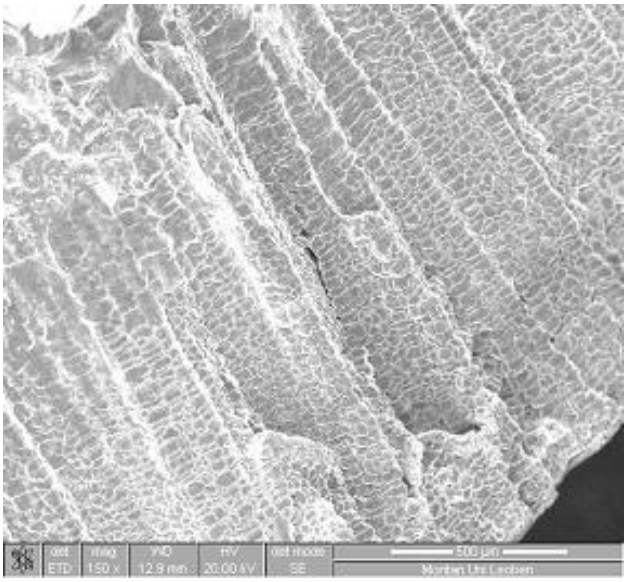
EVALUATION OF CRACK SURFACES

The crack formation in the first ductility trough is interdendritic, typically expected when the crack opens between the solid/liquid interface where liquid steel remains between the solidified dendrites. In the second ductility trough the expected crack formation is intercrystalline. The crack formation happens at grain boundaries due to proeutectoid ferrite formation and/or precipitations.

With the new test, all the light optical and electron microscopic evaluations show that the generated cracks are different to the expectations about crack formation in the first and second ductility trough. The generated cracks at a temperature range between 700 and 900°C are interdendritic and not (or only partly) intercrystalline.

Fig. 11 to **Fig. 13** show pictures of this evaluation. It can be seen that the generated crack surfaces are interdendritic, compare **Fig. 11** and **Fig. 12** and not intercrystalline at the austenite grain boundaries, as it would be expected at these temperatures. The cracks were generated in a very short time period and small temperature changes of 50°C during the test procedure. The interdendritic crack formation is typically related to higher temperatures in the range of the first ductility trough but the investigated cracks are generated in the temperature range of the second ductility trough, far below the solidification point of the composition. Some small regions are showing the typical microductile and intercrystalline crack formation as it would be expected at these testing temperatures, compared with **Fig. 13**. Therefore the crack formation characteristics have altered during crack growth, on these samples. This will be the content of further evaluations in the clarification of the crack formation in these small areas. It appears that the crack growth resistance of both characterised growth mechanism are on a similar level. Thus small deviations in parameters, such as temperatures, lead to altering characteristics. On the other hand, it might be that -influenced by defects- the nucleation of cracks start in several regions independently, which will be the content of further investigations.

The evaluated strain before the crack occurs with the IMC Tests is just a few percent. This is also different and much lower to the well-known results of other hot tensile tests.



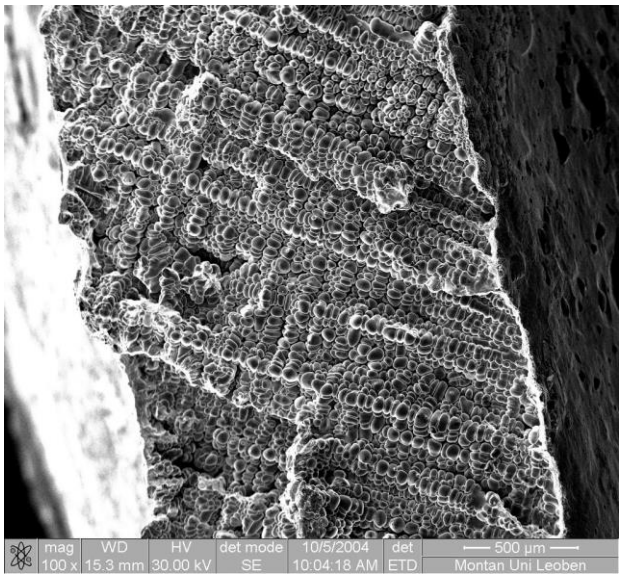


Fig. 11 and Fig. 12: Interdendritic crack surfaces of samples cracked at temperatures of 800 °C.

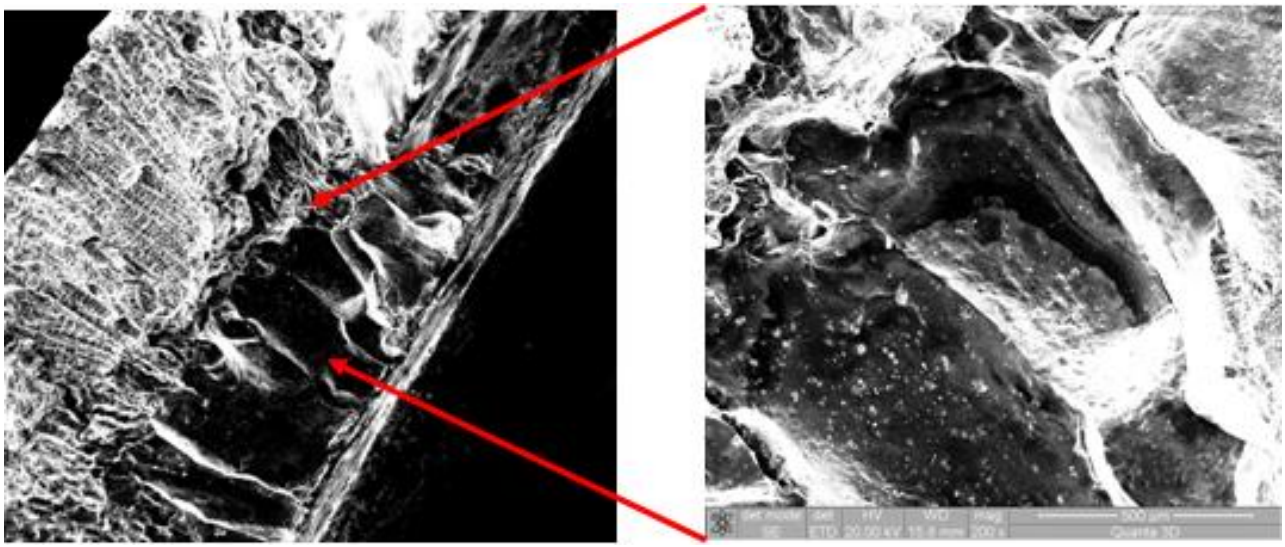


Fig. 13: An interdendritic crack with a small intercrystalline part

CONCLUSIONS AND OUTLOOK

The main differences of the IMC – test compared with other hot tensile tests is that the test body has a non reheated and deformed primary solidification structure and the heat removal direction is perpendicular to the load direction. Although the conditions are different to other hot tensile tests, they are equivalent to the continuous casting process.

The presented new IMC – Test shows a lot of different results compared to other well-known hot tensile test methods. It is assumed that these results are basically a consequence of the primary anisotropic non reheated or deformed casting structure. For conventional hot tensile tests, the crystal structure is equiaxed and hence local disturbances e.g. grain boundaries and precipitation have a minor influence to the material strength.

When reheated or partly solidified steel is tested with tensile tests, the grain structure of the steel sample is not the same as from steel samples, which are directly solidified and cooled. Inclusions have time to grow, alloy elements have time for diffusion and the grain structure is recrystallized. All these factors have a significant influence on the material properties, especially on the formation of cracks. For the continuous casting process it is very important to characterize material properties directly after solidification during cooling.

This was the reason for the development of a new kind of facility able to characterize material properties directly after solidification. The obtained data show that this reproducible test procedure present results in unexpected crack formation mechanism at temperatures typically related to intercrystalline crack growth at the austenite grain boundaries.

For the immediate future, the work will focus on further optimization of the sample geometry and standardisation of the IMC – testing procedure, to improve the simulation program and to go further with the practical testing of various alloyed carbon steel grades.

The IMC – Test has been developed to investigate steels which are very sensitive to crack formation during continuous casting. The IMC – Test is fully simulating the continuous casting process on a laboratory scale.

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