A laboratory test for simulation of solidification on Gleeble 1500D thermo-mechanical simulator

Laboratorijski test simulacije strjevanja na termo-mehanskem simulatorju Gleeble 1500D

Boštjan Bradaskja[1], Jurij Koruza[2], Matevž Fazarinc[2], Matjaž Knap[2], Radomir Turk[2]

[1]ACRONI d.o.o., Cesta Borisa Kidriča 44, SI-4270 Jesenice, Slovenia;
E-mail: bostjan.bradaskja@acroni.si
[2]University of Ljubljana , Faculty of Natural Sciences and Engineering,
Departmant of Materials and Metallurgy, Aškerčeva cesta 12, SI-1000 Ljubljana, Slovenia;
E-mail: jurij.koruza@ntf.uni-lj.si, matevz.fazarinc@ntf.uni-lj.si,
matjaz.knap@ntf.uni-lj.si, rado.turk@ntf.uni-lj.si

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Abstract: A highly repeatable solidification test was developed. It was designed to study the effect of process parameters on solidified micro and macrostructures. Different cooling rates were used for solidification and cooling of S 355 J2 construction steel. It was observed that cooling rate has a substantial influence on final grain size and on columnar-to-equiaxed transition zone (CET). Within the interval of constant thermal gradient, different macro- and micro-structural features have been provided, which consequently enables the study of dynamics of precipitation dependence on solidification processing parameters.

Izvleček: Razvit je bil visoko ponovljiv test strjevanja. Razvit je bil kot orodje, za preučevanje vpliva procesnih parametrov na lito mikro in makrostrukturo. Za strjevanje konstrukcijskega jekla S 355 J2 so bile uporabljene različne ohlajevalne hitrosti. Opažen je bil močan vpliv ohlajevalne hitrosti tako na končno velikost zrn, kot tudi na mejno plast med stebričastimi in ekviaksialnimi zrni. Znotraj intervala, kjer je bil temperaturni gradient konstanten, so bile s pomočjo različnih ohlajevalnih hitrosti, v vzorcih dobljene različne mikro in makrostrukturne značilnosti, kar omogoča študij dinamike izločanja v odvisnosti od parametrov strjevanja.

Key words: solidification test, macrostructure, cooling rate, Gleeble
Ključne besede: test strjevanja, makrostruktura, ohlajevalna hitrost, Gleeble

Scientific paper

INTRODUCTION

The solidification of molten steel is one of the most important processes in the processing path of steel manufacturing. The microstructure that derives from the solidified ingot or slab has a large influence on the characteristic of the produced material and process path that follows.

The micro and macrostructures in ingots usually show spatial variations of features and properties. This can be simulated with combination of computer simulation of ingot/slab cooling and high temperature testing with controlled solidification and cooling of the specimens.

The high temperature experiments on the Gleeble testing machine that were found in literature are mainly focused on characteristics of metal in the mushy zone or the mechanical properties of solidified specimens for continuous casting application and do not deal with the development of microstructure. The investigations have not been focused on influence of process parameters on development of micro and macrostructure. In these tests, the solidification process was only the first stage followed by tensile mechanical loading and no observations of solidified structure were made.

SEOL et al.[1,2] were studying the behavior of material under tensile loading of in-situ melted and solidified cylindrical specimens. The tensile strength and ductility of carbon steels was measured in the temperature range of mushy zone. Also the stress – strain relations in austenite and δ-ferrite phase regions at various temperatures and strain rates were analyzed. NA et al.[3] used the Gleeble system to investigate cracking occurrence during continuous casting of steels by tensile testing. In this work effects of carbon content, strain rate and sampling orientation on hot ductility were investigated. GLOWACKI et al.[4] used tensile test to study the deformation behavior of steel within the mushy zone. SUZUKI et al.[5-7] employed different methods of laboratory simulation of continuous casting and direct rolling of steels with special emphasis on embrittlement in dependence to chemical composition, thermal history, strain rate and fracture mode.

Nevertheless some investigations were made on development of solidified microstructure of aluminum alloys, using the Gleebe system, by KOSTRIVAS and LIPPOLD[8]. Although the testing procedure was similar, the difference between the physical properties of aluminum and steel brings up new problems and requires some different approaches.

Because of the above mentioned reasons it was decided that a highly repeatable solidification test should be developed with accurate knowledge and control of the temperature field in the specimen. The test was developed using a Gleeble 1500D thermo-mechanical simulator. This machine enables simultaneous, computer guided, thermal and mechanical loading of the specimen, within a very precise range. The main goal was to track the development of micro and macrostructure in dependence to testing parameters.

Experimental procedure and material

The laboratory test for controlled solidification has been developed using Gleeble 1500D thermo-mechanical simulator, which has been equipped with specially developed additional parts for solidification simulation.

Cylindrical specimens, 110 mm long with a diameter of 10 mm, as shown in Figure 1 were used for the experiments. A 10 mm screw-thread was engraved on each side of the specimen which enabled the fixation of the specimen into the jaws with aim to perform tensile and compression loadings. To support the melt zone a 30 mm quartz crucible with a 1 mm wall thickness has been placed around the center part of the specimen. Quartz material was used because of its high temperatures endurance, small linear coefficient of thermal expansion and negligible diffusion with the specimen material. Crucibles have been furnished with axially running 2 mm slot for insertion of the thermo couples and also to provide an opening for exiting gasses. To decrease the radial thermal gradient, refractory material was placed over the crucible. Additionally two special water-cooling ribs made from copper were placed on each side of the specimen, to attain sufficient thermal gradient in axial direction. Varying the position of the ribs enabled the control of the axial thermal gradient and consequently the width of the melting zone. Schematic plan of placing of the specimen is shown on Figure 2.

Temperature was controlled by 0.35 mm thick S-type (Pt10wt%Rh-Pt) thermocouple, spot-welded in the middle of the specimen and isolated with alumina tubes. An additional thermocouple stand was applied to prevent the motion of the leading thermocouple and to assure the correct temperature measurements during the upset of liquid phase. In order to minimize the surface oxidation and consequently the detachment of the welded thermocouple, all experiments were conducted in protective Ar atmosphere.

Before setting up the temperature program for the test, Thermo-Calc software was used to define the liquidus and solidus temperature of the material studied. The specimen was heated with the heating rate of 15 K/s up to 100 K below liquidus temperature. The heating rate was then decreased to 1 K/s for better control of temperature near the melting temperature. If necessary, the

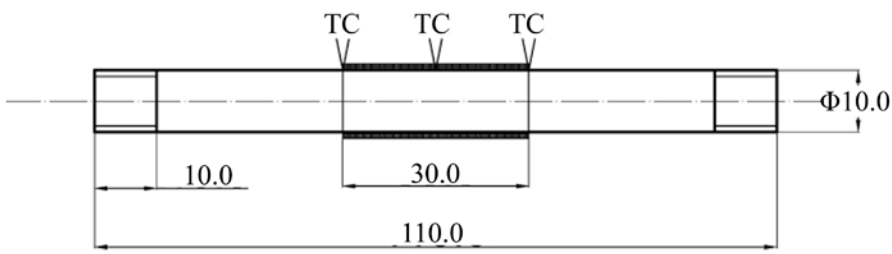

Figure1. Cross section of the specimen
Slika 1. Shematski načrt preizkušanca

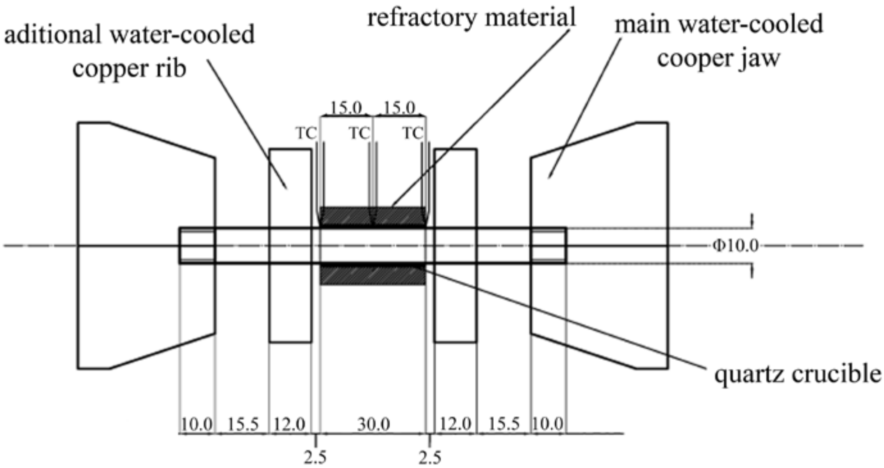

Figure 2. Schematic plan of the mounted specimen
Slika 2. Shematski načrt namestitve preizkušanca

temperature was raised manually to attain the desired width of the melted zone. For stabilization of melted zone the specimen was held at the liquidus temperature for 30 - 40 seconds. Afterwards it was cooled with a predefined cooling rate between 1 K/s and 10 K/s.

During the cooling an adequate mechanical program was applied with the movement of the main jaws to compensate solidification shrinkage and to prevent formation of cracks. Value of stroke depends on the material properties and the width of the melt zone. After the whole volume has been solidified, a constant compression loading was applied to prevent appearance of cracks caused by further temperature shrinkage of material.

In order to evaluate the developed equipment the construction steel, S 355 J2, with chemical composition listed in Table 1, was selected.

After the tests, the solidified samples were sectioned longitudinally and axially along their mid-plane for metallographical observation. To reveal the macrostructure and microstructure, 10 % HNO_3 and 2 % HNO_3 etching agents were used, respectively. The etched specimens were examined by means of optical microscopy using Olympus SZX12 binocular and Olympus BX51M optical microscope.

Results and discussion

The axial thermal gradient was adjusted by setting two additional cooling ribs at different positions from the center of the specimen. By this way the solidification conditions (steepness of the gradient) can be controlled. Figure 3 represents the measured axial temperature difference at different positions of cooling ribs: 17.5, 20.0 and 25.0 mm from each side of the center of the specimen. Thermocouples were spot

Table 1. Chemical composition of the S 355 J2 steel in wt. %
Tabela 1. Kemijska sestava jekla S 355 J2 v ut.%

Element	C	Si	Mn	P	S	Al	Nb
wt. %	0.15	0.45	1.03	0.012	0.001	0.028	0.019

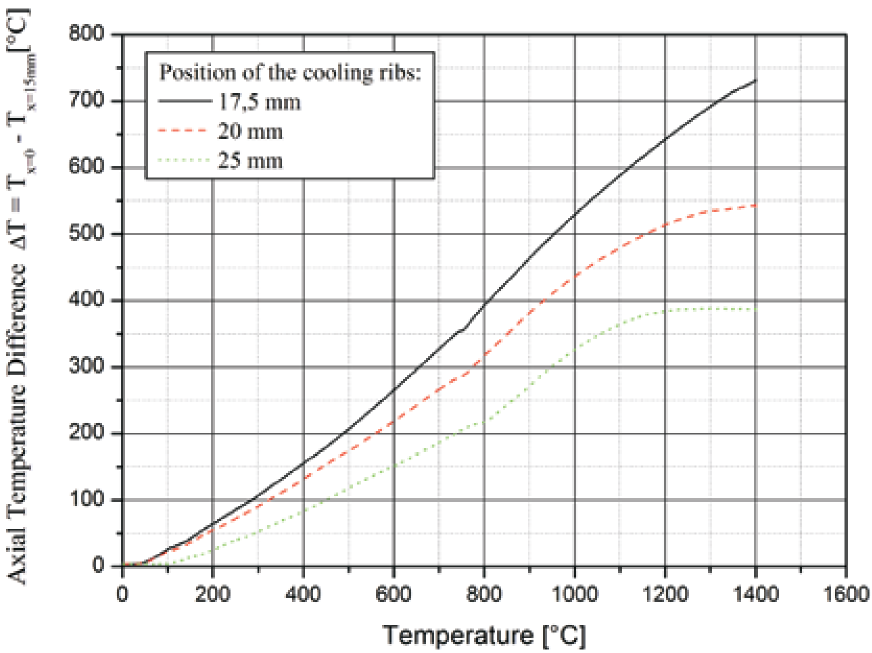

Figure 3. Comparison of axial temperature difference measured between the center part of the specimen and 15 mm from the center along the axis of the specimen, using different placements of the water-cooled copper ribs (17.5, 20, and 25 mm from the center of the specimen)
Slika 3. Primerjava temperaturnih razlik izmerjenih med sredino vzorca ter 15 mm od sredine vzorca v osni smeri vzorca, v odvisnosti od postavitve dodatnih hladilnih reber (postavitev 17,5, 20 in 25 mm od sredine vzorca)

welded on the surface of the specimen as could be seen on Figure 2. The specimen was isolated with refractory material.

The effect of the refractory material on the radial temperature difference between the surface and the center of the specimen was measured for the isolated and unisolated specimen. The results are shown in Figure 4. This difference was measured using the same technique as described by GLOWACKI et al.[4].

With isolation the heat loss through the surface was minimized and consequently more directional solidification achieved.

Four isolated specimens were melted and solidified using different cooling rates, 1 K/s, 3 K/s, 5 K/s and 10 K/s. For comparison, one experiment without isolation and with cooling rate of 3 K/s was carried out. The obtained macrostructures in axial and radial cross-section are presented in Figure 5a-d for isolated specimens and in Figure 5e for un-isolated specimen. Position of the cooling ribs was the same in all experiments that is 17.5 mm from the center on each side as shown on Figure 2. The width of the melted zone was between 12 and 15 mm and was surrounded by 5 to 8 mm wide heat affected zones on each side.

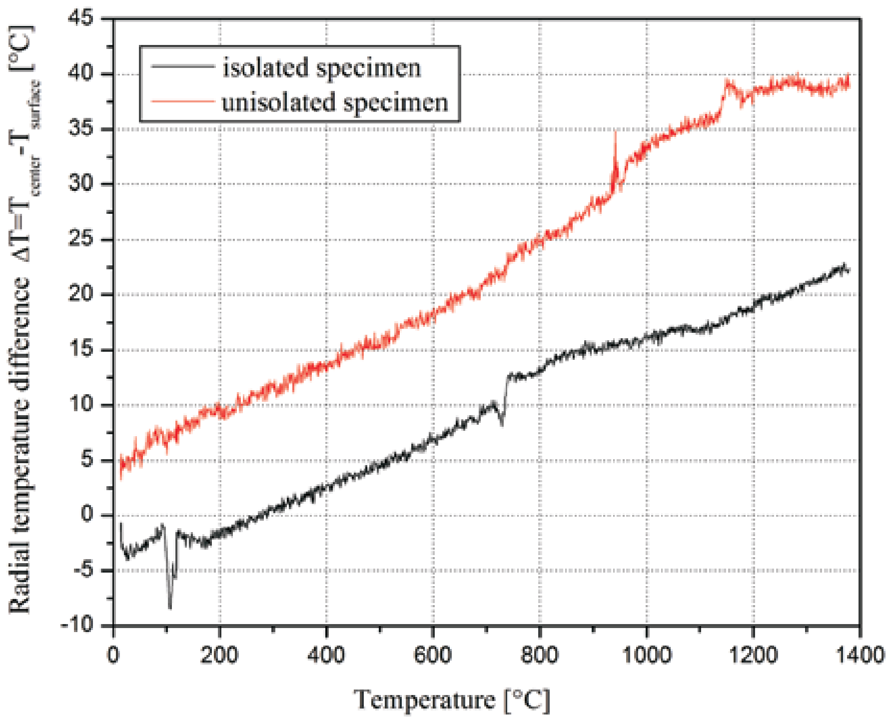

Figure 4. Comparison of the measured radial temperature difference between isolated and un-isolated specimens

Slika 4. Primerjava radialne temperaturne razlike med izoliranim in neizoliranim vzorcem

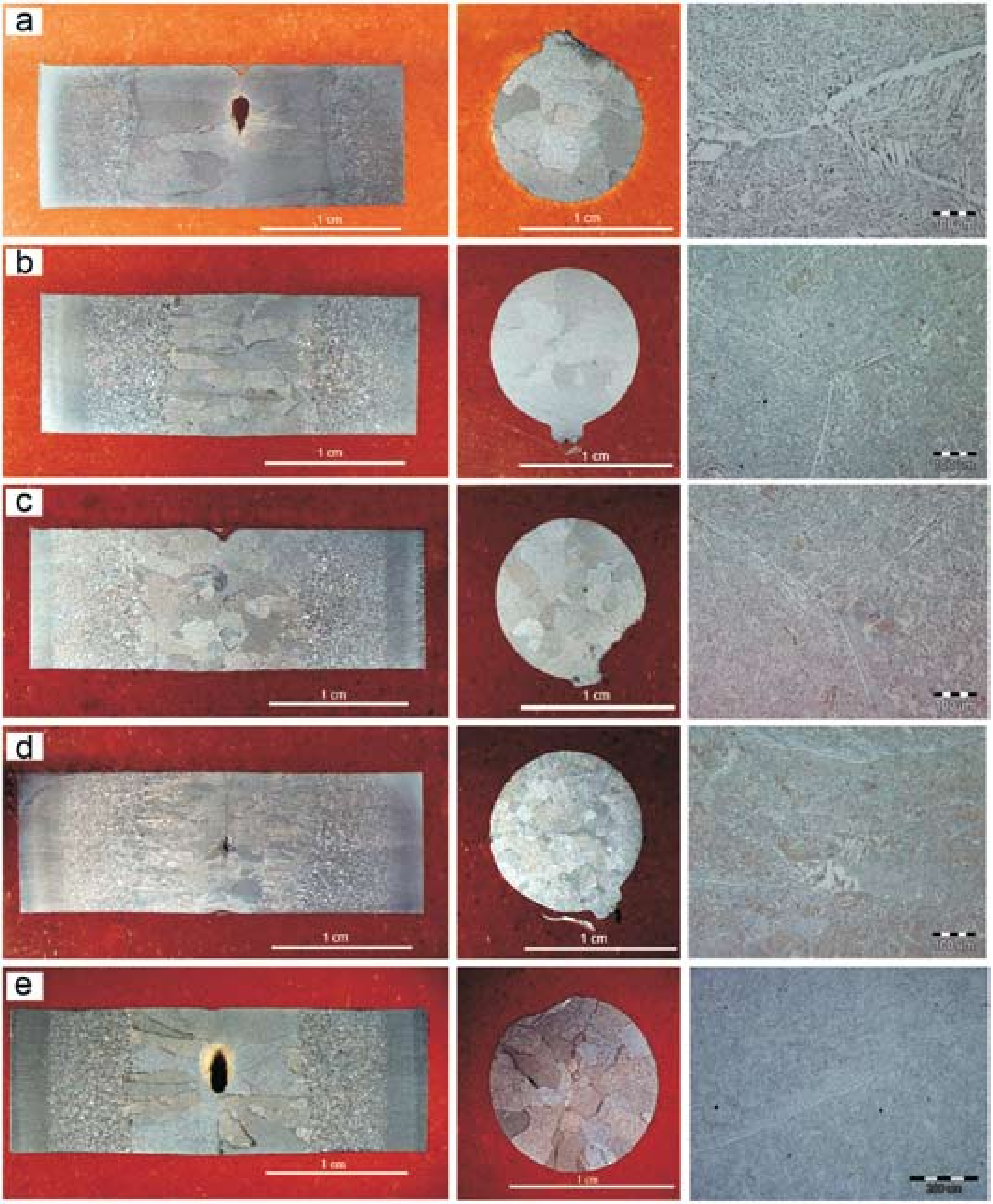

Figure 5. Longitudinal and axial cross-section of the isolated specimens with cooling rates 1 °C/s, 3 °C/s, 5 °C/s, 10 °C/s from a) to d) respectively and an un-isolated specimen e) with cooling rate 3 °C/s

Slika 5. Vzdolžni ter prečni prerez vzorcev s pripadajočo mikrostrukturo: a) hitrost ohlajanja 1 °C/s, b) hitrost ohlajanja 3 °C/s, c) hitrost ohlajanja 5 °C/s, d) hitrost ohlajanja 10 °C/s, e) hitrost ohlajanja 3 °C/s. Vzorci od a) do d) so bili izolirani, vzorec e) je bil strjen brez izolacije

Lower cooling rates caused the formation of columnar grains which can be observed in the macrostructures presented in the Figure 5a-d. These grains start to form on the solid material on the edge of the melted zone and grow into the center of the melt, in the opposite direction of the heat flow.

With increase of the cooling rate the solidification time shortens which resulted in increased number of smaller sized grains (Figure 5a-d). Above a certain threshold of cooling rate, as seen in Figure 5d, a relatively large amount of latent heat is momentarily set free. The heat flow is not sufficient thus the cooling rate of the remained liquid in the center of the specimen is reduced. Therefore the center of the specimen is solidified with a slower cooling rate. Consequently the observed solidified structure consists of two different grain structures where the CET zone is clearly seen. The first grains are columnar and are more numerous than those obtained from the specimens solidified with slower cooling rate. The second type of grains is more globular type and is solidified last. These grains did not solidify directionally.

In order to investigate the effect of the isolation on macro- and microstructure, one specimen was solidified without isolation. The cooling rate was 3 °C/s and the macrostructure is shown on Figure 5e. Because of the large radial heat loses due to radiation the solidification was not directional and there was also a large shrinkage cavity. The surface was solidified prior to the center of the specimen and the lack of the material appears because it could not be compensated by compression loading. The grain size does not differ from that obtained from the specimen which was solidified with isolation.

CONCLUSIONS

A solidification test was developed, using thermo-mechanical simulator Gleeble 1500D which enables simultaneous computer guided mechanical and thermal loading.

Additional cooling ribs were used to control the axial thermal gradient. With varying the position of these ribs different temperature fields were obtained. The radial temperature difference was minimized by means of refractory material, placed around the quartz crucible. With an additional test without refractory material it was proven that solidification was not unidirectional and a large shrinkage cavity was observed.

A comparison between four samples solidified and cooled with different cooling rates was made. It was observed that cooling rate has a substantial influence on final grain size. With increasing the cooling rate the grains are found to be smaller. CET zone was also observed in the sample that was cooled with the highest cooling rate.

POVZETEK

Laboratorijski test simulacije strjevanja na termo-mehanskem simulatorju Gleeble 1500D

Razvit je bil test za strjevanje kovinskih materialov. Test je bil razvit kot dodatek termo-mehanskemu simulatorju metalurških stanj, Gleeble 1500D, ki omogoča simultano računalniško vodenje temperaturnega in mehanskega programa obremenjevanja vzorca.

Z dodatnimi, vodno hlajenimi, bakrenimi, hladilnimi rebri je bil kontroliran aksialni temperaturni gradient. S premikanjem leteh je moč doseči različna temperaturna polja. Radialni temperaturni gradient je bil zmanjšan z uporabo ognjevzdržnega materiala, ki je obdajal kvarčno cevko. Z dodatnim testom, ki je bil izveden brez ognjevzdržnega materiala, je bilo pokazano, da strjevanje ni potekalo usmerjeno, oziroma ni bilo enoosno.

Narejena je bila primerjava med štirimi vzorci, strjenimi in ohlajenimi z različnimi ohlajevalnimi hitrostmi. Opaženo je bilo, da ima ohlajevalna hitrost močan vpliv na končno velikost zrn. Z povečevanjem ohlajevalne hitrosti so zrna postajala manjša, v vzorcu, ki je bil ohlajen z najvišjo hitrostjo, pa je moč opaziti prehodno cono med stebričastimi in ekviaksialnimi kristali.

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