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Vpliv sestave cepiva v bloku in velikosti zrn na stroškovno učinkovito cepljenje duktilne litine v formi

Influence of the Inoculation Block's Composition and Grain Size on the Cost- Effective Inmould Inoculation of Ductile Iron

Izvleček

Postopek cepljenja je posebno pomemben pri izdelavi varnostnih strojnih delov iz duktilne litine. Če cepljenje ni popolno ali ga sploh ni, se je treba na druge načine izogniti nastanku metastabilne mikrostrukture. Za zagotovitev strjevanja s stabilnimi fazami (npr. grafitom in ne cementitom) se v podružnicah GF uporabljajo dodatni bloki cepiva. Nameščena je bila poskusna naprava za izdelavo blokov cepiva iz rahlo povezanih cepiv in veziv. Cilj je bil zmanjšati stroške in odvisnost od dobaviteljev.

Osnovna tema prispevka je izdelava in ocena blokov cepiva iz štirih različno zrnatih cepiv z dvema različnima zrnavostma. Ocena učinkovitosti cepljenja je slonela na mehanskih lastnostih in analizi mikrostrukture preskušancev. Ta raziskava kaže, da so bile mehanske in metalografske lastnosti preizkušancev, cepljenih z doma izdelanimi bloki cepiva, primerljive s standardnimi bloki cepiva. Zato se lahko nadomesti na tržišču kupljeni izdelek.

Abstract

The inoculation process is of particular importance for the production of safety-related components made of ductile iron. In case of an incomplete or missing ladle inoculation, the formation of a metastable structure has to be avoided by other means. In order to guarantee solidification with stable phases (e.g. graphite and not cementite), additional inoculant blocks are used at GF-production sites. An experimental device was installed to produce inoculant blocks made of loose inoculant and inorganic binder. The aim is to reduce costs and supplier dependence.

The production and evaluation of inoculant blocks, made of four different granular inoculants with two distinct grain size distributions, are the main topics of the present study. The evaluation of the inoculation efficiency of the various inoculants is based on mechanical properties and microstructure analysis of the test specimens. The present study shows that the mechanical and metallographic properties of the test components achieved with self-made inoculant blocks are comparable with these using the standard inoculant block. Consequently, the commercially available product can be substituted.

1 Uvod

Zaradi velikega razmerja trdnost – masa, izvrstne livnosti in sorazmerno nizke cene je duktilna železova litina (DI) privlačen livarski material, ki postopoma nadomešča kovane in varjene jeklene dele v avtomobilih [1, 2]. Na mikrostrukturo in mehanske lastnosti duktilne litine vpliva predvsem njena kemična sestava, hitrost ohlajevanja in obdelava taline z dodajanjem cepiva v obliki magnezijeve predzlitine [3].

Dodatek magnezija v osnovno talino železove litine spreminja obliko lamelastega evtektičnega grafita v kroglasto obliko in tako močno zmanjša vpliv grafitne faze na nastajanje razpok. To v veliki meri izboljša mehanske lastnosti delov iz litine. Poleg tega to modificiranje z Mg ustvarja veliko število majhnih mikrovključkov, ki so osnova za učinkovito cepljenje, ki sledi [4-6]. Pri tem cepljenju se izločajo heksagonalne faze na nastalih Mg-delcih, ki so ugodna mesta za nukleacijo grafita zaradi faznih mej s koherentno/semikoherentno nizko energijo [6].

Cepljenje poveča število mest za nukleacijo grafita in vodi do minimalnega evtektičnega podhlajanja [7]; krogličavost in število kroglic se večata z manjšanjem deleža perlita in s tvorbo bele plasti zaradi velikega potenciala grafitizacije kroglastega grafita in manjše stopnje izcejanja zlitinskih elementov [2, 8-10]. Z izboljšanjem strjevalne morfologije cepljenje zmanjšuje delež poroznosti [11].

Ker cepilni učinek slabi, je Riposan s soavtorji predlagal, da je cepljenje železove osnovne taline kolikor mogoče tik pred ulivanjem [12]. Huerta in Popovski sta pokazala, da se učinek cepljenja drastično zmanjšuje in lahko nastopi v prvih dveh do šestih minutah po cepljenju [13]. Tako imenovano pozno cepljenje v formi je primerna metoda pri slabljenju cepilnega

1 Introduction

Due to its high strength to weight ratio, excellent castability, and comparatively low price ductile iron (DI) is an attractive casting material and therefore gradually replaces forged and welded steel components in automobiles [1, 2]. The DI's microstructure and mechanical properties are mainly influenced by its chemical composition, the cooling rate and the melt treatment, which consists of a magnesium and inoculant masteralloy addition [3].

The addition of Mg to a cast iron base melt leads to a shape change of the eutectic lamellar graphite to a nodular shape thus dramatically decreasing the crack-initiating effect of the graphite phase. This greatly enhances the cast iron component's mechanical properties. In addition, the nodularising Mg treatment creates a high number of small micro-inclusions providing a basis for an effective following inoculation [4-6]. By subsequent inoculation hexagonal phases are built on the formed Mg particles, which are favourable sites for graphite nucleation due to the coherent/semi coherent low energy interfaces [6].

Inoculation increases the number of graphite nucleation sites and leads to a minimised eutectic undercooling [7]; the nodularity and nodule count are increased leading to decreased pearlite content and formation of chill due to the high graphitisation potential of spheroidal graphite and a lower alloying element's segregation gradient [2, 8-10]. By improving the solidification morphology, inoculation decreases the amount of porosity [11].

Due to the inoculation effect's fading, Riposan et al. suggest that the inoculation of the iron basis melt should conducted as close as possible before the casting process [12]. Huerta and Popovski showed, that the effect of inoculant fade is drastic and can učinka [9]. Za ta namen se izdelajo uliti ali sintrani valjčki cepiva in položijo v spodnji formarski okvir.

Cepivo je navadno iz predzlitine FeSi z različnimi deleži Al [14], ki vsebuje enega ali več cepilnih elementov, npr. Sr, Ca, Ba, Zr in elemente redkih zemelj [7]. Sr-FeSi in Ca-FeSi kot cepivi za cepljenje duktilne litine v formi navadno ne delujeta dobro, imata pa koristen učinek kot snov za predhodno kondicioniranje taline [6]. FeSi-zlitine, ki vsebujejo Ba ali Zr v kombinaciji s Ca ne delujejo dobro pri cepljenju duktilne litine v formi. Na drugi strani pa so Ca,Ce,S,O-FeSi in Ca-FeSi ob predhodnem kondicioniranju taline z Al,Zr,Ca-FeSi ali brez kondicioniranja dobra cepiva za duktilno litino v formi [6, 15].

Kot že omenjeno, cepljenje v formi preprečuje v odvisnosti od kemične sestave bloka cepiva slabljenje učinkovitosti cepljenja, zato je ta način zelo učinkovit pri preprečevanju belega strjevanja in za povečanje števila kroglic grafita ter njihove kroglіčavosti [16]. Vendar so stroški izdelave ulitih ali sintranih blokov cepiva dva- do trikrat večji od standardnih zrnatih cepiv, ki se pogosto uporabljajo pri dodajanju cepiva v curek taline. V predstavljenem prispevku smo cepivo v bloku za cepljenje taline EN-GJS-400-15, ki še ni bila cepljena, v formi izdelali iz zrnatega cepiva različnih zrnavosti (ustreznih velikosti zrn ali z premajhnimi zrni) in veziva iz natrijevega silikata. Vpliv teh blokov na mikrostrukturo in mehanske lastnosti duktilne litine smo preverjali metalografsko in z nateznimi preskusi. Posebno nas je zanimal učinek premajhnih zrn cepiva na delovanje blokov cepiva, ker prahovi nastajajo pri mletju cepiva in se lovijo v sistemu filtrov mlina. Ti prahovi se do sedaj niso nikjer neposredno in koristno uporabljali, zato jih je na tržišču dovolj na razpolago in so zelo poceni.

occur within the first two to six minutes after inoculation [13]. The so called late inmould inoculation is an appropriate method against fading of the inoculation effect [9]. For this cast or sintered cylinders made from the inoculation agent are laid-up in the drag.

Inoculation agents, usually made from a FeSi base alloy with different amounts of Al [14], consist of one or more inoculation elements, e.g. Sr, Ca, Ba, Zr, and rare earth elements (RE) [7]. As inmould inoculation agents for ductile iron Sr-FeSi and Ca-FeSi alloys do not perform well, but have a beneficial effect as preconditioners [6]. Ba or Zr in combination with Ca bearing FeSi alloys also do not perform well for inmould inoculation of ductile iron. In contrast, Ca,Ce,S,O-FeSi and Ca-FeSi with or without Al,Zr,Ca-FeSi preconditioning of the melt are powerful inmould ductile iron inoculant agents [6, 15].

As mentioned above, inmould inoculation prevents, depending on the chemical composition of the inoculation block, the fading of the inoculation effect thus making this method very effective to prevent chilling and to increase the nodule count and nodularity [16]. However, the costs for the cast or sintered inoculation blocks are two to three times higher than the costs for conventional granular inoculation agents, which are often used in combination with the pouring stream inoculation method. In the present work, inoculation blocks made from a granular inoculation agent with different grain sizes (undersized and correctly sized particles) and a sodium silicate binder are used to realise an inmould inoculation of a completely not inoculated EN-GJS-400-15 melt. The influence of these blocks on the microstructure and mechanical properties of ductile iron are verified by metallography and tensile tests. Especially the effect of the undersized inoculation particles on the performance of the inoculation

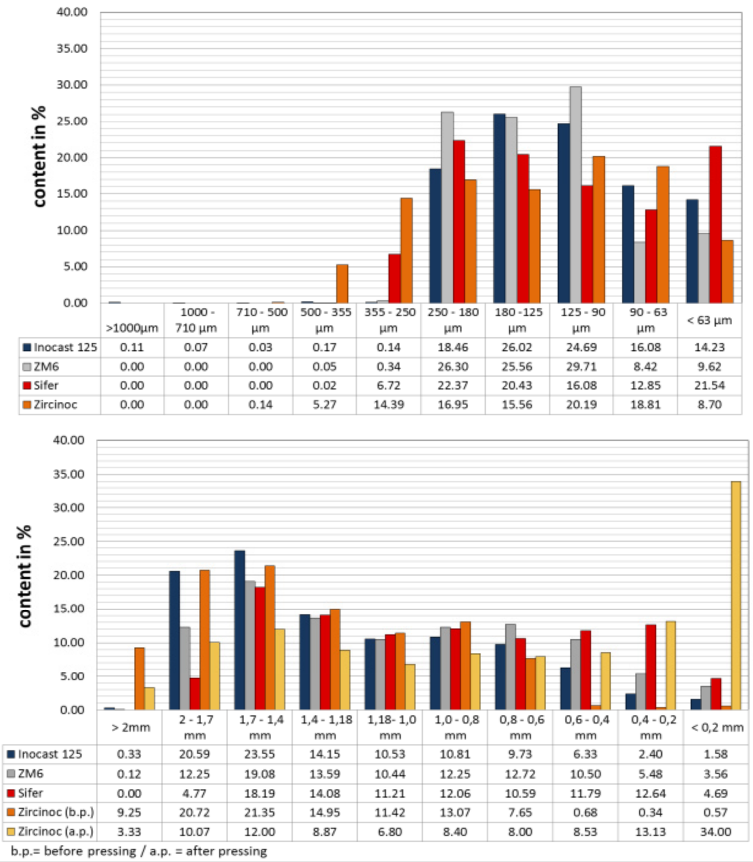
2 Materiali in metode

Trgovska cepiva Inocast125 (FerroPem Foundry Products, Francija), ZM6 (Foseco Nemčija pri Vesuvius GmbH, Nemčija), Sifer (TDR Legure, Slovenija) in Zircinoc (Elkem GmbH, Nemčija) za dodajanje cepiva v curek taline so bila s sejanjem v stolpu sit razvrščena v dva razreda velikosti zrn (0,0—0,4 mm in 0,4—2,0 mm) (slika 1), v nadaljevanju jih bomo imenovali ‘premajhna zrna’ in ‘zrna pravilne velikosti’.

blocks is of special interest since these powders are produced during milling of the inoculation agent, collected in the mill filter system. These find no direct and profitable application up to now, thus making them highly available and very cheap.

2 Materials and Methods

The commercially available pouring stream inoculation agents Inocast125 (FerroPem



	>1000µm	1000 - 710 µm	710 - 500 µm	500 - 355 µm	355 - 250 µm	250 - 180 µm	180 -125 µm	125 - 90 µm	90 - 63 µm	< 63 µm
Inocast 125	0.11	0.07	0.03	0.17	0.14	18.46	26.02	24.69	16.08	14.23
ZM6	0.00	0.00	0.00	0.05	0.34	26.30	25.56	29.71	8.42	9.62
Sifer	0.00	0.00	0.00	0.02	6.72	22.37	20.43	16.08	12.85	21.54
Zircinoc	0.00	0.00	0.14	5.27	14.39	16.95	15.56	20.19	18.81	8.70

	> 2mm	2 - 1,7 mm	1,7 - 1,4 mm	1,4 - 1,18 mm	1,18- 1,0 mm	1,0 - 0,8 mm	0,8 - 0,6 mm	0,6 - 0,4 mm	0,4 - 0,2 mm	< 0,2 mm
Inocast 125	0.33	20.59	23.55	14.15	10.53	10.81	9.73	6.33	2.40	1.58
ZM6	0.12	12.25	19.08	13.59	10.44	12.25	12.72	10.50	5.48	3.56
Sifer	0.00	4.77	18.19	14.08	11.21	12.06	10.59	11.79	12.64	4.69
Zircinoc (b.p.)	9.25	20.72	21.35	14.95	11.42	13.07	7.65	0.68	0.34	0.57
Zircinoc (a.p.)	3.33	10.07	12.00	8.87	6.80	8.40	8.00	8.53	13.13	34.00

b.p.= before pressing / a.p. = after pressing

Slika 1. Zrnavost cepiva iz premajhnih zrn (zgoraj) in iz zrn pravilne velikosti (spodaj)

Figure 1. Grain size distribution of the undersized (above) and of the correctly sized (below) particles

Razpredelnica 1. Podrobnosti izdelave blokov cepiva

Table 1. Details of the inoculation block's production.

velikost zrn / grain size	pravilna velikost / correctly sized	premajhna velikost / undersized
delež veziva / binder content [%]	1.5	4.0
masa vzorca Inocast / sample weight of Inocast 125, ZM 6 [g]	80	80
masa vzorca Sifer, Zircinoc / sample weight of Sifer, Zircinoc [g]	70	70

Razpredelnica 2. Kemična sestava uporabljenih cepiv in primerjalnega bloka

Table 2. Chemical compositions of the used inoculation agents and the reference block

Cepivo / Inoculation agent	velikost zrn / grain size [mm]	Si %	Zr %	Ca %	Mn %	Al %	Cr %	C%	Mg %
pravilna velikost / correctly sized									
Inocast 125	0,4-2	65.5	3.60	1.26	3.18	0.96	x	x	x
ZM6	0,6-2	65.5	3.20	1.25	3.36	0.61	x	x	x
Zircinoc	0,5-2	73.4	1.56	2.76	x	1.17	x	x	x
Si f e r	0,3-2	73.9	x	1.34	x	0.61	0.03	0.18	x
premajhna velikost / undersized									
Inocast 125	0-0,3	64.6	3.50	0.83	4.05	0.26	x	x	x
ZM6	0-0,4	64.2	3.30	0.70	3.20	0.50	x	x	x
Zircinoc	0-0,4	73.3	1.60	2.50	x	1.25	x	x	x
Si f e r	0-0,3	72.2	x	1.25	x	0.47	0.03	0.13	x
primerjalni vzorci / reference									
T 80	69	x	1.34	x	3.42	0.03	0.11	1.03	

Razpredelnica 3. Kemična sestava taline

Table 3. Chemical composition of the melt

%	C	Si	Mn	Cu	S	Cr	Al	Mg	P	CE
livna peč / casting furnance	3.66	2.44	0.14	0.12	0.003	0.04	0.007	0.058	0.03	4.48

Zrnata cepiva so bila zmešana z natrij-silikatnim vezivom HK 30 (Wöllner GmbH & Co. KG) in stisnjena v 45 mm ± 1.5 mm visoke bloke. Razpredelnica 1 prikazuje ostale podrobnosti izdelave blokov cepiva. Za primerjavo se je uporabil blok cepiva T80 (Tennant, Derbyshire, England). Kemična sestava cepiv je bila določena z mokrimi kemičnimi analizami (razpredelnica 2).

Foundry Products, France), ZM6 (Foseco Deutschland der Vesuvius GmbH, Germany), Sifer (TDR Legure, Slovenija) and Zircinoc (Elkem GmbH, Germany) were classified by a sieve tower into the two grain classes 0.0 - 0.4 mm and 0.4 - 2.0 mm (Figure 1), referred in the following as undersized and correctly sized particles.

Za izdelavo ulitkov in doseganje homogenega cepljenja se je uporabila simetrična kokila za brizgalno ulivanje z dvema gnezdoma, ki je vsako imelo 4 poglobitve. Kot poskusni vzorec je bil izbran ležaj tečaja s tankostenskim zavornim sedlom. Ko je bil vstavljen blok cepiva v spodnji okvir, smo formo zaprli in ulili necepljeno talino iz livne peči pri približni temperaturi 1420 °C (razpredelnica 3). Pred ulivanjem se je peč napolnila s talino iz 5 t GF-konvertorja, kjer je potekala obdelava z magnezijem. Da bi ustvarili neugodne razmere cepljenja, so bila poskusna ulivanja po premoru ob koncu tedna in po izmeni čiščenja. Ulilo se je nad 50 jedrnikov s 400 deli. Dodatno so bile z AccuVo-lončki (OCC GmbH, Nemčija) narejene še termične analize.

Iz vsakega poskusa z blokom cepiva je bilo v skladu s standardom DIN EN 50125 iz ležaja tečaja izdelanih s struženjem 16 nateznih preizkušancev. Kvazistatični poskusi so bili izvedeni v skladu s standardom DIN EN ISO 6892-1. Vzorci za metalografsko analizo so bili vloženi v dvokomponentno maso, nato brušeni na SiC-brusnih papirjih različnih stopenj (80 do 600) in polirani z Buehlerjevo polirno suspenzijo (ITW Test & Measurement GmbH, Nemčija). Izdelanih je bilo po osem obrusov za vsak blok cepiva in 9 fotografskih posnetkov v svetlobnem mikroskopu na vsakem obrusu. Obrusi so bili jedkani s HNO_3 30—60 s, da se je odkrilo razmerje ferit/perlit. Na nejedkanih mikroposnetkih mikrostrukture se je ugotavljalo število kroglic na mm^2 in njihova oblika ter velikostna porazdelitev.

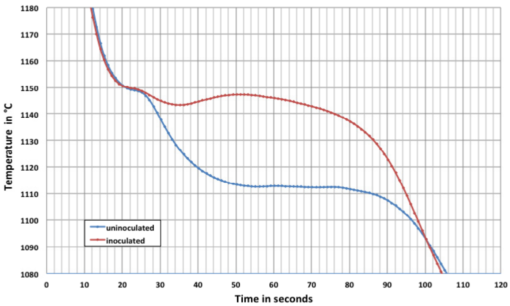
3 Rezultati

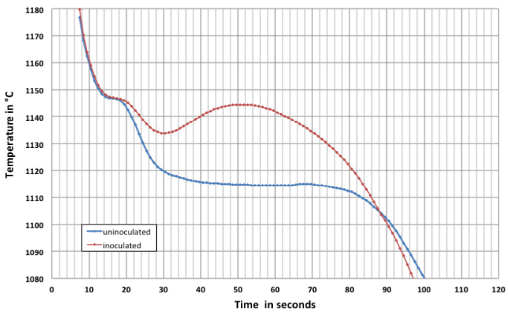
Slika 2 kaže krivulje termične analize taline, vzete iz poskusov litja (levo), in primerjalnega vzorca, vzetega iz serijske proizvodnje (desno). V obeh primerih

These were mixed with the soda silicate binder HK 30 (Wöllner GmbH & Co. KG) and pressed to 45 mm ± 1.5 mm high blocks. Table 1 summarises further details of the inoculation block's production. As reference the inoculation block T80 (Tennant, Derbyshire, England) was used. The chemical compositions of the inoculation agents were determined by means of wet chemical analyses (Table 2).

For the production of the casting mould by an impact moulding facility, a symmetric pattern was used to realise a homogenous inoculation of the two nests with four cavities each. As test part, a hinge bearing with a thin-walled break calliper was chosen. After the inoculation block's insertion in the drag the mould was closed and cast with the aid of a casting furnace at a temperature of approx. 1420 °C with a not inoculated melt (Table 3). Before casting, this furnace was filled with the aid of a 5 t GF converter, which was used for the magnesium treatment. To achieve hindered inoculation conditions the casting trials were conducted after a weekend break and cleaning shift. Overall 50 boxes with 400 parts were cast. In addition thermal analyses were taken with the aid of AccuVo crucibles (OCC GmbH, Germany).

Per inoculation block variant, 16 tensile specimens were machined from the hinge bearings according to DIN EN 50125 and tested by a quasi-static tensile test according to DIN EN ISO 6892-1. By embedding specimen sections in a two component embedding material, grinding them with SiC abrasive paper of different grade (80 to 600) and polishing them with a polishing suspension from Buehler (ITW Test & Measurement GmbH, Germany), 8 metallographic sections per inoculation block variant were prepared. After taking 9 photographs per metallographic section with a light microscope, they were etched





Slika 2. Termična analiza poskusov litja (zgoraj) in serijske proizvodnje (spodaj)

Figure 2. Thermal analysis of casting trials (above) and serial production (below)

so bile meritve narejene z necepljeno talino. Specifične vrednosti obeh krivulj so prikazane v razpredelnici 4. Zdi se, da so temperature likvidus obeh krivulj enake, toda evtektična podhladitev in rekalescenca sta bili pri serijski proizvodnji manjši kot pri poskusih litja.

30 to 60 s with HNO_3 to reveal the ferrite/pearlite ratio. With the aid of the unetched microstructures' micrographs, the number of nodules per mm² and their shape and size distributions were analysed.

3 Results

Figure 2 shows the thermal analysis of the melt taken during the casting trials (left) as well as a reference, taken under serial production conditions (right). In both cases, the measurements were done on uninoculated melt. The specific values of both curves are given in Table 4. The liquidus temperatures of both curves can be seen as equal, but the eutectic supercooling and recalescence under serial production conditions are lower compared to the values of casting trials.

Table 5 presents the results of the quasi-static tensile tests. In general, it can be seen that the minimal values for yield strength (250 MPa) and tensile strength (400 MPa), according to DIN EN 1563:2012-3, are achieved by the application of all types of inoculant blocks. In contrast to yield strength and tensile strength, the elongation at fracture can be considered as the critical property. The required minimal value of 15 % is just reached by four variants of inoculant blocks. These four variants achieve 15 % elongation at fracture in average, without taking the standard deviation into account.

Razpredelnica 4. Specifične vrednosti termičnih analiz

Table 4. Specific values of the thermal analyses

		T(liq)	T(sol)	TE(min)	TE(max)	Soopercooling	Recalescence
		[°C]					
Casting trials	uninoculated	1147	1137	1111.3	x	35.7	x
	inoculated	1147	1142	1133.8	1144.4	16.2	10.6
Normal production	uninoculated	1149	114	111.6	x	37.4	x
	inoculated	1150	1145	1143.3	1147.3	6.7	4

Razpredelnica 5 prikazuje rezultate kvazistatičnih nateznih poskusov. V splošnem se lahko vidi, da so bile najmanjše vrednosti napetosti tečenja (250 MPa) in natezne trdnosti (400 MPa), ki so ustrezale standardu DIN EN 1563:2012-3, dosežene pri vseh vrstah blokov cepiva. V nasprotju z napetostjo tečenja in natezno trdnostjo se raztezki pri pretrgu smatrajo kot kritične vrednosti. Zahtevana najnižja vrednost 15 % je bila komaj dosežena pri štirih blokih cepiva. Pri teh blokih je doseženi 15 %ni raztezek pri pretrgu predstavljal povprečje brez upoštevanja standardne deviacije. Če se upošteva še standardna deviacija, nekateri bloki cepiva niso izpolnjevali zahtev po raztezku pri pretrgu, ki jih predpisuje DIN EN 1563:2012-3.

Rezultati z blokom cepiva T-80 so predstavljali primerjavo za rezultate, dosežene z bloki cepiva lastne izdelave. Primerjava obojih rezultatov je pokazala, da so bile z bloki T-80 komaj dosežene povprečne vrednosti napetosti tečenja in

In consideration of the standard deviation, some variations of inoculant blocks do not fulfil the elongation at fracture required by DIN EN 1563:2012-3.

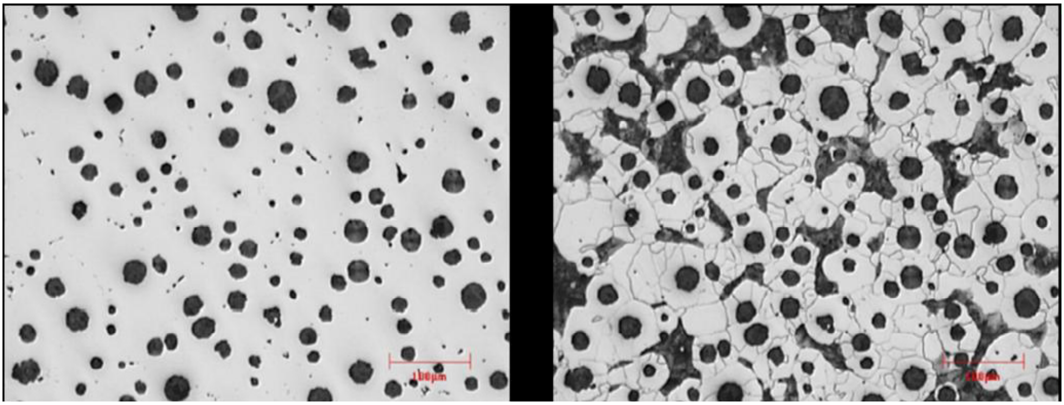
The results of the T80-inoculant block are used as a reference for the results achieved by the self- made inoculant blocks. A comparison of the results achieved by the commercial product and by the self-made blocks shows that by applying of the T80 block just average values concerning yield strength and tensile strength but highest elongation at fracture are achieved. The self-made inoculant blocks made of correctly sized Inocast125 and ZM6 reach an average elongation at fracture over 15 %, while undersized Inocast125-blocks reach the lowest value.

A typical microstructure of the samples (unetched and etched) is presented in Figure 3. The etched part of Figure 3 shows a mainly, but not complete, ferritic microstructure as it is typical for GJS-400-15. Single degenerated spherulites

Razpredelnica 5. Rezultati kvazistatičnih nateznih preskusov in meritev trdot

Table 5. Results of the quasi-static tensile tests and hardness measurements

Inoculation agent	Grain size		Yield strength [N/mm^2]	Tensile strength [N/mm^2]	elongation at fracture [%]	Hardness 5/750 [HB]
T80	reference	mean	317	509	15.7	166
		s +/-	10.4	23.1	2.2	7.4
Inocast 125	undersized	mean	334	537	12.2	183
		s +/-	9.6	15.1	3.2	11.4
ZM 6	undersized	mean	328	533	15	181
		s +/-	10.1	21.9	2.7	7.1
Sifer	undersized	mean	322	515	14	173
		s +/-	15.4	28.6	3.3	9.6
Zircinoc	undersized	mean	319	510	14.4	171
		s +/-	12.3	24.6	2.3	9.3
Inocast 125	correctly sized	mean	313	497	15.5	171
		s +/-	5.2	18	1.6	6.9
ZM 6	correctly sized	mean	313	499	15.6	167
		s +/-	3.9	10.7	1.9	9
Sifer	correctly sized	mean	318	506	13.9	170
		s +/-	12.7	17.8	2.2	5.9
Zircinoc	correctly sized	mean	325	518	14.4	181
		s +/-	14.4	21.1	3.3	7.1



Slika 3. Značilna mikrostruktura vzorcev: nejedkano (levo) in jedkano (desno), pov. 100 x

Figure 3. Typical microstructure of samples: unetched (left) and etched (right), 100x

Razpredelnica 6. Rezultati metalografskih analiz

Table 6. Results of the metallographic analyses

Inoculation agent	Grain size		nodule count	graphite content	perlit content	nodule shape			nodule size		
			[1/mm^2]	[%]	[%]	III [%]	V [%]	VI [%]	6 [%]	7 [%]	8 [%]
T80	reference	mean	464	11.5	42.3	6.9	54.4	38.7	14.9	27.9	57.2
		s +/-	137	0.3	9	2.6	8.1	10.5	7.6	8.2	2.0
Inocast 125	undersized	mean	414	11.7	46.2	7.3	53.7	38.8	17.2	25.2	57.5
		s +/-	101	0.4	9.5	2.3	6.4	8.7	7.2	8.4	2.2
ZM 6	undersized	mean	524	11.7	40.3	5.3	48.9	45.8	11.9	29.3	58.8
		s +/-	150	0.4	7.9	1.3	5.2	6.4	5.9	3.2	3.4
Sifer	undersized	mean	529	11.8	44.5	6.2	51.6	42.2	12.2	27.6	60.2
		s +/-	162	0.3	12.3	2.4	9.5	11.9	7.2	7.2	2.8
Zircinoc	undersized	mean	583	12.1	31.9	4.5	45.7	49.8	10.3	30.7	59.0
		s +/-	153	0.7	5.8	2.0	7.3	9.2	6.2	5.3	3.6
Inocast 125	correctly sized	mean	613	11.4	34.6	4.6	48	47.4	8.6	30.6	60.8
		s +/-	182	0.5	9.6	2.2	6.2	8.2	7.0	4.7	3.7
ZM 6	correctly sized	mean	602	11.7	35.9	3.9	46.8	49.3	8.6	31.0	60.4
		s +/-	121	0.7	6.4	2.2	4.2	5.7	4.1	3.1	3.5
Sifer	correctly sized	mean	518	11.6	38.9	5.8	48.7	45.5	10.7	31.5	57.8
		s +/-	113	0.4	8.4	1.4	5.3	6.1	4.8	3.3	2.4
Zircinoc	correctly sized	mean	525	11.7	41.7	5.6	52.8	41.5	11.4	28.0	60.5
		s +/-	127	0.6	11.3	2.8	8.6	11.4	6.1	7.4	3.6

natezne trdnosti, a raztezki pri pretrgu so bili večji. Z bloki cepiva lastne izdelave, narejenimi iz cepiv Inocast 125 in ZM 6 s pravo velikostjo zrn so bile doseženi povprečni raztezki nad 15 %, medtem ko so bili z bloki cepiva Inocast 125 iz premajhnih zrn dosežene najnižje vrednosti.

were detected during the metallographic examination, but mainly nodule shape V and VI is existent.

The results of the metallographic examination, according to DIN EN ISO 945-1:2010 are summarised in Table 6. For all types of inoculant blocks the content of

Značilno mikrostrukturo vzorcev (nejedkanih in jedkanih) prikazuje slika 3. Jedkani vzorci na sliki 3 kažejo predvsem, a ne v celoti, feritno mikrostrukturo, kot je značilno za litino GJS-400-15. Pri metalografski preiskavi so se našle tudi posamezne kroglice popačenih oblik, a so bile oblike kroglic predvsem razreda V in VI.

Rezultati metalografske preiskave po DIN EN ISO 945-1:2010 so povzeti v razpredelnici 6. Za vse vrste blokov cepiva je bilo kroglic oblik V in VI nad 90 % in kroglic na mm^2 med 414 in 613. Če je veliko število kroglic na mm^2 merilo za učinek cepljenja, so bili najboljši rezultati doseženi z bloki Inocast 125, narejenimi iz zrn pravilnih velikosti (613 kroglic na mm^2) in bloki ZM 6 (602 kroglice na mm^2) tudi narejenimi iz zrn pravilnih velikosti. Rezultati, doseženi s primerjalnim blokom T-80 (povprečno 464 kroglic na mm^2), so bili med najslabšimi.

4 Razprava

Glede na termične analize, ki so bile narejene med poskusi ulivanja, sta velika podhladitev (16,2 °C) in nizka evtektična temperatura TE_{min} nakazali nevarnost metastabilnega strjevanja taline. Specifične vrednosti v razmerah serijske proizvodnje so kazale na manjšo podhladitev in višjo evtektično temperaturo, kar povečuje verjetnost stabilnega strjevanja. Poleg tega so poskusi ulivanja potekali pri neugodnih razmerah (po premoru ob koncu tedna in po izmeni čiščenja), da bi preverili učinkovitost doma narejenih blokov cepiva pri 'najslabših razmerah'. Hladna oprema za ulivanje in hladen pesek so povečali nevarnost metastabilnega strjevanja. Zato se lahko velika podhladitev in nizka evtektična temperatura pripišeta neugodnim razmeram ulivanja. Z uporabo blokov

nodule shape V and VI is over 90 % and the number of nodules per mm² is between 414 and 613 spherulites/mm2. If a high number of nodules per mm² is taken to evaluate the inoculation effect, best results are achieved by correctly sized Inocast125-blocks (613 spherulites/mm2) and ZM6 (602 spherulites/mm2). With an average number of nodules per mm² of 464 spherulites/mm2 the T80-reference block reaches one of the worst results.

4 Discussion

Concerning the thermal analysis taken during the casting trials, the high supercooling (16,2 K) as well as the low eutectic temperature TE(min) indicate the risk of a metastable solidification of the melt. The specific values under serial production conditions have a lower supercooling and a higher eutectic temperature, which increases the probability of a stable solidification. Moreover, the casting trials were conducted under hindered conditions (after a weekend break and cleaning shift) in order to test the effectiveness of the self-made inoculant blocks under ´worst-case´ conditions. The cold casting equipment and sand system increase the risk of metastable solidification. Hence, the high supercooling and the low eutectic temperature can be related to hindered casting conditions. By using inoculant blocks a metastable solidification can be avoided.

A comparison of the mechanical properties of the self-made inoculant blocks with these of the T80-reference block shows, that the specific values of all variants reach comparable results. This was verified within a statistical check-up of the results by the Tukey-method (t-test). A significant distinction was observed between the tensile strength of correctly sized Inocast125- and cepiva se lahko izogne metastabilnemu strjevanju. Primerjava mehanskih lastnosti z doma narejenimi bloki cepiva in s primerjalnim blokom T-80 je pokazala, da so dobljene vrednosti pri vseh uporabljenih blokih dale primerljive rezultate. To je bilo statistično preverjeno s Tukeyevo metodo (t-test). Opazili smo občutno razliko med natezno trdnostjo, doseženo z bloki cepiva Inocast 125 in bloki cepiva Zircinoc, ki so bili narejeni iz pravilno velikih delcev. Poleg tega je natezna trdnost, dosežena z bloki cepiva Zircinoc, pokazala občutno razliko v primerjavi z enako izdelanimi bloki iz cepiv ZM6 in Inocast 125. Kar se tiče raztezka pri pretrgu kot kritične lastnosti, je treba upoštevali na eni strani, da so bili rezultati, dobljeni z vsemi uporabljenimi bloki, primerljivi, na drugi strani pa bi lahko velika odstopanja skrivala možen vpliv kemične sestave ali zrnavosti.

Primerjava rezultatov, dobljenih z bloki cepiva Sifer iz pravilno velikih zrn in iz premajhnih zrn, je dala podobne rezultate za vse mehanske lastnosti in ni odkrila vpliva zrnavosti cepiva. V nasprotju z bloki cepiva Sifer so bile z bloki cepiva Inocast 125 iz zrn premajhne velikosti dosežene večje natezne trdnosti in napetosti tečenja v primerjavi z bloki cepiva Inocast 125 iz pravilno velikih zrn, vendar je bil raztezek pri pretrgu pri njih manjši. Tako so bile z bloki iz premajhnih velikosti zrn dosežene slabše lastnosti ulitega materiala. Slabše lastnosti materiala se lahko pojasnijo z majhnimi deleži Al in Ca. Kot predlaga A. Henke v [17], je kumulativni delež Al in Ca v višini okoli 2 mas. % nepogrešljiv za uspešno cepljenje. Pri samo polovični količini Al in Ca se lahko slabe mehanske lastnosti, dosežene z bloki iz cepiva Inocast 125 iz premajhnih velikosti zrn, pripišejo nepopolnemu ali neučinkovitem cepljenju. Glede na podrobnosti izdelave blokov cepiva je treba omeniti različne mase blokov

Zircinoc inoculant blocks. Furthermore, the yield strength of correctly sized Zircinoc indicates significant differences compared to correctly sized ZM6 and Inocast125. With regard to the critical property, the elongation at fracture, it has to be considered that on one hand the results of all variations of inoculant blocks are comparable but on the other hand the large deviations can hide a possible effect of chemical composition or grain size distribution.

A comparison of correctly sized and undersized Sifer-inoculant blocks shows similar results for all mechanical properties, revealing no influence of grain size distribution. In contrast to Sifer- inoculant blocks, the undersized Inocast125 inoculant blocks achieve higher tensile strength and yield strength compared to the correctly sized Inocast125 blocks, but a decreased elongation at fracture. Therefore, lower material properties are achieved by the undersized blocks. The lower material properties can be explained by the low content of Al and Ca. As F. Henke suggested in [17], a cumulative content of about 2 % Al and Ca is indispensable for a successful inoculation. With just the half amount of Al and Ca, the low mechanical properties, generated by the undersized Inocast125 inoculant blocks, can be traced back to an incomplete or ineffective inoculation. Referring to the details of the inoculation block's production, the different weight of the blocks, and the different content of inoculation agent in the mould respectively have to be mentioned. Hence, the slightly lower values reached by Zircinoc and Sifer blocks, compared to the other types of inoculant blocks, can possibly be correlated to a 10-g discrepancy of inoculation agent.

Regarding the results of the metallographic examination, a statistical check-up of the results was not conducted, because of the large deviations and the oz. različne deleže cepiva v formi. Zato se malo nižje vrednosti, dosežene z bloki Zircinoc ali Sifer, v primerjavi z drugimi bloki cepiv lahko pripišejo 10 g odstopanjem pri količinah cepiva.

Kar se tiče rezultatov metalografskih preiskav, statistične analize teh rezultatov niso bile narejene zaradi velikih odstopanj in velikega števila vzorcev. Zato se ne da nakazati očiten vpliv kemične sestave ali zrnavosti. Pri ovrednotenju metalografskih rezultatov je bilo vzeto, da je potrebnih vsaj 350 kroglic na mm (kombinirano cepljenje v loncu in v formi) za potencialno uporabo v proizvodnji. Ta vrednost je bila določena s prejšnjimi preiskavami.

Z bloki cepiva Inocast 125 iz premajhnih zrn se je doseglo najmanjše število kroglic (414 ± 101 kroglic na mm^2), vendar je treba upoštevati majhen delež Al in Ca v tem cepivu. Druga najnižja vrednost števila kroglic na mm^2 je bila dosežena s primerjalnim blokom T-80 (464 ± 137 kroglic/mm^2). Najboljši rezultati so bili doseženi z bloki cepiva Inocast 125 iz pravilno velikih zrn (613 ± 182 kroglic/mm^2) in enako narejenih blokov cepiva ZM6 (602 ± 120 kroglic/mm^2).

Kar se tiče analize razmerja ferit/perlit, se je pričakovala popolnoma feritna mikrostruktura. Analiziran delež perlita 31.9 – 46.2 % je neznačilen za litino GJS 400-15 in kaže na nezadostno cepljenje. Zato je uporaba blokov cepiva učinkovita, da se izognemo nastanku metastabilne evtektične mikrostrukture, ne zadostuje pa za doseganje popolnoma feritne mikrostrukture, ki je z DIN EN 1563:2012-3 predpisana za litino GJS-400-15.

Zato so kombinirane metode cepljenja [18], kot se uporabljajo v standardni proizvodnji, koristne za izpolnjevanje vseh zahtev.

V primerjavi v primerjalnim blokom T-80 so dali vsi doma narejeni bloki

number of samples. Consequently, an obvious influence of chemical composition or grain size distribution cannot be indicated. In order to evaluate the metallographic results, a minimum number of 350 nodules per mm^2 is necessary for a potential application in production. This value has been defined during former investigations.

The undersized Inocast125 inoculant blocks yield the lowest number of nodules per mm^2 (414 ± 101 nodules/mm2), but the low Al and Ca content of this inoculation agent has to be kept in mind. The second lowest number of nodules per mm^2 is yielded by the reference block T80 (464 ± 137 nodules/mm^2). The best results are achieved by correctly sized Inocast125 (613 ± 182 nodules/mm^2) and ZM6 (602 ± 120 nodules/mm^2) inoculant blocks.

Concerning the analysis of ferrite/perlite ratio, a fully ferritic microstructure was expected. The analysed perlite content of 31.9 - 46.2 % is untypical for a GJS 400-15 and indicates an insufficient inoculation. Hence, the use of inoculation blocks is effective in order to avoid the formation of a metastable eutectic microstructure, but is deficient in order to produce a fully ferritic microstructure, required for GJS-400-15 by DIN EN 1563:2012-3. Consequently, a

After a combined ladle and inmould inoculation combination of inoculation methods [18], as applied in standard production, is beneficial in order to fulfil all requirements.

Compared to the T80 reference block, all types of self-made inoculation blocks achieve acceptable results. The partially large deviations of the results can be traced back to the number of samples on one hand and to process variations during casting trials on the other hand. Basically, a combined evaluation of mechanical and metallographic tests indicates best results for correctly sized Inocast125 and ZM6 cepiva sprejemljive rezultate. Delno veliko odstopanje rezultatov je bilo ugotovljeno na eni strani pri velikem številu vzorcev, po drugi strani pa se ga lahko pripiše procesnim spremembam pri poskusnih litjih. V osnovi kombinirano vrednotenje mehanskih preskusov in metalografskih preiskav daje najboljše rezultate za bloke cepiv Inocast 125 in ZM6 iz pravilno velikih zrn (največji raztezek pri pretrgu in največje število kroglic na mm^2), medtem ko dajeta bloka cepiv Sifer in Zircinoc iz zrn pravilne velikosti malo slabše rezultate. Kar se tiče kemične sestave cepiv Inocast 125 in ZM6, so bili dobri rezultati pričakovani zaradi povečanega deleža Zr in znanega dobrega učinka Zr na cepljenje [19, 20]. Nasprotno vir [17] predpostavlja, da učinek dodatkov cepivom pri cepljenju v formi ni tako pomemben kot učinek deleža Si. Zato večji delež Si v cepivih Sifer in Zircinoc lahko kompenzira manjši delež Zr. Če se upošteva to kompenziranje, so lahko nekoliko slabše vrednosti posledica manjše mase blokov cepiva in s tem manjšega dodatka cepiva. Poleg tega je možno, da se vpliv kemične sestave prekriva s kako motnjo.

Kar se tiče sprememb zrnavosti v odvisnosti od proizvodnega procesa, so bile razlike med uporabljenimi zrnavostmi premajhne, da bi lahko ugotovili njihov vpliv.

5 Sklepi

Kot so pokazali mehanski preskusi in metalografske analize ni očitne razlike med trgovskim izdelkom T-80 in doma narejenimi bloki cepiva. Poleg tega so nekateri doma narejeni bloki cepiva dali celo boljše rezultate kot T-80. Zato so vsi doma izdelani bloki primerni za uporabo v proizvodnji. Medtem ko sama uporaba blokov cepiva preprečuje metastabilno evtektično strjevanje, je

inoculant blocks (highest elongation at fracture and highest number of nodules per mm2), while correctly sized Sifer and Zircinoc blocks yield slightly decreased values. Considering the chemical composition of Inocast125 and ZM6, good results were expected, because of the raised Zr content and the high inoculation effect of Zr [19, 20]. In contrast, [17] suggests, that the effect of inoculation additives by the use of inmould inoculation is not as important as the effect of the Si content. Therefore, the higher Si content of Sifer and Zircinoc would equalize their lower Zr content. In case of equalization, the slightly decreased values can originate from the reduced weight of the inoculation blocks and thereby a reduced addition of inoculation agents. Moreover, it might be possible that the effect of the chemical composition is superimposed by any disturbance.

Concerning the change of grain size distribution caused by production process, the differences between the used grain size distributions are too small to identify any influence.

5 Conclusions

As shown by the mechanical and metallographic tests, there are no distinct differences between the commercial product T80 and the self-made inoculation blocks. Moreover, some of the self- made blocks even achieve better results than T80. Consequently, all variants are suitable for application in production. While the single use of inoculant blocks avoids a metastable eutectic solidification, a combination of inoculation methods is needed in order to produce safety related components.

A definite influence of chemical composition as well as of grain size distribution cannot be determined in this potrebna kombinacija postopkov cepljenja, da se zagotovi varna zanesljivost izdelanih strojnih delov.

S tem delom se ni dalo nedvoumno ugotoviti vpliv kemične sestave in zrnavosti cepiva. Zato menimo, da dodatki cepivom nimajo pomembne vloge pri cepljenju v formi. Poleg tega je možno, da se vpliv kemične sestave prekriva s kako drugo motnjo, npr. s spremembo dodatka cepiva. Tudi se ni dalo ugotoviti vpliva zrnavosti cepiva na učinek cepljenja taline duktilne litine. Sprememba zrnavosti, ki nastane pri izdelavi blokov cepiva lahko minimizira prvotne razlike. Če se povzame, je možna zamenjava trgovsko dostopnih blokov cepiva T-80 z doma narejenimi bloki iz zrnatih cepiv in natrij-silikatnega veziva. Poleg tega bo lahko firma Geoerg Fischer z zamenjavo trgovskega izdelka v prihodnosti postopoma zmanjšala svojo odvisnost od dobaviteljev.

6 Zahvale

Avtorji se zahvaljujejo upravnemu odboru firme Georg Fischer GmbH (Mettmann) za finančno podporo. Zahvaljujejo se tudi g. P. Hermannu in g. E. Thomasu za njuno podporo pri načrtovanju in izvedbi tega dela ter za analize in dokumentacijo.

work. Due to this it is assumed that the additives of inoculation agents play an insignificant role during inmould inoculation. Besides, it might be possible, that the effect of the chemical composition is superimposed by any disturbance, e.g. a variation of the inoculation amount’s addition. An influence of the grain size distribution to the inoculation effectiveness of a DI melt could neither be identified. A change of the grain size distribution, which is caused by the production process of the inoculant blocks, might have minimised the original differences. A decreased variation of grain size distribution could effect, that no influence can be identified. To sum up, a substitution of the commercial available inoculation block T80 by the self-made inoculant blocks made of granular inoculation agents and soda silicate binder is possible. Moreover, by substituting the commercial product in the future, Georg Fischer will be able to reduce the supplier dependence step by step.

6 Acknowledgements

The authors gratefully acknowledge the financial support of the management board of the Georg Fischer GmbH (Mettmann). They also extend their gratefulness to Mr. P. Herrmann and Mr. E. Thomas for their support during the planning, realisation, analysis and documentation of the presented study.

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