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INFLUENCE OF LOW-ALLOY CAST STEEL MODIFICATION ON PRIMARY STRUCTURE REFINEMENT, TYPE AND SHAPE OF NON-METALLIC INCLUSIONS

WPLYW MODYFIKACJI NISKOSTOPOWEGO STALIWA NA ROZDROBNIENIE STRUKTURY PIERWOTNEJ, RODZAJ I KSZTAŁT WTRĄCEŃ NIEMETALLICZNYCH

In the article there are presented methods and results of investigation which main aim were determination of influence of melting technology (gas extraction, vacuum refining, slag refining and extraction, deoxidation and degassing) and type of used modifiers on the type and shape of non-metallic inclusions and the primary structure refining. Low alloy cast steel melted in laboratory conditions, in an inductive furnace was investigated. Additions of FeNb, FeV, FeTi and FeZr modifiers were applied. The contents of oxygen and nitrogen in obtained cast steel were determined.

The most advantageous impact on refining of the primary structure of has been found for the modifiers FeTi+FeZr. In cast steel with the addition of Zr the conglomerates from a different non-metallic inclusions have been observed. Zirconium probably plays role of the nucleus – creating for other inclusions present in the conglomerates. Clusters of inclusions due to their size affect the crystallization and grain growth processes reducing grain size of the primary structure.

Keywords: steel cast, primary structure, non-metallic inclusions, modification, grain refinement

W pracy przedstawiono założenia, metodykę i wyniki badań, których celem było określenie wpływu technologii wytopu (ekstrakcji gazowej, rafinacji próżniowej, rafinacji i ekstrakcji żuźlowej, odtleniania i odgazowania) i rodzajów zastosowanych modyfikatorów na postać i rodzaj wtrąceń niemetalicznych oraz rozdrobnienie struktury pierwotnej. Analizie poddano niskostopowe staliwo wytopione w warunkach laboratoryjnych w piecu indukcyjnym. Zastosowano dodatek modyfikatorów FeNb, FeV, FeTi i FeZr. W uzyskanym staliwie określono zawartość O i N oraz jego skład chemiczny.

Stwierdzono najkorzystniejsze, ze względu na rozdrobnienie struktury pierwotnej, oddziaływanie modyfikatora FeTi+FeZr. Zaobserwowano występowanie konglomeratów różnych wtrąceń niemetalicznych w staliwach z dodatkiem Zr, który prawdopodobnie działa zarodkotwórczo na pozostałe wtrącenia wchodzące w skład konglomeratów. Skupiska wtrąceń ze względu na swoje rozmiary wpływały na krystalizację i wzrost ziaren staliwa rozdrabniając jego strukturę pierwotną.

1. Introduction

Mechanical properties of cast steel are, first of all, function of chemical composition and solidification process, that is of the, primary structure. The microstructure formation process influence external factors connected with casting technology which forces kinetics of solidification (mass of casting, average wall thickness, thermal properties of mold, way of pouring) as well as internal factors, generally understood as metallurgical and chemical quality of liquid alloy [1, 2].

The chemical purity is connected with chemical composition in context of the content of harmful elements, particularly sulfur and phosphorus. The significance of chemical purity increases when charge of recycled steel and cast steel is used, especially in the process carried on in the electric foundry furnaces [3].

The metallurgical purity is associated with the occurrence of non-metallic inclusions, which negatively affect the casting

process. The most common non-metallic inclusions in the cast steel are oxides, sulfides and nitrides [4-6].

To the group of the internal factors should also be included substances which are inserted purposely in modification process into the liquid metal. Change of crystallization process due to modification allows obtaining fine equiaxed structure and high mechanical properties. This change depends mainly on type of the modifier used [7-9].

The refinement of primary structure is explained by two mechanisms which may occur separately or simultaneously. The first mechanism consists in increasing the rate of nucleation, which is obtained by introducing into the liquid metal particles of high-melting metals, their compounds or microadditives, which react with the components of liquid metal. As the result phases form in the melt which make possible initiation of heterogeneous nucleation. The effect of the modifier is associated with reduction of the creation energy of critical nuclei on surfaces of the modifier particles, which are substrates of heterogeneous nucleation. Under such conditions,

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the crystallization proceeds at much lower undercooling than it would be necessary for homogeneous nucleation [7]. The initiation of nucleation on surfaces of foreign particles probably may be governed by the similar mechanisms as the widely described ones for non-ferrous base alloys, e.g., aluminium based or zinc-aluminium based cast alloys [8-11].

The second mechanism consisting in reducing the critical growth rate of crystal nucleus is explained basing on the phenomenon of adsorption, according to which modifiers or their surface-active compounds adsorbing on the edge of nucleus create a kind of barriers that inhibit the exchange of atoms between the growing crystal nucleus and the liquid, thereby preventing their growth. The adsorption does not occur on all sides of crystals evenly, leading to the stop of growth of individual crystal facets and changing its shape. In addition to inhibition of crystal growth rate the number of centers of crystallization increases, which results in fragmentation of the structure. Such modification can be explained also through a mechanism that assumes the presence of microadditives concentration gradient directly at the crystallization front. Thus, the layer of solution liquid alloy with higher microadditive content (concentration barrier) created at the front of the crystallization makes it difficult to provide so-called building material to interfacial surface which inhibits the growth of crystals and leads to a reduction of its size [12].

In many publications beneficial effect of small additives of various elements, the so-called microadditives is claimed, without explaining it by their modifying influence on the crystallization process. The microaddition of Nb and V increase mechanical properties of cast steel, especially after heat treatment, which is greater than the expected one from the degree of change of the chemical composition [13-14]. Scientists dealing with theories of modifications related to refining as well as other theories, consider that the base of classification are only two groups of all the theoretical hypotheses of explanation the phenomenon of modification, which are counted to theories of effect on the crystallization.

The hypotheses included in the first group explain the process of modification by the creation of new nucleation substrate, which can be the introduced into the liquid metal with elements (modifiers) or chemical compounds and intermetallic as well as with the compounds formed in the metal bath after a certain period of time from the modifiers (oxides, sulfides, nitrides, carbides and hydrides).

To the second group of hypotheses are numbered to modifiers acting through their influence on the reduction of crystal growth. Many researchers believe that in the modification processes two types of phenomenon occurs, parallel each to other, i.e. artificial nucleation and inhibition of crystal growth. Therefore it can be concluded that the modification of alloys could be perform with microadditives affecting the crystallization process: nucleus -creating or surface -active.

In current research carried out at the Department of Foundry Silesian University of Technology, there has been made an effort to perform the melting in laboratory conditions with parameters as close to the industrial ones as possible. For such conditions, during the series of the melts carried out with varying technological parameters, an attempt has been made to establish mentioned above correlation between gas content, modifiers used and refinement of the primary structure.

Basing on the data available in literature it can be concluded that the optimal conditions for obtaining low-alloy cast steel, not worse in terms of mechanical properties of equivalent steel, are: high chemical and metallurgical purity as well as fine grain primary structure. The first condition requires the high quality of charge materials, technical culture and background, that makes possible to obtain a cast steel with chemical composition exactly as planned on one hand, and free from gaseous and metallic impurities on the other hand. The second condition is connected with properly selected (both for the chemical composition of alloy and the geometry of the casting) the modifying additive having a broad spectrum of action.

However the task greatly complicates not fully understood and explained mechanisms of modification, interaction of elements in cast steel with the so-called modifiers as well as gases dissolved in cast steel.

2. Methods of investigation

The cast steel investigated in the present work, was subjected to the classic procedures of dephosphorization, using high basic and oxidizing slag, and desulfurization connected with the diffusive deoxidation. In order to decrease amount of non-metallic inclusions in the investigated cast steel, the diffusive, sedimentary and vacuum deoxidation treatment was applied during the melting. In frame of this work, aimed at primary structure refining of the investigated cast steel, the modification with additives of Nb, V, Zr, Ti was carried out.

Two stages of low-alloy cast steel meltings in the laboratory crucible inductive furnaces were carried out. The melts were subjected to dephosphorization, desulfurization, diffusive deoxidation, argoning, sedimentary deoxidation and vacuum refining as well as modification with FeNb, FeV, FeZr, FeTi additives. During each melting the samples of cast steel were taken directly from the furnace and a number of test castings were made. The chemical composition of melted cast steel as well as O and N content have been determined. The investigation of micro- and macro structure in terms of quality and type of non-metallic inclusions as well as grain size of the primary structure have been carried out.

In the first stages of the research three melts in an open inductive furnace were carried out, melts were marked in the following part of work 1_x, 2_x, 3_x (where x represents: A – sample taken just after charge had been melted, B – sample taken after metallurgical treatments, 1, 2, 3 – mark of test castings modified with various additives). Within each melt three test castings have been cast, each portion of cast steel for the individual casting was modified with various additives. The main elements of the modifiers are marked in Table 1. The chemical composition of cast steel was investigated with LECO GDS 500A. In Table 1 chemical composition, in weight percent, of cast steel on the each stage of melt are presented.

The analogous method of evaluating the primary structure refinement as in [14] was applied. The specimens were etched by Oberhofer's reagent in order to disclose the macrostructure. The macrostructures of the examined cast steels are shown in Figures 1 – 4; the images were made with using a common optical scanner. The largest effect on the primary structure

TABLE 1

Chemical composition of investigated cast steel on each characteristic melt stage

No	C [wt.%]	Si [wt.%]	Mn [wt.%]	P [wt.%]	S [wt.%]	Cr [wt.%]	Mo [wt.%]	Ni [wt.%]	Al [wt.%]	Co [wt.%]	Ti [wt.%]	Zr [wt.%]	Nb [wt.%]	V [wt.%]	O [ppm]	N [ppm]
1A	0,16	0,08	0,28	0,021	0,024	0,84	0,71	1,26	0	0,026	0,008	0,015	0,06	0,054	-	-
1B	0,28	0,93	1,03	0,024	0,022	0,82	0,7	1,28	0,009	0,037	0,026	0,019	0,06	0,05	54	188
1.1	0,34	0,835	1,03	0,022	0,017	0,83	0,79	1,24	0,036	0,025	0,008	0,008	0,038	0,05	105	210
1.2	0,27	0,8	1,1	0,02	0,019	0,83	0,77	1,22	0,009	0,022	0,006	0,007	0,043	0,247	33	238
1.3	0,246	0,77	1,02	0,018	0,016	0,8	0,75	1,2	0,004	0,029	0,009	0,01	0,191	0,093	43	277
2A	0,15	0,18	0,58	0,016	0,022	0,97	0,77	1,22	0	0,024	0,005	0,008	0,063	0,088	-	-
2B	0,31	0,55	0,95	0,016	0,021	0,95	0,74	1,21	0,004	0,031	0,007	0,011	0,071	0,086	0	240
2.1	0,32	0,72	0,97	0,016	0,024	0,95	0,78	1,23	0,063	0,028	0,007	0,035	0,073	0,088	44	243
2.2	0,31	0,6	0,96	0,014	0,014	0,94	0,78	1,22	0,037	0,027	0,222	0,008	0,07	0,088	46	245
2.3	0,3	0,69	0,97	0,014	0,022	0,94	0,76	1,22	0,019	0,025	0,022	0,009	0,06	0,087	-	-
3A	0,11	0,14	0,13	0,013	0,025	0,96	0,81	1,15	0	0,021	0,003	0,004	0	0,02	-	-
3B	0,227	0,767	1,02	0,016	0,022	1,08	0,77	1,12	0,005	0,025	0,007	0,008	0,001	0,023	100	174
3.1	0,24	0,95	1,01	0,015	0,027	1,12	0,78	1,11	0,056	0,028	0,064	0,029	0,002	0,025	50	178
3.2	0,23	0,88	0,99	0,017	0,028	1,17	0,78	1,11	0,019	0,027	0,016	0,011	0,104	0,122	51	211
3.3	0,22	0,86	0,94	0,016	0,026	1,14	0,77	1,12	0,007	0,027	0,01	0,011	0,016	0,038	-	-
1Bz.A	0,13	0,22	0,18	0,013	0,025	0,92	0,81	1,19	0	0,024	0,002	0,005	0	0,02	154	190
1Bz.B	0,26	1,18	1,28	0,015	0,024	0,97	0,79	1,15	0,024	0,03	0,008	0,01	0,006	0,023	49	247
1Bz	0,24	1,44	1,16	0,014	0,029	0,9	0,73	1,14	0,015	0,034	0,067	0,034	0,009	0,023	52	159
2Bz.A	0,14	0,16	0,17	0,016	0,026	0,94	0,74	1,08	0	0,015	0	0,007	0	0,016	175	215
2Bz.B	0,16	1,03	1,28	0,017	0,026	0,91	0,7	1,06	0,032	0,023	0,003	0,011	0,002	0,019	65	245
2Bz	0,23	1,3	1,08	0,017	0,027	0,95	0,69	1,03	0,01	0,028	0,053	0,041	0,008	0,02	68	164
3Bz.A	0,14	0,15	0,17	0,016	0,021	0,92	0,81	1,2	0	0,024	0,001	0,005	0,008	0,02	119	228
3Bz.B	0,34	1,19	1,34	0,018	0,02	0,96	0,75	3,03	0,017	0,03	0,013	0,013	0,009	0,023	91	270
3Bz	0,32	1,52	1,17	0,018	0,024	0,97	0,78	3,02	0,01	0,03	0,064	0,051	0,015	0,025	74	133
4Bz.A	0,13	0,08	0,06	0,016	0,02	0,95	0,82	1,27	0	0,021	0	0,005	0	0,019	201	164
4Bz.B	0,34	1,07	1,2	0,019	0,02	0,92	0,77	1,23	0,016	2,02	0,008	0,015	0,009	0,22	85	187
4Bz	0,33	1,34	1,04	0,019	0,024	0,91	0,76	1,28	0,01	2	0,058	0,053	0,01	0,023	65	121
5Bz.A	0,15	0,08	0,11	0,018	0,026	0,935	0,8	1,22	0	0,04	0,004	0,011	0,017	0,018	230	195
5Bz.B	0,29	0,95	1,39	0,018	0,023	0,92	0,77	1,74	0,034	1,95	0,009	0,019	0,017	0,02	64	201
5Bz	0,35	1,37	1,26	0,022	0,032	0,94	0,83	1,75	0,009	1,9	0,057	0,047	0,026	0,024	60	108

refining was obtained for the simultaneous addition of Ti and Zr. Therefore in the second stage of the investigation only simultaneous addition of ferroalloys of these two elements was used.

In the second stage of the study, five meltings in a vacuum inductive furnace were carried out, almost identically to the meltings of the first stage. The melting was carried out in open vacuum chamber, and only as a last treatment the keeping under reduced pressure to vacuum degassing was used. These melts are marked in the following part of this work as 1Bz_x – 5Bz_x: where x represents: A – sample taken just after charge had been melted; B – sample taken after metallurgical treatments but before vacuum degassing; marks without A or B represents test castings that were cast immediately after vacuum degassing with modification during the tapping. In cast steels 3Bz, 4Bz and 5Bz contents of Ni, Co, and Ni + Co, respectively, were increased.

The next stage of the research was microanalysis of the structure, shape and chemical composition of non-metallic inclusions in the melted cast steels, which was made at the Insti-

tute for Ferrous Metallurgy in Gliwice. Investigations were carried out with Inspect F scanning electron microscope equipped with an Energy Dispersive Spectroscopy (EDS) detector to analyze the chemical composition of microregions. The inclusions were imaged using Back Scattered Electrons (BSE) contrast.

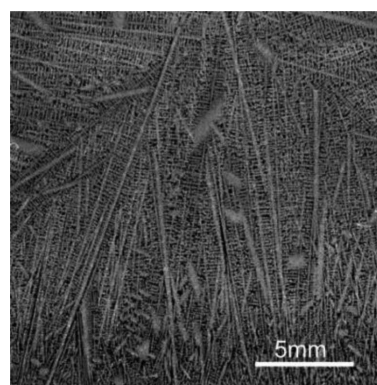


Fig. 1. Macrostructure of cast steel 2.1

3. Results

Macrostructures of the obtained cast steel in the second stage of the investigation are presented in Figures 5-9.

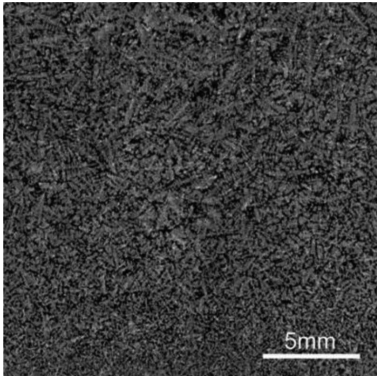


Fig. 2. Macrostructure of cast steel 2.2

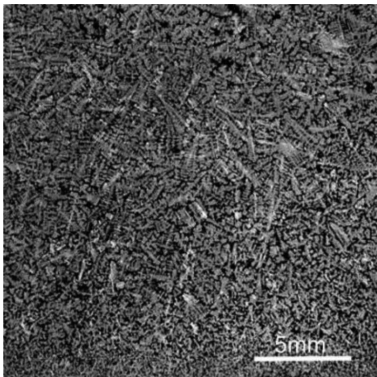


Fig. 3. Macrostructure of cast steel 3.1

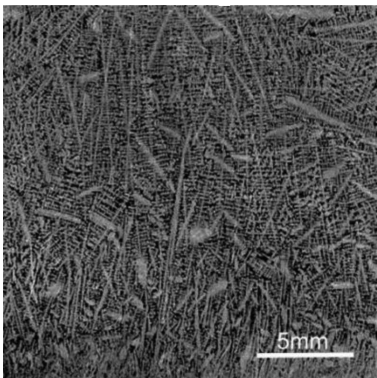


Fig. 4. Macrostructure of cast steel 3.2

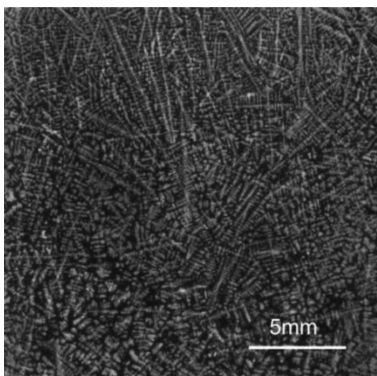


Fig. 5. Macrostructure of cast steel 1Bz

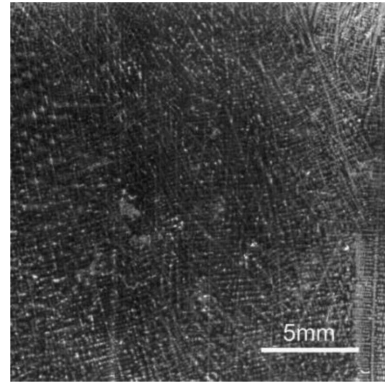


Fig. 6. Macrostructure of cast steel 2Bz

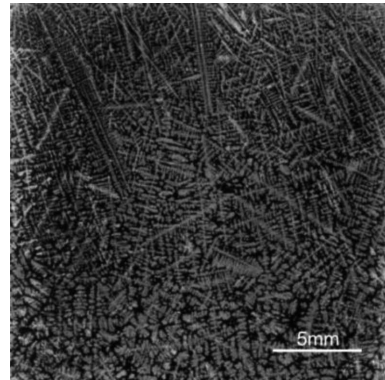


Fig. 7. Macrostructure of cast steel 3Bz

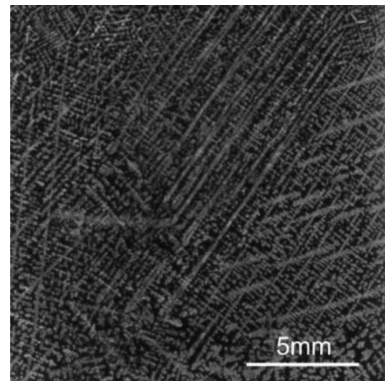


Fig. 8. Macrostructure of cast steel 4Bz

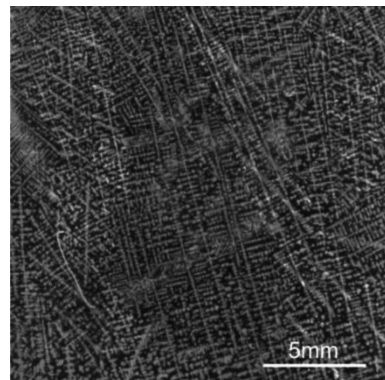


Fig. 9. Macrostructure of cast steel 5Bz

The results of the structures microanalysis, given as images of inclusions and their chemical composition are shown in Figures 10-18.

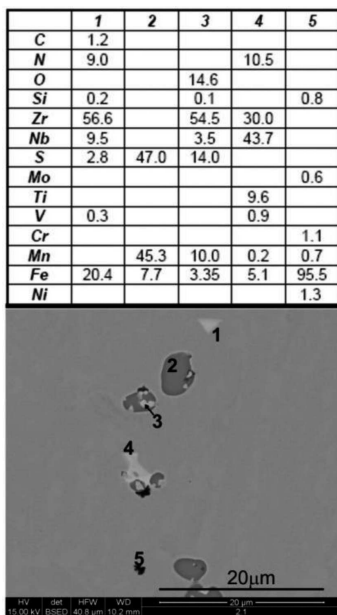


Fig. 10. The shape and chemical composition (wt.%) of inclusions – cast steel 2_1

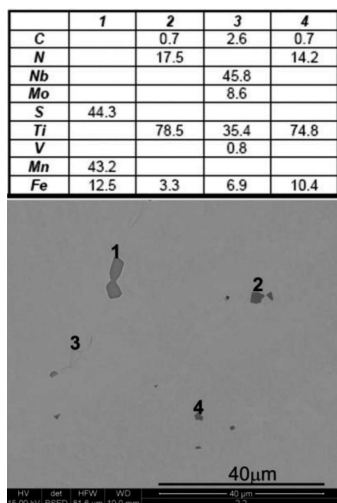


Fig. 11. The shape and chemical composition (wt.%) of inclusions – cast steel 2_2

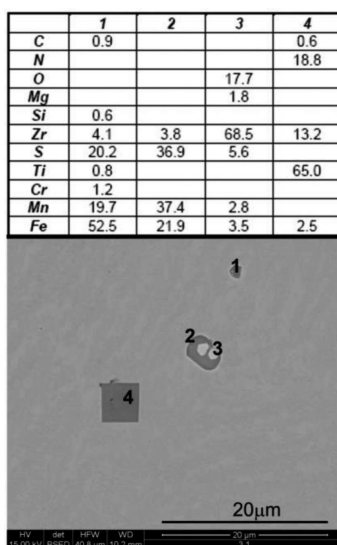


Fig. 12. The shape and chemical composition (wt.%) of inclusions – cast steel 3_1

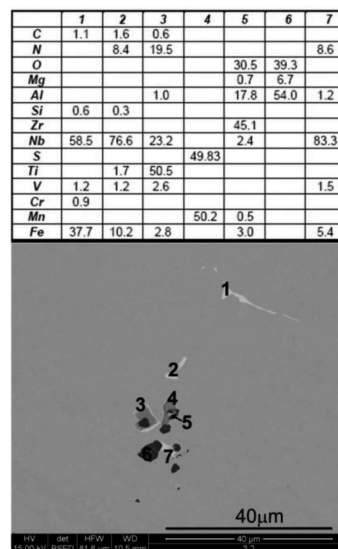


Fig. 13. The shape and chemical composition (wt.%) of inclusions – cast steel 3_2

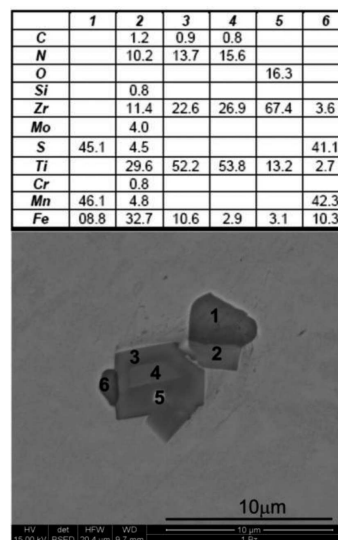


Fig. 14. The shape and chemical composition (wt.%) of inclusions – cast steel 1Bz

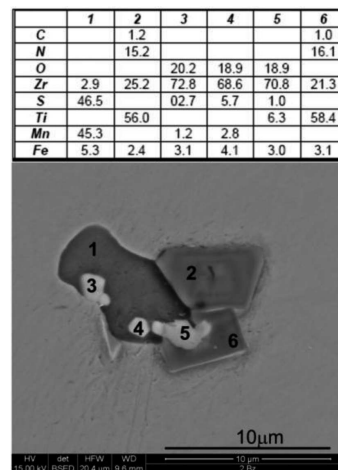


Fig. 15. The shape and chemical composition (wt.%) of inclusions – cast steel 2Bz

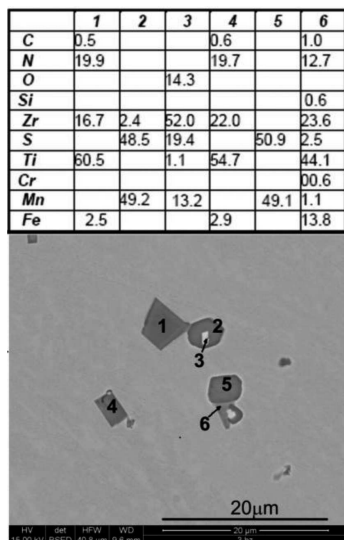


Fig. 16. The shape and chemical composition (wt.%) of inclusions – cast steel 3Bz

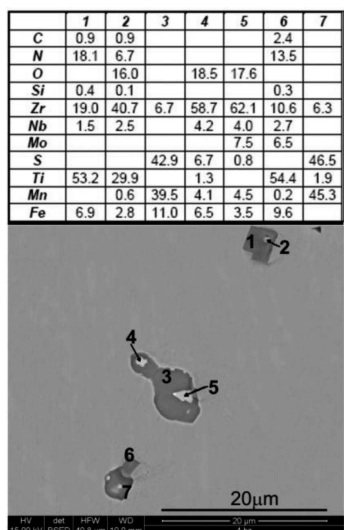


Fig. 17. The shape and chemical composition (wt.%) of inclusions – cast steel 4Bz

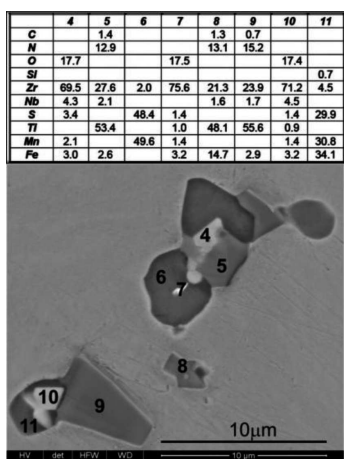


Fig. 18. The shape and chemical composition (wt.%) of inclusions – cast steel 5Bz

4. Discussion

Comparing cast steel macrostructures of the first and second stage of investigation it can be seen that no casting of the second stage has macrostructure refined as the cast steels 2.2 or 3.1 (Figures 2 and 3), though the same modifiers were used. The samples 1Bz, 2Bz and 3Bz are comparable to the steel cast modified with Nb and V (cast steel 3.2) taking into account their primary structure refining (Figures 5, 6 and 7). The macrostructure of cast steels with higher contents of Co (cast steels 4Bz and 5Bz; Figures 8 and 9) indicates lack of the modifying effect of Ti and Zr caused by Co addition. The degree of the primary structure refining after modifier addition could be explained by the appearance in significant amount of high melting fine inclusions of carbides, oxides, nitrides etc., generally non-metallic inclusions that disturb the crystals growth. In vacuum degassed cast steels the contents of N is lower (Table 1) and therefore the degree of the primary structure refining of vacuum degassed cast steels is noticeable.

In Figures 10-18 there are presented representative non – metallic inclusions found in the samples of melted cast steel. These inclusions are formed in the melt from the components intentionally introduced (i.e. modifiers) and those already existing in it (dissolved gases, components of the alloy). The clusters (conglomerate) of different inclusions are the common picture of the non – metallic inclusions. During the analysis of the structure of conglomerates of non – metallic inclusions one regularity is observed, which occurs even in cast steel with little amount of Zr and Ti (without intentional addition). Namely, the compounds of zirconium, most likely the oxide ZrO_2 of the ratio Zr to O about 2:7 (e.g. Figure 18: inclusions 10, 7 and 4, Figure 17: inclusions 5, 4 and 2), always occurs in the center of the inclusions. The rest of compounds, first of all sulfides (mainly MnS) and, nitrides (TiN) and oxides crystallize on them. The melt 2.2 (Figure 11) is an exception where percentage of zirconium is 0,008% and there are no characteristic clusters of non – metallic inclusions. In this cast steel the sulfides, nitrides and oxides clearly appear in some distance one from another and the inclusions containing titanium (it is said on the base of chemical constitution, Figure 11: inclusions 2 and 4) is the nitride of titanium (TiN). Even uniform inclusions, with similar shape and color as nitride of titanium, contain significant amount of zirconium in the cast steel with the addition of Zr (e.g. Figure 16, inclusions 1 and 4; Figure 14, inclusions 3 and 4). It would indicate complex compound of N, Ti, Zr and C. The occurrence of characteristic clusters of non – metallic inclusions in cast steel with the addition of zirconium could explain the fact, that the addition of FeZr + FeTi has bigger influence on the structure refinement than has only the addition of FeTi.

The relatively big clusters of non – metallic inclusions in liquid metal (the crystallization temperatures of all compounds of the clusters are higher than liquidus temperature of cast steel) cannot become the crystal nucleus. However, they effectively “block” the growth of dendrites by refining primary structure of cast steel due to their size and interaction. The influence of zirconium on forming clusters of non – metallic inclusions would indicate two – stage mechanism of the modification. The compound ZrO_2 (melting temperature $2700^\circ C$) appears at the beginning and it works as the nucle-

ation substrate of crystallization for the rest of inclusions, first of all, sulfides and nitrides. The inclusions of titanium oxides were not observed in the structure of examined alloys with even 74 ppm content of oxygen.

5. Conclusions

From the results of the investigations of low-alloy cast steel with different microadditions the following conclusions can be drawn:

- the biggest refining effect of primary structure of low-alloy cast steel was observed for the simultaneous additions of Ti (Fig. 2) and Ti+Zr (Fig. 3). The clusters of inclusions formed under the influence of zirconium refine the structure stronger than single inclusions,
- even little amount of zirconium causes the appearance of the clusters of sulfides, nitrides and oxides inclusions in cast steel; it could indicate the nuclei creation action of ZrO_2 for the rest of inclusions,
- vacuum degassing caused the decrease of the nitrogen content even by 50% but did not cause decrease of the oxygen content. Probably oxygen did not occur in its free form after earlier performed diffusion deoxidizing and the falling one, with the use of Al,
- the modifying additions used in the examination were not effective for cast steel with higher content of cobalt. It indicates a no nuclei creation mechanism of the primary structure refining, but rather the change of conditions of grain growth process caused by conglomerates of non – metallic inclusions. The influence of modifiers was compensated by cobalt action.

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