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EVALUATION OF THE CHEMICAL COMPOSITION AND MICROCLEANLINESS OF THE STEEL SAMPLES FROM THE HEAVY FORGING INGOT

OCENA SKŁADU CHEMICZNEGO I MIKROCZYSTOŚCI PRÓBEK STALOWYCH Z PRÓBEK STALOWYCH Z CIĘŻKICH WLEWKÓW PRZEZNACZONYCH DO KUCIA

The paper presents new results obtained from the evaluation of the chemical composition, microcleanliness and structure of the 90-ton heavy ingot cast in two successive heats, in which the content of Cu and Ni was intentionally modified in order to assess the degree of mutual mixing of the two heats in the ingot volume during the steel casting and solidification. For determination of chemical composition, spectral analysis and LECO were used. Microcleanliness evaluation was carried out on a Hitachi microanalytical complex equipped with the energy-dispersive spectrometer Vantage. To assess the composition of oxide non-metallic inclusions ternary diagrams were used. Structure of the basic steel matrix was induced by etching. The evaluation showed that in the casting of two successive heats, a certain degree of inhomogeneity of chemical composition, especially in the lower part of the ingot can be assumed in case of different composition. A greater segregation of sulphur in the central top part of the ingot was also detected. However, microcleanliness of the entire ingot is in general very good with low proportions of non-metallic inclusions.

Keywords: heavy steel ingot, microcleanliness, inclusion, microstructure, macrostructure

W pracy tej przedstawiono, wyniki uzyskane z oceny składu chemicznego, czystości w skali mikro i struktury 90 tonowych wlewków odlanych w dwóch kolejnych sekwencjach, w których zawartości Cu i Ni celowo zmieniono, dla oceny stopnia wzajemnego mieszania się dwóch wytopów w objętości wlewka podczas odlewania i krzepnięcia stali. Dla określenia składu chemicznego zastosowano analizę spektralną i LECO. Badania mikroczystości przeprowadzono na zespole urządzeń do mikroanalizy Hitachi wyposażonym w spektrometr energii rozproszonej Vantage. Dla oceny składu tlenkowych wtrąceń niemetalicznych użyto trójskładnikowych układów równowagi. Struktura podstawowej osnowy stali została ujawniona za pomocą trawienia. Ocena wykazała, iż przy odlewaniu dwóch kolejnych odlewów, można zakładać pewien stopień niejednorodności składu chemicznego, zwłaszcza w dolnej części wlewka, zwłaszcza przy różnych składach chemicznych. Stwierdzono również większą segregację siarki w centralnej części wlewka. Jednakże, mikroczystość całego wlewka jest na ogół bardzo dobra, przy niskich proporcjach wtrąceń niemetalicznych.

1. Introduction

Despite the ever-increasing volume of continuous steel casting, production of steel ingots for forgings and machine components is irreplaceable. Steel casting into the ingots even allows for the production of oversized components weighing up to several hundred tons. The main precondition of the competitiveness of any steel plant is production of a consistently high quality. However, despite significant advances in the technology of production of steel ingots, we can observe the defects in the final forgings that may be caused due to the non-uniform cast microstructure of an ingot as well as the macrostructure [1], which is the result of plastic deformation during the subsequent process of the forming. The solution to material weaknesses of forgings, or the final machine components, consists from a complex optimization of the steel casting process as well as of subsequent heat treatment up to the actual process of forming.

One of the ways to monitor and optimize the production steps from the casting to the forming process is the use of methods of experimental casting together with experimental evaluation of microstructure and numerical modelling [2-5]. As it is evidenced by the results of the model and experimental studies performed e.g. by the authors [6-9], the size of the central defect is strongly dependent on the boundary conditions of the casting and on the geometry of the mould. Attention has been also paid not only to optimization of the boundary conditions of casting, but also to the verification of the causes of possible secondary contamination of steel during casting due to erosion of the pouring ceramics [10, 11], to secondary

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re-oxidation or entrainment of casting powder by dynamics of the inlet casting flow at the early stages of casting [12].

The presented paper devoted to the evaluation of the chemical composition and microcleanliness of the steel samples from the heavy forging ingot produced in VÍTKOVICE HEAVY MACHINERY a.s. (hereafter as VHM). VHM is a traditional producer of large machinery components. The typical products of the company include crankshafts, propeller and connecting shafts, rotor shafts for wind power plants, forged parts for the container of pressurizers, steam generators, heat exchangers and collectors for both conventional and nuclear power engineering. For these products it is necessary to cast ingots weighting up to 200 tons. The steel plant of VHM is equipped with EAF, LF, VD and VOD facilities. Ingots from 1.7 up to 200 tons are bottom-casted. Typically, steel grades for these products are structural carbon-manganese, low alloyed, middle alloyed and tool steels. The EAF capacity is 70 tons, so the larger ingots are accumulated from two or three heats. To seek the solutions to problems of ingot casting and solidification, VHM cooperates with Technical University of Ostrava. The project MPO FR-TI 3/243 "Experimental development and optimization of production technology of heavy forging ingots to improve utility properties of special engineering components with higher added value" has included a complex experiment, consisting of a 90-ton ingot cast from two steel heats, in which the content of Cu and Ni was modified in order to assess the degree of mutual mixing of the two melts in the volume of ingot during casting and solidification. The cast ingot was then cut up along the height, then in five height cross-sections.

Thusly obtained sections were used inter alia to assess the overall steel microcleanliness along the height and cross-section of the cast ingot. The sampling was carried out on the sections 1, 3, and 5 as shown in Fig. 1.

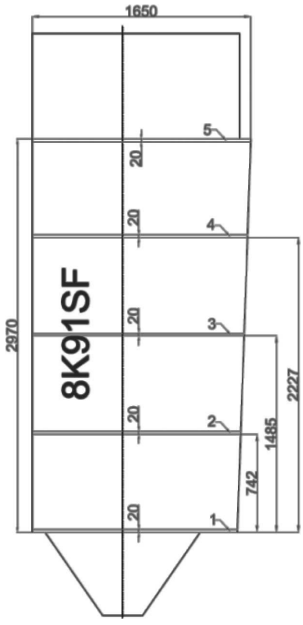

Fig. 1. Position of sampling for evaluation of microcleanliness of the cast ingot

2. Evaluation of chemical composition of steel samples

Spectral analysis was applied to determine the chemical composition of the samples, in case of C and S a combustion method on the LECO device was used. The chemical composition of both heats including the weighted average is shown in Table 1.

Table 1 clearly shows that in case of Cu and Ni, a targeted change of their content in individual heats from 0.13 to 0.46%, or from 0.506 to 0.118% has occurred. These contents allowed a more accurate identification of the areas that correspond to the composition of the two heats, or their resulting composition after mixing them together. The content of carbon and other elements were targeted at the same value in both heats.

TABLE 1
Chemical composition of two successive heats and their weighted average in the casting of one 90-ton steel ingot

Component	Heat No.1	Heat No.2	Weighted average
C	0.194	0.200	0.196
Mn	1.30	1.27	1.29
Si	0.26	0.27	0.26
P	0.008	0.009	0.008
S	0.0008	0.0010	0.0009
Cu	**0.13**	**0.46**	0.26
Ni	**0.506**	**0.118**	0.353
Cr	0.11	0.14	0.12

2.1. Discussion of results

The results of determination of the chemical composition of samples from all three evaluated sections are shown in Fig. 2. The samples 1 through 15 correspond from the left to the right to the edge and centre of the ingot. In the evaluation of the chemical composition results in terms of the height in individual sections of the ingot, it should be noted that changes in the composition may be caused not only by mixing two heats of different chemical composition, but also due to the segregation processes, especially in the elements with a low distribution coefficient, such as S and C. On the other hand, it may be also assumed that, given the value of the distribution coefficients of Cu and Ni, their concentration in the ingot regarded mainly may be considered a result of mutual mixing of both heats without significantly affecting segregation processes. The analysis of the graphs in Fig. 2 has resulted in the following findings:

- The content of Ni and Cu in subsurface samples No. 1 through 5 from the sections 1 and 3 (1_1 through 1_5, or 3_1 through 3_5) corresponds to the first heat. In the case of section 5 the subsurface layer (5_1 through 5_5) has already been influenced by the second heat, the Ni content gradually decreases and Cu content increases toward the centre of the ingot (in the samples 1-5).
- In samples 6-10 from the mid-distance between the surface and the centre of the ingot a significant increase in the

content of Cu and decrease in the content of Ni in section 1 is visible, practically from the composition of the first heat through the final, average composition of the two heats. Samples 6-10 of the section 3 and 5 correspond to the final, average composition of both heats.

• Samples 11-15 from the central part of the ingot correspond to a final average composition of the two heats in all three sections.

From the above it is evident that in the ingot casting from the two heats, the subsurface layers of the ingot are significantly affected by the composition of the first heat, and it is so even in section 5 (2.970 mm from the bottom of the ingot). The most significant changes in the chemical composition have been detected in the mid-distance between the surface and the centre of the ingot. The above behaviour is probably related to the nature of the ingot filling by a central bottom nozzle.

Regarding the behaviour of sulphur and carbon in the individual sections of the ingot, the following findings can be defined:

• A more significant macro-segregation of carbon in the section 3 can be observed, where the content to 0.287 wt.% was detected in its central part (which is 0.087 wt.% higher than in the composition of the second heat). In section 1 no macro-segregations of carbon have been observed.

• Also, a more significant segregation of sulphur can be observed in section 3, particularly in the samples from the centre of the ingot, where the content of sulphur up to 0.0015 (which is 0.0005% more than in the composition of the second heat) was detected.

3. Evaluation of microcleanliness of collected samples

The microcleanliness evaluation was carried out on a Hitachi microanalytical complex equipped with an energy dispersive spectrometer Vantage in TŽ, a.s. Ninety visual fields at a magnification of 500x were evaluated on each sample, which represents a summary evaluation area 5.625 mm^2. A total number of inclusions, their density, division to distribution groups, and areal proportion of inclusions (summary area of inclusions / total area of evaluated sample*100) were evaluated. Due to the possibility to also generally evaluate the chemical composition of the inclusions, classification of oxide inclusions into the ternary diagrams $CaO+MgO+MnO-Al_2O_3-SiO_2$ was performed. The oxide inclusions were also in the given ternary diagram categorized into areas A, B and C, where A is the area limited by the Al_2O_3 content of up to 30%, area B then includes inclusions containing 30-70% Al_2O_3, and area C the inclusions with Al_2O_3 content exceeding 70%. This classification is consistent with the microcleanliness assessment of carbon steels according to the Pirelli norm using the HW method. As for the sulphide inclusions due to their complex composition the ternary diagrams $CaO+MgO+MnO+Al_2O_3-SiO_2-MnS$ were used.

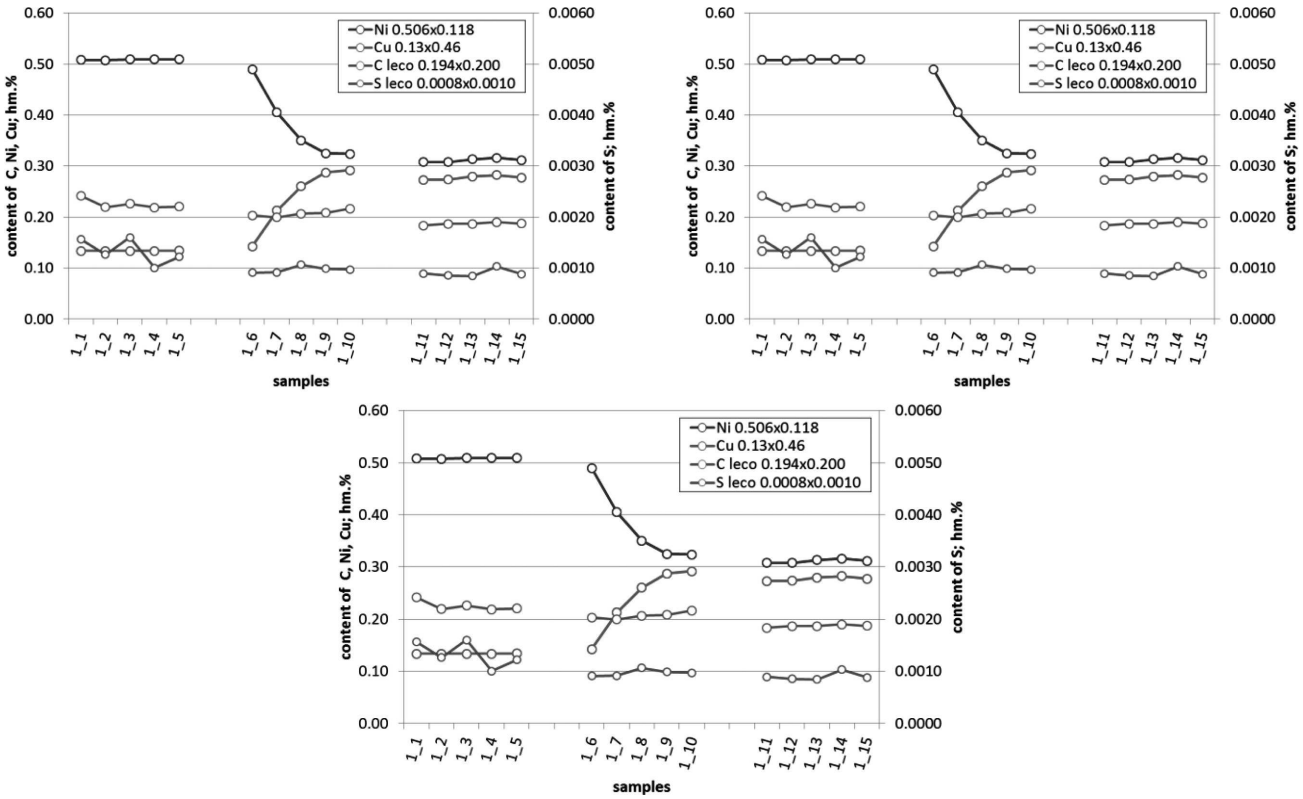

Fig. 2. Chemical composition of the samples taken from cross-sections 1, 3, 5 (see Fig. 1)

3.1. Discussion of results

To start this evaluation it is necessary to state that almost all the samples showed very good microcleanliness, measured areal proportions of oxide inclusions rarely exceeded a value of 0.015%, sulphide ones then 0.010%, especially in the central regions of the ingot. It shows, among other things a mastery of the production technology and casting of the steel as well as favourable conditions for the separation of inclusions during the steel solidification. It is therefore difficult to find a stronger connection between microcleanliness and, for example chemical composition of the steel, or other parameters.

Overall comparison of average densities and areal proportions of the inclusions (oxide and sulphide), always from the five samples (i.e., 1-5, 6-10 and 11-15) from the surface to the centre of the ingot, and along the height of the ingot (in the sections 1, 3 and 5) is shown in Fig. 3.

Based on the analysis of the graphs in Fig. 3 the findings can be defined as follows:

- Density of oxide inclusions is the highest in subsurface areas and towards the centre of the ingot decreases.
- The highest densities of oxide inclusions were detected in the subsurface layers of the bottom part of the ingot (section 1, samples 1 through 5).
- The lowest densities were detected in the section 3, both in subsurface samples and in other samples towards the centre of the ingot.
- Density of sulphide inclusions decreases towards the centre of the ingot too; however these changes are not as visible as in the oxide inclusions.
- Areal proportion ”does not copy” the tendencies of the inclusion density. This is due to different distribution of inclusions in different size groups. Larger inclusions (above 10μm) were captured in the central part of the ingot, and their area has become more visible in the percentage areal proportion of the inclusions.
- The highest areal proportions of oxides were detected in the central part of the ingot in the bottom section (samples 11 through 15, section 1) – up to 0.01%, which could be evidently related to the presence of a sedimentation cone, in which are normally found oxide inclusions torn by descending metal crystals.
- The areal proportions of the inclusions are reduced towards the top of the ingot; these proportions are even along the cross-section, not exceeding the value of 0.007%, which is very low.
- Areal proportion of sulphide inclusions was very low in almost all samples. It did not exceed a value of 0.0042%. The only exception was the samples from section 5 from the central part of the ingot, in which the value was determined to be 0.0114%.
- The noted increased incidence was associated with a higher segregation of sulphur in this area and therefore with more favourable conditions for the formation of larger sulphide inclusions.

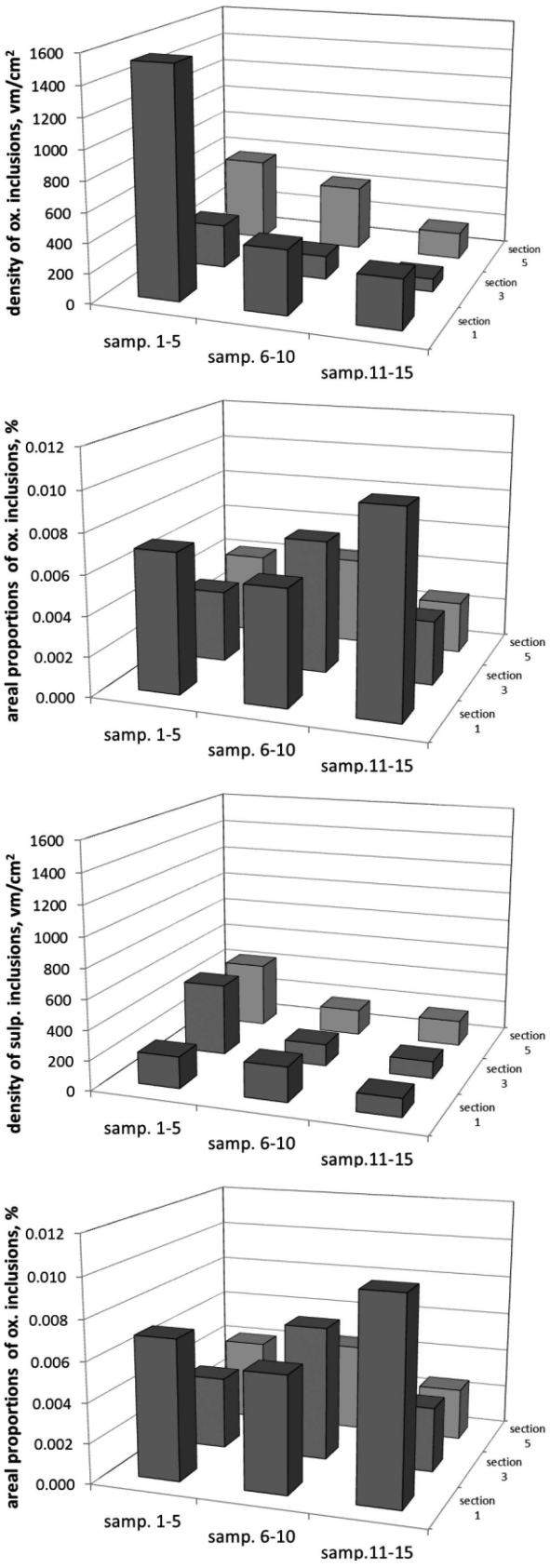

Fig. 3. Comparison of average densities and areal proportions of the oxide and sulphide inclusions in samples from the surface to the centre of the ingot and along the ingot‘s height

4. Composition and morphology of non-metallic inclusions

To assess the composition of oxide non-metallic inclusions, the above-mentioned ternary diagrams CaO + MgO + MnO − Al_2O_3−SiO_2 were used. As for the sulphide inclusions, the ternary diagrams CaO + MgO + MnO + Al_2O_3 − SiO_2 − MnS were used due to their complex structure. Fig. 4 shows an example of a ternary diagram of the composition of oxide inclusions in the "central" sample 13 in section 3. Fig. 5 shows an example of ternary diagram of the composition of sulphide inclusions in the "central" sample 13 in the section 3.

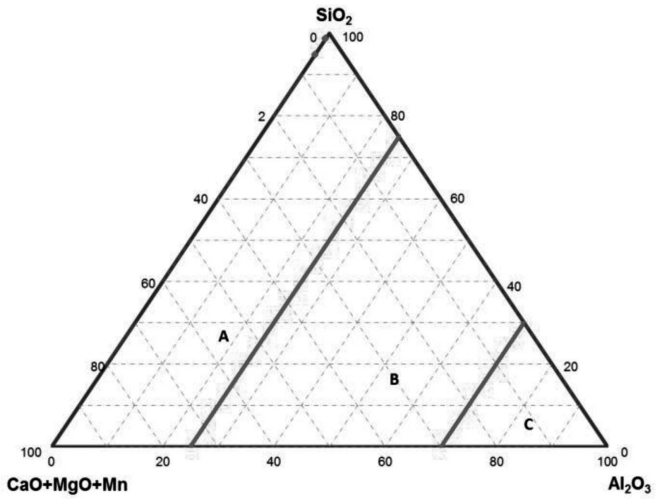

Fig. 4. Ternary diagram of the composition of oxide inclusions in the "central" sample 13 of the section 3

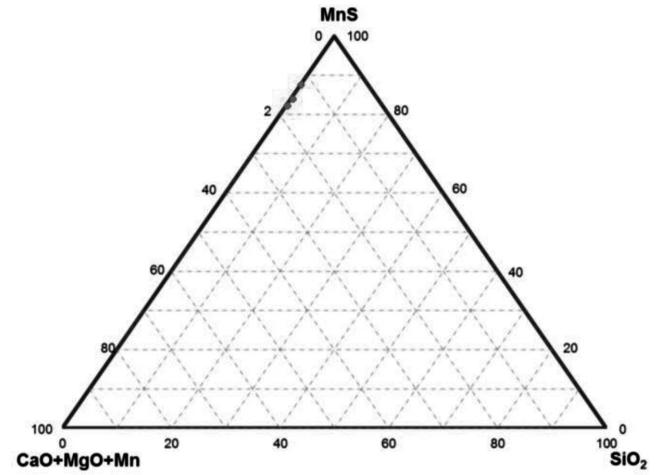

Fig. 5. Ternary diagram of the composition of sulphide inclusions in the "central" sample 13 of the section 3

4.1. Discussion of results

Based on the analyses of all ternary diagrams the obtained findings can be summarized as follows:

- The samples contain complex oxide inclusions based on oxides of Ca, Mg and Al (Fig. 6).
- Heterogeneous inclusions with a higher proportion of CaO were detected in the top part of the ingot (Fig. 7).
- The diagrams show that there are no significant differences between the compositions of sulphide inclusions. They were mostly Mn-Ca-S-based sulphides that excreted on the oxide particles based on Al, Mg and Ca – see Fig. 8, 9.

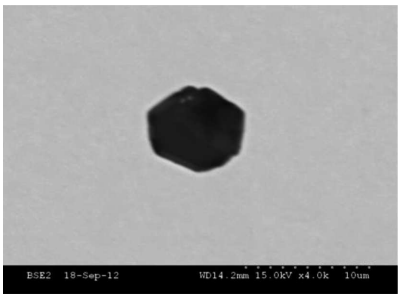

Fig. 6. Homogeneous oxide inclusions in the sample 1_5; Composition: 7% Mg, 30% Al, 12% Ca, 45% O

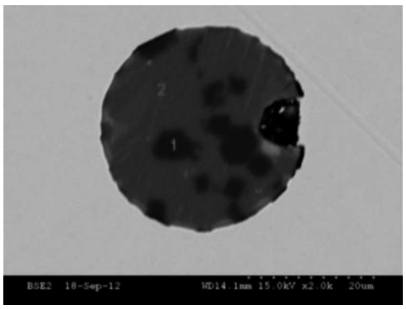

Fig. 7. Heterogeneous oxide inclusions in the sample 3_14 Composition1: 14%Mg, 40%Al, 35%Mn, 43%O; Composition2: 2% Mg, 23% Al, 36% Ca, 36% O

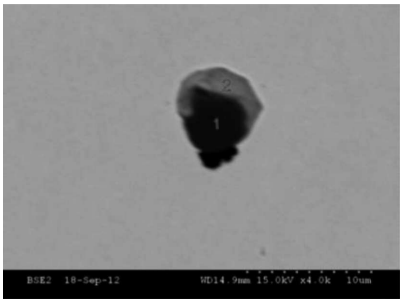

Fig. 8. Complex heterogeneous oxide-sulphide inclusion in the sample 5_6: Composition1: 12% Mg, 41% Al, 44%; Composition2: 36% Mn, 28% S, 21% Ca, 7% O

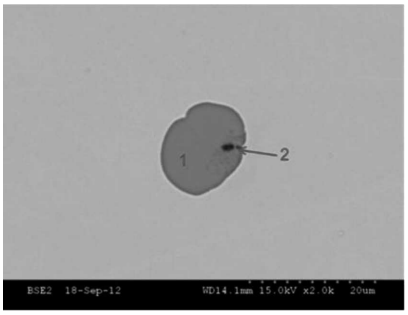

Fig. 9. Complex heterogeneous oxide-sulphide inclusion in the sample 3_14: Composition1: 57%Mn, 4%Mg, 31%S; Composition2: 35%Mn, 8%Mg, 21%S, 19%O

5. Structure of the basic steel matrix

To assess structure of the basic steel matrix, the selected samples were etched by the NITAL chemical agent (2% HNO_3 and hydrogen peroxide solution). The images are oriented in such a way that the left vertical edge is parallel to the vertical wall of the mould. It is in fact the vertical, an approx. 2 cm high cut.

The image of sample 1_2 in Fig. 10 shows dendritic structure corresponding to the edge of the ingot. Individual dendrites are mostly oriented perpendicular to the wall (dark stripe on the right edge of the image), and their size is up to 2 mm.

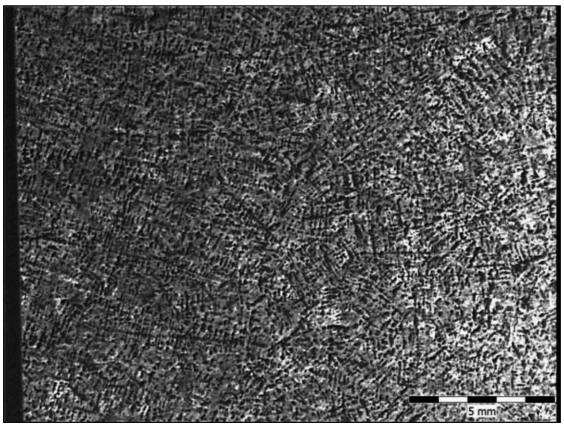

Fig. 10. The dendritic structure of edge of the ingot sample 1-2

The structure of the sample 1_12 Fig. 11 corresponds to the centre of the ingot. The grains are already oriented irregularly; it is also easier to see their boundaries.

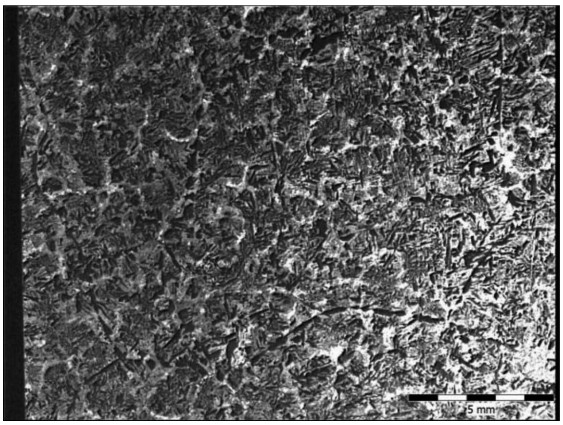

Fig. 11. Macrostructure in the centre of the ingot

A similar structure is shown in Fig. 12 and Fig. 13 on the samples 3_2 and 3_12, where there is a more pronounced dendritic structure with a dendrite size up to 15-20 mm. The grains in the centre of the ingot are undirected again.

The described general evaluation of the structures will serve as a basis for optimal sampling for the evaluation of micro- and macro-segregations in the body of the ingots.

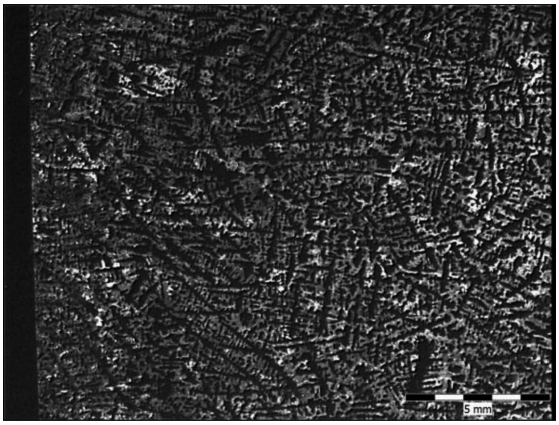

Fig. 12. Macrostructure of the sample 3_2

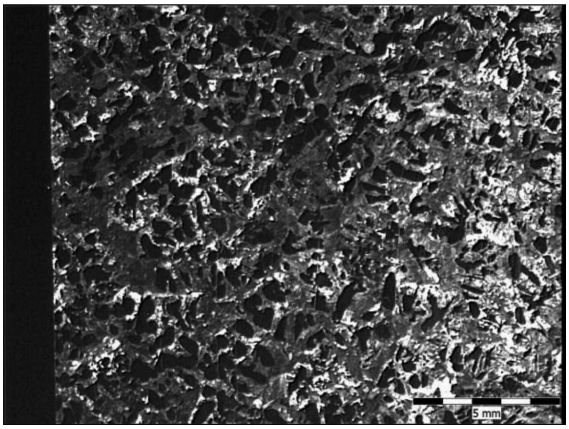

Fig. 13. Macrostructure of the sample 3_12

6. Conclusion

Evaluation of the chemical composition, microcleanliness, and structure of the 90-ton ingot cast from two heats, which had an intentionally modified Cu and Ni content was carried out in order to assess the degree of mutual mixing of both heats in the ingot volume during its casting and solidification.

The evaluation showed that during casting of the ingot from two successive heats, in case of their different composition, a certain degree of inhomogeneity of chemical composition can be assumed, which will especially show in a lower half of the ingot where there is no mutual mixing of the two heats. Also, a greater segregation of sulphur in the central top part of the ingot was recorded.

From the standpoint of achieved microcleanliness, some differences along the cross-section of the ingot and increased incidence of sulphides in a top central part can also be reported. However, in general, microcleanliness of the entire ingot is very good with low areal proportions of the non-metallic inclusions.

Acknowledgements

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