EVALUATION OF CHEMICAL HETEROGENEITY OF A 90-TON FORGING INGOT

Pavel MACHOVČÁK\textsuperscript{a}, Aleš OPLER\textsuperscript{b}, Zdeněk CARBOL\textsuperscript{a}, Antonín TREFIL\textsuperscript{b}, Karel MERTA\textsuperscript{b}, Jakub Zaoral\textsuperscript{b}, Markéta TKADLEČKOVÁ\textsuperscript{c}, Karel MICHALEK\textsuperscript{c}

\textsuperscript{a} VÍTKOVICE HEAVY MACHINERY a.s., Ruská 2887/101, 706 02 Ostrava – Vitkovice, Czech Republic, pavel.machovcak@vitkovice.cz

\textsuperscript{b} VÍTKOVICE TESTING CENTER s.r.o., Pohraniční 584/142, 709 00 Ostrava – Hulváky, Czech Republic, karel.merta@vitkovice.cz

\textsuperscript{c} VŠB – Technical University of Ostrava, Faculty of Metallurgy and Materials Engineering, Department of Metallurgy and Foundry, 17. listopadu 15, 708 33 Ostrava – Poruba, Czech Republic, marketa.tkadleckova@vsb.cz

Abstract

Heavy forging ingots are used in metallurgy engineering e.g. for the production crankshafts for marine engines, production of special parts for conventional, hydro and power engineering, rotor shafts, steam generators, heat exchangers and collectors for both conventional and nuclear power engineering. A production of heavy forging ingots is usually accompanied by a segregation of elements in the structure of steel. The segregation of the elements causes anisotropy of mechanical properties. But these mechanical parts must meet the strictest criteria, and therefore must be practically free of defects. In order to achieve the best possible quality of heavy forging ingots, the project focused on improving the useful properties of special machine parts in VÍTKOVICE HEAVY MACHINERY a.s. together in cooperation VHM and FMMI VŠB. An integral part of the project is numerical simulations of casting and solidification. The ingot was cut and macrostructure and chemical heterogeneity of the ingot was evaluated in detail. This article describes performance of the experimental ingot casting, the way of cutting, the methodology of chemical analysis and the results of that investigation. The gained knowledge is also used to specification of the setting of boundary conditions of the numerical simulations, which should help to optimize the production technology of casting heavy forging ingots and minimize the range of segregation in ingots.

Key words: heavy steel ingot, macro-segregation, chemical heterogeneity

1. INTRODUCTION

VÍTKOVICE HEAVY MACHINERY a.s. is traditional producer of large engineering components with a strong position in selected segments of machinery production. Heavy forging ingots weighing up to 200 tons are used for this production. These are mainly carbon, low and medium-alloy structural steel as well as tool steel. Due the fact that production of these heavy forging ingots is usually accompanied by a segregation of several elements it was decided to initiate work focused on reducing of the occurrence of these undesirable segregations. 90-ton forging ingot was cast and cut for the determination the current state of distribution of segregation of individual elements in the cast ingot. This project has been solved mainly in cooperation VHM and FMMI VŠB. An integral part of the project is numerical simulations of casting and solidification of the ingots. [1 - 3]
2. DESCRIPTION OF PRODUCTION AND CUTTING OF THE EXPERIMENTAL INGOT 8K91SF

An experimental ingot 8K91SF was cast at steel plant VHM a.s. so that the extent of the chemical heterogeneity can be determined. It is octagonal forging steel ingot weighing 87.5 tons. The whole production process was completely monitored. The steel for this production was melted at EAF followed by processing at LF and VD. The EAF capacity is approximately 70 tons so the experimental ingot was cumulated from two heats. Structural carbon manganese steel (S355J2G3 according to EN 10 250) was chosen for this trial. Each of these two heats had intentionally different content of copper and nickel in order to determine mixing of these two heats in solidified ingot. Content of the other elements was target at the same level. Chemical analyzes of both heats and the weighted average of the both heats are shown in **Table 1**. The course of ingot casting and solidification was monitored using thermal camera. Ingot was removed from the mold in hot conditions as usual. Stress relieving annealing was carried out due to the elimination of occurrence of possible cracks during cutting.

**Table 1** Chemical analysis of both used heats and the weighted average of these both heats

<table>
<thead>
<tr>
<th>Heat No.</th>
<th>weight (tons)</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Cu</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>52522</td>
<td>53.1</td>
<td>0.194</td>
<td>1.30</td>
<td>0.26</td>
<td>0.008</td>
<td>0.008</td>
<td>0.11</td>
<td>0.13</td>
<td>0.51</td>
</tr>
<tr>
<td>52523</td>
<td>34.5</td>
<td>0.200</td>
<td>1.27</td>
<td>0.27</td>
<td>0.009</td>
<td>0.0010</td>
<td>0.14</td>
<td>0.46</td>
<td>0.12</td>
</tr>
<tr>
<td>w.average</td>
<td>87.6</td>
<td>0.196</td>
<td>1.28</td>
<td>0.26</td>
<td>0.009</td>
<td>0.0009</td>
<td>0.12</td>
<td>0.26</td>
<td>0.36</td>
</tr>
</tbody>
</table>

To obtain a longitudinal cross section of the experimental ingot was at first considered on the technology band-saw cutting to save processing time. Unfortunately band saw can hold a maximum dimension of work piece 1700 mm because the spacing of its columns, which is insufficient for our case because the maximum diameter of ingot is 1935 mm. Milling technology was finally chosen for cutting of the experimental ingot. The conventional horizontal boring machine WD250 was chosen for this procedure. The maximum speed of this machine is 630 min\(^{-1}\). Special roughing milling with replaceable cutting edged was bought due to accelerate receive of large amounts of excess material. Cutting parameters were set with regard to the state of machine and clamping components. The processing time does not exceed 80 hours. The required quality of the machined surfaces was Ra 3,2 µm.

3. EVALUATION OF INGOT HETEROGENEITY

The evaluation of chemical composition, Baumann imprint and penetration test were carried out on the cross section of the experimental ingot in order to find the extent of heterogeneity.

3.1 Macroscopic examination by sulfur prints and fluid penetration testing

Macroscopic examination by sulfur prints (Baumann imprint) is used to detect macroscopic distribution of sulfur in iron alloys. Sulfur in steel is excreted in the form of sulfides FeS and MnS. The principle of the imprint is based on the reaction of sulfides with sulfide acid (1). This reaction is accompanied by the formation of hydrogen sulfide. If it operate at photographic paper surface, silver bromide is decomposed and silver react with sulfur and form very stable compound Ag\(_2\)S which is brown. (2)

\[
\text{FeS} + \text{H}_2\text{SO}_4 = \text{FeSO}_4 + \text{H}_2\text{S} \quad (1)
\]

\[
\text{H}_2\text{S} + 2\text{AgBr} = \text{Ag}_2\text{S} + 2\text{HBr} \quad (2)
\]

Performed Baumann imprint showed isolated significant sulfide segregations length up to 2 mm. These sulfides formed locally clusters or were directed into lines. The stronger lines of sulfides were occurred mainly in the head of ingot and in the quarters of the width, ie. in expected areas of “A” segregations. In the lower third of the ingot height were sulfide segregations very weak and quite uniform.
Liquid penetration testing (further LPI) is a widely applied inspection method used to locate surface-breaking defects in all non-porous material. LPI is based upon capillary action, where low surface tension fluid penetrates into clean and dry surface-breaking discontinuities. Penetrant may be applied to the test component by dipping, spraying or brushing. After adequate penetration time has been allowed, the excess penetrant is removed and developer is applied. The developer helps to draw penetrant out at the flow where an invisible indication becomes visible for the inspection. Inspection is performed under ultraviolet or white light depending upon the type of dye used – fluorescent or nonfluorescent (visible).

The small cavity especially in the upper half of the ingot in the central area and in the area of expected “A” segregations were detected after the performed penetration test. Dimension of identified cavity was relatively small.

3.2 Evaluation of chemical heterogeneity
Chemical analyses were performed on half the area of cut ingot as the ingot can be considered as rotation solid and the second half is the mirror image of the first half. Chemical analysis had two main aims. Firstly, determine the extent of segregation of the individual elements and secondly, mixing of both heats needed for experimental ingot casting. To achieve these aims has been chosen determination of the following elements: carbon, sulfur, manganese, copper, nickel, phosphorous and silicon. Chemical composition was analyzed along the height of the real axis of the ingot and then in the next four straight lines that were parallel to the axis. The distance of these parallel lines were 200, 450, 650 and 850 mm far form the central axis. Two main vertical lines to the ingot axis were chosen; at the interface of the body of the ingot and its head and at the interface of the body of the ingot and its heel. Other seven vertical lines were analyzed between these two main vertical lines. Other three vertical lines were analyzed through the head of the experimental ingot and other three vertical lines were analyzed through the heel of the ingot. Chemical analyses were performed totally at 15 vertical lines. Coordinate system was established due to a clear description of places that were analyzed. This system's starting point is at the intersection of ingot axis and the plane dividing the body and heel of the ingot. Analyzed places are shown in Fig. 1.

The standard method used in metallurgical analytics – analyses using optical emission spectrometers – was not applicable due to the large number of required analyzes. Thus, the mobile optical spectrometer SPECRTOTEST was used for analysis of Mn, Si, Cu a Ni content. Its great advantage was that the analysis could be carried out directly on the ingot without sampling. Due to the low content of sulfur was evident from the very beginning that there is no other alternative than to laboratory analysis of a combustion analyzer, with the lowest possible detection limit and the greatest accuracy. Sample of chips weighting from 0,5 to 2,0 g was need for this method. Thus the surface of ingot cross-section was drilled and chips were analyzed on an automatic analyzer LECO CS-600. Sulfur and carbon were analyzed on this unit. Phosphorus content was also determined on samples taken from the chips. The larger sample weight of chips was needed for this analysis, at least 2 grammes.

![Fig. 1. Analyzed lines throughout the ingot](image-url)
For all the elements of each method was determined experimentally possible accuracy achieved by measuring the control sample under repeatability conditions. Table 2 shows the values of accuracy of the method that were calculated as half of the difference between the upper and lower quartile.

4. DISCUSSION OF RESULTS

A total of 1279 points were analyzed. Copper and nickel were analyzed on the cross-section of the ingot to find mutual mixing of both used heats. These analyses were performed on half of ingot and are shown in Fig. 2 and Fig. 3. These figures shows that there is already layer of solidified steel during the casting of the first heat at the wall of the mould. Layer thickness decreases with the distance from the bottom part of ingot as is evident from the right part of Fig. 2. The copper content is in the contrast with the nickel content near the wall of the mold. However, a very good mix of both heats is achieved in the middle of ingot as show in Fig. 2 and Fig. 3.

Table 2: Average concentration of element in sample and accuracy of each used method

<table>
<thead>
<tr>
<th>Element</th>
<th>Method</th>
<th>Average concentration of element in the control sample (wt %)</th>
<th>Accuracy of method</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>LECO CS-600</td>
<td>0.175</td>
<td>± 0.0015</td>
</tr>
<tr>
<td>C</td>
<td>OES - Spectrometry</td>
<td>0.175</td>
<td>± 0.01</td>
</tr>
<tr>
<td>Mn</td>
<td>Chemical determination</td>
<td>1.15</td>
<td>± 0.003</td>
</tr>
<tr>
<td>Mn</td>
<td>OES - Spectrometry</td>
<td>1.15</td>
<td>± 0.01</td>
</tr>
<tr>
<td>Si</td>
<td>OES - Spectrometry</td>
<td>0.26</td>
<td>± 0.01</td>
</tr>
<tr>
<td>P</td>
<td>Chemical determination</td>
<td>0.0087</td>
<td>± 0.006</td>
</tr>
<tr>
<td>S</td>
<td>LECO CS-600</td>
<td>0.0007</td>
<td>± 0.0001</td>
</tr>
<tr>
<td>Cu</td>
<td>OES - Spectrometry</td>
<td>0.11</td>
<td>± 0.01</td>
</tr>
<tr>
<td>Ni</td>
<td>OES - Spectrometry</td>
<td>0.18</td>
<td>± 0.01</td>
</tr>
</tbody>
</table>

Fig.2 Ni content on the half of ingot cross section

Fig. 3 Cu content on the half of ingot cross section
The second aim of this investigation was to determine the extent of segregation of the individual elements on the ingot cross section. The evaluation was mainly focused on the content of C, Mn, S and P. Figures 4 – 7 shows the courses of content of these elements along the height of the ingot axis. In the case of carbon were found only very minimal negative segregation at the bottom part of ingot. The maximum carbon content in the ingot body, 0.30 %, was found at the interface ingot body – ingot head, while carbon content in the ladle was 0.20 % (see Fig. 4). Manganese content in the ladle analysis was 1.28 %. The lowest content of manganese, 1.18 % was detected at the bottom part of ingot. The highest Mn content in the axis of the ingot body was 1.42 %, extremely high value, 1.62 %, was found in the head of the ingot (see Fig. 5).

Fig. 4 Carbon content in the ingot axis

Fig. 5 Manganese content in the ingot axis

Fig. 6 Sulfur content in the ingot axis

Fig. 7 Phosphorus content in the ingot axis
In case of sulfur and phosphorus was found only positive segregation in the ingot axis. The highest content of sulfur which was found in the axis of the ingot body was 0.0018 % while sulfur content in the ladle was 0.0009 % (see Fig. 6). The highest content of phosphorus was 0.013 % while ladle analysis was 0.009 % (see Fig. 7).

According to expectations, content of elements, mainly such as carbon, manganese, sulfur and phosphorus increased towards the axis of the ingot and towards from bottom to top part of the experimental ingot.

5. CONCLUSION

It was performed detailed analysis of heterogeneity the 90-ton experimental ingot that was produced at steel plant VÍTKOVICE HEAVY MACHINERY a.s. The main aim of this work was to obtain information about current state of distribution of macro-segregations. Mutual mixing of two heats needed for the production of this ingot was verified. The extent of segregation of individual elements was determined. These results are also used for improving numerical simulations of casting and solidification of heavy steel ingot. The final aim is minimize occurrence of macro-segregations.

ACKNOWLEDGMENT

We would like to express thanks to the Ministry of Industry and Trade of the Czech Republic for the financial support within the frame of the project TIP No. FR-TI3/243.

LITERATURE

