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Scanning Electron Microscopy as a Tool for Castings Quality Analysis

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Abstract

Nowadays, the best castings' manufacturers have to meet very demanding requirements and specifications applicable to mechanical properties and other characteristics. To fulfill those requirements, more and more sophisticated methods are being used to analyze the internal quality of castings. In many cases, the commonly used Non-Destructive Methods, like X-ray or ultrasonic testing, are not enough to ensure precise and unequivocal evaluation. Especially, when the properties of the casting only slightly fail the specification and the reasons for such failures are very subtle, thus difficult to find without the modern techniques. The paper presents some aspects of such an approach with the use of Scanning Electron Microscopy (SEM) to analyze internal defects that can critically decrease the performance of castings. The paper presents the so-called bifilm defects in ductile and chromium cast iron, near-surface corrosion caused by sulfur, micro-shrinkage located under the risers, lustrous carbon precipitates, and other microstructure features. The method used to find them, the results of their analysis, and the possible causes of the defects are presented. The conclusions prove the SEM is now a powerful tool not only for scientists but it is more and more often present in the R&D departments of the foundries.

Keywords: Castings defects, Castings quality, Internal defects, Bifilm, Scanning Electron Microscopy

1. Introduction

Complicated devices without castings inside are very rare, and thus their quality is a very important issue [1]. Taking into consideration the growing quality of machines or working devices, their components should be of the highest possible quality, too. That is why the quality of castings is the most important aspect for every foundry. The overall quality is a matter of dimensional accuracy, surface finish, and internal soundness. To achieve the desired and exceptional range of mechanical properties, casting should be free of internal defects, both in macro and micro scale [2-6]. To find the first group of defects, the Non-Destructive Testing (NDT) methods like Ultrasonic Testing (UT) or Radiography Testing (RT) can be employed, which nowadays is quite a traditional approach [7-9]. Computer Tomography (CT) becomes increasingly popular, and some leading foundries have bought such devices quite recently [10]. The evaluation of the second group of problems is much more difficult, therefore more and more sophisticated methods have to be developed, and Scanning Electron Microscopy (SEM) is one of them. For many years, the method has been successfully developed, and nowadays the method itself and the laboratory devices became less complicated, therefore they may be used not only in research labs but in factory R&D departments as well [4, 11-13]. The article presents some selected aspects of the use of SEM for the internal castings' quality evaluation. It shows the potential of electron microscopy as a day-by-day quality improvement approach which helps to find and fight the root causes of critical failures of castings. What is more, it helps to understand the links between the macro defects reported in the foundry with their micro causes, which are hard to find without the help of the SEM examinations. The paper presents examples of so-called bifilm defects in cast iron of various grades, near-surface corrosion caused by sulfur, micro-shrinkage defects located under the risers, lustrous carbon precipitations, and other microstructure features.

2. Materials and Methods

The experiments have been carried out on castings made in a laboratory and industrial conditions, too. They were made of a variety of alloys including typical ductile iron grade GJS-400-15 presented in Table 1 (two melts), high chromium cast iron with the addition of titanium obtained based on EN-GJN-HV600(XCr18), presented in Table 2, and the EN-GJS-SiMo50-10 presented in Table 3. The alloys have not been selected purposely to check any relations between them. The aim was to present the castings quality evaluation examples with the use of the EM methods since the analyzed examples featured some interesting castings defects impossible to present without such a tool.

Table1.
Chemical composition of GJS-400-15 iron

	Chemical composition, wt. %						
	C	Si	Mn	P	Mo	S	Mg
Melt 1	3.47	2.40	0.206	0.085	0.003	0.007	0.054
Melt 2	3.63	2.95	0.06	0.03	0.001	0.012	0.042

Table 2.
Chemical composition of EN-GJN-HV600(XCr18)

Chemical composition, wt. %						
C	Cr	Ti	Mn	Si	Ni	Mo
3.09	19.6	1.08	0.346	0.817	1.46	0.594
Al	V	Zr	S	P	Nb	Cu
0.167	0.172	0.283	0.036	0.053	0.141	0.037

Table 3.
Chemical composition of EN-GJS-SiMo50-10 ductile cast iron

Chemical composition, wt. %						
C	Si	Mn	P	Mo	S	Mg
3.04	4.94	0.11	0.02	1.1	0.005	0.031

All castings were subjected to quality check including the metallographic examinations with the use of a Phenom-ProX scanning microscope with an EDS system. Electron microscopy methods are perfect for the examination of the internal quality of engineering alloys. Their use facilitates deeper analysis of samples and allows to obtain results unattainable in the case of other methods, such as light microscopy (LM). A vital feature is the ability to analyze the fracture surface of samples. It can be used after a mechanical properties examination (UTS, KV) as a further step allowing for a more precise description of the material. Additionally, it can be used as a failure cause detection mechanism. For instance, if casting fails during operational use, EM methods can be applied to find the root cause of the failure. White chromium cast iron (Table 2) is a commonly used alloy for machine parts that work in peculiar conditions where special properties are required like, for example, corrosive or abrasive resistance. The ability to work in a particular environment depends on the chemical composition of the alloy that determines the characteristic microstructure formation. To achieve the best wear resistance of chromium cast iron, it is essential to obtain the microstructure reach in fine eutectic M_7C_3 carbides that guarantee extraordinary wear properties. Many researchers conducted works about the influence of Ti addition on the crystallization of M_7C_3 carbides in chromium white cast iron. According to [14-16], titanium added into melt causes refinement of chromium cast iron microstructure, since the TiC precipitation creates crystallization underlays for primary austenite. Those authors also state that the addition of a Fe-Ti-RE-Bi mixture changes the morphology of eutectic carbides. The other studies [17-21] also show the microstructure improvement after Ti inoculation, causing better wear properties. Unfortunately, it is not commonly known that this ingredient, when intentionally added to the melt, may cause a lot of problems with the casting quality.

3. Results and Discussion

As was mentioned earlier the castings and the alloys used are quite different but the link between them are the EM methods employed to check their internal quality. The results have been divided into three groups of defects and discussed separately.

3.1. Inclusions defects in cast iron

As it is widely known internal non-metallic inclusions can affect the casting property badly. For instance, an analysis of Figure 1 a) and b) provides interesting information about two samples of ductile iron. Both samples feature the same chemical composition, (Melt 1 in Table 1) were cast at the same pouring temperature and presented similar solidification times, and yet one featured 50% more elongation than the other. A thorough analysis of the fracture surface indicated the less ductile sample b) presented much more non-metallic inclusions and cleavage facets than a dimple-rich fracture of the more ductile sample a). Even without the tensile tests, based on the comparison of these two images, it would be possible to estimate and predict which of those two samples presented more ductility and which would be more prone to failure.

EM analysis also allows indicating defects associated with reoxidation of the metal which is commonly connected with turbulent filling while using inadequate gating system design. Those defects introduced by John Campbell – bifilms and bubbles – are often omitted and commonly misinterpreted during the failure analysis, nevertheless, their presence is undoubtedly a necessary condition for initiation of the failure.

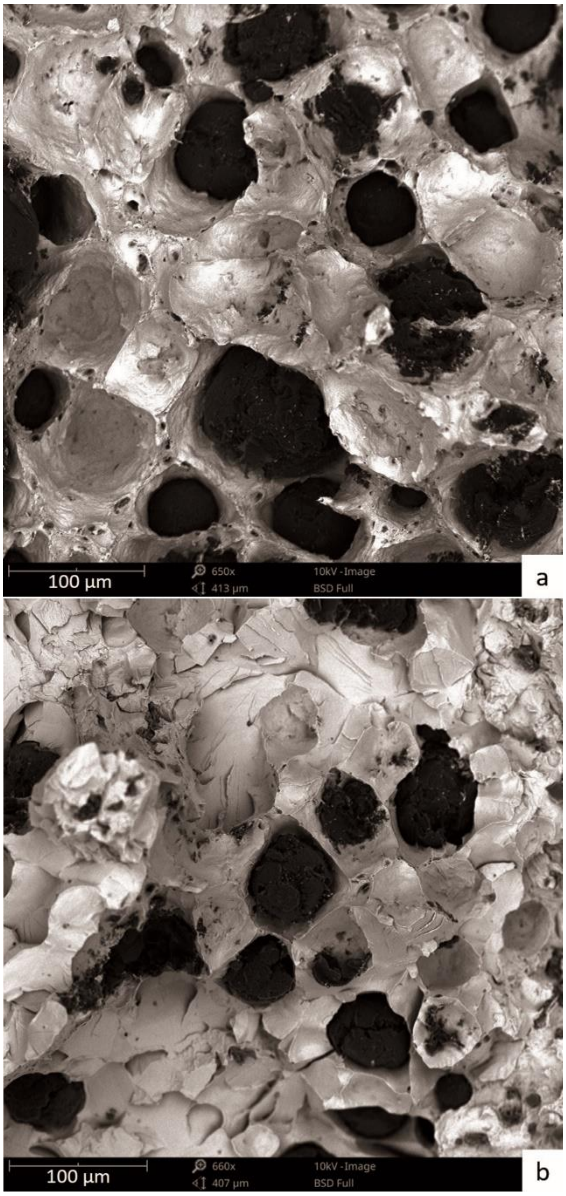


Fig. 1. Two ductile iron samples: more ductile a) more brittle b)

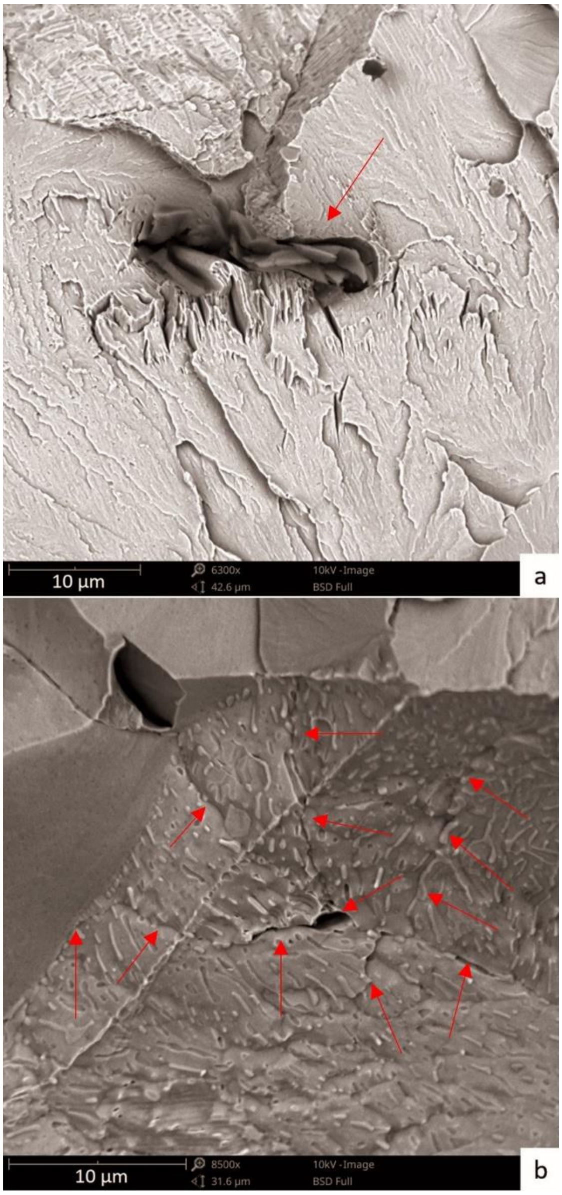


Fig. 2. Furled a) and unfurled b) bifilms on the fracture surface of a ductile iron sample

Figure 2 presents examples of double oxide films entrained into the alloy in its liquid state, in case of a) metal surrounding the inclusion solidified relatively fast, partially keeping the furled morphology of the inclusion, whereas in case of b), slow solidification of the metal surrounding the defect allowed the solidification front to straighten the previously furled inclusion. Red arrows mark the ‘path’ of the bifilm, an inattentive observer could incorrectly interpret such defect as a crack.

Alongside bifilms, bubbles are often remnants embedded into the matrix of the alloys, being the testament of inadequate filling of the molds cavity.

Figure 3 presents a partially collapsed gas bubble entrained into the liquid metal during the filling of the mold which did not present sufficient buoyancy to reach the top of the casting. As its way up was hindered and finally stopped by the forming crystals, it can be seen how its surface is deformed by the dendrites. Again, an inattentive observer could incorrectly identify such defects as gas defects of homogenous origin associated with a high content of gasses in the melt, however, such formation mechanism was proven impossible [22, 23]. Without the application of EM, such a defect on a section prepared for LM would look like ordinary porosity; however, only the depth of the SEM image allows to correctly classify this defect. Interestingly, the oxidized and wrinkled surface of the bubble contains a large number of inclusions that can be analyzed, for instance, with the use of EDS.

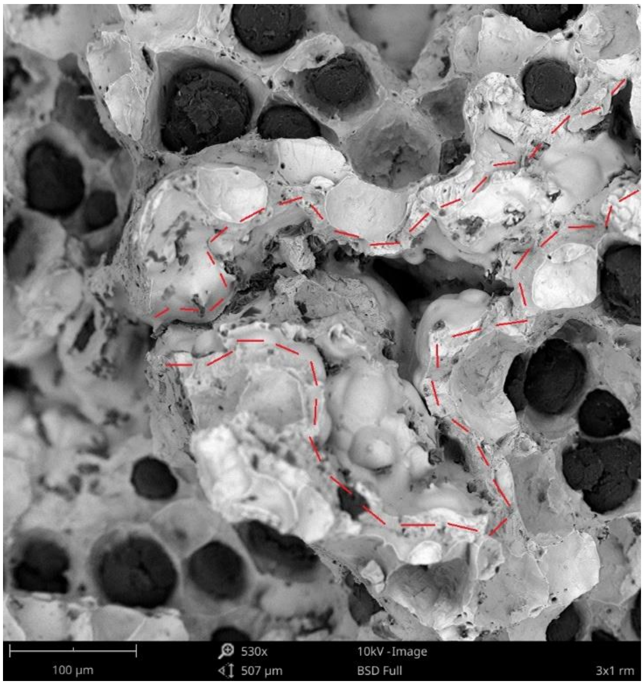


Fig. 3. Collapsed bubble in ductile iron sample

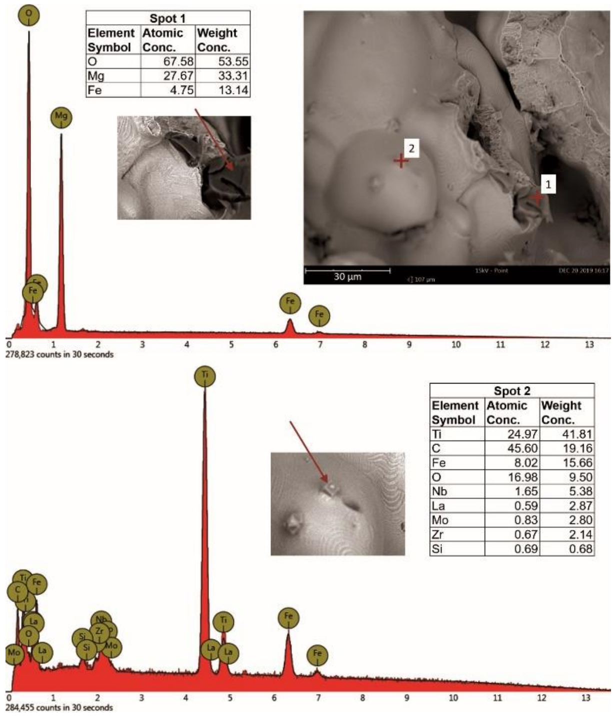


Fig. 4. EDS analysis of the collapsed bubble surface in the ductile iron sample

Quantitative analysis of the square-like inclusions visible in Figure 4 on the surface of the bubble allowed to state that they contain large amounts of titanium and carbon, which indicate that the inclusion is titanium carbide. On the other hand, it can be assumed that irregular inclusions with darker colors, also visible in Figure 4, contain lighter elements. Quantitative analysis indicated significant content of magnesium as well as oxygen, based on which it can be assumed that those inclusions could be magnesium oxides, which would be quite common for ductile iron. In such a case, diffraction analysis could not be used to identify the inclusions (phases) as their content is insufficient. Of course, TEM could be used for precise identification and description of the inclusion; however, due to the high price of the TEM examination, as well as a long preparation time, the analysis based on the SEM can be sufficient for the majority of the cases in well-examined materials that are most commonly used in the foundries, such as cast steels, cast irons, aluminum alloys and copper alloys. In such cases, an experienced SEM operator can usually identify the root cause of the defect without the necessity to use TEM, thus saving precious time and money.

It should also be mentioned that, in case of cast iron, EM offers amazing capacity for evaluation of graphite precipitations. For instance, in case of ductile iron not only the nodularity can be analyzed, but also the subsequent layers, thus informing a thorough observer about the growth kinetics of the nodule. Figure 5 presents an agglomeration of degenerated graphite nodules visible in the fractured surface of the ductile iron tensile sample.

Such an example perfectly shows that EM methods can be applied not only for analysis of the objects with characteristic dimensions close to nanometers but also larger objects as the presented agglomeration was visible on the sample with the naked eye as a dark spot.

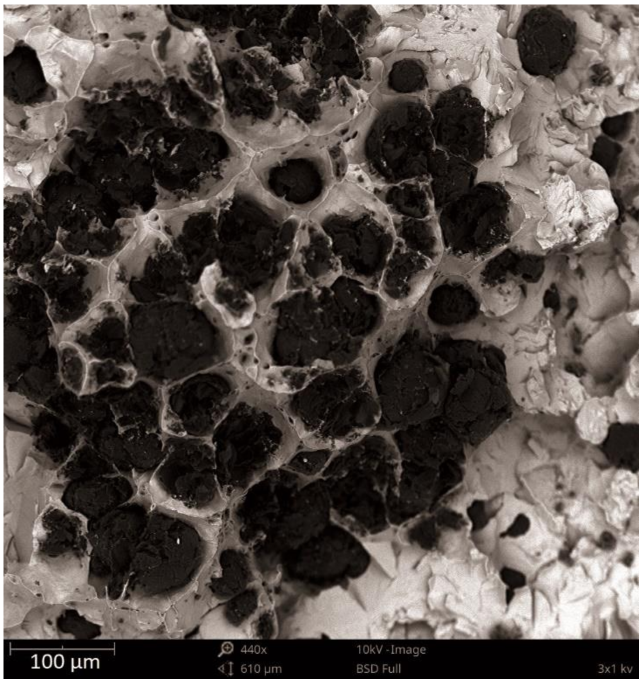


Fig. 5. Agglomeration of degenerated graphite nodules in the ductile iron sample

3.2. Defects and phase distribution in chromium white cast iron

Another issue is the improper distribution of the complex compounds in the alloyed grades of cast iron. In the case of the high chromium iron (see Table 2 for chemical analysis) research, it showed that the addition of titanium as an underlay forming element for M_7C_3 carbides crystallization can agglomerate in large clusters. The application of scanning electron microscopy SEM allowed the identification of numerous anomalies related to the inappropriate use of modifying additives. Figure 6 presents how improper distribution of TiC hard carbides may form in the casting microstructure. The sample was taken from the experimental casting of 30 mm diameter.

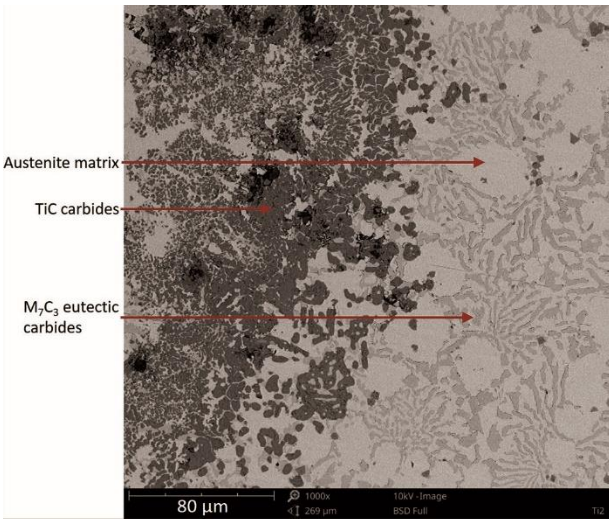


Fig. 6. Micrograph of 20% chromium white cast iron with the addition of 2% Ti, SEM, unetched

On the left side of the micrograph in Figure 6, a huge cumulation of TiC carbides can be seen, while on the right side, there are few titanium carbides surrounded by austenite + M_7C_3 eutectic. There is no denying that such a cumulation of the hard phase in the microstructure is less than optimal. A deeper analysis of this kind of area will allow us to notice that the agglomerates of TiC carbides are usually adjoined with the space inside the casting. Figure 7 shows this type of situation in the higher magnification on two SEM micrographs. According to the previous research, those empty areas in the castings where the titanium carbides seem to agglomerate were classified as bifilm defects [24].

In the previous work based on prof. J. Campbell's theories [25] and after SEM analysis, the authors connected this phenomenon to the hypothesis of the TiC closure mechanism in inclusions (bifilms). But the analysis of other samples and other SEM micrographs, like those present in Figure 7, shows that TiC phases may not only be closed in bifilm inclusions but also crystalize on them. Bifilm inclusion can provide crystallization underlay for titanium carbides.

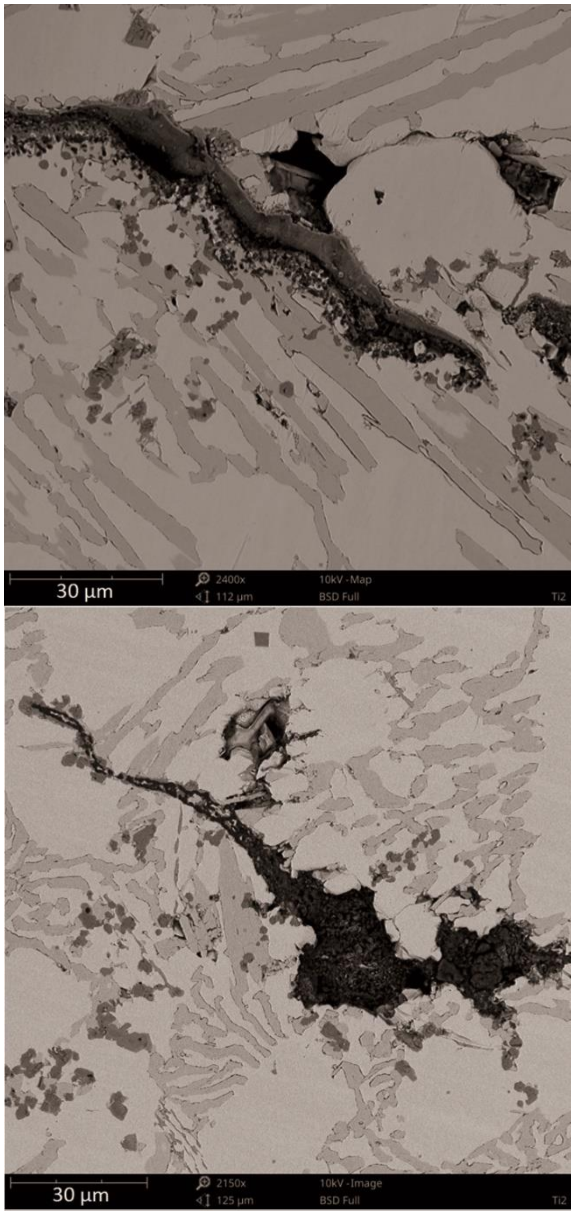


Fig. 7. Bifilm defects with TiC attached in 20% chromium white cast iron sample with 2% of Ti addition, SEM, unetched

Unfortunately, this situation is not favorable. Bifilm inclusions, accompanied by titanium carbides, are transported with waves of liquid metal and on the crystallization front and most generally they can stick in the middle of the casting, for instance, in the shrinkage cavity. Figure 8 presents the typical shrinkage cavity in chromium white cast iron experimental casting of 10x10 mm dimensions from the fracture surface of the Charpy test sample. Closer examination of the cavity found TiC phases (see Figure 9). These phases further reduce the strength of the casting.

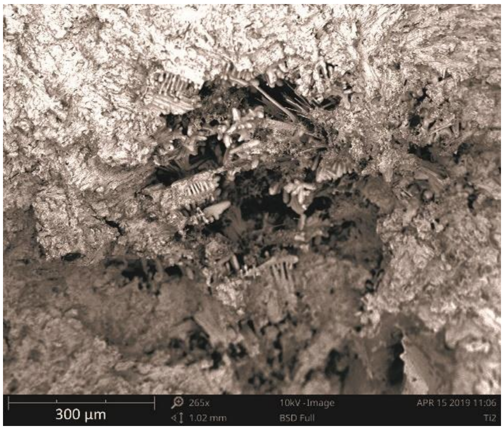


Fig. 8. Shrinkage cavity in chromium cast iron sample, SEM

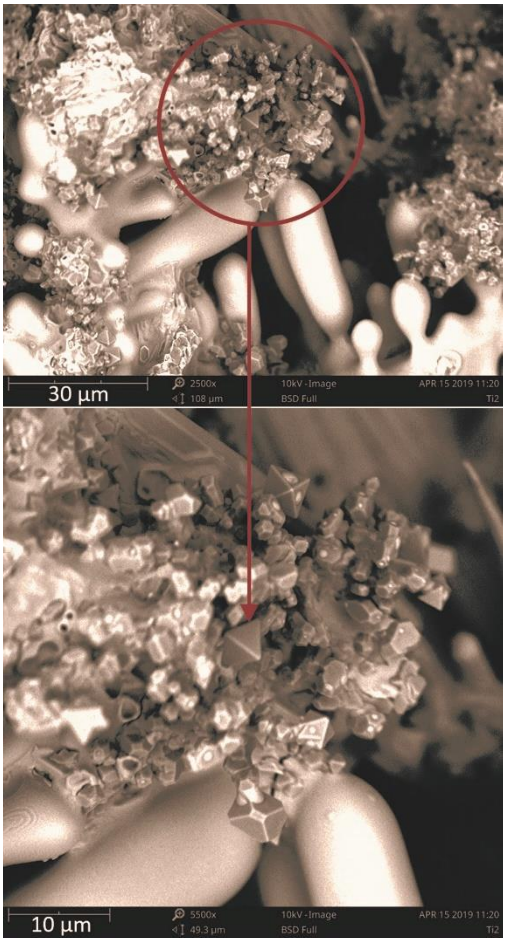
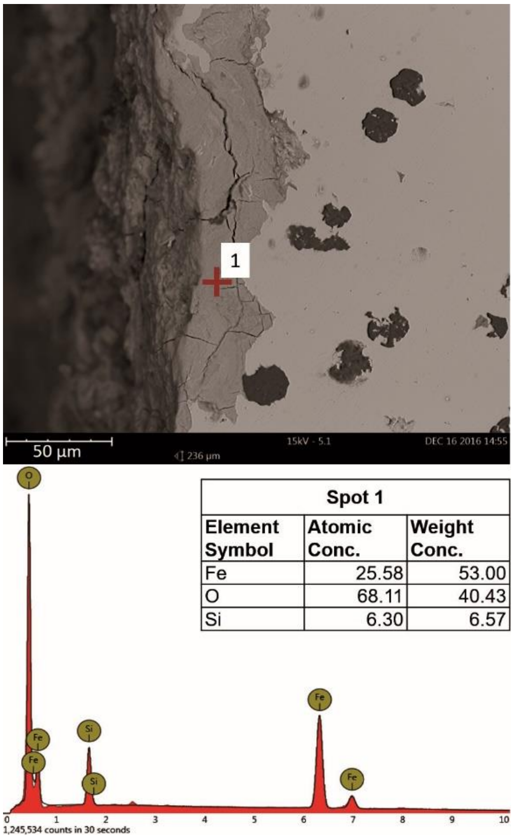


Fig. 9. Agglomeration of TiC phases in the shrinkage cavity, SEM

By analyzing the micrographs shown in Figure 9, we can tell that the huge amount of TiC phases, which were supposed to be crystallization underlays for M_7C_3 carbides, were transported into the place where they no longer serve their purpose. Moreover, in this place, the hard TiC phase also cannot improve the wear properties of chromium cast iron. This is significant for the foundry industry for economic reasons. The presented SEM studies conducted for chromium cast iron show how bifilm inclusions affect the distribution of phase that is supposed to have a key role in casting crystallization. Besides, it shows that the simple SEM analysis of phase distribution may be useful for casting quality examination.

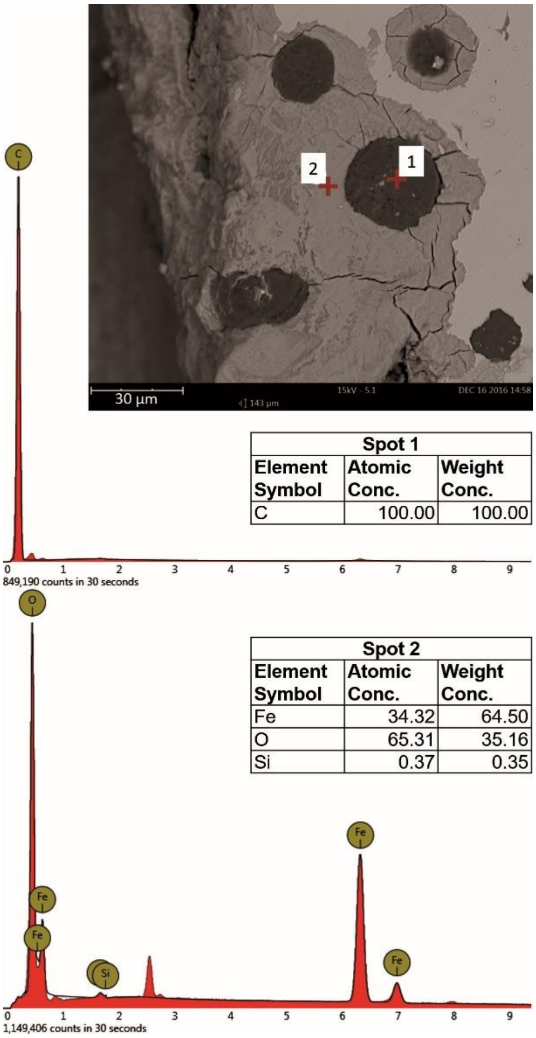
3.3. Intermetallic phases and surface corrosion of ductile iron

The next issue described in the article was the intermetallic phases distribution and surface corrosion of the analyzed castings. Metallographic tests were carried out on a sample cut from the casting made of the Melt 2 grade cast iron, Table 1. The specificity of the spheroidization process of castings using the in-mold method does not create conditions for the removal of chemical reaction products to the slag. These products remain in the mold cavity, reducing the quality of the casting. In the presented work, defects were diagnosed in the sub-surface layer of the casting in the form of intermetallic phase precipitates – fayalite (Fe_2SiO_4) [26-29]. These defects cause heterogeneity of the metal matrix while reducing the quality of the finished element. Figures 10-11 show the defects of the microstructure in a form of fayalite (Fe_2SiO_4) precipitates.



Spot 1		
Element Symbol	Atomic Conc.	Weight Conc.
Fe	25.58	53.00
O	68.11	40.43
Si	6.30	6.57

Fig. 10. View of the sub-surface layer of the tested casting. The dark area of fayalite (Fe_2SiO_4). The EDS point analysis site is marked



Spot 1		
Element Symbol	Atomic Conc.	Weight Conc.
C	100.00	100.00

Spot 2		
Element Symbol	Atomic Conc.	Weight Conc.
Fe	34.32	64.50
O	65.31	35.16
Si	0.37	0.35

Fig. 11. Separation of spheroidal graphite in the near-surface layer of the casting surrounded by fayalite (Fe_2SiO_4). The marked point 1 on the EDS point analysis – spheroidal graphite, the marked point 2 of the EDS point analysis – fayalite

Another disadvantage is the surface corrosion of ductile iron castings made with the in-mold method. Figure 12 shows the surface layer of two ductile iron castings with a very similar chemical composition. The samples have not been placed in a corrosive environment. The samples corroded in the laboratory room where they were stored. The cast on the left side (surface corroded) was made with the in-mold method, while the cast on the right side, with a clear lack of any signs of corrosion, was made via “sandwich” spheroidization. In the in-mold method, the sulfur compounds that are formed during the spheroidization process are not removed from the metal bath. They float to the surface of the casting, causing surface corrosion over time. An example of such a phenomenon is presented in Figure 13.

Fig. 12. Ductile iron casting surface. On the left side, a casting made in the "in-mold" technology. On the right, the casting surface is made in the traditional sandwich spheroidization method

The analysis of the corroded surface using a scanning microscope revealed an image of uniform corrosion with local cracks in the oxide layer (Figure 13).

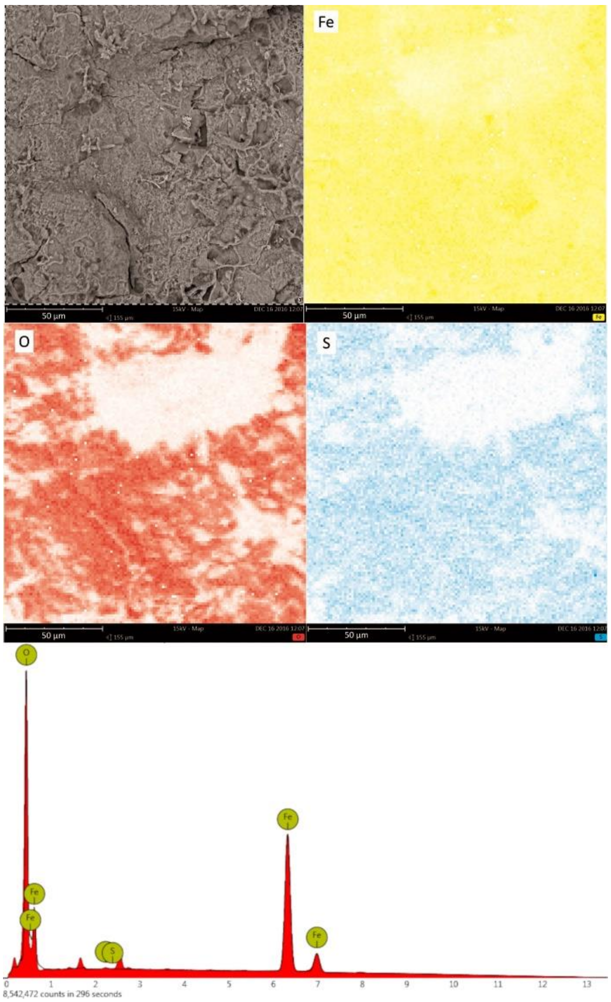


Fig. 13. Corrosion products on the surface of in-mold ductile iron

The EDS analysis revealed sulfur in the oxide layer. This is one of the reasons for the appearance of a corroded layer on the surface of the castings. Such a defect reduces the commercial quality of the casting. Some surfaces of the castings are left unfinished. The presence of sulfur compounds in the surface layer causes corrosion of these surfaces, despite their protection with a layer of protective paint.

Another analyzed case of a defect formed in casting is shrinkage porosity in castings made of SiMo cast iron (see Table 3).

The view of the shrinkage porosity of SiMo cast iron is presented in Figure 14. This defect is caused by the reduced carbon content in the alloy. Due to the designation of SiMo castings for operation at elevated temperatures, the carbon content should be at the level of 3%. The relatively low proportion of carbon reduces the phenomenon of pre-contraction expansion of cast iron as a result of the graphitization process, and thus the ability to self-supply the casting.

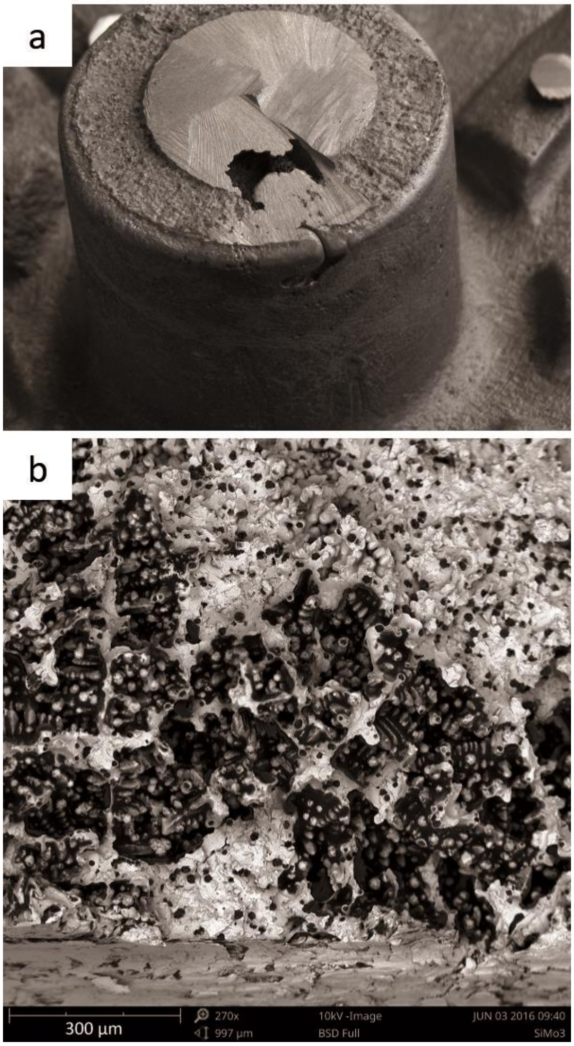


Fig. 14. Shrinkage cavity located under the riser in SiMo cast iron a), Shrinkage porosity in SiMo cast iron, SEM b)

As a result of the analysis, clear traces of corrosion were observed on the surface of the inmold casting. It should be noted that corrosion phenomena appear in the upper part of the casting. The reason for this is the MgS, CeS [30, 31] compounds formed as a result of the reaction of sulfur contained in cast iron with spheroidizing reagent, which cannot be removed from the liquid metal. This disadvantage can be eliminated by using a metal charge with reduced sulfur content. The examinations of the SiMo castings using the SEM led us to some conclusions. Firstly, the elements that reduce the quality of castings are compounds of iron, silicon, and oxygen (fayalite – Fe_2SiO_4). For the in-mold spheroidization method, the occurrence of this type of intermetallic inclusions cannot be eliminated. Secondly, the shrinkage porosity in SiMo cast iron castings is difficult to eliminate due to the reduced carbon content. The only way to avoid defects of this type is to design an appropriate technology of the production of these castings.

4. Summary

The article shows how powerful a tool for internal castings quality evaluation, Electron Microscopy, is. The examinations like these presented here help the foundries to continuously improve the quality of their products. The presented results were then discussed and the recommendations have been proposed. They covered the changes in the alloy chemical composition (inside the standard specification), casting technology (including the gating system re-design), process temperature, etc. In some cases, the use of SEM helped to understand the physical phenomena and to prove certain theories, like John Campbell's bifilms theory. Of course, we must be aware that some results, like those of the EDS analysis, should be confirmed utilizing other, more sophisticated and precise research methods.

Acknowledgments

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