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Examination of Internal Defects in the Casting Process by Using a Scanning Electron Microscopy

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Abstract

In today's manufacturing landscape, stringent requirements and specifications for mechanical properties and other characteristics have become crucial for top casting manufacturers. This paper focuses on using Scanning Electron Microscopy (SEM) as a powerful tool to analyze internal defects that significantly impact the performance of castings. Specifically, it delves into various aspects of this approach, including the identification of bifilm deficiencies in ductile and chromium cast iron, near-surface corrosion induced by sulfur, micro-shrinkage under risers, remnants of lustrous carbon, and other microstructural features. The paper elucidates the methodology employed to detect these defects, presents the results of their analysis, and explores potential causes behind their occurrence.

Keywords: Mechanical Property, Scanning Electron Microscopy, Internal Defect, Casting, Chromium Cast Iron

INTRODUCTION

In light of the increasing quality standards for machines and working devices, the significance of ensuring superior component quality cannot be overstated, particularly considering the rarity of intricate devices devoid of castings [1]. Consequently, the utmost importance lies in the quality of castings for any foundry, as it directly influences the overall quality through factors such as dimensional accuracy, surface finish, and internal integrity. In order to attain the desired and exceptional mechanical properties, it is imperative that castings are devoid of both macro and micro-scale internal defects [2-6]. To identify the first category of defects, traditional Non-Destructive Testing (NDT) methods such as Ultrasonic Testing (UT) or Radiography Testing (RT) have been conventionally employed [7-9]. Computer Tomography (CT) has become increasingly popular, and some leading foundries have bought such devices recently [10]. The evaluation of the second group of problems is much more difficult. Therefore, more sophisticated methods have to be developed, and Scanning Electron Microscopy (SEM) is one of them. The technique has been successfully created for many years, and nowadays, the method and laboratory devices have become less complicated. Therefore, they may be used not only in research labs but in factory R&D departments as well [4, 11-13]. The paper highlights specific aspects of utilizing SEM for evaluating the internal quality of castings, emphasizing its potential as an effective approach for continuous quality improvement. By utilizing electron microscopy, it becomes possible to identify and address the root causes behind critical casting failures. Furthermore, SEM examinations help establish connections between reported macro defects in foundries and their underlying micro-level causes, which can be

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challenging to identify without the aid of SEM analysis. The article presents instances of bifilm defects in various grades of cast iron, near-surface corrosion induced by sulfur, micro-shrinkage defects situated beneath risers, lustrous carbon precipitations, and other noteworthy microstructural features.

MATERIALS AND METHODS

The study encompassed experiments conducted on castings produced under both laboratory and industrial conditions. These castings were fabricated from a diverse range of alloys, including the commonly used ductile iron grade GJS-400-15 (two melts) as presented in Table 1. Additionally, high chromium cast iron containing titanium, designated as EN-GJN-HV600(XCr18) and illustrated in Table 2, along with EN-GJS-SiMo50-10 showcased in Table 3, were deliberately chosen for assessment purposes. The selection of these alloys aimed to establish correlations and provide examples of quality evaluations for castings using electron microscopy techniques. The incorporation of EM methods was vital as it facilitated the identification and demonstration of intriguing casting defects observed in the analyzed models, which would have been otherwise challenging to present without such a tool.

Table 1: 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	0.176	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
3.14	4.94	0.11	0.02	1.1	0.005	0.031

Electron microscopy techniques, such as metallographic examinations using a Phenom-ProX scanning microscope with an EDS system, are highly effective in assessing the internal quality of engineering alloys. Unlike light microscopy (LM), electron microscopy allows for more comprehensive analysis of samples, enabling the acquisition of results that are otherwise unattainable. One significant advantage is the capability to examine fracture surfaces, which can be utilized following mechanical properties testing (e.g., UTS, KV) to provide a more detailed characterization of the material.

Moreover, electron microscopy methods serve as a valuable tool in identifying the causes of failures. In the event of a casting failure during operational use, these techniques can be applied to determine the root cause of the problem. White chromium cast iron, as presented in Table 2, is a commonly employed alloy in machine parts that operate under challenging conditions requiring specific properties such as resistance to corrosion or abrasion. The ability of an alloy to perform in a specific environment is dependent on its chemical composition, which in turn influences the formation of its characteristic microstructure. In the case of chromium cast iron, achieving optimal wear resistance relies on obtaining a microstructure rich in fine eutectic M7C3 carbides, which impart exceptional wear properties. Numerous researchers have explored the impact of titanium (Ti) addition on the crystallization of M7C3 carbides in chromium white cast iron. As reported in references [14-16], the introduction of titanium into the molten metal leads to a refinement of the microstructure of chromium cast iron. This is attributed to the precipitation of titanium carbides (TiC), which act as nucleation sites for primary

austenite. These authors also note that the addition of a mixture containing iron (Fe), titanium (Ti), rare earth elements (RE), and bismuth (Bi) alters the morphology of the eutectic carbides. Other studies [17-21] have similarly demonstrated the improvement in microstructure and resulting enhanced wear properties following Ti inoculation. However, it should be noted that intentional addition of titanium to the melt can introduce challenges to the quality of the casting, a fact that is not widely known.

RESULTS AND DISCUSSION

Although the castings and alloys used may differ, the common thread between them lies in the utilization of electron microscopy (EM) methods to assess their internal quality. The obtained results have been categorized into three distinct groups of defects, which have been discussed individually.

INCLUSIONS DEFECTS IN CAST IRON

It is widely recognized that internal non-metallic inclusions can have a detrimental impact on casting properties. For example, the analysis of Figures 1 (a) and (b) provides intriguing insights into two ductile iron samples. Despite having the same chemical composition (Melt 1 in Table 1), being cast at identical pouring temperatures, and exhibiting similar solidification times, one sample demonstrated 50% greater elongation compared to the other. A detailed examination of the fracture surface revealed that the less ductile sample (b) exhibited a higher presence of non-metallic inclusions and cleavage facets, whereas the more ductile sample (a) displayed a fracture surface characterized by a greater abundance of dimples. Even without conducting tensile tests, a comparison of these two images would allow for an estimation and prediction of which sample exhibited greater ductility and which one was more susceptible to failure. Moreover, EM analysis can identify defects associated with metal reoxidation, which is often attributed to turbulent filling during the implementation of a preliminary gating system design. These defects, known as bifilms and bubbles, were introduced by John Campbell.

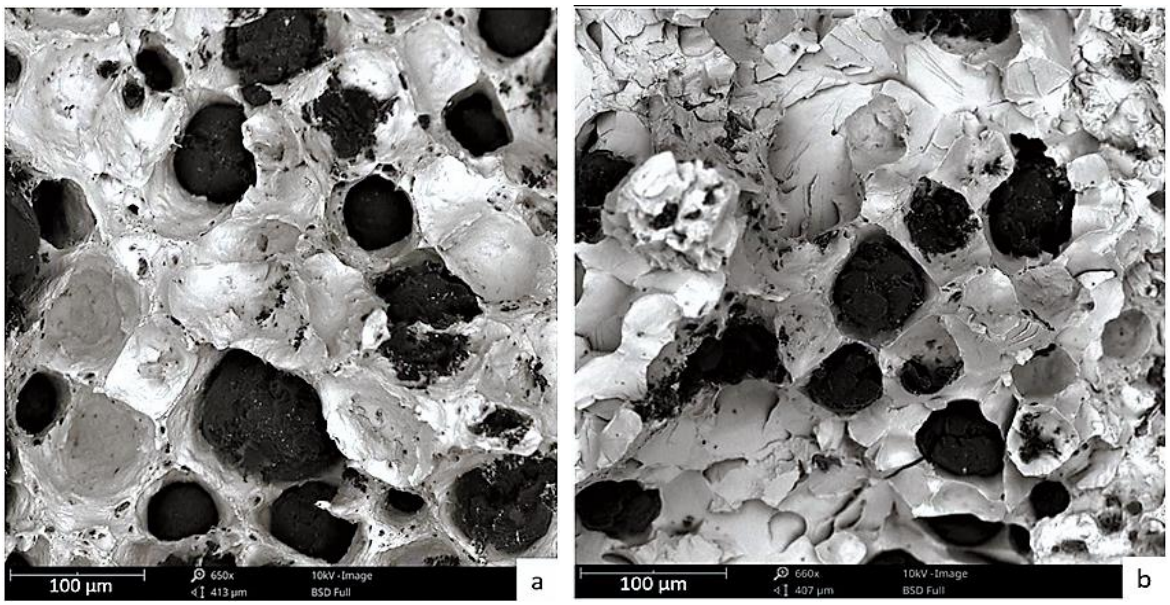


Figure 1. Two ductile iron samples: (a) more ductile, (b) more brittle.

Figure 2 showcases instances of double oxide films that have become entrained within the alloy during its liquid state. In case (a), the metal surrounding the inclusion solidified relatively quickly, preserving the folded morphology of the inclusion to some extent. Conversely, in case (b), the slow solidification of the metal surrounding the defect allowed the solidification front to straighten the previously folded inclusion. The red arrows indicate the "path" of the bifilm, which, if not observed carefully, could be mistakenly interpreted as a crack. In addition to bifilms, bubbles are often present as remnants embedded within the alloy's matrix, indicating inadequate filling of the mold's cavity.

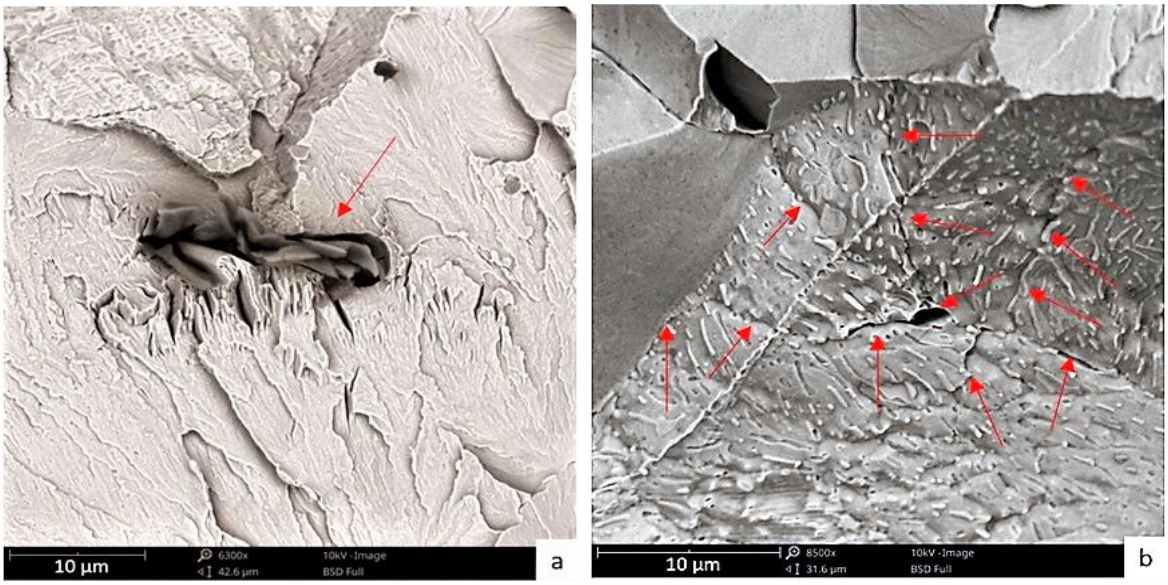


Figure 2. Furled (a) and unfurled (b) bifilms on the fracture surface of a ductile iron sample

Figure 3 illustrates a partially collapsed gas bubble that became entrained within the liquid metal during the filling process but did not possess sufficient buoyancy to rise to the top of the casting. As it encountered obstacles and was eventually halted by the growing dendrites, its surface underwent deformation. It is worth noting that an inattentive observer may mistakenly attribute such defects to gas-related issues originating from high gas content in the melt. However, extensive research has proven this particular formation mechanism to be impossible [22, 23]. Without the application of electron microscopy (EM), such a defect on a section prepared for light microscopy (LM) would appear as ordinary porosity. Only the depth provided by the SEM image enables us to accurately classify this defect. Interestingly, the oxidized and wrinkled surface of the bubble contains numerous inclusions that can be analyzed, for example, using energy-dispersive X-ray spectroscopy (EDS).

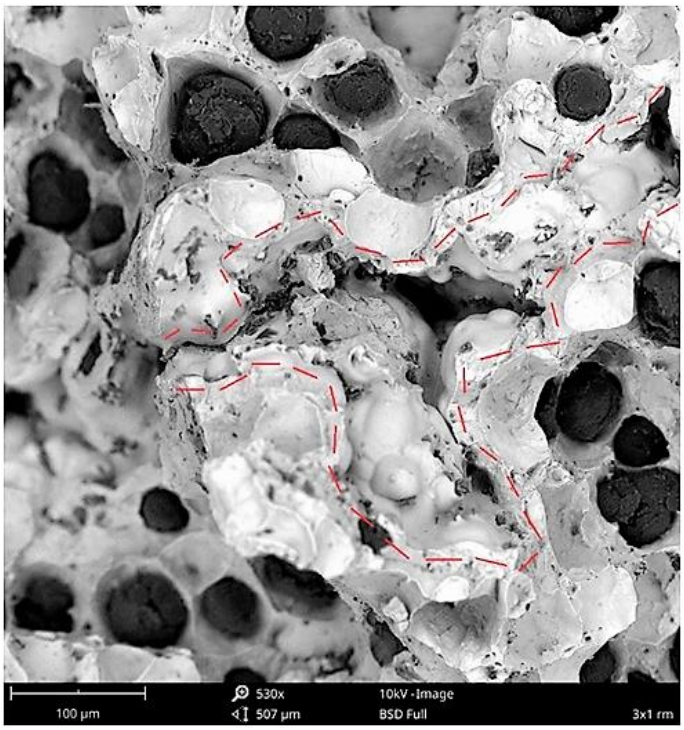


Figure 3. Collapsed bubble in ductile iron sample

The quantitative analysis of the square-like inclusions observed on the surface of the bubble in Figure 4 revealed significant amounts of titanium and carbon, suggesting that these inclusions are titanium carbides. On the other hand, the irregular inclusions with darker colors seen in Figure 4 are presumed to contain lighter elements based on quantitative analysis, which indicated substantial levels of magnesium and oxygen. These inclusions could potentially be magnesium oxides, a common occurrence in ductile iron. However, the low content of these inclusions makes it impractical to identify them using diffraction analysis. While transmission electron microscopy (TEM) could provide precise identification and characterization of the inclusions, its high cost and lengthy preparation time often make SEM analysis sufficient for most cases involving well-examined materials commonly used in foundries, such as cast steels, cast irons, aluminum alloys, and copper alloys. Skilled SEM operators can typically identify the root cause of the defect without the need for TEM, thereby saving valuable time and resources.

It is worth noting that electron microscopy (EM) techniques provide exceptional capabilities for evaluating graphite precipitations in the case of cast iron. In ductile iron, EM allows not only the analysis of nodularity but also provides insights into the subsequent layers, thereby enabling a comprehensive understanding of nodule growth kinetics. Figure 5 demonstrates an accumulation of degenerated graphite nodules observed on the fractured surface of a ductile iron tensile sample.

This example illustrates that EM methods can be employed to analyze objects with characteristic dimensions ranging from nanometers to larger structures. In the case presented, the agglomeration of degenerated graphite nodules was visible to the naked eye as a dark spot on the sample, highlighting the ability of EM to investigate both micro- and macro-scale features.

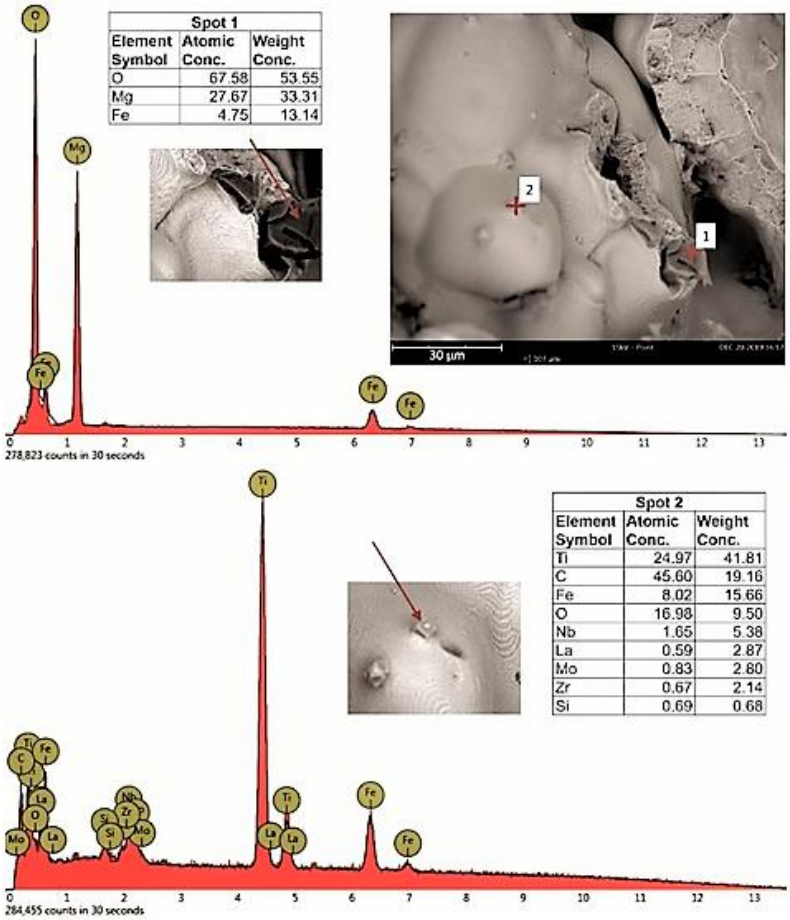


Figure 4. EDS analysis of the collapsed bubble surface in the ductile iron sample.

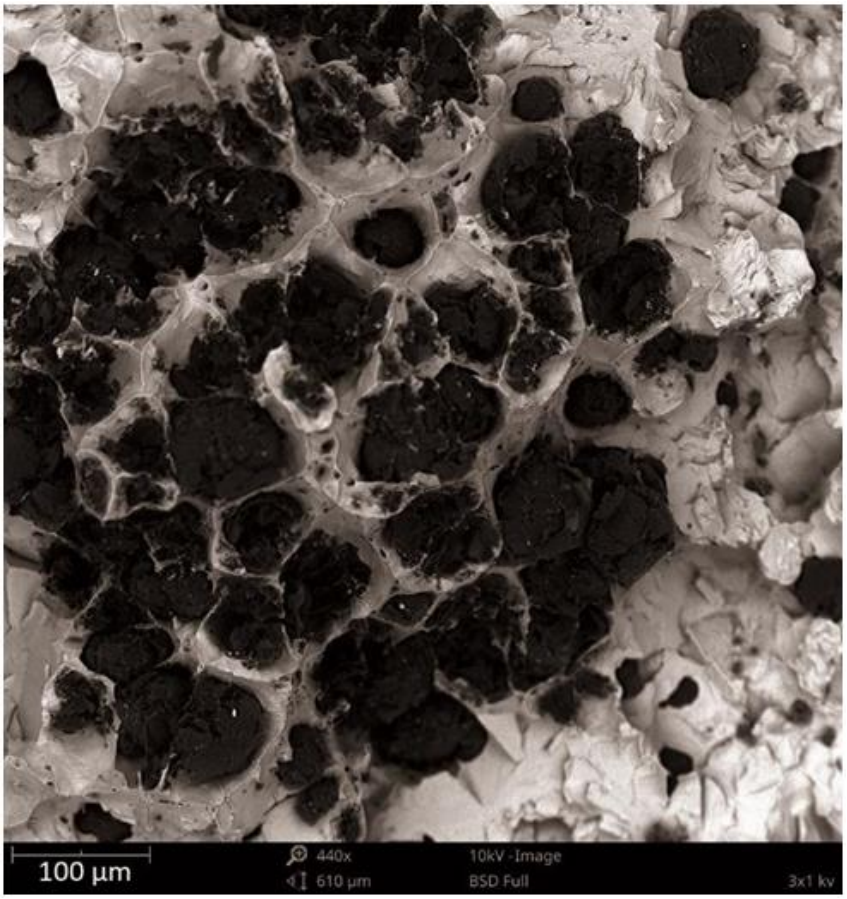


Figure 5. Agglomeration of degenerated graphite nodules in the ductile iron sample.

DEFECTS AND PHASE DISTRIBUTION IN CHROMIUM WHITE CAST IRON

The distribution of complex compounds in alloyed grades of cast iron is a significant concern. Research on high chromium iron (refer to Table 2 for chemical analysis) has demonstrated that the inclusion of titanium as an underlying element for the crystallization of M7C3 carbides can lead to the formation of large clusters. By utilizing scanning electron microscopy (SEM), various abnormalities associated with the improper utilization of modifying additives have been identified. Figure 6 illustrates the potential formation of TiC hard carbides in the casting microstructure due to inadequate distribution. The sample analyzed in this case was extracted from a 30 mm diameter experimental casting.

Figure 6 exhibits a notable contrast in the micrograph, where a significant accumulation of TiC carbides is observed on the left side, while on the right side, there are only a few titanium carbides surrounded by austenite + M7C3 eutectic. Undoubtedly, such an accumulation of the hard phase in the microstructure is suboptimal. A more detailed analysis of this region allows us to observe that the agglomerates of TiC carbides are typically found adjacent to the internal voids within the casting. Figure 7 provides a closer examination of this situation through higher magnification in two SEM micrographs. Previous research has classified these empty areas in the castings, where titanium carbides tend to agglomerate, as bifilm defects [24].

In earlier studies, based on the theories proposed by Prof. J. Campbell [25] and subsequent SEM analysis, the authors attributed this phenomenon to the hypothesis of the closure mechanism of TiC inclusions (bifilms). However, the analysis of additional samples and other SEM micrographs, such as those depicted in Figure 7, suggests that TiC phases can crystallize not only within bifilm inclusions but also on their surfaces. Bifilm inclusions can serve as a substrate for the crystallization of titanium carbides.

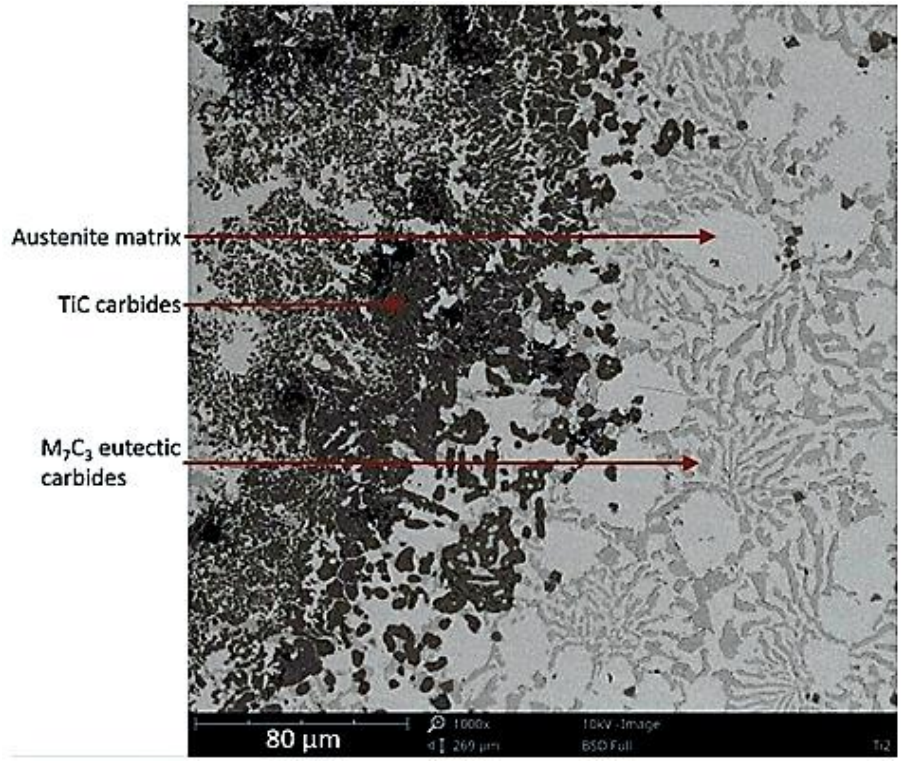


Figure 6. Micrograph of 20% chromium white cast iron with the addition of 2% Ti, SEM, unetched

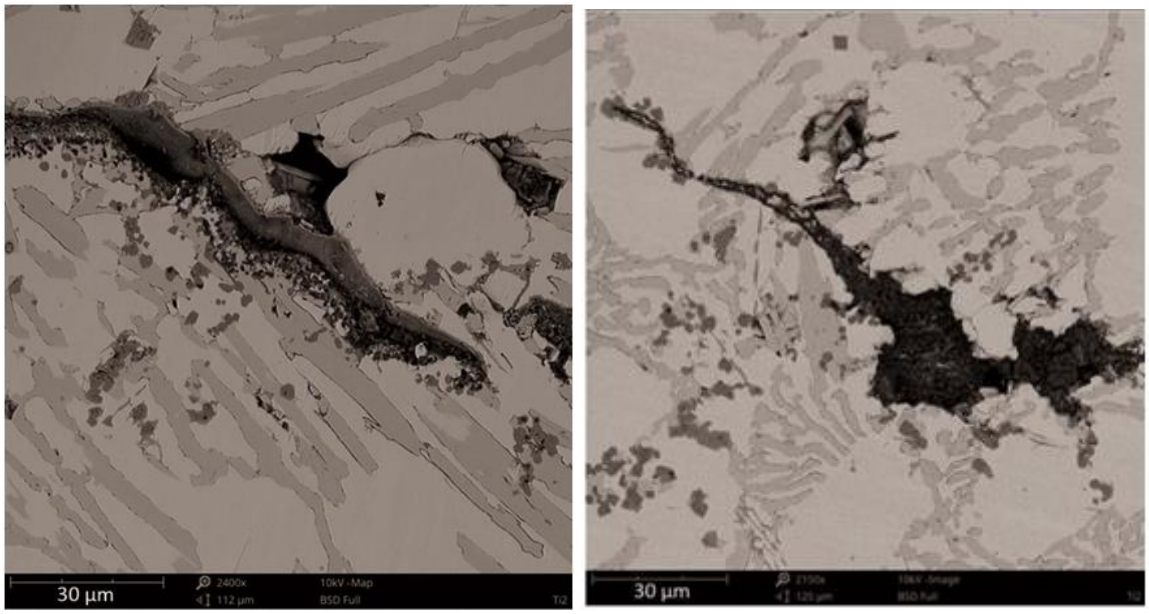


Figure 7: Bifilm defects with TiC attached in 20% chromium white cast iron sample with 2% of Ti addition, SEM, unetched

The current situation is unfavorable due to the presence of bifilm inclusions and titanium carbides. These impurities are carried along by waves of liquid metal during the casting process and can become trapped in various locations, such as the shrinkage cavity. A specific example is illustrated in Figure 8, showing a typical shrinkage cavity in a 10x10 mm chromium white cast iron experimental casting, as observed from the fractured surface of a Charpy test sample. Upon closer inspection of the cavity, TiC phases were discovered (refer to Figure 9). These phases significantly contribute to the reduction in the casting's overall strength.

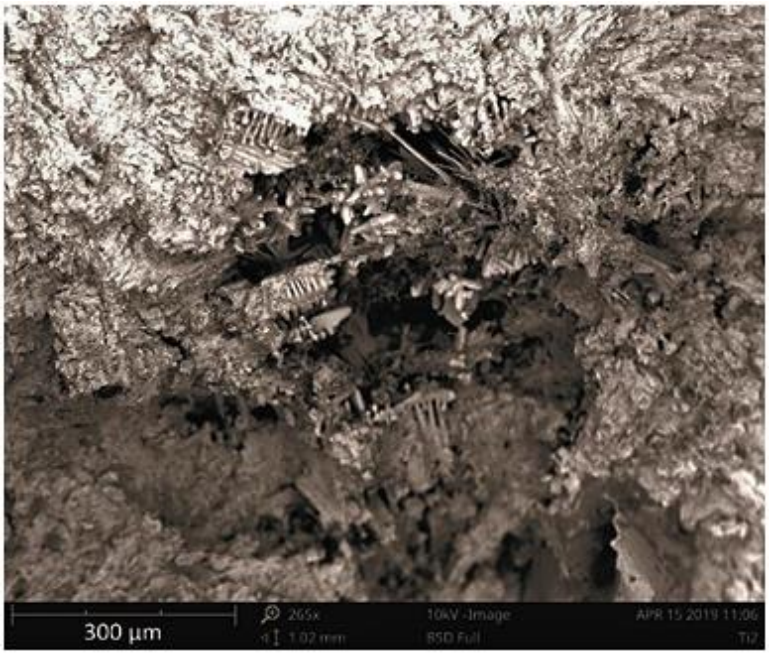


Figure 8: Shrinkage cavity in chromium cast iron sample, SEM.

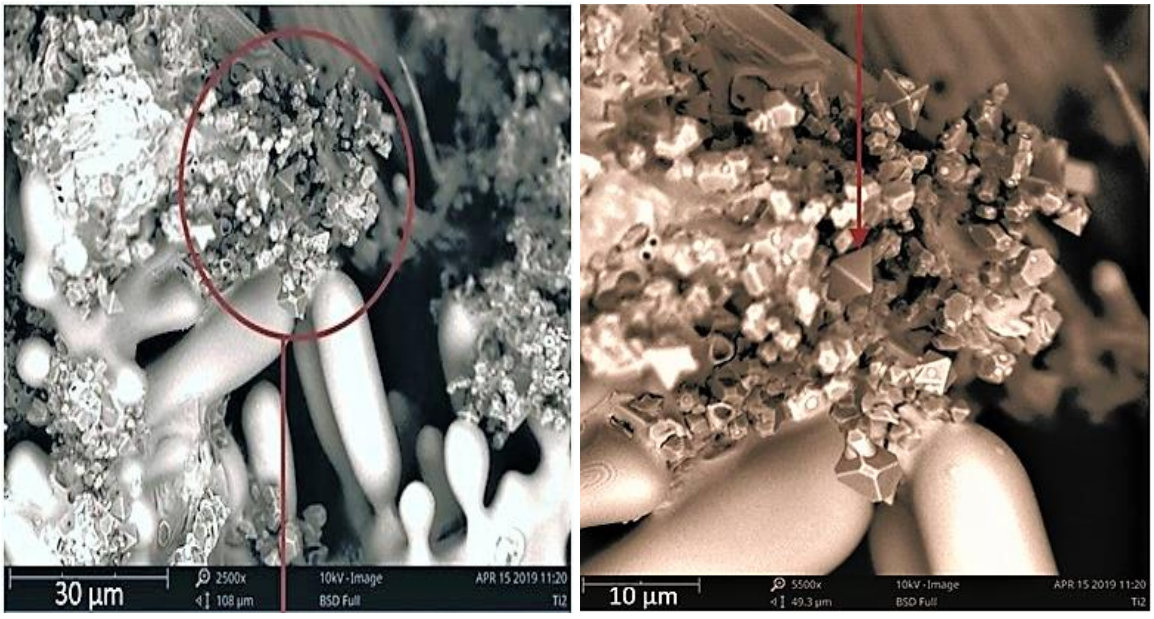


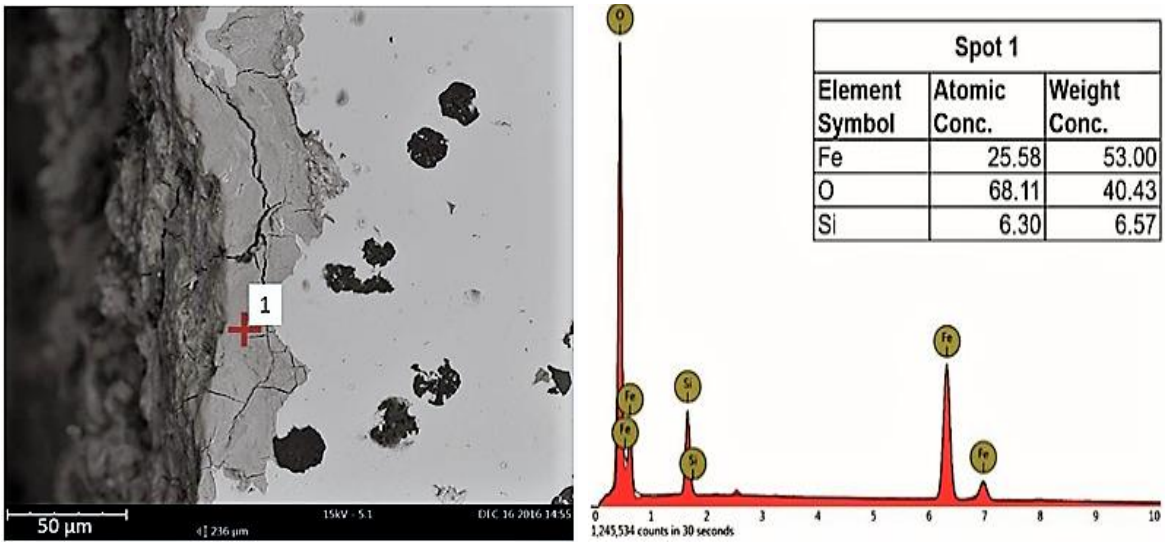
Figure 9: Agglomeration of TiC phases in the shrinkage cavity, SEM

The analysis of the micrographs in Figure 9 reveals that a large number of TiC phases, intended as crystallization underlays for M7C3 carbides, have been transported to a location where they no longer fulfill their intended function. Furthermore, in this particular area, the challenging TiC phase fails to enhance the wear properties of chromium cast iron. This finding holds significant implications for the foundry industry from an economic standpoint. The SEM studies presented for chromium cast iron demonstrate how bifilm inclusions impact the distribution of the phase that plays a crucial role in casting crystallization. Additionally, it illustrates that a simple SEM analysis of phase distribution can be valuable for assessing casting quality.

INTERMETALLIC PHASES AND SURFACE CORROSION OF DUCTILE IRON

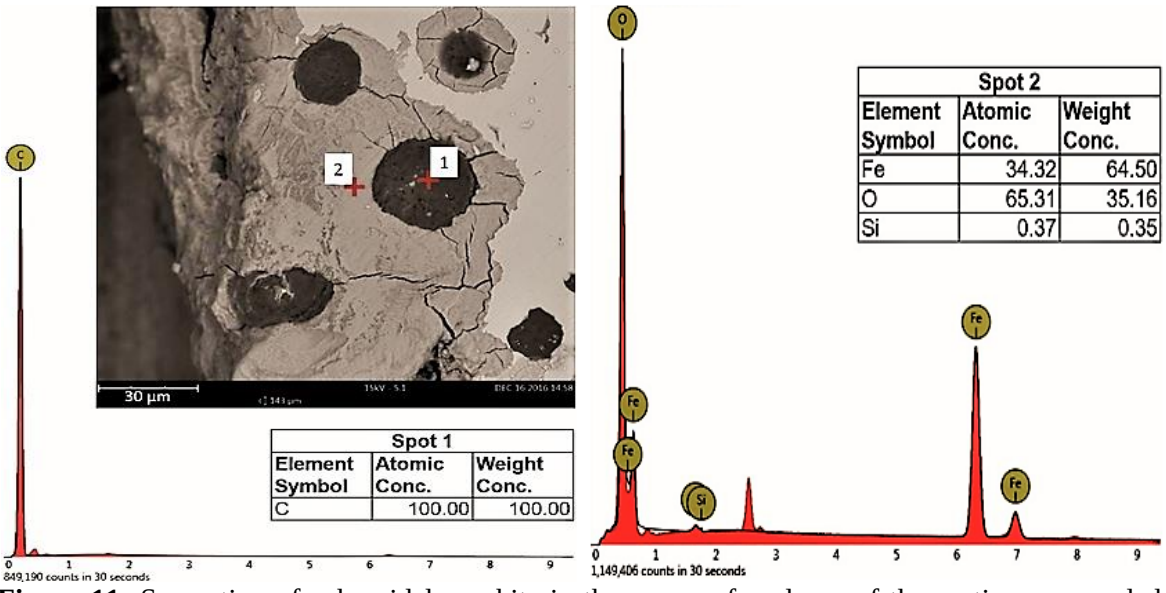
The article discusses another issue related to the distribution of intermetallic phases and surface

corrosion in the examined castings. To investigate this, metallographic tests were conducted on a sample taken from a casting made of Melt 2 grade cast iron, as indicated in Table 1. The specific nature of the spheronization process used in the casting, employing the in-mold method, does not provide conditions for eliminating chemical reaction byproducts from the slag. As a result, these byproducts remain in the mold cavity, which adversely affects the casting's quality. In the presented study, defects were identified in the sub-surface layer of the casting, taking the form of precipitates of an intermetallic phase known as fayalite (Fe2SiO4) [26-29]. These defects introduce heterogeneity to the metal matrix, thereby diminishing the quality of the final component. Figures 10-11 depict the presence of fayalite (Fe2SiO4) precipitates, illustrating the flaws in the microstructure.



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

Figure 10: View of the sub-surface layer of the tested casting. The dark area of fayalite (Fe2SiO4). The EDS point analysis site is marked



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

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

Figure 11: Separation of spheroidal graphite in the near-surface layer of the casting surrounded by fayalite (Fe2SiO4). The marked point 1 on the EDS point analysis – spheroidal graphite, the marked point 2 of the EDS point analysis – fayalite

Surface corrosion of ductile iron castings made using the in-mold method is another drawback. Figure 12 illustrates the surface layer of two ductile iron castings, both having a similar chemical composition.

These samples were not exposed to a corrosive environment but were stored in a laboratory room. The casting on the left side, made with the in-mold method, exhibits noticeable surface corrosion, while the casting on the right side, produced through the "sandwich" spheroidization technique, shows no signs of corrosion. In the in-mold method, sulfur compounds generated during spheronization are not eliminated from the metal bath. Instead, they rise to the surface of the casting, leading to surface corrosion over time. Figure 13 provides an example of this phenomenon.

Figure 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 examination of the corroded surface under a scanning microscope unveiled an image depicting homogeneous corrosion along with isolated cracks in the oxide layer (refer to Figure 13).

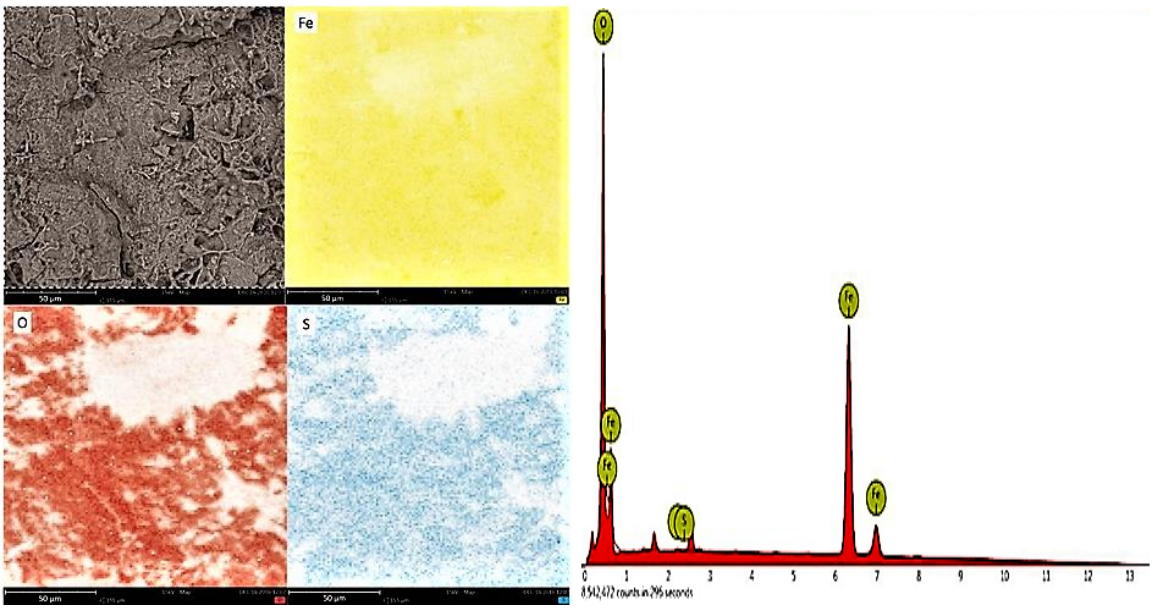


Figure 13: Corrosion products on the surface of in-mould ductile iron

The EDS analysis identified the presence of sulfur within the oxide layer, which contributes to the formation of the corroded layer on the surface of the castings. This defect significantly diminishes the commercial value of the castings. Additionally, certain characteristics of the castings require further

refinement. Despite being coated with a protective layer of paint, the presence of sulfur compounds in the surface layer leads to corrosion of these surfaces.

Table 3 presents another examined instance of a defect observed in castings made of SiMo cast iron, known as shrinkage porosity.

The visual representation of shrinkage porosity in SiMo cast iron is depicted in Figure 14. This defect arises as a result of the decreased carbon content in the alloy. For SiMo castings intended for high-temperature applications, the carbon content should ideally be around 3%. However, the relatively low proportion of carbon diminishes the pre-contraction expansion of the cast iron during the graphitization process, consequently reducing its self-supplying capability for the casting.

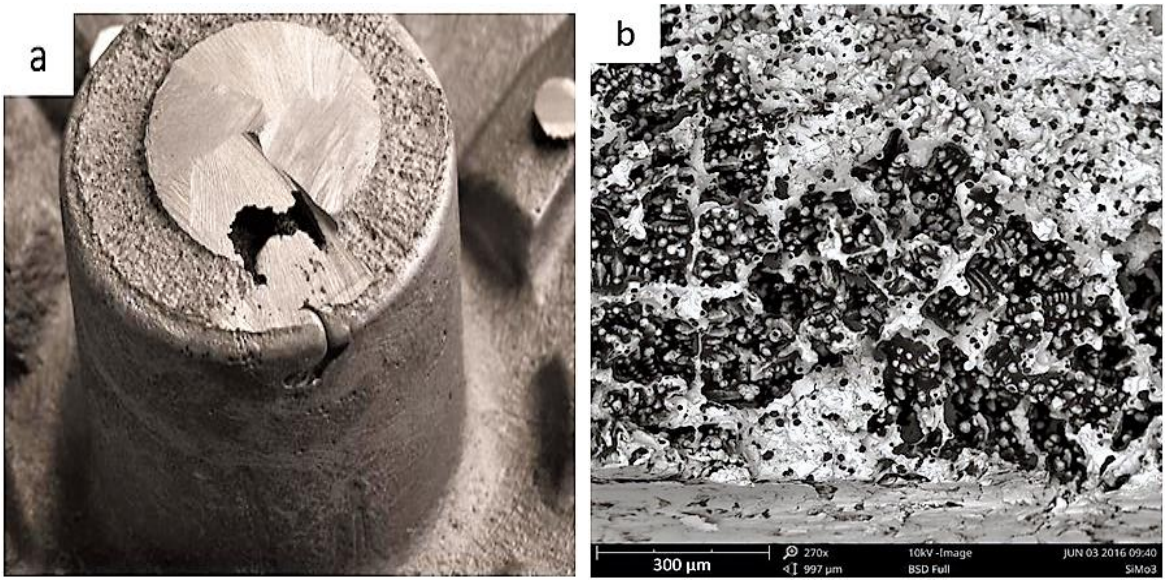


Figure 14. (a) Shrinkage cavity located under the riser in SiMo cast iron, (b)Shrinkage porosity in SiMo cast iron, SEM.

The analysis revealed clear signs of corrosion on the surface of the mold casting, primarily in the upper part. This corrosion is caused by the formation of MgS and CeS [30, 31] compounds resulting from the reaction between sulfur in the cast iron and the spheroidizing reagent, which cannot be removed from the liquid metal. However, this issue can be resolved by using a metal charge with lower sulfur content. SEM examination of the SiMo castings yielded several conclusions. Firstly, the presence of iron, silicon, and oxygen compounds (such as fayalite – Fe_2SiO_4) negatively affects the casting quality, and these intermetallic inclusions cannot be completely eliminated with the in-mold spheroidization method. Secondly, due to the reduced carbon content, controlling shrinkage porosity in SiMo cast iron castings is challenging. The only solution to prevent such defects is to develop an appropriate production technology for these castings.

CONCLUSION

The article highlights the significance of Electron Microscopy as a powerful tool for evaluating the internal quality of castings. Through examinations like the ones presented, foundries can continuously enhance the quality of their products. The discussed results and recommendations propose various changes, such as adjusting the alloy chemical composition (within standard specifications), re-designing the gating system, and modifying process temperature. In certain cases, using SEM aids in comprehending physical phenomena and substantiating theories, such as John Campbell's bifilms theory. However, it is important to acknowledge that certain results, like those obtained from EDS analysis, should be verified using other more advanced and precise research methods.

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