Comparison of Nonmetallic Inclusion Characteristics in Metal Samples Using 2D and 3D Methods

Yong Wang,* Andrey Karasev, and Pär G. Jönsson

The characteristics of different shapes of nonmetallic inclusions (NMIs) are determined using different methods. In this respect, various shapes of NMIs, including spherical, octahedral, elongated, bar-like, plate-like, polyhedral, and irregular inclusions, are observed in different steels and ferroalloys. The inclusions are investigated using three methods: 1) 2D investigations on a polished cross section (2D method); 2) 3D investigations on a film filter after electrolytic extraction and filtration (EE method); and 3) 3D investigations on a metal surface after electrolytic extraction (MS method). In addition, scanning electron microscopy (SEM) with an energy-dispersive spectrometer (EDS) is used for the determination of the chemical composition of inclusions. The advantages and limitations of different methods for investigations of different shaped inclusions are discussed. The results show that the 2D method is less precise to detect the morphology, size, and number of inclusions; however, the EE and MS methods are used to determine a more accurate morphology. Furthermore, the MS method is found to be more advantageous in detecting large-sized inclusions. This study also shows that the results on both the film filter and metal surface should be grouped together to obtain more comprehensive information of the inclusion characteristics.

1. Introduction

Nonmetallic inclusions (NMIs) are a matter of concern with respect to several properties of steels, such as fatigue, formability, toughness, machinability, and corrosion resistance. How to effectively investigate NMIs in steel is very important, so the research focusing on the determination of NMIs has always drawn a lot of attention. Thus, studies of fast, accurate, and direct determinations of NMIs have been the research focus for a long time. Zhang et al. reviewed over 20 different detecting methods and found that most methods have their own specific limitations, especially for detecting large-sized NMIs. It is well known that especially the largest NMIs have significant harmful effects on the mechanical properties of final products and are therefore important indicators of the quality of the steel. Although the large NMIs are very rare, their total volume fraction may be large. Therefore, the production of clean steel involves not only to control the mean inclusion content in steel but also to avoid NMIs larger than the critical size harmful to the product.

Various quantitative analysis methods of NMIs have been recommended by different research studies. Traditionally, the inclusion characteristics are investigated in two dimensions on polished cross sections of metal samples (2D method). This method is widely applied today, but the very low probability of finding large-sized NMIs strongly increases the number of fields that have to be examined. Moreover, it cannot show the real shapes of inclusions and the measured sizes of inclusions are smaller than their actual sizes. The difference is particularly larger for the irregularly shaped inclusions. Therefore, 3D investigations of the inclusion characteristics have become more important and interesting to use. The precise characterization of the shape requires an extraction of the inclusions from the steel matrix. Bulk sample electrolysis (slime) is one common method to investigate the 3D morphologies of inclusions. However, the number and size measurements are inaccurate because the inclusions are located together and therefore hard to separate. Furthermore, it can cause harm to inclusions due to its acidic electrolytic solution. Another disadvantage of this method is that the cluster inclusions possibly break into separate particles after extraction from steel. According to the previous studies, the 3D investigations of inclusions are widely applied using the chemical extraction (CE) method and the electrolytic extraction (EE) method. The CE method has a higher efficiency; however, some inclusions such as CaO, MgO, and REM oxides might be partially or completely dissolved during extraction. Garcia et al. reported that this method was not useful to identify inclusions having sulfur as part of their chemical composition. On the contrary, the EE method is a softer dissolution method, which has been proved to have major advantages in comparison with the CE and 2D methods. This is especially true for the evaluation of large-sized NMIs and clusters. The location of inclusions cannot be observed in the EE method because inclusions are removed from the metal matrix. Previous studies...
of the EE method usually considered the extraction of inclusions in the solution. However, the results on the metal surface (hereinafter referred to as MS method) are also very meaningful and interesting.

Recently, some researchers have paid attention to the inclusions present on the metal surface. Partial acid extraction of steels is one of the methods used to obtain the information of inclusions on the metal surface.\(^{18,19}\) However, it has the same disadvantages as the CE method. Zhang et al.\(^{20}\) reported that the 3D structure of sulfides in steels could effectively be exposed through electrolytic etching using nonaqueous solutions. According to the shapes and sizes of inclusions in different steel grades, the electrolytic parameters can be adjusted to obtain the real 3D morphologies of inclusions. In addition, this etching method was also applied for the determination of the morphology evolution of the inclusions in Mg-treated 16MnCr55 steels.\(^{21}\) Therefore, the inclusions on the metal surface can also give us useful information. In this case, inclusions that remain on the metal surface after a completed extraction should also be investigated regardless if some inclusions are partially exposed or remain as holes on the metal surface.

Steel samples obtained from specific steel grades are commonly used for inclusion analysis to evaluate cleanliness. However, there is not much attention paid to the cleanliness of ferroalloys. Therefore, the chemical composition of NMIs in ferroalloys is also very important to control as NMIs in ferroalloys can be inherited as NMIs in the steel and thereby decrease the steel cleanliness. However, the effect of the cleanliness of ferroalloys on steel quality is quite complicated and indefinable. It depends on different factors, such as the addition sequence, addition stage, and the behavior of inclusions after ferroalloy addition. These aspects will be investigated more in depth in a separate future study.

In this study, three types of steel and ferroalloy samples were studied using the 2D, EE, and MS methods. In addition, a pure iron (Fe) sample was used as a reference. The results of the characteristics of NMIs, such as morphology, composition, size, and number, obtained from these methods are compared. The objective of this work is to identify the most suitable methods for the investigation of different types of inclusions. Based on the comparison results, the limitations and advantages of the application of each method for analysis of NMIs are discussed.

### 2. Experimental Section

The investigations of NMIs were carried out using 11 metal samples: pure commercial Fe, low-alloy steel (LAS; lollipop-type sample taken from the liquid steel during ladle treatment), 42CrMo (42A-as-cast sample from ingot and 42C casting sample after rolling taken in rolling direction), and commercial FeV, FeCr (three samples of FeCr alloys), and FeSi (three samples of FeSi alloys). The samples were selected based on the obtained typical morphologies of inclusions. The typical chemical compositions of these samples are shown in Table 1.

First, each sample was polished and prepared to enable 2D observations of inclusions on a cross section of the metal surface using scanning electron microscopy (SEM) in combination with an energy-dispersive spectrometer (EDS). Then, the polished surface was dissolved using the EE method in a 10% AA electrolyte (10 v/v% acetylacetone–1 w/v% tetramethylammonium chloride–methanol). The following parameters were used for the electrolytic dissolution of the selected samples: an electric current between 28 and 70 mA, a voltage between 2.6 and 5.2 V, and an electric charge of 500 and 1000 C. The electrolyte after EE, which contained undissolved inclusions, was filtered using a polycarbonate (PC) membrane film filter with an open pore size of 0.4 µm. Thereafter, the characteristics of NMIs (such as morphology, composition, size, and number distribution) collected on the film filters were evaluated. The surface of the metal samples (MS) after EE was also used for inclusion investigations.

The equivalent diameter of a nonspherical inclusion was estimated as the diameter of a circle having the same area as an investigated inclusion.\(^{22}\) The average size of a nonspherical inclusion \((d_A)\) was calculated according to Equation (1). The harmonic mean diameter of the inclusions \((\bar{d}_A)\) measured on a cross section and the mean spatial diameter of the inclusions\(^{11}\) \((\overline{d}_A)\) were calculated using Equation (2) and (3), respectively.

\[
d_A = \frac{L_{\text{max}} + W_{\text{max}}}{2}
\]

\[
\overline{d}_A = \frac{n}{\sum \frac{1}{d_{Ai}}}
\]

### Table 1. Typical compositions of different samples investigated in this study (wt%).

| Sample   | Si  | Mo | Cr  | V  | Al | Mn | Mg | Ti | Ca | O  | C  | S  | P  | Fe  |
|----------|-----|----|-----|----|----|----|----|----|----|----|----|----|----|-----|
| Fe       | 0.01| –  | –   | –  | 0.11| –  | 0.005| –  | 0.18| 0.085| –  | 0.023| 0.009| 99.57|
| LAS      | 0.3 | 1.0| –   | –  | 0.05| –  | –   | –  | <0.001| <0.001| 0.18| 0.002| 0.011| 98.45|
| 42CrMo   | 0.28| 0.18| 1.05|<0.01| 0.04| 0.77| –   | 0.03| 0.021| 0.001| 0.42| 0.022| 0.018| 97.15|
| FeV      | 1.2 | –  | –   | 80.4| 3  | –   | 0.04| –  | 0.25| 0.714| 0.201| 0.021| 0.018| 14.15|
| FeCr-1   | 0.41| 71.8| –  | 0.05| 0.25| 0.006| –  | 0.04| 0.078| 0.025| 0.002| 0.09 | 27.25|
| FeCr-2   | 0.82| 0.04| 72.5| –  | 0.03| 0.65| 0.005| 0.01| 0.02| 0.065| 0.055| 0.008| 0.02 | 25.77|
| FeCr-3   | 0.65| 70.3| –  | 0.01| 0.55| 0.005| –  | 0.04| 0.069| 0.057| 0.007| 0.04 | 28.07|
| FeSi-1   | 72.3 | –  | –   | <0.3| 0.18| 0.22| 0.004| 0.065| 0.007| 0.127| 0.84 | 0.015| 0.013| 25.92|
| FeSi-2   | 72.8 | –  | –   | –  | 0.05| 0.25| 0.004| 0.1 | 0.011| 0.078| 0.13 | 0.012| 0.035| 26.53|
| FeSi-3   | 73.6 | –  | –   | –  | 0.24| 0.33| 0.003| 0.02| 0.008| 0.045| 0.35 | 0.017| 0.011| 25.37|
\[ d_v = \frac{\pi d_A}{2} \]  
\[ L_{\text{max}} \text{ and } W_{\text{max}} \text{ are the maximum length and width of the investigated inclusion measured by ImageJ software, respectively. Furthermore, } n \text{ is the total number of observed inclusions and } d_A, \text{ is the average size of ith inclusion.} \]

The numbers of inclusions per unit area (N_A) and per unit volume (N_V) were calculated using Equation (4) and (5), respectively. The N_V value can be recalculated from the N_A value according to Equation (6)

\[ N_A = \frac{n}{A_{\text{observed}}} \]  
\[ N_V = n \times \frac{A_{\text{filter}}}{A_{\text{observed}}} \times \frac{\rho_{\text{metal}}}{W_{\text{dissolved}}} \]  
\[ N_V = \frac{N_A}{d_v} \]

where \( A_{\text{filter}} \) is the area of the film filter with inclusions (1200 mm\(^2\)), \( A_{\text{observed}} \) is the total observed area on the film filter, \( \rho_{\text{metal}} \) is the density of the metal matrix, and \( W_{\text{dissolved}} \) is the dissolved weight of the metal during extraction.

The length, width, and height of each sample were measured before the extraction experiments. Also, an average dissolved depth (\( D_{\text{diss}} \)) of a sample was determined by using Equation (7)

\[ D_{\text{diss}} = \frac{W_{\text{dissolved}}}{\rho_{\text{metal}} \times A_{\text{surface}}} \]

where \( A_{\text{surface}} \) is the surface area of a metal sample, which is exposed to extraction solution.

3. Results and Discussion

3.1. Dissolution of Metal Samples during EE

It is well known that the EE method is a soft and stable extraction method, which is used for the dissolution of small amounts of metal samples (0.05–0.5 g).\(^{[12]}\) Figure 1 shows the dissolution rates and the dissolved depths obtained during the EE of the different samples investigated in this study.

It is shown in Figure 1a that Fe has the highest dissolution rate compared with the other samples, which indicates that Fe is easily electrolyzed. Moreover, the dissolution rates for ferroalloys are lower than those for steel samples. It is known that the metal sample is used as the anode, where the reaction at the anode is Fe \( \rightarrow \) Fe\(^{2+} \) + 2e\(^-\). In this case, the lower dissolution rate of ferroalloys can be explained by the fact that the Fe content in ferroalloys is much smaller than that in steels. FeCr (\( \approx 27\% \text{ Fe} \)) and FeV (\( \approx 14\% \text{ Fe} \)) alloys have similar ranges of dissolution rates (0.3–0.5 mg min\(^{-1}\)). The dissolution rates for steel samples vary from 0.7 to 0.9 mg min\(^{-1}\). In addition, it is clearly shown that the three FeSi alloys (\( \approx 25\% \text{ Fe} \)) have the lowest dissolution rates, which also correspond to the smallest dissolved depths (12–18 μm), as shown in Figure 1b. Even though the charge became larger (an increase from 500 to 1000 C), the dissolved weight of FeSi alloys did not increase significantly. The possible reasons for this will be discussed in Section 3.2. Clearly, a more stable dissolution rate is preferred to obtain a more reliable prediction of the aimed dissolved weight of the sample.\(^{[12]}\) However, the situation differs a little for ferroalloys because the values of the dissolution rates become smaller at the end of the electrolysis process for some ferroalloy samples, such as FeV and FeCr. Further systematic research is needed to explain this phenomenon.

The smallest dissolved depth was 73 μm, except for the FeSi alloys, as shown in Figure 1b. The obvious differences between the dissolved depths of different samples under similar dissolved weights were due to the different surface areas of the metal samples. Overall, these results can be used for the planning and controlling of future extraction experiments for different metal specimens, in which the required depth of a metal sample should be dissolved at a given time.

3.2. Investigation of Inclusion Morphology

The morphology of NMIs is an important characteristic, which provides information on their state of existence in the melt. The accurate investigation of the inclusion morphology can be used

![Figure 1](attachment:image.png)
for better investigations of the formation and transformation of inclusions and the effect on the mechanical properties of steels.

Previously it was reported\(^2\) that the conventional 2D method was less efficient to determine the morphology of inclusions compared with the 3D method. Moreover, the inclusions on the metal surface are highlighted because the initial electrolytic potential of the inclusions is higher than that of the metal matrix.\(^2\)\(^1\) The typical SEM images of inclusions obtained by the 2D, EE, and MS methods in Fe and different steel samples are compared in Figure 2.

It is clear from Figure 2 that the irregular Ca–Al–S–O inclusion in Fe determined using the 2D method is shown to have a true 3D morphology when using the EE method. Moreover, it is apparent that a large gap existed between the inclusion and the metal matrix when using the MS method. The possible explanation for this is that the dissolution of a metal matrix easily occurred around the inclusions, due to their different properties of the metal. Similar results were obtained for the Ca–Al–Mg–S–O inclusions in LAS. In most cases, these inclusions were removed from the matrix and remained in holes on the metal surface after EE. It has been reported that spherical inclusions can easily fall off.\(^2\)\(^8\) Only partial inclusions were visible on the metal surface after a completed extraction. Therefore, the precise information of morphology and size cannot be obtained accurately using the MS method. This indicates that the disadvantage of the MS method is more pronounced with respect to the observation of such globular-shaped inclusions.

The MnS inclusions in an as-cast sample of 42 CrMo steel were more of the regular polygon type, when they were determined using the 2D method. Here, they were randomly cut on the surface. However, they present their real octahedral or irregular shape when determined using the EE method. When using the MS method, almost a complete 3D morphology can be observed, although small partial inclusions cannot be visible. A previous research\(^2\)\(^4\) showed that a perfect octahedron with eight (111) crystal faces was always achieved. However, not all MnS inclusions reach the equilibrium state with a minimum surface energy and some even tend to appear to have irregular shapes.\(^2\)\(^5\) Thus, the different shapes of MnS inclusions found in this study can be explained from this point of view.

In the deformed steel of the 42 CrMo steel, the sample was cut in the parallel direction to the rolling direction, to accurately observe the elongated MnS inclusions when using the 2D method. It is clearly seen that some parts of the inclusions were exposed while some were invisible. Therefore, it was hard to distinguish whether they belonged to one single inclusion or if they were separate inclusions which were located closely together. In addition, some elongated inclusions had curvatures due to

| Sample | 2D | EE | MS |
|--------|----|----|----|
| Fe     | ![2D Image](image1) | ![EE Image](image2) | ![MS Image](image3) |
| Ca-Al–O | Ca-Al–S–O | Ca-Al–S–O |
| Ca–S   | 2 μm | 4 μm | 2 μm |
| LAS    | ![2D Image](image4) | ![EE Image](image5) | ![MS Image](image6) |
| Ca-Al–Mg–S–O | Ca-Al–Mg–S–O | Ca-Al–Mg–S–O |
| 42A    | ![2D Image](image7) | ![EE Image](image8) | ![MS Image](image9) |
| MnS    | MnS | MnS | MnS |
| 5 μm   | 30 μm |
| 42C    | ![2D Image](image10) | ![EE Image](image11) | ![MS Image](image12) |
| MnS    | MnS | MnS |
| 30 μm  | 60 μm |

Figure 2. Typical morphology of NMI analyzed using the 2D, EE, and MS methods in pure Fe and different steel samples.
the change of the deformation direction. Thereby, a precise measurement of the original sizes of them using the 2D method cannot be obtained. However, the real lengths of elongated inclusions can be accurately measured using the EE method. In the MS method, inclusions were not completely seen because some parts of inclusions dropped off after extraction, as shown in Figure 2. This can be explained by the observations that thin elongated inclusions can be broken during sample preparation, extraction, and washing of the steel specimen during 3D investigations. However, it was reported that about 70% of the elongated inclusions were undamaged after the extraction of this steel sample.

The typical SEM images of inclusions obtained by the 2D, EE, and MS methods in different ferroalloy samples are shown in Figure 3.

In FeCr alloys, polygonal and rectangle Cr–Mn–O inclusions observed in the 2D method showed their true polyhedral and bar-like shapes when determined using the EE method. The shapes of inclusions observed using the MS method were similar to those when using the EE method because the inclusions were almost entirely exposed to the surface. Furthermore, it should be pointed out that Si–Al–Ca–Mg–O, Al₂O₃, and Cr–O inclusions were also observed when using the EE method. However, they were not observed when using the 2D and MS methods. It means that the EE method can be used for detecting more types of inclusions that exist in alloys compared with the 2D and MS methods.

When it comes to FeV alloys, irregular Al₂O₃ inclusions located close to each other were easily observed by using the 2D method. However, they were found to be plate-like inclusions when using the EE method. Also, single plate-like Al₂O₃ inclusions were found to be located close to the metal surface after extraction, but they had different orientations. In the case of carbides of vanadium (VC), they look like clusters on a polished cross section. However, they had a rod-like shape when observed on a film filter after EE. In reality, they combined together in the

![Figure 3. Typical morphology of NMIs analyzed by the 2D, EE, and MS methods in different ferroalloy samples.](image-url)
matrix, which was clearly seen when using the MS method even though they were only partially visible. This illustrates that the MS method can give more information than the other two methods on morphology and location for some types of inclusions in the metal. In addition, the results showed that some irregular \( \text{Al}_2\text{O}_3-\text{CaO}, \ \text{Al}_2\text{O}_3-\text{MgO}, \ \text{and Al}_2\text{O}_3-\text{SiO}_2 \) inclusions were observed when using the EE method,\(^{14} \) but not when using the 2D and MS methods.

From the results of the 2D method, it was seen that the matrix of FeSi alloy was loose and porous and contained many cracks. The matrix mainly consisted of two phases, namely, a dark one consisting of a pure silicon phase and a light phase consisting of a ferrosilicon phase (54–71% Si), as shown in Figure 3. In general, the 2D investigations of inclusions in FeSi alloys were difficult to carry out due to the easy crushing and removal of inclusions during the polishing of ferroalloys. It should be mentioned that by the EE method, three types of inclusions were observed, namely, pure SiO\(_2\), SiC, and Si–Al–Mg–Ca–O complex oxides. The same inclusion types have been reported by other researchers.\(^{10,13} \) Also, when using the MS method, irregular SiO\(_2\) oxides were easily observed and they were located close to each other. Most SiO\(_2\) were distributed in a narrow area and further research is needed to explain this phenomenon.

An easy oxidation of Si on the metal surface and a formation of a thin layer of SiO\(_2\) might prevent the deep electrolysis of FeSi alloys. It should also be noted that the inclusions remaining on the metal surface have larger sizes than those obtained when using the EE method. A detailed comparison is discussed in Section 3.4.

On the basis of obtained results, it can be concluded that the EE method can detect more inclusion types than the 2D and MS methods. Moreover, the real morphologies of inclusions observed using the 3D method (EE and MS) are different from those using the 2D method. For instance, what seems to look like a rod-shaped inclusion in 2D may actually be a plate-like or an acicular inclusion when using the 3D method. However, the MS method can show the real locations and orientations for some inclusions in the metal matrix, especially for some alloys which cannot be extracted to large depths.

### 3.3. Analysis of Inclusion Composition

It is very important to know the exact composition of inclusions with respect to different analysis methods. It is known that the metal matrix can influence the composition determination when using the 2D method.\(^{16} \) Therefore, the contents of Fe obtained from the metal matrix in the inclusion composition as a function of inclusion size and the distributions of inclusion compositions in the (CaO–CaS)–Al\(_2\)O\(_3\)–MgO phase diagram are compared for different methods in Figure 4.

A previous research\(^{16} \) showed that the size 4 \( \mu \text{m} \) was an inclusion size above which the determination became more reliable for the 2D and EE methods. It is shown in Figure 4a that the content of Fe in inclusions decreases significantly with an increasing inclusion size in both the 2D and MS methods. This indicates that the effect of metal matrix on the composition analysis is higher for smaller inclusions (<6 \( \mu \text{m} \)). In addition, the contents of Fe in the composition results of inclusions obtained from the 2D method are much higher (about 4 times) than the results from the MS method for inclusions smaller than 6 \( \mu \text{m} \). This might be explained by the presence of cavities between the inclusions and the steel matrix, which decreased the effect of the matrix on the results during the composition determination when using the MS method.

To compare the accuracy of different methods, the compositions of Ca–Al–Mg–O–S inclusions obtained using the three methods are projected onto the (CaO–CaS)–Al\(_2\)O\(_3\)–MgO phase diagram, in which small amounts of CaS (6 mass % on average) are treated as being part of the CaO content. As shown in Figure 4b, the composition distribution of inclusions obtained from the 2D method deviates significantly from the liquid area, whereas most of the inclusions analyzed by the EE and MS methods are located within this area. This might be explained by the higher content of Al compared with the Ca and Mg contents in the steel matrix, which results in an underestimated concentration of Al\(_2\)O\(_3\) in small inclusions analyzed using the 2D method. Thus, the effect of the metal matrix on the composition determination for small-sized inclusions decreases in the following order: 2D, MS, and EE methods.

![Figure 4](image-url) a) Contents of Fe obtained from the metal matrix in inclusions with different diameters and b) composition of complex inclusions in LAS sample presented in a (CaO–CaS)–Al\(_2\)O\(_3\)–MgO ternary phase diagram for different methods.
When it comes to heterogeneous inclusions, it is difficult in many cases to determine the accurate composition of a surface layer with a thickness smaller than 1 μm using the 2D method. In this case, the EE method can be used for more precise determinations of the surface compositions of the inclusions without being influenced by the matrix. However, it is difficult to know their inner morphologies and compositions using the EE and MS methods. In this case, the 2D method is more preferable to determine the compositions of different phases inside the inclusions. One typical complex heterogeneous inclusion in Fe is shown in Figure 2. It clearly shows that the inclusion consists of CaO–Al₂O₃ and CaS phases.

3.4. Determination of Inclusion Sizes and Numbers

The size of inclusions is also a particularly important feature affecting steel properties, especially for large-sized inclusions. According to previous research, it is clear that the EE method has a major advantage in comparison with the 2D method with respect to the precise measurement of inclusion sizes. It is especially important for the investigation of elongated inclusions and clusters when their sizes depend on the morphology and cutting angle of the metal samples, when using the 2D method. Therefore, aspect ratios (ARs = length/width) are plotted against lengths for different shapes of inclusions obtained by the three methods in Figure 5. The corresponding characteristics (such as length range, \( N_A \), and \( N_V \)) of different types of inclusions are shown in Table 2.

It is shown in Figure 5a,b that the ARs for small-sized irregular and spherical inclusions are close to 1 for the 2D and EE methods, whereas they are larger for the MS method. One interesting point is that the percentage of inclusions (\( P = \) the number of inclusions whose length is larger than the average length obtained by the EE method/total number of inclusions) is nearly 3 times higher for the EE method than that of the 2D and MS methods. This might be explained by only random cross sections of the inclusions being observed and measured when using the 2D method. This resulted in the fact that the measured sizes were smaller than their real sizes. Furthermore, only partial sizes of the inclusions were measured because they were not completely exposed on the metal surface, in the MS method as shown in Figure 2. The 2D and EE methods show similar \( N_V \) values, which are significantly larger (about 30 times in Fe and 5 times in LAS) than those of the MS method. It is because most of the inclusions fell off from the metal surface when using the MS method. To conclude, the EE method is more recommended for the determination of small-sized inclusions compared with the other two methods.

Figure 5. A comparison of sizes of NMIs obtained in the pure Fe sample and different steel samples by the 2D, EE, and MS methods.
The three methods show similar ranges of ARs (1–3) for octahedral MnS inclusions, as shown in Figure 5c. The lengths of the observed inclusions vary from 1.9 to 38.6 μm, according to the three methods. In addition, almost the same results are obtained for the average length and P (Table 2) when using the 2D and EE methods, which are smaller than that of the MS method. It should be noted that only few smaller inclusions were observed when using the MS method based on the N_A and N_V values. It means that the MS method can observe only large-sized octahedral inclusions.

For elongated MnS inclusions shown in Figure 5d, the largest range is found when using the EE method (3.8–234.6 μm). For the 2D method, the size range is 1.6–84.6 μm. The AR, maximum length, and P values decrease in the following order: EE, MS, and 2D methods. The N_A values obtained by the 2D method are almost twice as large as those of the MS method. The numbers of inclusions in the 2D method might be overestimated due to single inclusions being considered as multiple inclusions because the visible parts of the inclusions are not continuous, as shown in Figure 2. To conclude, the EE method is more suitable for the investigation of elongated inclusions, although some inclusions can be broken during sample preparation or extraction. The reasons for the phenomenon of broken inclusions will be investigated more in depth in a separate study.

The main characteristics of different NMIs observed in various ferroalloys are shown in Figure 6 and Table 3. 

Cr–Mn–O inclusions in FeV alloys are divided into two groups based on their morphology, namely, polyhedron and bar-like shape inclusions. The polyhedral Cr–Mn–O inclusions are similar to the octahedral MnS inclusions (Figure 5c), and they show the same range of ARs (1–3) when determined using the three methods. In addition, the average length and P values also decrease in the order of MS, EE, and 2D methods. It should be pointed out that some Cr–Mn–O inclusions found using the MS method were broken (Figure 7a) or bonded together (Figure 7b). Therefore, they should be treated as one big inclusion, which corresponds to the points of MS stacked in Figure 6a. In this case, the lengths of multiple inclusions were easily measured by the MS method. However, only few multiple inclusions were found using the EE method. One possible reason might be that single Cr–Mn–O inclusions were bonded by precipitated Fe–Cr intermetallic phases, as shown in Figure 7b,c. These intermetallic phases can be dissolved during the EE. As a result, the inclusions were distributed randomly on the film filter after EE.

Thus, the location and maximum size of inclusions can be precisely investigated using the MS method, whereas the EE method is less suitable to use in this case.

In the case of bar-like Cr–Mn–O inclusions, similar average ARs (3.7–3.9) are found for the EE and MS methods, where the real morphologies can be observed. The MS method shows the largest length (70.6 μm) and P (60%), followed by the EE method and the 2D method. However, the smallest values of N_A and N_V for the MS method because most of them fell off from the metal surface during EE. It means that the MS method can help to find larger bar-like inclusions compared with the other methods, but at the same time it is more time-consuming.

In FeV alloys, the ARs of most plate-like Al_2O_3 inclusions determined by the EE method are smaller than those determined using the 2D and MS methods. The higher AR values obtained by the 2D method were due to the cutting surface along with the width of plate inclusions, whereas in the MS method, this might be due to different orientations of plate-like inclusions on the metal surface, which can cause an underestimated width of inclusions, as shown in Figure 3. In this case, it is important to determine the length of inclusions, which is more accurate. The 2D method shows the largest length of inclusions (491.3 μm), which is three times larger than that of the EE method (159.4 μm). One possible reason might be that these thin and large plate-like inclusions can easily be broken during the extraction of a specimen. Another explanation might be that these inclusions were located closely together or overlapped when they were observed using the MS method. Therefore, they can be overestimated when using the 2D method. Further research should be conducted with respect to this point. The average length and P values obtained by the MS method are almost twice of those obtained when using the EE method. It is also easier to observe the Al_2O_3 inclusions using the MS method based on the larger N_A and N_V values than that of the EE method.

When it comes to rod-like VC inclusions, the largest length increases in the following order: 2D, EE, and MS methods. The same trend is seen for the average length and P values. However, the average AR of inclusions for the MS method is smaller than that of the EE method. This can be explained by single VC inclusions being measured separately when using the EE method. In reality, a set of these inclusions is located together on the metal surface after an extraction. This, in turn,
results in a larger width of inclusions, as shown in Figure 3. The $N_A$ and $N_V$ values are significantly larger when using the 2D method compared with when using the other two methods. They cannot reflect the real situation of VC inclusions in the metal, because the number of VC inclusions is dramatically overestimated.

It can clearly be seen that the ARs of SiO$_2$ inclusions in FeSi alloys show similar ranges, whereas the lengths of them obtained by the MS method (up to about 186.5 μm) are significantly larger than those determined using the EE method (up to about 40.3 μm). One possible explanation is the difficulty of the electrolytic dissolution of FeSi alloy matrix according to the very low dissolved weight (<0.01 g) which resulted in the fact that most SiO$_2$ inclusions remained on the metal surface and only small-sized inclusions were extracted during EE and observed on film filter. Therefore, the MS method is more recommendable than

![Figure 6](image-url)
the EE method to be used for the investigation of larger inclusions, when the metal samples are hard to dissolve during the extraction.

In accordance with the obtained results of inclusion morphology, size, number, and location, the application of three methods with respect to different types of inclusions is shown in Table 4. It is shown that the EE method is the most preferred method to use for determination of the morphology of inclusions, which is followed by the MS method in most cases. For the size measurement of small inclusions, the EE method is the most recommended whereas the MS method is almost unusable. In addition, the EE method can successfully be applied for the investigation of elongated inclusions.

It should be noted that the size of inclusions determined by the MS method may be smaller than the real size because only the size of the exposed part of the inclusion can be measured. However, from the perspective of detecting the large-sized and maximum size of inclusions, the MS method is preferable compared with the 2D and EE methods. Moreover, the MS method can be successfully applied for the investigation of inclusions when their real information is only shown on the metal surface. Therefore, more focus should be paid to metal surface investigation after EE when we consider large-sized inclusions. For the inclusion number measurement, the EE method is the most suitable except for the cases of samples that are hardly dissolved.

Table 4. Application of three methods for investigations of different types of inclusions in metal samples.

| Type           | Morphology   | Size                 | $N_A$ ($N_V$) | Others                                      |
|----------------|--------------|----------------------|---------------|---------------------------------------------|
|                |              | Small                | Largest       |                                             |
| Spherical      | MS<2D<EE     | MS<2D<EE             | MS<2D<EE      | 1) For inner composition analysis of heterogeneous inclusions: MS<EE<2D. |
| Octahedral     | 2D<MS<EE     | 2D<EE<MS             | MS<2D<EE      | 2) For composition analysis of all small inclusions (<6 μm): 2D<MS<EE.   |
| Polyhedral     | 2D<MS<EE     | 2D<EE<MS             | MS<2D<EE      |                                             |
| Elongated      | 2D<MS<EE     | 2D<MS<EE             | MS<2D<EE      | 3) For size analysis of inclusions which locate together: EE<2D<MS.      |
| Bar            | 2D<MS<EE     | 2D<EE<MS             | MS<2D<EE      |                                             |
| Plate          | 2D<MS<EE     | 2D<EE<MS             | EE<2D<MS      |                                             |
| Rod (cluster)  | 2D<EE<MS     | 2D<EE<MS             | 2D<MS<EE      |                                             |
| Hard to dissolve | 2D<MS<EE   | 2D<EE<MS             | 2D<EE<MS      |                                             |
4. Conclusions

A study of the inclusion characteristics in steel and ferroalloy samples, such as morphology, composition, size, and number, was conducted using three investigation methods: 1) 2D observations of NMIs on a polished cross section of the metal specimen (2D method), 2) 3D investigations of NMIs on the surface of the film filter after EE and filtration (EE method), and 3) partial 3D investigations of NMIs on the surface of the metal sample after EE (MS method). The main merits and disadvantages for the applications of the 2D, EE, and MS methods for different types of inclusions were compared. According to the results obtained in this study, the following specific conclusions were obtained: 1) The 2D method can be applied for fast investigations of the inclusion characteristics. It is less accurate to detect the real morphology, size, and number of inclusions. This method is more suitable to use for the chemical composition determination of multiphase inclusions. 2) The EE method is more recommendable to determine the number and accurate morphology of inclusions and detect the accurate size of small-sized spherical inclusions and elongated inclusions. Furthermore, this method is more preferable to use to obtain an accurate chemical composition for small-sized inclusions (<6 μm) due to a smaller effect of the metal matrix. 3) A partial or full 3D morphology of the NMIs can be observed when using the MS method, depending on the specific conditions. This method is more advantageous to use when the aim is to detect the largest inclusions, such as polyhedral MnS and Cr–Mn–O inclusions, bar-like Cr–Mn–O inclusions, and plate-like Al2O3 inclusions. Sometimes the real locations or existence form of inclusions can only be obtained using the MS method, such as multiple Cr–Mn–O inclusions and sets of VC inclusions. Moreover, the advantage is more obvious for the investigation of inclusions using this method when the metal has hardly been dissolved, as for FeSi alloys. However, this method cannot be successfully used for the investigation of small-sized inclusions or to determine the number of inclusions. 4) In general, the EE method can show the complete types of inclusions in a sample, whereas the MS method can only detect inclusions that are relatively large in size and number. Overall, the results obtained by the EE and MS methods should be combined together to obtain more accurate information of the inclusion characteristics.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords

electrolytic extraction, ferroalloys, metal surfaces, nonmetallic inclusions

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[1] A. B. Schmiedt, H. H. Dickert, W. Bleck, U. Kamps, Acta Mater. 2015, 95, 1.
[2] X. L. Zhang, L. Zhang, W. Yang, Y. Zhang, Y. Ren, Y. C. Dong, Metall. Res. Technol. 2017, 114, 113.
[3] L. F. Zhang, B. G. Thomas, ISIJ Int. 2003, 43, 271.
[4] P. Kaushik, H. Pielet, H. Yin, Ironmak. Steelmak. 2013, 36, 561.
[5] J. Cheng, R. Eriksson, P. G. Jönsson, Ironmak. Steelmak. 2013, 30, 66.
[6] T. Hansén, P. G. Jönsson, presented at The 24th Electric Furnace Conf., Phoenix, AZ, USA, November 2001.
[7] T. Hansén, P. G. Jönsson, S. E. Lundberg, K. Törrøsvoll, Steel Res. Int. 2006, 77, 177.
[8] W. Yan, H. C. Xu, W. Q. Chen, Steel Res. Int. 2014, 85, 53.
[9] L. F. Zhang, B. G. Thomas, presented at Proc. of XXIV National Steelmaking Symp. Morelia, Michoacan, Mexico, November 2013, pp. 138–183.
[10] Y. Y. Bi, A. V. Karasev, P. G. Jönsson, Steel Res. Int. 2014, 85, 659.
[11] R. Inoue, S. Ueda, T. Ariyama, H. Suito, ISIJ Int. 2011, 51, 2050.
[12] D. Janis, R. Inoue, A. V. Karasev, P. G. Jönsson, Adv. Mater. Sci. Eng. 2014, 7.
[13] M. M. Pande, M. Guo, X. Guo, D. Gyesen, S. Devisscher, B. Blanpain, P. Wollants, Ironmak. Steelmak. 2010, 37, 502.
[14] Y. Wang, A. Karasev, P. G. Jönsson, Metals 2019, 9, 687.
[15] M. Nabeel, M. Alba, A. V. Karasev, P. G. Jönsson, N. Dogan, Metall. Mater. Trans. B 2019, 50, 1674.
[16] H. Doostmohammadi, A. V. Karasev, P. G. Jönsson, Steel Res. Int. 2010, 81, 398.
[17] M. Fernandes, N. Cheung, A. Garcia, Mater. Charact. 2002, 48, 255.
[18] Y. Ren, Y. F. Wang, S. S. Li, L. F. Zhang, X. J. Zuo, S. N. Lekakh, K. Peaslee, Metall. Mater. Trans. B 2014, 45, 1291.
[19] S. F. Yang, J. S. Li, Z. F. Wang, J. Li, L. Lin, Int. J. Min. Met. Mater. 2011, 18, 18.
[20] D. Zhang, P. Shen, J. B. Xie, J. M. An, Z. Z. Huang, J. X. Fu, J. Iron Steel Res. Int. 2019, 26, 275.
[21] J. B. Xie, D. Zhang, Q. K. Yang, J. M. An, Z. Z. Huang, J. X. Fu, Ironmak. Steelmak. 2019, 46, 564.
[22] A. V. Karasev, H. Suito, Metall. Mater. Trans. B 1999, 30, 259.
[23] X. Li, M. Wang, Y. P. Bao, L. D. Xing, J. H. Chu, Ironmak. Steelmak. 2018.
[24] W. Kurz, D. J. Fisher, Fundamentals of Solidification, Trans Tech Publications, Zurich 1989.
[25] Y. Liu, L. F. Zhang, H. J. Duan, Y. Zhang, Y. Luo, A. N. Conejo, Metall. Mater. Trans. A 2016, 47, 3015.
[26] Y. Kanbe, A. V. Karasev, H. Todoroki, P. G. Jönsson, Steel Res. Int. 2011, 82, 313.
[27] Y. Y. Bi, A. V. Karasev, P. G. Jönsson, ISIJ Int. 2013, 53, 2099.