Degree project in Materials Science and Engineering

Second Cycle, 30 credits

Investigation of Non-Metallic Inclusions In Inconel 718 by Using Conventional Cross-Sectioned (CS) Method and Experimental Extraction Metal-Surfaced (MS) Method

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Abstract

Inconel 718 is a nickel-based alloy, predominantly used in the oil and gas industry, in aircraft engines as well as in gas turbine engines. It provides exceptional properties like high strength, creep resistance, corrosion resistance and long fatigue life at high temperatures as well as in cryogenic environments. However, presence of non-metallic inclusions in the metal matrix affects these properties and reduces the quality of alloy. Therefore, the determination of inclusion characteristics like morphology, chemical composition, number, and size is very important to control the product quality. The purpose of this study was to find an effective method for investigating non-metallic inclusions characteristics in a short period of time. Two investigation methods were used: i) Conventional cross-sectioned (CS) method in which the sample is grinded and polished for 2D cross-sectional metallographic examinations under a scanning electron microscope (SEM), ii) New experimental extraction metal-surface (MS) method in which the sample is electrolyzed in a non-aqueous solution for a short period of time, followed by 3D metallographic examinations under a scanning electron microscope (SEM). Four types of non-metallic inclusions were observed in tested samples of Inconel 718 alloy named as; carbides, sulphides/carbosulphides, carbonitrides/nitrides and intermetallic carbides. It was also found that medium and large inclusions appeared more on MS and provides good precision of morphology and size compared to CS. However, it is not useful in finding the total number of inclusions, including small-sized inclusions. Inclusions compositional analysis showed no effect of metal matrix element on inclusions >6μm in both testing methods. From the findings, it is suggested to use MS method for analyzing medium and large-sized inclusions. But to achieve more accurate information, results from both methods should be combined.
Sammanfattning

1. Introduction
1.1 Non-Metallic Inclusions
Quality of finished products especially in the steel industry measured in terms of mechanical properties, fatigue life, toughness, corrosion resistance and formability [1]. These quality factors could be deteriorated by the presence of harmful non-metallic inclusions in the form of oxides, sulphides, carbides or nitrides in any type of steel and alloys. For instance, sulphide inclusions in high-strength steels reduce mechanical properties [2], oxide inclusions reduce fatigue strength in tool steels [3], nitrides and carbides inclusions located at grain boundaries decrease corrosion resistance in nickel-based alloys, Inconel 718 [4]. Thus, the control of non-metallic inclusion characteristics during production process and its evaluation in the finished product is very crucial for improving the processes and product quality.

The characteristics of non-metallic inclusions are mostly evaluated in terms of size distribution, number of particles, morphology and chemical composition [1-7]. These characteristics have either positive or negative effects on the final properties of steel product. For example, in superalloys, small inclusions like NbC located at grain boundaries prevent grain growth during heating and large size inclusions initiate crack and form fractures at high temperatures and low stresses [5]. Similarly, small nitride inclusions are not affected by harsh environments but nitride inclusions larger than 10μm decrease corrosion resistance in Inconel 718 alloy [6].

Different on-line and off-line methods have been used for the determination of non-metallic inclusions characteristics and found that each method has its own advantages and limitations [7–11], listed down in Table 1. However, the analysis accuracy obtained from electrolytic extraction (EE) is higher compared to all other methods [1]. At the same time, this method requires a long duration of time for extraction of inclusions [7, 12]. The other few disadvantages are: the location of inclusions cannot be observed because of observing inclusions on filter paper [1,7, 12], small inclusions on film filter might mix together to form an agglomerate [12,13], and difficult to obtain inner composition of inclusions [1,14,15].

Few researchers recently study non-metallic inclusions on a metal surface (MS) after short electrolytic extraction on low alloy steels and ferroalloys (FeCr, FeSi) [1], EP718 and Inconel 718 [4,6], high sulphur 1215 steel grade [16], and Incoloy 825 [17], to obtain reliable results in a short period of time. In this method, the sample is electrolyzed for a few minutes in a non-aqueous solution by adjusting charge and voltage parameters. It is less time-consuming and provides good information regarding the
location, size distribution, chemical composition and morphology of inclusions [1,4,6,16]. However, more studies are required regarding inclusion characteristics on metal surfaces of different types of steels and alloys for the accuracy validation of this method.

<table>
<thead>
<tr>
<th>Method</th>
<th>Analysis Time</th>
<th>Test Sample Area/Volume</th>
<th>Advantages</th>
<th>Disadvantages</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total Oxygen (on-line testing method) [7,9,10,11]</td>
<td>~ 1-2 min</td>
<td>1 - 3 g</td>
<td>Short analyzing time, quick and easy method</td>
<td>Only applicable for oxide inclusions, provides no information on morphology and chemical composition of inclusions, represent micro-inclusions</td>
</tr>
<tr>
<td>Ultrasonic test (on-line) [7,9,11]</td>
<td>1-2 hr</td>
<td>Large volume</td>
<td>Provides information about inclusions amount, size and location</td>
<td>Time-consuming, lacks details of inclusion morphology and chemical composition, identify defects as inclusions</td>
</tr>
<tr>
<td>Automatic Image Analysis Analysis [7,9,11]</td>
<td>2-3hr</td>
<td>10-20mm²</td>
<td>Large quantitative data regarding size, location, number of particles</td>
<td>Not suitable for very small inclusions, microscope features could limit the analysis</td>
</tr>
<tr>
<td>Electrolytic Extraction(EE) + SEM (off-line) [7,9,10,11]</td>
<td>3hr-17hrs</td>
<td>0.1-3g</td>
<td>Large quantitative information of inclusions size, number, morphology and composition, high accuracy</td>
<td>Time consuming, lacks details of inclusions location in test sample, intensity of labor</td>
</tr>
<tr>
<td>Metallographic Examination +SEM (off-line)[7,9,10,11]</td>
<td>~ 1-2 hr</td>
<td>Thin Section</td>
<td>Large quantitative information of inclusions size, number, morphology and composition</td>
<td>less accuracy of size and morphology, influence of operator specifications in inclusion observations</td>
</tr>
</tbody>
</table>

1.2 Nickel-Based Alloy

Nickel-based alloys are widely used superalloys in industries like aerospace, petrochemical, oil and gas, medical, space industry and nuclear plants [18–21]. In fact, the total weight of aircraft engines constitutes 50% of nickel-based alloys [18]. The reason for wide application is because of providing unique properties of fatigue strength, creep strength, and resistance to corrosion at high temperatures and under extreme conditions [18–21].
1.2.1 Inconel 718

Among many nickel-based alloys, Inconel 718 is one of the most commonly used alloy in the oil and gas industry as drilling equipment [21] due to its ability to form a passive layer over the surface under heat and pressure in cryogenic environments, providing excellent resistance to corrosion. It is also used in aircraft engines as disks, casing, rings, blades, shafts and in gas turbine engines [19,22] due to the exceptional properties it possesses like high strength, creep resistance and long fatigue life at high temperature (~750°C) [18,19]. CF-6 engine of US Air force aircraft exhibits 34% of Inconel 716 compared to other alloys, shown in Figure 1.

![Figure 1 Wt.% of Inconel 718 in CF6 Engine for US Aircraft w.r.t. Other Alloys]([5])

1.2.2 Production of Inconel 718

Conventional production of Inconel 718 involves melting and casting, followed by heat treatment. Melting is carried out in a vacuum induction furnace (VIM), Electroslag remelting (ESR) or Vacuum arc remelting (VAR) [19]. In vacuum induction furnace (VIM), raw-material such as scrap, reactive materials and virgin materials (material never used in vacuum melting before)[18] are melted in the furnace by the application of induction current, producing melt of required chemical composition. It is then poured into a mould for ingot casting (mostly known as electrodes) while still under vacuum. This semi-finished electrode is either remelted in ESR/VAR or used for further processes. The main advantage of VIM is that it considerably reduces oxygen, hydrogen and nitrogen gas contents in the alloy [19].
In electroslag remelting (ESR), the electrode obtained from VIM is remelted and refined by the heat produced from electrical resistance in slag. Its main advantage is the capability of controlling the solidification process and chemical homogeneity simultaneously in the ingot [19,23].

In vacuum arc remelting (VAR), electrodes from VIM/ESR are remelted under vacuum atmosphere. It produces highly homogenized and cleaned material because of controlled melting and solidification process [18]. The main difference between VAR and other processes is the absence of slag. Main production melting route for Inconel 718 is either using double-melt method i.e., VIM+ESR, VIM+VAR, or triple-melt method i.e., VIM+ESR+VAR [18,19] because of the requirement homogeneity structure and high quality of alloy. Later, the mould casting electrodes undergo one or more heat treatments to homogenize the compositional gradient and produce the preferred microstructure [23].

Heat treatment of Inconel 718 is performed by solution annealing, followed by precipitation hardening [5]. In solution annealing, alloy is heated up to 1065°C for a time duration of approximately 4 hours, followed by air/nitrogen cooling [5,24]. Its objective is to dissolve laves phase or other precipitates (e.g. TiN) by atomic diffusion back into steel matrix, that may be formed during alloy production. Precipitation hardening also known as ageing or age hardening is performed after solution annealing. Its purpose is to allow the formation of secondary phases (e.g., $\gamma'$, $\gamma''$) within the microstructure to strengthen the alloy. Figure 2 shows the heat-treatment process for Inconel 718.

![Figure 2 Heat-Treatment Process for Inconel 718][5]

1.2.3 Phases in Inconel 718

The chemical composition of Inconel 718 has a lot of alloying elements, producing a very complex microstructure in age-hardened conditions. It contains primary and secondary phases. Primary phases are $\gamma$, carbides (MC) and Laves phases, which forms during the solidification of alloy. While the
secondary phases (also known as intermetallic phases) are \( \gamma' \), \( \gamma'' \) and \( \delta \), which precipitates after solidification, in post-heat treatment processes. \( \gamma' \) and \( \gamma'' \) provide strength to the alloy. The composition of \( \gamma'' \) and \( \delta \) is same but different crystal structure, as mentioned in Table 2. Above 750 °C during aging, \( \gamma'' \) starts to convert to \( \delta \), a stable brittle phase and mostly formed at grain boundaries [5]. Sometimes \( \delta \) helps in pinning grain boundaries but its higher amount in alloy would reduce Nb in metal matrix and thus prevent formation of \( \gamma'' \), decreasing the strength [5] and fatigue life [26] of alloy.

<table>
<thead>
<tr>
<th>Phase</th>
<th>Crystal Structure</th>
<th>Formula</th>
<th>Solvus Temperature, °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \gamma )</td>
<td>FCC</td>
<td>Ni</td>
<td>1260-1364</td>
</tr>
<tr>
<td>( \gamma' )</td>
<td>FCC</td>
<td>Ni(_3)(Al,Ti)</td>
<td>850-910</td>
</tr>
<tr>
<td>( \gamma'' )</td>
<td>BCT</td>
<td>Ni(_3)Nb</td>
<td>910-940</td>
</tr>
<tr>
<td>( \delta )</td>
<td>Orthorombic</td>
<td>Ni(_3)Nb</td>
<td>1020</td>
</tr>
<tr>
<td>MC</td>
<td>Cubic</td>
<td>(Nb,Ti)C</td>
<td>1267-1305</td>
</tr>
<tr>
<td>Laves</td>
<td>Hexagonal</td>
<td>Fe(_2)Nb, Fe(_2)Ti, Fe(_2)Mo</td>
<td>1150</td>
</tr>
</tbody>
</table>

Precipitate phases like Laves and carbides are critical to Inconel 718 properties. Laves phases are produced by segregation of Nb during solidification and decrease the alloy strength by inhibiting \( \gamma'' \) formation. However, Laves phase disappears in post-heat treatment processes at temperature 1150° C with increasing homogenization time (~28h) and this happens by the back-diffusion of atoms from Laves phase into the metal matrix without local melting [25,34]. Primary carbides (MC) are mixed of (Nb,Ti) C or NbC in Inconel 718, that start precipitating when the alloy is almost 50% [35] or 70% [33] solidified. The precipitation is driven by the segregation of carbon and Nb during primary solidification. Figure 3 [36] shows the solidification pattern of MC carbides in Inconel 718. It begins with the formation of \( \gamma \) dendrites and enriching the liquid with Nb and C. As the \( \gamma \) grows and enrichment of Nb and C exceeds their solubilt, NbC particles starts to precipitate in the interdendritic regions at carbide precipitating-start temperature and continues precipitating until it reaches \( \gamma/NbC \) eutectic temperature (L=\( \gamma+NbC \)) [33]. This eutectic reaction consumes majority of C, therefore the remaining liquid forms \( \gamma \) again. With growing \( \gamma \), segregation of Nb in remaining liquid promote another eutectic reaction L=\( \gamma+\)Laves at which solidification
terminates. $\gamma$/NbC does not appear at lower temperatures, since the available carbon is not enough for the formation of NbC \cite{36}.

In addition, the size of carbides appeared within the temperature range of carbide precipitating-start temperature and $\gamma$/NbC eutectic is usually that to be found in end alloy product \cite{33,36-38}. This is because the size-growth of carbides particles depends upon the local solidification time (cooling rate) and the trapping ability of carbide forming elements in the inter- secondary $\gamma$ dendrite arms and in the bottom mushy zone \cite{37,38}. For instance, the carbides tend to grow in the liquid until it is completely trapped by $\gamma$ dendrites \cite{37}. This also affects carbide morphology, changes from octahedron to discrete blocky shapes or irregular shapes \cite{35-38}

The primary carbides are reported to be distribute in a non-uniform manner within the grains and at the grain boundaries as well \cite{35-39}. In one study it is reported that $\gamma$ phase get super-saturated by carbon during solution annealing and quenching and subsequent aging relieved this phase by the formation of carbide precipitates, exclusively nucleated on grain boundaries \cite{39}. The carbides in the grains also preferred to nucleate at dislocations, hindering their movement. It is also reported that MC carbides tend to nucleate on TiN particles during primary solidification \cite{34-39}.

Carbide particles have either positive or negative effects on Inconel 718 properties. Small carbides at grain boundaries prevent the growth of grains during heat-treatment \cite{18,23}. However, a large number of carbides in alloy would reduce Nb content in metal matrix and inhibit the formation of $\gamma'$ and $\gamma''$ phase, reducing the strength of alloy \cite{5}. Another reason is that these carbides are difficult to diffuse
back into the metal matrix without remelting material because of high solvus temperature (1260-1300°C) [5, 26]. It is also reported that large size and more amount of carbide particles on grain boundaries would reduce ductility and toughness because of easily propagation of microcracks along carbides during plastic deformation [40]. In another study, carbides tend to crack during machining, as NbC is 4 times harder than TiC [27]. This surface cracking tendency reduce fatigue life of alloy 718. Therefore, carbides in the shape of small equiaxed are preferred in conventional nickel-based alloys [28].

Several studies have focused on non-metallic inclusions in Inconel 718, that plays an important role in deteriorating strength and corrosion resistance properties [4, 6, 15, 21, 25, 26, 28, 40], thus reducing the quality of finished alloy. Some of the non-metallic inclusions found in this alloy are nitrides, carbonitrides, secondary carbides (M_{23}C_6, M_6C), primary carbides (MC) and sulphides. In one study, the effect of these inclusions on the corrosion property of alloy is found in the following order: sulphides > M_{23}C_6 > M_6C > Primary carbide (Nb, Ti)C > Nitrides(Nb,Ti)N [21]. M_{23}C_6 appears on dislocations at very low temperatures and promotes hydrogen embrittlement in extreme hydrogen-containing environments [26]. While primary carbides prefer to precipitate on grain boundaries and dislocations during primary solidification. They found to be distributed heterogeneously in alloy and reduces ductility and toughness of alloy because of propagation of microcracks along carbides [40]. However, it is also reported that large size carbides and nitrides in the size range of 2-30 μm and 9-27 μm respectively affect deleteriously the corrosion resistance of alloy Inconel 718 [6].

In another study [15], carbonitrides as non-metallic inclusions in Inconel 718 were found in the size range of 5 to 15 μm, which are harmful to the alloy during deformation processes. By adding Ca in the liquid melt of Inconel 718 during electroslag refining, the amount of carbonitrides is minimized, because it modifies the morphology of oxides and reduces the nucleating sites. Hence, inclusions could be modified in the liquid melt by investigating their characteristics and could contribute to enhancing the quality of the finished alloy. Therefore, the focus of this study is also to understand the characteristics of non-metallic inclusions in terms of their size and composition.

1.3 Comparison of 2-Dimensional and 3-Dimensional Method

In Table 1, few of the current testing methods for characterization of non-metallic inclusions are summarized. Each method has its own advantages and disadvantages. The testing method is considered best for the investigation of inclusions characteristics when it provides statistically accurate and reliable results in a short period of time. Therefore, two testing methods i.e., conventional cross-
sectioned (CS) and new experiment extraction on metal surface (MS), are investigated and compared in this study, since various methods present various results sometimes on the same samples.

1.3.1 CS-Method (2-Dimensional)
The conventional way of investigating inclusion characteristics is a two-dimensional method, in which the sample is grinded and polished for cross-sectional metallographic examinations under a scanning electron microscope (SEM). This method is named as CS-method in this study. It is reported that this method provides good information regarding the location and inner composition of inclusions [14]. However, the metal matrix has a higher effect on inclusion compositional data for inclusions smaller than 6μm using CS method [1]. Another few limitations are that the true size and morphology of inclusions are difficult to obtain by CS-method [1,6,7,16], because the inclusions are cut cross-sectionally during grinding and polishing and it shows apparent size instead of inclusion true size during metallographic examinations, as shown in Figure 4.

![Diagram of CS-Method](image)

Figure 4 Schematic illustration of apparent size of single and cluster inclusions in CS-Method

1.3.2 MS-Method (3-Dimensional)
MS method is predominantly used for three-dimensional analysis of inclusions [7]. In this method, a sample is electrolyzed in a non-aqueous solution for a short period of time. The metal matrix dissolved in electrolyte and inclusions remains intact on the metal surface. This method is named as MS-method in this study.

It is reported that the MS-method is more reliable for analyzing morphology and relative-true size of large inclusions [1], as the partial size and morphology is exposed on the metal surface by the dissolution of metal matrix during electrolytic extraction, shown in Figure 5. But the total number of inclusions is difficult to obtain in MS, as small inclusions might fall into electrolyte during electrolysis [1,7,16]. Therefore, this method is not advantageous for the determination of total number of inclusions.
Moreover, it provides the real location and orientation of inclusions in metal matrix [1,16]. It is also reported that the MS is a better method for analyzing the chemical composition of inclusions larger than 4μm compared to the CS-method because of the cavity created by the dissolution of metal matrix around inclusion and shows less or no effect of metal matrix elements [1].

Figure 5 Schematic Illustration of Inclusions on Metal Surface after Electrolytic Extraction (MS)

1.4 Aims and goals
In this study, two samples of Inconel 718 from different manufacturing companies were investigated for non-metallic inclusions characteristics.

The purpose of this study is to:

i. Investigate non-metallic inclusions characteristics (morphology, size distribution, number, location and chemical composition) on the cross-sectional surface of polished samples (CS) and on metal surface after short electrolytic extraction (MS)

ii. Comparison between CS-method and MS-method

iii. Finding out which method is better for investigating non-metallic inclusion characteristics

1.5 Environmental Aspect
The quality of steel depends upon non-metallic inclusions characteristics like size, morphology, number, composition and distribution because they affect mechanical properties, fatigue life, toughness, corrosion resistance, formability and many more. For instance, microcracks easily propagate along carbides located on grain boundaries, that in turn will reduce ductility and toughness of Inconel 718 alloy. Their precipitation in the alloy during solidification would be suppressed by lowering carbon content in liquid melt. Thus, analyzing their characteristics will help process operators in controlling the chemical composition of liquid melt on the melting platform. From an environmental perspective, this will help in reducing the energy required in post-heat treatment processes.
2 Experimental Procedure

2.1 Sample Material

In this study, two similar grades of Nickel-based Alloy (Inconel 718) produced by different manufacturing companies were examined for non-metallic inclusions characteristics by using CS-method (2D) and MS-method (3D). The samples were taken from the finished alloy. Typical chemical composition of the Inconel 718 is mentioned in Table 3 [6,42].

<table>
<thead>
<tr>
<th>Wt %</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Nb</th>
<th>Ti</th>
<th>Al</th>
<th>C</th>
<th>P</th>
<th>S</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wt %</td>
<td>50-55</td>
<td>17-21</td>
<td>2.80-3.30</td>
<td>4.75-5.50</td>
<td>0.65-1.15</td>
<td>0.20-0.80</td>
<td>≤0.08</td>
<td>≤0.015</td>
<td>≤0.015</td>
</tr>
</tbody>
</table>

The exact elemental composition of the tested samples from the manufacturers was not known. All the experimental procedures were performed in KTH laboratories. Initially, samples were prepared before performing any experiments and metallographic examination on them.

2.2 Sample preparation

The purpose of sample preparation is to provide ease for handling samples while doing experiments like polishing and metallographic examination afterwards. Each sample is prepared in a Mounting press machine using thermosetting powder (bakelite). The procedure for mounting sample is mentioned in Appendix A.1.

2.2.1 Cross-Sectioned Method (Polishing)

After mounting, samples were polished for cross-sectional metallographic examination of non-metallic inclusions.

The purpose of polishing is to remove scratches and stains on the sample surface that may be produced during cutting. The second reason is to produce a flat reflective surface for subsequent accurate metallographic examinations.

Initially, samples were coarse grinded on a rotating disc, and water is used to prevent sample heating while grinding. They were grinded on 280, 460, 620 and 1000 grit emery papers. Once the scratches were reduced and noticed lesser, the samples were polished afterwards to give a shiny and mirror finish to the surface. While polishing, a diamond suspension of 3µm and 1µm was used.

After polishing, the samples were examined under scanning electron microscope for metallographic examinations.
2.2.2  Metal-Surfaced Method (Electrolytic extraction)

For MS method, electrolytic extraction method was used. For electrolytic extraction, the polished samples were initially dismounted from bakelite powder. Afterwards, the length, height and diameter of metal samples were measured using a digital vernier caliper as can be seen in Figure 6. Square shape sample is sample A and circle one is sample B. Both samples were washed with acetone and benzene solvent in the ultrasonic bath. This cleaning is carried out to remove any remained bakelite powder particles or other stains on a sample surface. After proper cleaning and drying, sample weight was measured using digital weight balance (error ≈ 0.00005g).

Figure 6 Sample A and B after cleaning

Figure 7 displays set-up for the electrolytic extraction experiment. Before setting this apparatus, the beaker, platinum ring, tong and lid were cleaned three times with tap water, distilled water and methanol in order to prepare dust-free equipment for electrolysis. The beaker was filled with 250 ml of 10% AA electrolyte (10% acetylacetone+ 1% tetramethylammonium chloride + rest methanol). Inclusions like carbides, nitrides and sulphides are not dissolved in this electrolyte, as they remain intact with metal surface and the metal matrix around inclusions dissolved [6,29-30].

The level of electrolyte in beaker was kept higher than the sample to perform electrolysis on the whole surface. One-side dissolution was used, which means that only polished surface of sample was exposed for dissolution of metal matrix. In Figure 7a, discolouration of electrolyte indicates the start of dissolution of the metal matrix from sample A surface. For sample B, the same electrolyte was used as for sample A after short extraction of sample A, that’s why it appears in darker brown colour.

This extraction took place around 8-12 mins with the following electrical parameters: an electric current 43mA, a voltage 3.3 and an electric charge up to 20 C. After extraction, the final weight of both samples was measured again to determine the dissolved depth ($D_{dis}$).
The total weight of dissolved metal for samples A and B after electrolytic extraction was approximately 0.0048g and 0.0039g respectively, which corresponds to 2.8μm and 3.6 μm of the dissolved metal layer.

Figure 8 shows the surfaces of samples A and B after electrolytic extraction.

2.3 Sample analysis

For metallographic examinations, samples were analyzed using a scanning electron microscope (S-3700N Hitachi).
2.3.1 *Scanning Electron Microscope (SEM)*

Scanning Electron Microscope (Hitachi S-3700N) was utilized to investigate inclusion characteristics like the morphology and chemical composition. For compositional analysis, an integrated system of SEM known as an energy dispersive spectrometer (EDS) was used. While taking pictures on SEM, back-scattered electron (BSE) mode was utilized because it helps in analyzing the surface structure of inclusions and distinguishing different phases. Typically, the heavier element appears as a bright phase and the lighter element appears as a dark phase on SEM image. This is because the heavier element emitted more back-scattered electrons due to their bigger nuclei compared to lighter elements [11]. Figure 9 shows a typical SEM image on the polished surface and metal surface of a sample after short electrolytic extraction using BSE mode.

![Figure 9 SEM image using BSE mode on polished surface(a)and metal surface after short electrolytic extraction(b)](image)

For analysis of inclusions, two magnifications were selected (500X and 1000X) because it is dependent upon inclusion number, size and area analyzed. At lower magnification, more large inclusions can be analyzed accurately because it provides a wider area to assess more inclusions compared to large magnifications. At large magnification, small size inclusions can be analyzed more accurately as it provides a smaller area to assess inclusions more closely. The results of both magnifications were combined to get the estimated final size distribution of inclusions. The working distance was around 11.5 mm and the accelerating voltage was 15kV.

During investigation, systematic and subjective observation method was used. Systematic observation method means that the pictures were taken continuously from the bottom towards top zone of a sample. This approach will help in reducing observer error and give more accurate and representative data for analysis of number and size distributions of typical inclusions. Subjective observation method means that few typical inclusions were selected subjectively in different zones of the observed sample and examined their chemical composition by EDS.
In general, inclusions analyzed in the center by EDS system provides a more precise elemental composition, as can be observed in Figure 10 (b). However, the elemental composition analyzed at the outer surface of inclusion can be affected by steel matrix elements. This can be seen in Figure 10 (c), which shows spectrums for Cr, Fe and Ni. Few other limitations of SEM-EDS are that it does not measure the correct amount of light elements like nitrogen, carbon and oxygen [11]. Sometimes, it is also hard to quantify the composition of micro-inclusions similar or smaller to the size of electron interaction beam and gives often inaccurate results [11,12].

![Figure 10](image1.png)

*Figure 10 (a) SEM image of nitride inclusion covered by NbTi-C on polished surface, (b) and (c) EDS Spectrum*

### 2.4 Measurement of Size and Number of Inclusions

After taking pictures from SEM, the area of inclusions was measured using software called ImageJ. The Polygon Selection Tool was used to measure the inclusion area manually, as shown in Figure 11. The dark black shaded region around inclusion(crater) shows a gap between inclusion and metal matrix in Figure 11(b), which indicates the successful dissolution of the metal matrix around inclusion during electrolytic extraction.

![Figure 11 Schematic Illustration of Inclusion Area Measurement in CS(a) and MS(b)](image2.png)
To classify different inclusions based on size, equivalent diameter ($d_{eq}$) is calculated using below equation.

$$d_{eq} = \sqrt{\frac{4 \times A}{\pi}}$$  \hspace{1cm} (1)

$A$ = Projected area of inclusion calculated by ImageJ software

The equivalent diameter is used to project the size of irregular particles and clusters to a sphere of equivalent area. It gives the estimated equivalent diameter of spherical inclusions.

The number of inclusions per unit area ($Na$) in the sample is calculated using below equation.

$$N_a = \frac{n_i}{A_{obs}}$$  \hspace{1cm} (2)

where,

$n_i$ represents number of inclusions observed and $A_{obs}$ is the total observed area.

The dissolved depth ($D_{dis}$) of each sample is calculated by following equation.

$$D_{dis} = \frac{W_{dis}}{\rho_{me} A_{sur}}$$  \hspace{1cm} (3)

where $W_{dis}$ is the weight dissolved of sample during electrolytic extraction, $\rho_{me}$ is density of sample Inconel 718 (0.00817 g/mm$^3$) [31,32], $A_{sur}$ = surface area of sample
3 Results
3.1 Methodology Analysis

Methodology analysis helps to find out how many inclusions/particles should be observed in samples to get an error ($\%A_{\text{cum.Na}}$) of less than 5%. Further, it will set a requirement for 3D investigation of inclusions after electrolytic extraction on samples surface (MS).

Polished sample A and sample B were systematically analyzed two-dimensionally (2D) for non-metallic inclusions. The investigation of inclusions was carried out at two magnifications i.e., 500X and 1000X. The size of inclusion was then measured manually using ImageJ software because it is open-platform software and operator can easily distinguish between hole and inclusion for measuring its exact size manually. The minimum size of analyzed inclusions was set to 1\(\mu\)m. Figure 12 shows typical inclusions found on polished surface of sample A at 500X and 1000X.

![Figure 12 Typical inclusions on Polished surface of Sample A at (a)500X and (b)1000X](image)

The typical particle size distribution of inclusions by the CS method for sample A and sample B is shown in Figure 13. It can be seen that at 1000X number of small inclusions(<2\(\mu\)m) per area appeared more compared to 500X in both samples. It is obvious that larger magnification helps in analyzing smaller inclusions more precisely because of analyzing smaller area more closely. At 500X, inclusions larger than 5 \(\mu\)m are analyzed more in sample B comparatively at 1000X. This is because smaller magnification provides a wider area to analyze numerous and big inclusions precisely. However, in sample A, the particle size distribution for inclusions larger than 6 \(\mu\)m is scattered at both magnifications. It may be due to the inhomogeneous distribution of inclusions in sample A. The second reason might be the total number of inclusions per area in sample A is less compared to sample B which suggests that sample A is cleaner steel compared to sample B.

In order to get a good estimate of the total number of inclusions per unit area (T.Na), a combined curve of particle size distribution was produced for both samples as shown in Figure 13. From a
statistical point of view, higher magnification helps in measuring a sufficient number of smaller inclusions more accurately and lower magnification helps in analyzing larger inclusions more accurately. Therefore, T.Na for inclusions less than 2\( \mu \text{m} \) for both samples is calculated by using 1000X and for inclusions larger than 5\( \mu \text{m} \) is calculated using 500 magnification. T.Na for inclusion size between 2-5 \( \mu \text{m} \) is calculated using equation 4, to get an average value of inclusions in the mentioned range. The combined curve for sample B shows more inclusions larger than 6 \( \mu \text{m} \) compared to sample A. This also suggests that sample A is clean steel comparatively sample B.

\[
T \cdot Na = \frac{n_{(500X)}+n_{(1000X)}}{A_{obs(500X)}+A_{obs(1000X)}} \quad (4)
\]

Based on particle size distribution, more area for sample A is analyzed at 500X (i.e., up to 2.84mm\(^2\)) because of inhomogeneous distribution of inclusions. This gave an understanding of how much inclusions should be observed in order to get an error of less than 5 \% for subsequent investigations. The error percent of cumulative Na (\(\%\Delta_{cum,Na} \)) is calculated by the following equation.

\[
\%\Delta_{cum,Na} = \frac{\overline{Na} - Na_{cum}}{\overline{Na}} * 100 \% \quad (5)
\]

where \(\overline{Na} \) is the average number of inclusions per area, \(Na_{cum} \) is the cumulative number of inclusions observed per successive increasing area.

Figure 14 illustrates typical SEM images of inclusions with different sizes in sample A at 500X in CS method.
Figure 15 shows $Na_{cum}$ and $%\Delta_{cum,Na}$ as a function of a number of inclusions observed ($n_{inclusions}$) and each figure in respective rows correspond to one size range. It can be observed in Figure 15a that for inclusions with the size of 2-3μm, $Na$ values are widely spread out from $\overline{Na}$ for below 110 observed inclusions. It may be because these small inclusions formed during solidification in ingot, and were unable to move freely, therefore not homogeneously distributed. However, with increasing observed inclusions, it comes closer to $\overline{Na}$. Meanwhile, less than 5 percent of error for cumulative Na ($%\Delta_{cum,Na}$) is achieved for number of inclusions observed above 120, which means that observing more than 120 number of inclusions for 2-3 μm size inclusions would be statistically enough for further analysis.

With increasing inclusion size like 3-4μm and 4-5μm, the $Na_{cum}$ values spread closely near to $\overline{Na}$, which means that $\overline{Na}$ is representative of the data and it shows less than 5% error of $Na_{cum}$ for number of inclusions observed above 84 (Figure 15d) and 86 (Figure 15f) respectively. This indicates that the number of inclusions observed in the size range of 3-4μm and 4-5μm is statistically significant for analysis and distributed more homogenous in the sample compared to small inclusions.

For inclusion sizes like 6-7μm and 8-9 μm, the $Na_{cum}$ is a bit scattered out from $\overline{Na}$ but with an increasing number of observed inclusions, it comes closer to $\overline{Na}$, as can be seen in Figure 15 (g) and (i). The reason for some variation in data may be that the inclusions are not homogenously distributed, shown in Figure 16, which displays the uneven distribution of observed inclusions around $\bar{n}$ with increasing distance. Another reason could be that the large inclusions are not well-dispersed like medium inclusions (3-5μm), for instance, large inclusions like carbides are mostly precipitated when the melt is almost 70% solidified [33,5], thus finding it difficult to move freely in a solid-state. Furthermore, they are found in small numbers compared with small inclusions. The error of less than 5% of $Na_{cum}$ for 6-7μm and 8-9μm is achieved for number of observed inclusions above 35 and 18 respectively. However, future investigation regarding this topic is necessary.

Thus, with the consideration of number of inclusions observed, it is decided to set 2 μm as the minimum critical size for detected inclusions and analyze up to 2.16 mm² of sample area (i.e., 48 photos) at 500X to draw statistically significant conclusions with error less than 5% or close to this value for further three-dimensional (3D) analysis method.
Figure 15 $\text{Na}_{\text{cum}}$ and $\%\Delta_{\text{cum}, \text{Na}}$ with respect to no. of inclusions ($n_{\text{inclusions}}$) observed (Sample A)
3.2 Classification of Non-Metallic Inclusions

For classification of typical non-metallic inclusions and determination of their compositions, subjective observation method was used during investigation under scanning electron microscope (SEM) for CS-method and MS-method. Their chemical composition was determined by an energy dispersive spectroscopy (EDS) method which is an integrated system of SEM.

The inclusions are classified into four types, based on their morphology and chemical composition. It is observed that all four type inclusions in both samples A and B are similar with slight differences in their chemical compositions and morphology.

3.2.1 Non-Metallic Inclusions in Sample A

Table 4 shows the typical SEM images of inclusions, their composition, and size range (d_{eq}, μm) for sample A on the polished cross-sectional surface (CS), as well as on the metal surface (MS) after electrolytic extraction.

Type A1 are carbide inclusions denoted as (Nb,Ti)C. It also contains W in the range of 0.3-5 wt%. They are found as irregular clusters and small agglomerates of irregular particles located close to each other and as single particles. Most of the clusters and big particles are observed on grain boundaries but they were also present inside the grains. In both CS and MS-method, few carbides appeared to be surrounded by some ferroalloy material. It is assumed that this ferroalloy is added during secondary refining might not be melted properly. The ratio of Nb to Ti, R_{Nb/Ti} (average value ± σ) in CS and MS was found in the range of 0.78 – 1.62 (1.21 ± 0.28) and 0.95 – 1.79 (1.28 ± 0.35) respectively.
**Table 4 Typical Inclusions observed in CS-method and MS-method of sample A**

<table>
<thead>
<tr>
<th>Type</th>
<th>CS Composition (mass%)</th>
<th>MS Composition (mass%)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>A1</strong> (Nb,Ti)C</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nb 35 - 51</td>
<td></td>
<td>Nb 38- 54</td>
</tr>
<tr>
<td>Ti 32 - 45</td>
<td></td>
<td>Ti 30 - 42</td>
</tr>
<tr>
<td>W 0.3 - 5</td>
<td>C* 12 – 15</td>
<td>W 1 – 5</td>
</tr>
<tr>
<td>d_eq (µm) 1-12</td>
<td></td>
<td>C* 13 – 16</td>
</tr>
<tr>
<td><strong>A2</strong> (Ti,Nb,Zr)S,C</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nb 14 - 24</td>
<td>Ti 34 – 47</td>
<td>Nb 9 -20</td>
</tr>
<tr>
<td>Zr 7 -10</td>
<td>S 14 -15</td>
<td>Zr 5 -11</td>
</tr>
<tr>
<td>C* 2 -9</td>
<td>W 0 – 1</td>
<td>S 8 -15</td>
</tr>
<tr>
<td>d_eq (µm) 1.8</td>
<td></td>
<td>C* 5 -11</td>
</tr>
<tr>
<td><strong>A3</strong> (Ti,Nb) N,C</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nb 1 - 46</td>
<td>Ti 35 - 75</td>
<td>Nb 1 – 42</td>
</tr>
<tr>
<td>N* 0 – 21</td>
<td>C* 2 –14</td>
<td>Ti 37 - 75</td>
</tr>
<tr>
<td>W 0 – 4</td>
<td>Mo 0 – 5</td>
<td>N* 0 – 22</td>
</tr>
<tr>
<td>d_eq (µm) 1-11</td>
<td></td>
<td>C* 3 –13</td>
</tr>
<tr>
<td><strong>A4</strong> MoCrWNbTi-C</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nb 1 – 4</td>
<td>Ti 1 – 3</td>
<td>Nb 0-3</td>
</tr>
<tr>
<td>W 1 – 16</td>
<td>Mo 5 – 28</td>
<td>W 0 – 4</td>
</tr>
<tr>
<td>Cr 14 –15</td>
<td>C* 0 – 3</td>
<td>Mo 0-3</td>
</tr>
<tr>
<td>d_eq (µm) 0.6-1</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*%C and %N obtained from EDS are not accurate, as SEM-EDS do measure light elements accurately.

Type A2 are sulphides and carbo-sulphides inclusions containing Ti, Nb, Zr, W, S and C. Mostly they are found as pure sulphides in both CS and MS method and sometimes sulphides are observed to be precipitated on carbide inclusions in CS method. They are observed in elongated bar-like shapes in CS and as a plate-like shape in MS-method. The \( R_{\text{Nb/Ti}} \) of sulphides and carbides+sulphide in CS method found in the range of 0.31 – 0.71(0.48 ± 0.18) and 0.73 – 1.62 respectively. The \( R_{\text{Nb/Ti}} \) of sulphides in MS method found in the range of 0.39 – 0.61(0.50 ± 0.1).
Type A3 are carbonitride and nitride inclusions. Carbonitrides are observed as irregular particles as well as clusters in CS and MS methods. Figure 17 shows clusters of carbonitrides and nitrides that were found in CS and MS method. These inclusions have two phases, one phase is nitride, and the other is carbide phase. Usually, the nitride phase is surrounded by the outer carbide phase. Nitride phase has higher mass percent of Ti and N, along with smaller mass percent of Nb and C. While carbide phase has higher mass percent of Nb and C, along with smaller mass percent of Ti and N. Meanwhile, nitride inclusions are observed in a single phase with a higher percentage of Ti and N and contains a very small amount of C and Nb. They are found in regular particles in the shape of square, triangle, diagonal/diamond or irregular shapes in the form of cluster. Nitride inclusions have a single phase. The R_{Nb/Ti} of carbonitrides and nitrides in CS found in the range of 0.02 – 1.32 and 0.02 respectively. The R_{Nb/Ti} of carbonitrides and nitrides in MS found in the range of 0.03 – 1.14 and 0.01 – 0.13 (0.05 ± 0.04) respectively.

Type A4 are intermetallic inclusions containing carbon. They are observed occasionally on the surface of sulphide inclusions and mostly on grain boundaries in CS. The population of these inclusions is very high, but they are found in very small sizes as circular/irregular and rod-like small shapes in the size range of 0.67- 1 μm, and up to 0.67 μm respectively. Therefore, they are not considered in this study. On MS images, they are not investigated because of small population of very tiny sizes up to 1 μm.

3.2.2 Non-Metallic Inclusions in Sample B

Table 5 shows the typical SEM images of inclusions, their composition, and size range (d_{eq, μm}) for inclusions observed in CS-method and MS-method for sample B.
<table>
<thead>
<tr>
<th>Type</th>
<th>CS Composition (mass%)</th>
<th>MS Composition (mass%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>B1 (Nb,Ti)C</td>
<td>Nb 51–54 Ti 29 – 32 W 1 – 3 C* 12 – 13 $d_{eq}$ (μm) 2 – 13</td>
<td>Nb 49-55 Ti 28 – 34 W 1 – 4 C* 10 – 12 $d_{eq}$ (μm) 1-14</td>
</tr>
<tr>
<td>B2 (Ti,Nb,Zr)S,C</td>
<td>Nb 10 – 28 Ti 27 – 40 Zr 4 – 6 S 11 -14 C* 7 – 9 W 0 – 2 $d_{eq}$ (μm) 1-8</td>
<td>Nb 6 – 12 Ti 17 – 46 Zr 2 – 4 S 7 -14 C* 6 – 13 $d_{eq}$ (μm) 1-7</td>
</tr>
<tr>
<td>B3 (Ti,Nb) N, C</td>
<td>Nb 1 – 38 Ti 43 – 78 N* 0 – 22 C* 2 – 12 W 0 – 3</td>
<td>Nb 1 – 47 Ti 35 – 78 N* 0 – 19 C* 2 – 11 W 0 – 5 $d_{eq}$ (μm) 2-15</td>
</tr>
<tr>
<td>B4 MoCrWNbTi- C</td>
<td>Nb 2 – 16 Ti 3 – 5 W 9 – 11 Mo 12 – 32 Cr 17 - 20 C* 3 – 4 $d_{eq}$ (μm) 0.33-4</td>
<td>Nb 4 – 5 Ti 3 – 6 W 2 – 13 Mo 8 – 19 Cr 15 - 19 C* 3 – 5 $d_{eq}$ (μm)</td>
</tr>
</tbody>
</table>

*%C and %N obtained from EDS are not accurate, as SEM-EDS do measure light elements accurately*

Type B1 inclusions are found in the same morphology as for sample A i.e., irregular clusters, small agglomerates particles and single irregular particles within the grain and grain boundaries. The ratio of Nb to Ti ($R_{Nb/Ti}$) in CS and MS was found in the range of 1.58 -1.86(1.69 ± 0.14) and 1.43 – 1.93 (1.63 ± 0.22) respectively.

Type B2 inclusions are observed in the similar morphology of type A2 in both CS and MS method. However, they are found attached to carbides in MS method as well. The $R_{Nb/Ti}$ of sulphides and carbides+sulphide in CS method found in the range of 0.26 – 0.60(0.43 ± 0.17) and 0.39 – 1.58.
The $R_{Nb/Ti}$ of sulphides and carbides+sulphide in MS method found in the range of 0.36 and 0.27 – 1.93 (1.14 $\pm$ 0.77) respectively.

Type B3, carbonitride inclusions have appeared as irregular single particles both in CS and MS. Meanwhile few nitrides are observed as irregular clusters and as separate irregular particles, both in CS and MS. The $R_{Nb/Ti}$ of nitrides in CS found in the range of 0.01 – 0.88. The $R_{Nb/Ti}$ of nitrides and carbonitrides in MS found in the range of 0.04 – 1.35 and 0.01 – 0.02 (0.02 $\pm$ 0.001) respectively.

Type B4 inclusions in sample B are observed both in CS and MS images. Mostly they have appeared on grain boundaries containing Mo, Cr, W, Nb, Ti and C. They are also not considered in the further study because of their higher population in small sizes (<2$\mu$m).

### 3.3 Effect of Metal Matrix

Although similar types of inclusions were found in both tested samples of Inconel 718 alloy using CS and MS method but their inclusion compositional data obtained from SEM-EDS shows a slight variation in elemental compositions, as can be seen in Table 4 and 5. Therefore, the effect of metal matrix elements especially Cr and Ni on inclusion compositional analysis for sample A and B is compared in Figure 18. In both samples, %Cr and %Ni is decreasing with increasing inclusion size, which shows that inclusions larger than 6 $\mu$m have no or are very less effect of metal matrix elements. However, inclusions below 6$\mu$m display some variation in data, which means that their compositional data is affected by metal matrix in both CS and MS methods. Therefore, both CS and MS methods are good to obtain precise inclusion compositional data for inclusions greater than 6 $\mu$m. Composition analysis of inclusions smaller than 6 $\mu$m should consider and exclude an effect of metal matrix.

Another way of checking effect of different analyzing methods on inclusion compositional data is by finding the ratio of Nb to Ti. Table 6 displays the summary of the ratio of Nb to Ti, $R_{Nb/Ti}$ (average value $\pm \sigma$) for different types of inclusions. For type A1, B1, and A2(sulphides), the difference in $R_{Nb/Ti}$ between CS and MS is below 0.1, which means that the data obtained from inclusion compositional analysis is significant. However, for types A2(carbosulphides), B2(sulphides/carbosulphides), A3 and B3, the measured inclusions were very few. Therefore, this data shows only some tendencies but for accurate results, more inclusion compositional data should be analyzed in future work.
Table 6 Summary of $\frac{R_{Nb/Ti}}{r}$ (average ± cf) for different type inclusions

<table>
<thead>
<tr>
<th>Sample</th>
<th>A1 (Carbides)</th>
<th>A2 (Sulphides)</th>
<th>A3 (Carbosulphides)</th>
<th>A4 (Carbonotrides)</th>
<th>A5 (Nitrides)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample A</td>
<td>CS</td>
<td>MS</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>0.78–1.62</td>
<td>0.95–1.79</td>
<td>0.31–0.71</td>
<td>0.73–1.62</td>
<td>0.02–1.32</td>
</tr>
<tr>
<td></td>
<td>(1.21 ± 0.28)</td>
<td>(1.28 ± 0.35)</td>
<td>(0.48 ± 0.18)</td>
<td></td>
<td>0.02</td>
</tr>
<tr>
<td>Sample B</td>
<td>B1</td>
<td>B2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>1.58–1.86</td>
<td>1.43–1.93</td>
<td>0.26–0.60</td>
<td>0.39–1.58</td>
<td>0.04–1.35</td>
</tr>
<tr>
<td></td>
<td>(1.69 ± 0.14)</td>
<td>(1.63 ± 0.22)</td>
<td>(0.43 ± 0.17)</td>
<td>(1.00 ± 0.56)</td>
<td>0.01–0.02</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>(0.02±0.001)</td>
</tr>
</tbody>
</table>

3.4 Number and Particle-Size Distribution

The total number of non-metallic inclusions are investigated in CS-method and MS-method for both samples. Afterwards, a comparison is made for total particle-size distribution for total inclusions, as well as for different types of inclusions.

3.4.1 Number of Inclusions

Table 7 shows a summary of observed inclusions size distribution in CS-method and MS-method for sample A and B. The density of inclusions per area (Na) was found to be higher in MS-method for
both samples compared to CS-method. Simultaneously, more large inclusions up to 18µm and 15µm were observed in MS method of sample A and B respectively. Smaller area for sample B is analyzed because the distribution of inclusions was higher.

Table 7 Summary of number of inclusion

<table>
<thead>
<tr>
<th>Sample</th>
<th>Method</th>
<th>Dissolved metal layer, µm</th>
<th>Observed area, mm²</th>
<th>Number of NMI observed, n</th>
<th>Size range, µm</th>
<th>Na, mm²²</th>
</tr>
</thead>
<tbody>
<tr>
<td>A(AK-SP5)</td>
<td>CS(63)*</td>
<td>-</td>
<td>2.835</td>
<td>592</td>
<td>1-14</td>
<td>208</td>
</tr>
<tr>
<td></td>
<td>MS(48)*</td>
<td>2.8</td>
<td>2.16</td>
<td>578</td>
<td>1-18</td>
<td>268</td>
</tr>
<tr>
<td>B(AK-SP4)</td>
<td>CS(32)*</td>
<td>-</td>
<td>1.44</td>
<td>455</td>
<td>1-14</td>
<td>316</td>
</tr>
<tr>
<td></td>
<td>MS(32)*</td>
<td>3.6</td>
<td>1.44</td>
<td>590</td>
<td>1-15</td>
<td>410</td>
</tr>
</tbody>
</table>

(*) = number of observed SEM pictures

Figure 19 displays the number percentage of each type of inclusions that have appeared on the polished surface (CS) and the metal surface (MS). In sample A, 69.78% and 24.91% of A1 and A3 inclusions are found in MS compared to CS. Meanwhile, A2 inclusions are observed up to 5.2%. The reason might be small inclusions fallen out from surface during electrolytic extraction.

In sample B, B1 inclusions are observed approximately in the same percentage both in CS (52.22%) and MS (51.22%) method. Meanwhile, B2 inclusions appeared to be 32.93% in MS compared to 26.27% in CS.

Overall, carbide inclusions (A1 and B1) appeared to be prominently higher compared to sulphide/carbosulphide (A2, B2) and carbonitride/nitride (A3, B3) inclusions.

3.4.2 Particle-Size Distribution

Figure 20 displays the comparison of particle size distribution for a total number of inclusions observed per unit area (T.Na) in sample A and Sample B for CS and MS method. In Sample A, inclusions below 2µm are observed more in CS method. However, inclusions in the size range of 3 µm to 18 µm are
observed higher in MS method. In sample B, inclusions have appeared in the range of 2 µm to 13 µm in CS. While in MS, inclusions are observed in the size range of 2 µm to 15 µm. Overall, large inclusions up to 18 µm are observed in MS method compared to CS.

![Figure 20](image1.png)  
**Figure 20** Particle-Size Distribution in Sample A and Sample B

Figure 21 shows a comparison of particle size distribution with respect to Na for different types of inclusions analyzed in sample A and B. In sample A, large type A1 inclusions up to 18 µm are analyzed in MS in the form of irregular clusters, agglomerates of small particles as well as single particles. A3 inclusions in CS-method have observed up to 11µm and in MS-method up to 12 µm. Meanwhile, small size A2 inclusions up to 6 µm appeared more on polished surface (CS) and large size sulphide inclusions in the size range of 6µm to 11µm have appeared in MS-method. However, the amount of A2 (sulphide) inclusions is less in sample A but their sizes are big up to 11µm.

In sample B, small size as well as large size inclusions up to 14µm are observed in MS-method for B1 type inclusions. For B2 type inclusions, inclusions up to 5µm are observed more in MS-method, while large inclusions up to 8 µm have appeared in CS-method. The total amount of B2 inclusions in sample B is higher, however, their size is less than 9 µm. For B3 inclusions, the particle size distributions show a random variation both in CS and MS-method. Most of the carbonitrides are found as irregular single crystals in both CS and MS, while few large nitride clusters have appeared in MS-method up to 15 µm.
Table 8 represents overall summary of data for different types of inclusions in sample A and B obtained in CS-method and MS-method.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Method</th>
<th>Type of Inclusion</th>
<th>Size range, ( \mu m )</th>
<th>( \bar{d}_{eq} \pm \sigma, \mu m )</th>
<th>Na, mm(^2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample A</td>
<td>CS</td>
<td>A1</td>
<td>1-12</td>
<td>4.42(\pm)2.52</td>
<td>109</td>
</tr>
<tr>
<td></td>
<td></td>
<td>A2</td>
<td>1-8</td>
<td>2.65(+)1.10</td>
<td>49</td>
</tr>
<tr>
<td></td>
<td></td>
<td>A3</td>
<td>1-11</td>
<td>4.51(+)2.46</td>
<td>50</td>
</tr>
<tr>
<td></td>
<td>MS</td>
<td>A1</td>
<td>1-18</td>
<td>4.90(+)2.99</td>
<td>187</td>
</tr>
<tr>
<td></td>
<td></td>
<td>A2</td>
<td>2-11</td>
<td>5.63(+)2.15</td>
<td>14</td>
</tr>
<tr>
<td></td>
<td></td>
<td>A3</td>
<td>2-12</td>
<td>5.19(+)2.11</td>
<td>67</td>
</tr>
<tr>
<td>Sample B</td>
<td>CS</td>
<td>B1</td>
<td>2-13</td>
<td>3.39(+)2.18</td>
<td>165</td>
</tr>
<tr>
<td></td>
<td></td>
<td>B2</td>
<td>1-8</td>
<td>2.88(+)1.46</td>
<td>83</td>
</tr>
<tr>
<td></td>
<td></td>
<td>B3</td>
<td>2-13</td>
<td>4.75(+)2.44</td>
<td>68</td>
</tr>
<tr>
<td></td>
<td>MS</td>
<td>B1</td>
<td>1-14</td>
<td>4.35(+)2.22</td>
<td>210</td>
</tr>
<tr>
<td></td>
<td></td>
<td>B2</td>
<td>1-9</td>
<td>2.70(+0.96)</td>
<td>135</td>
</tr>
<tr>
<td></td>
<td></td>
<td>B3</td>
<td>2-15</td>
<td>4.58(+)2.20</td>
<td>65</td>
</tr>
</tbody>
</table>

\( \sigma \) = standard deviation
4 Discussion

Two samples of Inconel 718 from different manufacturing companies are investigated for non-metallic inclusions by CS-method and MS-method. The SEM analysis and ImageJ results are discussed for inclusion characteristics like composition, number, size, morphology and compared between the testing methods.

4.1 Accuracy of Measurements

Number of inclusions in samples A and B are investigated by CS-method at two magnifications initially i.e., 500X and 1000X. Based on particle-size distribution in Figure 12, approximately 2.84mm² area of sample A is examined at 500X to obtain less than 5% error of cumulative Na for different sized inclusions. Inclusions smaller than 3 µm and medium inclusion in the size range of 3-5 µm, less than 5% error of Na cum is obtained and was statistically significant. The reason is that they are found to be distributed less heterogeneously compared to large inclusions (<6µm) and observed in more numbers within the grains, as well as on grain boundaries. Another reason would be that few inclusions might form before solidification. For instance, nitride particles tend to precipitate in liquid melt of Inconel 718 before solidification [34-36].

For large inclusions greater than 6 µm, error percent of cumulative Na is relatively close to 5%. It’s because they are observed in a non-uniform distribution in metal matrix. Other few reasons would be; (i) they precipitate in the inter-dendritic regions in final moments of solidification, thus finding harder to move freely (ii) large and cluster inclusions observed distributed heterogeneously on grain boundaries as well. For instance, inclusions like carbides are mostly start precipitating when the metal is 70% solidified [33] in Inconel 718. Other few anomalies that would be reason for less accuracy in measurement is that the systematic analysis was done at two different magnifications from bottom towards top zone at 500X first, then at 1000X and again at 500X, as shown Figure 22. Therefore, further investigations either using only one magnification or by increasing observed area is required in future to improve error analysis for large-sized inclusions.

For sample B, the area of inclusions is investigated from the center position (1) of bottom zone towards middle zone. For more observational area, location near to the center (2) is selected because during the investigation, we did not fix the position of last picture taken at 1000X in position 1. Using different locations would be the possibility of getting more number of inclusions under small
observational area compared to sample A. In future, further investigation is required by doing systematic analysis continuously for comparison purpose.

Figure 22 Direction of Analyzed area for Non-metallic Inclusions at magnification 500 and 1000 in CS-method

4.2 Types of Inclusions

Four similar types of inclusions are found in both samples of Inconel 718, named Type A1, B1(carbide inclusions), Type A2, B2(sulphide and sulphide+carbide inclusions), Type A3, B3(carbonitride and nitride inclusions) and Type A4, B4 (intermetallic carbide inclusions). The observation of these inclusions in the studied alloy is consistent with the findings of earlier researchers [4,6,15,21,25-28]. However, the compositional mass percent and size range of inclusions analyzed in CS and MS method differ slightly. This would be because of the differences in using measuring methods and the number of inclusions examined.

Type A1 and B1 are carbide inclusions containing Nb, Ti and C, appeared in a non-uniform distribution within the grains and on grain boundaries. They are found in the same morphology as irregular clusters, small agglomerates of irregular particles located close to each other and as single particles in both samples. The previous researchers [34-39] have found that carbides are rich in Nb and Ti and are formed by segregation of high content of Nb and carbon in the liquid zone within a short temperature range (from precipitating-start temperature (L+γ+NbC) till γ/NbC eutectic (L=γ+NbC)) during primary solidification. They prefer to nucleate on dislocations, in the inter-secondary γ dendrite arm, the bottom mushy zone and on TiN particles as well [37-38]. It is also reported that γ/NbC does not appear at lower temperatures, since the carbon is almost consumed during γ/NbC reaction [36-38]. However, in post-heat treatment processes, for example, during solution annealing and quenching, γ phase becomes super-saturated by carbon and further ageing relives this phase by forming carbide precipitates which exclusively nucleated on grain boundaries.
Thus, it is assumed that the carbides formed during primary solidification and post-heat treatment processes would coincide to produce carbide clusters. Since the carbides formed during primary solidification are difficult to diffuse back into the metal matrix because of high solvus temperature (1260-1300°C) [5,26].

Another reason for having large-sized carbide particles within grains and grains boundaries would be because of the size-growth of particles, dependent upon the cooling rate and the trapping ability of carbide forming elements in the inter-secondary γ dendrite arm and in the bottom mushy zone [37-38]. For instance, in one study, mostly carbides in the directional solidified part of Inconel 718 are found to be >10 μm because the carbides were partially trapped by the solid-liquid interface and continues growing towards the liquid until it is completely trapped by γ dendrites [37]. This trapping ability also affects carbide morphology, changing from octahedron to discrete blocky shapes or irregular shapes [37-38].

Few A1 inclusions in sample A are appeared to be surrounded by some ferroalloy material both in CS and MS methods. It is assumed that this material might be a non-melted alloy added during the melting stage. Meanwhile, no ferroalloy particles are found in sample B.

Type A2 and B2 are pure sulphides and carbosulphides inclusions which are found either in the shape of elongated bars or irregular elongated clusters in CS-method. While in MS-method, they have appeared as plate-like shapes. In both samples, titanium sulphides with small amount of niobium and zirconium were detected and few sulphides were found to be precipitated on carbide inclusions. It is reported that sulphide inclusions in Inconel 718 tend to enhance pitting corrosion in chloride or hydrogen sulphide environments [6,21].

Type A3 and B3 contain carbonitride and nitride inclusions. Carbonitrides observed in the same morphology for both samples in CS and MS methods in sample A. However, in sample B, irregular single particles of carbonitrides have been observed. Meanwhile, nitride inclusions are observed as regular particles in the shape of a square, triangle, diagonal/diamond and irregular shapes in the form of clusters in both samples. It is reported that oxide inclusions in liquid superalloy act as nucleating sites for nitrides during solidification and with decreasing temperature, these pre-formed nitrides act as nucleating sites for carbide which subsequently form two phases as a result of interdiffusion with time [15]. However, in the samples of tested alloy, Al2O3 inclusions did not appear. The reason may be because nitrides and carbides might get enough time to precipitate over the oxide surface and completely hide them. And the reason for single phase nitride with similar
chemical composition would be less availability of Nb in metal matrix to form another phase around it.

During the CS method, few single Al₂O₃ particles are found in both samples in the form of irregular plate-like shapes. It is assumed that they might be penetrated onto the sample surface from the cutting disc during sample cutting. These particles are not observed in MS method because of not being deeply penetrated into the metal matrix and easily removed during electrolysis.

Type A4, B4 are intermetallic carbide inclusions, observed mostly on grain boundaries in both samples. They are not analyzed in this study because of their higher population in small sizes (<3µm). It is assumed that these inclusions mostly precipitated after solidification and during heat-treatment based on location, composition, and morphology [6].

4.3 Number and Particle-Size Distribution

According to Table 7, the total number of inclusions per area (T.Na) are observed higher in MS method compared to CS method. At the same time, large size inclusions up to 18 µm and 15 µm are also found in MS method. It’s because the dissolution of metal matrix around inclusions during extraction exposed large sized inclusions on metal surface and can clearly analyze their sizes.

According to Figure 19, 5.2% of type A2 inclusions are found less in MS-method compared to CS-method. It’s because 3µm of metal layer dissolved into electrolyte during extraction in sample A and inclusions <3µm are easily removed from metal surface, Figure 21. Overall, sample A has more carbide (A1, 69.7%) and carbonitride/nitride (A3, 24.9%) inclusions and sample B has more carbide (B1, 51.2%) and sulphide/carbosulphide(B2, 32.9%) inclusions.

In sample A, type A1 (carbide) inclusions are found in large size up to 18 µm compared to type A2 and A3 in MS method. The reason may be the carbides in the inter-dendritic region of γ are not properly trapped by dendrite arms and continues growing towards the liquid until it is completely trapped by γ dendrites [37], producing large size carbides. It is reported that large size carbides in the size range of 2-30 µm affect the corrosion resistance of alloy Inconel 718 very badly [6]. And during plastic deformation, microcracks easily propagate along large-sized carbides located on grain boundaries, reducing ductility and toughness of Inconel 718 [40]. In another study, carbides tend to crack during machining, as NbC is 4 times harder than TiC [27]. This surface cracking tendency reduce fatigue life of alloy 718. Meanwhile, small carbides at grain boundaries prevent the growth of grains during heat-treatment [18,23]. Therefore, carbides in the shape of small equiaxed are preferred in conventional nickel-based alloys [28].
In sample B, type B3 (carbonitrides/nitrides) inclusions are found in the size range of 2-15µm compared to type B1 and B2. In one study [15], carbonitrides were found in the size range of 5 to 15µm, which are harmful to the alloy during deformation processes and its morphology was modified by adding calcium during the electroslag remelting process.

In addition, the percentage frequency of type B2 (sulphide) inclusions is higher in sample B compared to sample A. However, they are found in the size range of 1-11µm and 1-9 µm in sample A and B respectively. This indicates that sample A can be more prone to corrosion because of large-sized sulphide and large sized carbide inclusions as well. In one study, the effect of inclusions in Inconel718 on the corrosion property is reported in the following order: sulphides > Primary carbide (Nb, Ti)C > Nitrides(Nb,Ti)N [21]. However, in another study, it is also reported that large size nitrides (10-27µm) also affect deleteriously the corrosion resistance of alloy Inconel 718 [6].

4.4 Comparison between CS and MS method
Elemental composition data for inclusions obtained by EDS in CS-method and MS-method for samples A and B show slightly different values. The reason for differences might be the effect of metal matrix elements because inclusions are mostly surrounded by the metal matrix in CS-method and in MS-method, most of the metal matrix around inclusions dissolved into the electrolyte, thus reducing the effect of metal matrix elements [1]. According to Figure 18, there is no influence of the metal matrix elements on inclusions larger than 6 µm in CS and MS method. However, effect of metal matrix should be considered for inclusions < 6 µm, because $R_{\text{Nb/Ti}}$ is different in inclusions and in metal matrix. In the tested samples, most of inclusions are < 6 µm, that’s why average value of $R_{\text{Nb/Ti}}$ in the Table 6 for different inclusions is not accurate. Therefore, more compositional data of inclusions needed to be investigated in future to obtain accurate results.

The morphology of carbides (A1, B1) and carbonitrides/nitrides (A3, B3) inclusions are found to be similar in CS and MS-method. Meanwhile, for sulphide/carbide+sulphides (A2, B2) inclusions it was different. In CS they have appeared as elongated bar-like shapes, while in the MS method they appeared in plate-like shapes. Sometimes in CS-method, it is hard to detect carbide inclusions because of same colour to metal matrix. In MS method, inclusions clearly appeared on metal surface, can be easily detected, and analyze their morphology. Therefore, MS is a much better method to obtain better morphology of all types of inclusions compared to CS [1,16].

Large-sized inclusions are observed higher in MS compared to CS. The reason is that the dissolution of metal matrix around inclusions during electrolytic extraction exposed the large size inclusions
more. Meanwhile in CS method, inclusions get cut cross-sectionally during grinding and polishing and the area observed is a sectioned area of inclusions, which is smaller than the real size of inclusions. Therefore, CS is not a good method for analyzing the true size of inclusions [1,7,12,16] as well as large size inclusions. Another possibility of getting wrong size of inclusions in CS is when particles located very close to each other and they appeared as cluster on SEM images, as shown in Figure 4. However, MS also does not provide the true size of inclusions because the part of inclusion that embedded on metal surface is hidden, as shown in Figure 5 and it only provides the near-true size of inclusions.

CS method is better in determining the total number of inclusions including small sized-inclusions compared to MS, because inclusions smaller than the dissolved depth layer dropped down into electrolyte from metal matrix [1,7,16] and underestimate the small-sized inclusions.

From the results obtained regarding inclusions characteristics, it is suggested to use MS method for analyzing size, morphology, number, location and orientation of medium and large-size inclusions.

Table 9 shows the comparison summary of non-metallic inclusions characteristics for CS and MS method obtained from the results.

<table>
<thead>
<tr>
<th>Method</th>
<th>Composition</th>
<th>Morphology</th>
<th>Number</th>
<th>Size</th>
<th>Location</th>
</tr>
</thead>
<tbody>
<tr>
<td>CS</td>
<td>Good for inclusions larger than 6µm</td>
<td>Fairly good</td>
<td>Very Good</td>
<td>Not good</td>
<td>Good</td>
</tr>
<tr>
<td>MS</td>
<td>Good for inclusions larger than 6µm</td>
<td>Very good</td>
<td>Good, but underestimation of small-sized inclusions</td>
<td>Good</td>
<td>Good and provides the exact orientation of inclusions</td>
</tr>
</tbody>
</table>
5 Conclusions

This study investigated two Inconel 718 samples for non-metallic inclusions morphology, composition, number, and size, obtained from different manufacturing companies. The investigation methods used were i) Conventional CS-method, in which the cross-sectional surface of polished samples was analyzed under SEM, ii) New experimental MS-method, in which the metal surface of samples after short electrolytic extraction was analyzed under SEM. In the end, a comparison was made between CS and MS to find out which method is more effective for accurately analyzing inclusions in a short period of time. Based on the obtained results, the following conclusions have been derived:

1. According to measurement accuracy in sample A, small (< 2µm) and medium size (3-5µm) inclusions at 500X provides less than 5% error (%Δcum.Na ) because of less heterogenous distribution within grains and grain boundaries compared to large inclusions (6 µm). However, for inclusions larger than 6µm, error (%Δcum.Na ) is relatively close to 5% because of non-uniform distribution in metal matrix or precipitate in the final moments of solidification, thus finding harder to move freely.

2. Four types of inclusions were found in sample A and B, named Type A1 and B1(carbide inclusions), Type A2 and B2 (sulphide and carbosulphides inclusions), Type A3 and B3 (carbonitride and nitride inclusions) and Type A4, B4 (intermetallic carbide inclusions).

3. A1, B1 and A3,B3 inclusions have appeared in the same morphology as irregular single particles and irregular clusters. However, in sample B, only irregular single particles of B3 have been observed. All these inclusions in the shape of clusters are observed mostly on grain boundaries but they are also found within the grains. At the same time, A2 and B2 inclusions are found in the shape of elongated bars or irregular elongated clusters in CS-method. While in MS-method, they have appeared in plate-like shapes. Few carbide inclusions are found to be attached with sulphide inclusions in type A2 and B2 as well. Type A4 and B4 inclusions are observed mostly on grain boundaries in very small sizes (<2µm).

4. The chemical composition and size range of inclusions analyzed in CS and MS methods showed some variation because of the differences in testing methods.

5. The total number of observed inclusions (>2µm) per area (T.Na) are observed higher in MS (approximately 56%) compared to CS method. At the same time, large-sized inclusions up to 18µm are found in MS method.
6. In sample A, type A1, A2 and A3 inclusions are found in the size range of 1-18 μm, 1-11μm and 1-12 μm respectively. In sample B, type B1, B2 and B3 inclusions are found in the size range of 1-14 μm, 1-9μm and 2-15 μm respectively.

7. By comparing two testing methods, MS is a good method for analyzing the morphology and size of medium (3-6μm) and large inclusions (>6 μm). It also provides the exact location and orientation of inclusions on the metal surface. However, it underestimates the number of small-sized inclusions (<3μm) because of their removal from metal matrix during electrolytic extraction. Meanwhile, CS is a better method for only finding the total number of inclusions, including small-sized inclusions. For chemical composition, both methods show no influence of the metal matrix on inclusions larger than 6μm.

8. MS is an effective method for estimating the size, morphology, location and orientation of medium(3-6μm) and large size inclusions (>6 μm). Therefore, it is suggested to used MS method for analyzing medium and large sized inclusions. However, results for CS and MS methods can be combined to achieve more accurate information.
6 Future Work

To improve the accuracy of results, the following points are recommended for future research.

1. More systematic analysis either by lowering magnification or increasing observed area for large sized inclusions (>6 µm) in sample A is required, to improve error analysis.

2. Nitrides and carbonitrides should be analyzed as separate inclusions to find out the distribution pattern of both particles in metal matrix and investigate its effect on alloy properties separately.

3. More inclusions are needed to be analyzed for elemental composition to get more accurate results, and to find out which analyzing method (CS or MS method) is effective and efficient.

4. In this study, only CS and MS method is investigated, which are effective methods for analyzing non-metallic inclusions characteristics, but they do not provide the true size of inclusions. Therefore, other testing methods are needed to be performed to find out the true size of inclusions.

5. The reason for the formation of sulphides and carbosulphide inclusions has not been discussed in this study. Therefore, the reason for the formation and its effect on the alloy quality properties needed to be investigated in future work.

6. For measuring size of inclusions ImageJ software is used which is very tedious and time-consuming method. Automated programs like INCA or ASPEX are suggested to use in future research for analyzing inclusions.
7 Acknowledgement

I would like to express my deepest gratitude to my supervisor Docent Andrey V. Karasev (KTH/Material Science and Engineering) for kind guidance, support, patience and time during the whole thesis work. Without his help, I wouldn’t be able to finish the experimental work and SEM investigations. Under his supervision, I have learned the scientific method for doing research and guided me in the right direction during the whole project. He is a very good mentor and his mentoring helped me in finding a path towards professional career. I am very grateful for his mentorship.

I also would like to express my gratitude to Wenli Long (KTH/Material Science and Engineering), who gave her precious time to train me on scanning electron microscope.

Finally, I also want to thank my husband and my son for giving me a lot of time and support during the research work.
8 References


Appendix

A.1 Sample Mounting

The following steps were performed during mounting.

1. First, the mounting press machine was inspected thoroughly to become familiarized with the apparatus and its tools.
2. The water pipeline is connected to the machine, and it’s turned on to keep the sample cooled while mounting.
4. Raised the bottom ram to the top of the mould cylinder and placed the sample in the centre position at the bottom ram.
5. Lowered the bottom ram along with the sample by pressing the downward arrow on the machine.
6. When it is lowered enough, almost two spoons of bakelite powder were added to it.
7. Closed the upper lid and screwed it tightly on the top of the mould cylinder.
8. Set the pressure up to 290 bar and time. Pushed the start button and wait for almost 9-10 minutes. The temperature will rise to 150°C, and it will sinter the powder within mentioned time.
9. Once it is pressed and sintered, the machine beeped to alert that it is ready to remove the sample.
10. Pressed the ram downward for a few seconds by pressing the downward arrow on the machine and then unscrewed the upper lid.
11. Raise the ram to the top of the mould cylinder again and take the sample off the stage.
12. Cleaned any excess bakelite powder on the machine afterwards.
A.2 Observed SEM Pictures for Non-Metallic Inclusions

Sample A
Inclusion: A1, A2, A3 and A4  
Method: CS

<table>
<thead>
<tr>
<th>SEM photo</th>
<th>EDS Spectrum</th>
<th>Type of Inclusion</th>
</tr>
</thead>
</table>
| ![SEM photo](image1) | ![Spectrum 1](image2) | Type:A1 (Carbide)  
Spectrum 1 |
| ![SEM photo](image3) | ![Spectrum 19](image4) | Type:A2 (Sulphide)  
Spectrum 19 |
| ![SEM photo](image5) | ![Spectrum 14](image6) | Type:A2 (Carbosulphide)  
Spectrum 14 |
Type: A3 (Nitride)
Spectrum 27

Type: A3 (Carbonitride)
Spectrum 29

Type: A4 (Intermetallic Carbide)
Spectrum 22
Sample A  
Inclusion: A1, A2 and A3  
Method: MS

<table>
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<td><img src="image" alt="EDS Spectrum" /></td>
<td><strong>Type:A2</strong> (Sulphide)</td>
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</table>

Un-melted Feroalloy  
Spectrum 34
Type: A3 (Single Nitride)
Spectrum 30

Type: A3 (Nitride cluster)
Spectrum 8

Type: A3 (Carbonitride cluster)
Spectrum 3

Spectrum 6
Un-melted Feroalloy

Spectrum 15
Sample B
Inclusion: B1, B2, B3 and B4

<table>
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<tr>
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<td><img src="image4" alt="EDS Spectrum" /></td>
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<td><img src="image5" alt="SEM photo" /></td>
<td><img src="image6" alt="EDS Spectrum" /></td>
<td>Type:B2 (Carbosulphide) Spectrum 23</td>
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<tr>
<td><img src="image7" alt="SEM photo" /></td>
<td><img src="image8" alt="EDS Spectrum" /></td>
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</table>
Type: B3
(Nitride Cluster)
Spectrum 13

Type: B4
(Intermetallic Carbides)
Spectrum 3

Un-melted Feroalloy
Spectrum 28
Sample B
Inclusion: B1, B2, B3 and B4
Method: MS

<table>
<thead>
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</table>
Type: B3  
(Carbonitride)

Spectrum 14

Type: B4  
(Intermetallic Carbide)

Spectrum 18