

Possibilities to Obtain Feedback on Inclusion Characteristics during Ladle Refining

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Abstract: The paper will discuss possibilities to control impurity elements and inclusion characteristics during ladle refining. Here, practical aspects such as steel sampling for suitable inclusion characteristics need to be considered. In addition, it is essential to study methods for rapid determinations of inclusions such as the OES/PDA and the LA-ICP-MS techniques. Finally, off-line methodologies such as the electrolytic extraction method in combination with metallographic methods to determine more in-depth information with respect to the specific inclusion characteristics.

Key words: PDA/OES, Inclusions, Ladle, refining, Sampling

1. Introduction

For niche products the inclusion characteristics as well as the impurity element contents have a large influence on the final material properties. Thus, it is essential to design the ladle refining, casting, working and heat treatment operations so that these quantities are well controlled. Here, it should be noted that especially secondary refining in ladles is of outmost importance, since if it is well performed less problems with inclusion characteristics and impurity elements will occur in the later process steps.

In production of clean steel it is important to have process knowledge on the following three parameters: i) elements that dissolve into steel, ii) steel temperature and iii) inclusions characteristics, as shown in Table 1. The elements that dissolve into the steel can, in-turn, be divided into two groups. The first are the valuable alloying elements that are added to a steel to obtain the desired material properties for a specific steel grade. The second are impurity elements such as S, H and N that need to be kept below a certain value to guarantee that the desired material properties are met in the final product. The way the dissolved elements are controlled during production is that sampling is carried out on a regular interval. Thereafter, optical emission spectroscopy (OES) is used to analyze the dissolved alloy element content. Therefore, it is possible to get a rapid feedback to determine if the dissolved alloy content stays within the specified range per each element during production in the ladle. Often, steel samples are also taken in the tundish, mold (sometimes) and in the metal working mill. However, at these later stages it is not possible to change the dissolved alloying content too a large degree.

The second important control parameter is the steel temperature. This is measured in on-line throughout the steel making process. Normally, it is measured several times in the ladle as well as in the tundish. Furthermore, it is often measured indirectly and continuously in the mold and during metal working in a rolling or forging mill.

The third important control parameter is the inclusion characteristics. A major disadvantage is that no on-line feedback of inclusion characteristics is available during steelmaking, which makes process control impossible. At

the same time, it is well known that inclusion characteristics affect the material properties of the finished steel product. Thus, there is a strong driving force to develop a means of rapid obtaining information on existing inclusion types and contents during the different stages of steelmaking, which would make it possible to optimize steel production in on-line. Customer requirements regarding material properties could then better be fulfilled. Moreover, casting problems related to inclusions in the ladle could be decreased.

Table 1 Parameters controlled in liquid steel on different stages of steel production

Controlled parameters	Ladle	Tundish	Mold	Metal working mill
Elements that dissolve in steel	yes	yes	yes	yes, not necessary
Temperature	yes	yes	yes	yes
Inclusion characteristics	no	no	no	yes, afterwards

15-20 years ago the steelmaking industry had not access to analytical techniques for reasonably fast and accurate determinations of inclusions and clusters in steel samples. Therefore, the samples taken from liquid steel during ladle treatment and casting were used for on-line analysis of total chemical composition of steel (by using various methods based on principles of spectrometry) and for estimation of total content of O, S and N. In addition, the inclusion characteristics (such as composition, number, size and morphology) in steel samples usually were determined by using different time consuming methods. Some of these off-line analytical techniques are listed in Figure 1. The results obtained by investigation of inclusions and clusters in steel samples were used for off-line correction and optimisation of technological processes.

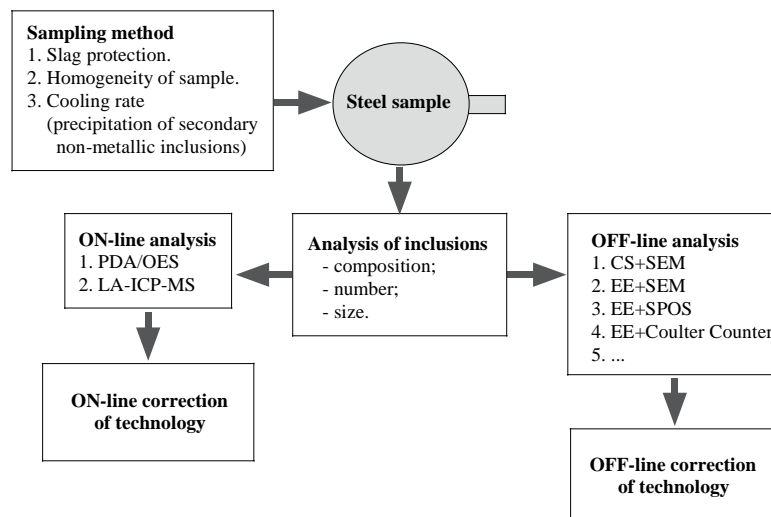


Fig. 1 Schematic illustration relations between steel sampling, investigation of inclusion characteristics and correction of technology parameters during steelmaking

Due to tightened requirements of the steel quality (particularly for high quality steel grades as stainless and tool steels) in recent years, it has become necessary to obtain feedback of inclusion characteristics in liquid steel in order to improve the steel quality design with respect to inclusion characteristics. Therefore, some new and improved methods for rapid analysis of inclusions in steel samples (such as Pulse Discrimination Analysis by Optical Emission Spectroscopy, PDA-OES, and Laser Ablation - Inductively Coupled Plasma - Mass Spectrometry, LA-ICP-MS) have

been developed during the recent decades. These methods were tested and used for rapid evaluation of inclusions and clusters in steel samples from laboratory scale experiments as well as from industrial samples from different steel grades. Obtained results have been compared to data from conventional two-dimensional (2D) observations of inclusions on polished cross-sections of steel samples (CS+SEM) and from three-dimensional (3D) investigations of inclusions. For the latter, electrolytic extraction (EE) has been used to extract the inclusions from the steel matrix in and analytical techniques such as EE+SEM, EE+SPOS (Single-Particle Optical Sensing or Photo-Scattering method) have been used to determine the inclusion characteristics.

This paper focuses on experimental approaches to obtain more information on inclusion characteristics. Initially, some aspects of methods to collect samples from liquid steel, which can effect on inclusion characteristics in steel samples, will be discussed. Thereafter, some advantages, disadvantages and limitations of the current available techniques to determine inclusion characteristics will be discussed.

2. Sampling method

A determination of the total oxygen content in a steel sample provides a rough idea of the oxidic steel cleanness. Moreover, by studying the different zones of the metal sample it is possible to provide a rough estimation of the dispersion of oxide inclusions in a sample. This is important in order to determine what part of the sample that is most appropriate to use in the determination of the inclusion characteristics. Below some results from two sample types lollipop (with a 6mm and a 12 mm thickness) and a Björneborg sampler will be discussed. In addition, data from the following two slag protection techniques will be discussed: metal-cap protection and argon protection.

The body, pin and inlet parts of the industrial metal samples from three heats of the S32304 stainless steel grade were cut into small specimens (0.5~1 g). The analyzed zones of the body, pin and inlet in the different samples are shown in Figure 2. The specimens were prepared by filing, cleaning in alcohol in an ultrasonic bath, and drying. Thereafter, they were analyzed by the inert gas fusion method using a Leco TC600 instrument.

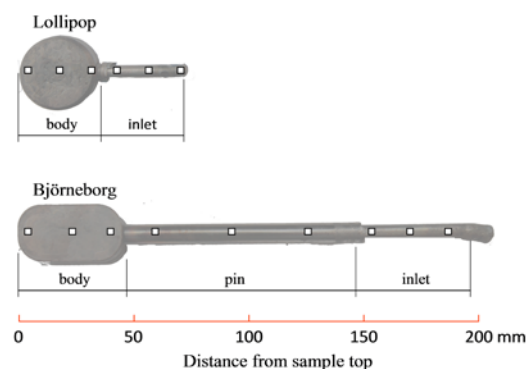


Fig. 2 Schematic illustration of the different zones (white squares) for determination of the total oxygen content in the metal samples.

Figure 3 shows the average total oxygen content in the different samples depending on geometry and type of slag protection method.^[1] The error bars in this figure represent the arithmetic standard deviation values. As seen, most of

the argon-protected samples contain on average 25-35 ppm of oxygen. However, the total oxygen content in the metal-cap-protected samples is significantly higher, particularly in the Lollipop samples (on average 45-80 ppm). Moreover, the standard deviations values for the results obtained with metal-cap-protected samplers are significantly larger in comparison to the argon-protected samplers. In addition, it was found that the oxygen content in the end part of the inlet in all the samples was drastically higher. This is most likely due to reoxidation and/or entrapment of top slag during withdrawal of the sampler from the melt. Therefore, the end part does not represent the melt and those values should not be used. As seen in Figure 3, the average value of the total oxygen content increases significantly with a decreased sample weight in the metal-cap-protected samples. However, for the argon-protected samples the value of the total oxygen content is independent of the sample geometry.

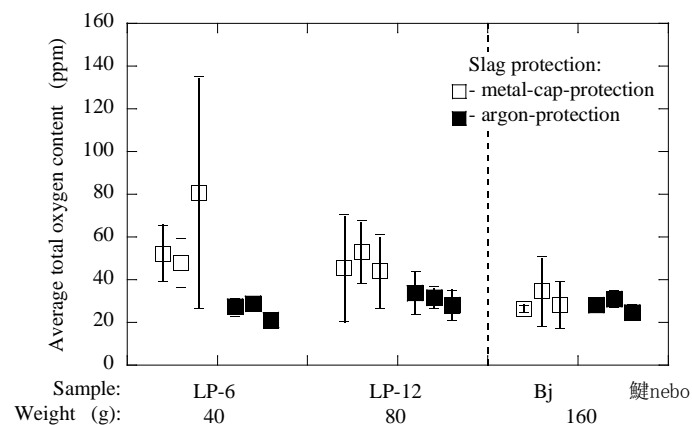


Fig. 3 Average total oxygen content in LP-6, LP-12 and Björnebo samples depending on type of slag protection

It is important to have access to a reliable sampling procedure in order to obtain samples that as much as possible represents the inclusion characteristics, which exists in liquid steel at ladle refining temperatures. Here, it should first be noted that it is necessary to minimize the effect of reoxidation during sampling. Here, Ericsson *et al.*^[1] has shown that the use of argon flushing is beneficial to push away the top slag during the immersion of the sampler in to the steel melt. This method also removes the need to use a steel cap at the sampler inlet, whose alloy and inclusion composition can influence the metal sample during sampling. It is also necessary to minimize the turbulence during filling in order to minimize the growth of inclusions due to collisions. This can be done by lowering of the inlet velocity and thereby the turbulence, as illustrated by Zhang *et al.*^[2,3] This, in turn requires the use of a controlled back pressure during sampling of the liquid steel.

The implementation of a good sampling procedure is a very important component for reaching this goal. By using argon flushing in combination with a controlled back pressure, the reoxidation will be minimized. Furthermore, the turbulence will be decreased so that the inclusion distribution existing at the ladle refining temperatures will be determined more accurately compared to in samples filled by a higher turbulence. Ericsson *et al.*^[1] has also shown that it is important to carry out the inclusion determinations in the lower part of the Lollipop sample, where the inclusion distribution is most homogeneous.

3. On-line estimation of inclusion characteristics

PDA/OES. An illustration of the Optical Emission Spectroscopy (OES) method is shown in Figure 4. The basic

principle is that an electric discharge hits an inclusion in a steel sample which gives rise to a light intensity. These light pulses are broken up by a grating into their respective wavelengths and then detected by separate units for each element (Al, Mg, Ca, etc.). By analyzing the information from each spark, the size distribution and composition of inclusions can be determined. In addition, by analyzing the average information from a longer sparking sequence, the contents of the elements dissolved in the steel can also be determined. The lower part of Figure 4 shows the output signals from one of the detection units for a longer sparking sequence. The base line represents the dissolved content of the metal in the steel sample and the peaks correspond to the metal content in inclusions. More details regarding the analysis equipment, data acquisition and data processing can be found in earlier publications.^[4,5]

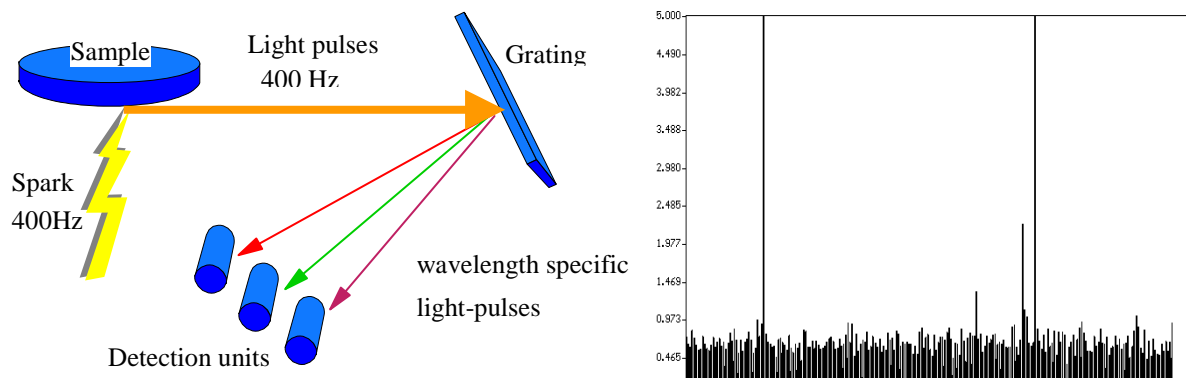


Fig. 4 Basic principles of the OES method (left diagram) and typical pulse chart (right diagram)

When using the PDA/OES method it is necessary to determine the window for which the method can provide reliable information on inclusion characteristics. It has been shown that the accuracy of the analyses is different for different elements and depends on the soluble content of these elements. This means that there will be different lower limits for the smallest inclusion size that is necessary in order to provide reliable results for a specific element, as shown in Figure 5. It can be seen that the number of non-metallic inclusions obtained from PDA/OES and EE+SEM agrees satisfactorily well within the size range of inclusions from 2 to 6 μm .

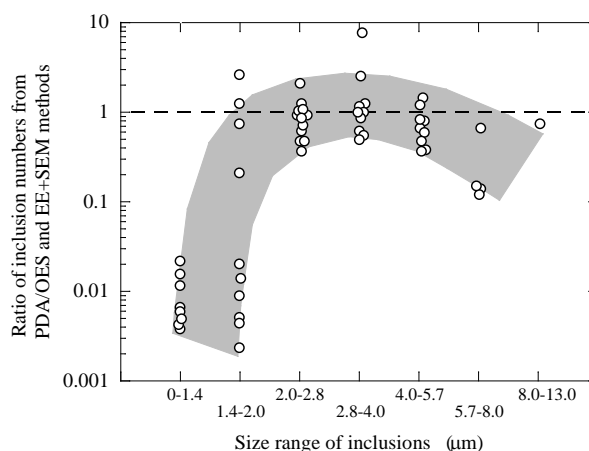


Fig. 5 Ratio between inclusion numbers obtained from PDA/OES and EE+SEM methods for different size ranges of inclusions

LA-ICP-MS. Another perspective method of fast determination of inclusion characteristics, from our point-of-view, can be a Laser Ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS) method.^[6-10] Until recent times the LA-ICP-MS was used for direct analysis of bulk composition of steels and alloys, spatial and surface distribution of elements, and for mapping of inclusions in metal samples. However, in lab scale studies it was found that the compositions of MgO-Al₂O₃-CaO and MnO-SiO₂ particles determined by the LA-ICP-MS, EE+SEM and chemical analysis agreed well.

A schematic illustration of LA-ICP-MS system is shown in the left diagram of Figure 6.^[8] In this method, a surface of a metal sample in an ablation chamber is irradiated and melted by a Nd:YAG laser beam. Then, a fine sample aerosol is formed by the laser beam, transported by the stream of Ar carrier gas to the ICP torch and ionised in an Ar plasma. The number of ions having different mass is determined in a mass spectrometer. The process of inclusion ablation on a sample surface can be directly controlled visually on a computer monitor by using a CCD system. The main principle of this method implies that the mass of each element consisted in ablated inclusion is proportional to the area of the ion intensity peak for this element on a time chart, as shown in the right diagram of Figure 6. As seen, the complex inclusions contain two or more ion intensity peaks for respective components of this inclusion. The peak area of each ion can be used for a determination of the composition and size of the ablated inclusions. For this purpose, relationships between the average ion intensities and the concentrations of corresponding element in standard samples can be used as a calibration line for a determination of the inclusion composition. The calibration lines for determination of inclusion sizes were obtained by laser ablation of particles with known sizes and compositions.

Laboratory tests were done to estimate the Al₂O₃ size distributions by using different analytical techniques. More specifically, hot-pressed iron samples with a 0.1mass% Al₂O₃ content were prepared by using the standard (JIS Z 8901) white fused Alumina powders with known size distributions ($d_v = 6.8 \pm 1.7$ and $13.1 \pm 2.9 \mu\text{m}$ for powders No.3 and No.4, respectively).^[9] After hot-pressing of mixtures of Fe and Al₂O₃ powders at 1000°C under a 30 MPa pressure in an Ar atmosphere, the samples were analysed by the LA-ICP-MS method. The samples were also electrolytically extracted before being studied in three dimensions by using SEM (EE+SEM) and SPOS (EE+SPOS).

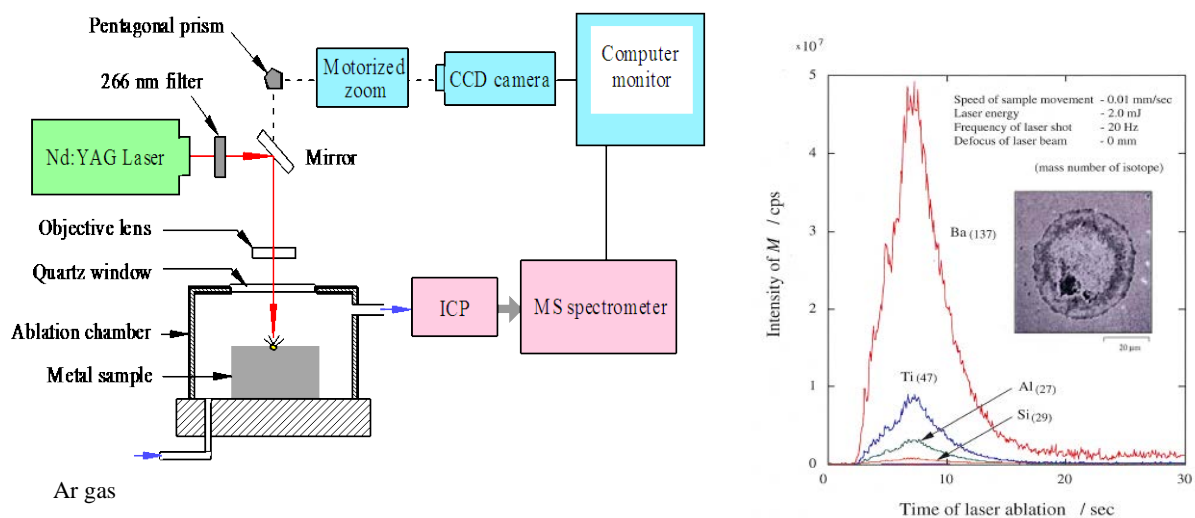


Fig. 6 Schematic diagram of LA-ICP-MS system (left diagram) and typical intensity peaks for different ions by laser ablation of complex oxide inclusion (right diagram)

The comparison of the particle size distributions in Fe-0.1mass% Al₂O₃ laboratory samples measured by the EE+SEM, EE+SPOS and LA-ICP-MS methods is shown in Figure 7. It is apparent that the size distributions of Al₂O₃ particles in both lab scale samples obtained by using LA-ICP-MS, EE+SEM and EE+SPOS methods agreed satisfactorily well in the particle size range of 1 to 25 μm.

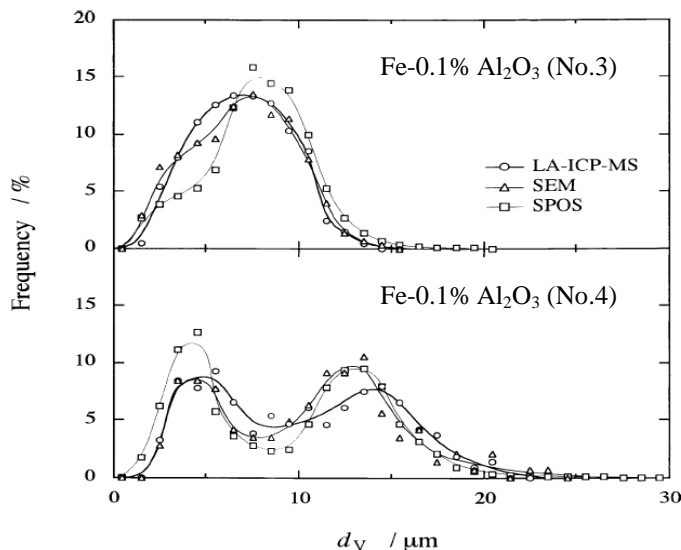


Fig. 7 Size distributions of Al₂O₃ particles in hot-pressed iron samples obtained by LA-ICP-MS, EE+SEM and EE+SPOS methods

The size distributions of non-metallic inclusions (MgO, MgO-Al₂O₃ and Al₂O₃) in a melted Fe-10%Ni alloy deoxidised with (0.05-0.1%) Mg and (0.05-0.1%) Al were also investigated using the LA-ICP-MS and the EE+SEM methods.^[10] It was found that the PSD for inclusions larger than 1 μm obtained by the LA-ICP-MS method is in good agreement with the data from the EE+SEM method in most experiments.

Based on the obtained results, it is believed that the LA-ICP-MS method has a very good potential to be used for rapid analysis of inclusions and clusters in steel samples. Thus, it may provide online control of steel quality on different stages of steelmaking processes. Depending on the cleanness of steel, the main characteristics (such as composition, number and size) of 40-200 non-metallic inclusions in the size range 1-40 μm can be determined during 5-20 minutes of the LA-ICP-MS analysis.

4. Off-line estimation of inclusion characteristics

A determination of inclusion characteristics (such as composition, size and number) in steel samples was carried out by using a conventional two-dimensional (2D) observation of inclusions on polished cross section of steel samples (CS+SEM) and from developed three-dimensional (3D) investigation of inclusions after electrolytic extraction (EE) from steel matrix.

Electrolytic extraction. Specimens (15×10×4 mm) were cut from metal samples taken at different stages of steel production to carry out 3D investigations of inclusions and clusters in different steel grades. The electrolytic extractions of inclusions from these specimens were carried out by using 10% AA (1 w/v% tetramethylammonium chloride - 10

v/v% acetyl acetone - methanol) or 2% TEA (2 v/v% triethanolamine - 1 w/v% tetramethylammonium chloride - methanol) electrolytes. After electrolytic extraction, the solution with un-dissolved non-metallic inclusions and clusters was filtrated through polycarbonate (PC) film filters with open pore sizes of 0.05, 0.4 or 1 μm . Thereafter, 3D investigations of inclusions on a surface of film filter were carried out by using SEM (EE+SEM method). The weight of the metal dissolved during the extraction varied from 0.05 to 0.40 g. In addition, the depth of a dissolved layer on surface of metal specimens (50 - 500 μm) depends on the specific steel grade and purposes of electrolytic extraction. Overall, the total number of analysed inclusions for determination of the particle size distribution in each specimen was more than 300.

Along with conventional methods for two-dimensional (2D) observations of non-metallic inclusions on polished cross section of steel samples (CS+SEM), contemporary methods are in recent years widely applied for three-dimensional (3D) investigations of inclusions after chemical or electrolytic extraction from steel samples. One such perspective method is the electrolytic (potentiostatic and galvanostatic) extraction techniques (EE). In this case, a conductive steel matrix can be dissolved electrolytically. However, non-conductive non-metallic inclusions do not dissolve in the electrolyte. Therefore, they can be collected on a surface of film filter after filtration of a solution. After electrolytic extraction, the characteristics of inclusions and clusters (such as morphology, size, number and composition) can be investigated and analysed by using SEM. The SEM typical inclusions and cluster are shown in Figure 8.

Several studies^[11-13] showed that the 3D investigation of particle size distribution (PSD) and morphology of inclusions and clusters after electrolytic extraction is more accurate in comparison with usual 2D CS+SEM method, particularly by investigation of clusters and small size inclusions.

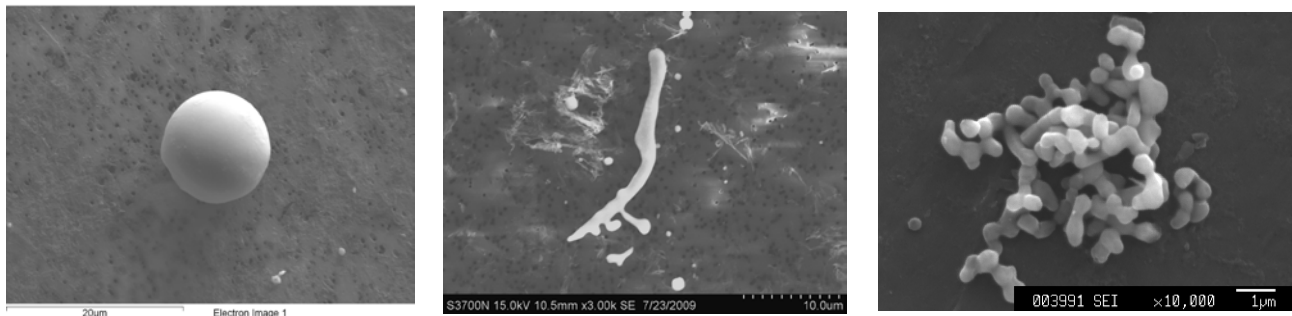


Fig. 8 Typical spherical $\text{CaO-Al}_2\text{O}_3\text{-MgO-SiO}_2$, elongated MnS inclusions and ZrO_2 cluster on surface of film filter after electrolytic extraction (EE+SEM method)

The EE method cannot be used as a rapid method for determination of inclusion characteristics because it needs an additional time for preparation of samples, electrolytic extraction and filtration. However, this method in combination with other investigation techniques (such as SEM, Photo Scattering Analysis or Coulter Counter) can be used as a reference method for determination of inclusions and clusters in steel samples, as shown in the present study.

Table 2 Successful applications of electrolytic extraction (EE+SEM) for 3D investigation of non-metallic inclusions and clusters in different industrial steels and alloys

Steels and alloys	Content (%)	Inclusion types	Morphology	Composition	PSD (μm)	SEV*	Ref. **
Low carbon low alloyed steels (samples: liquid steel, ingot, rolled steel and final product)	0.01-0.2%C, < 1.5%Cr	Oxides, sulfides, nitrides, complex NMI. Sulfides	OK	CaO, MgO, Al ₂ O ₃ , SiO ₂ , TiO _x , TiN, S	0.05-20	OK	up
			OK	MnS	2-250	OK	13, up
High silicon stainless steel (samples: liquid steel)	0.2-0.5%C, 19-24%Cr, 11-20%Ni	Oxides, sulfides, complex NMI	OK	MgO, SiO ₂ , Al ₂ O ₃ , CaO, S	1-25	OK	14
253MA stainless steel (samples: liquid steel, nozzle zone)	0.08-0.1%C, 19-21%Cr, 9-11%Ni	Oxides, complex NMI	OK - clusters	REM _x O _y , Al ₂ O ₃ , SiO ₂ , S	3-25	OK	up
316L stainless steel (samples: liquid steel)	0.02%C, 17%Cr, 10%Ni	Oxides, sulfides, complex NMI	OK	Al ₂ O ₃ , MgO, CaO, SiO ₂ , S	0.05-6		15, up
Tool steel – AISI H13 (samples: liquid steel, ingot)	0.3-0.4%C, 4-6%Cr, 1-2%Mo	Oxides, sulfides, complex NMI	OK	CaO, MgO, Al ₂ O ₃ , SiO ₂ , S	2-30	OK	16, up
Alloy-powder metallurgy (samples: liquid steel, final product)	0.3-0.5%C, <3%Cr, <0.5Mo	Oxides, sulfides, complex NMI.	OK	SiO ₂ , Al ₂ O ₃ , CaO, MgO, TiO _x , MnS	1-25	OK	up
High carbon steel	0.8-0.9%C	Oxides, sulfides, complex NMI.	OK	SiO ₂ , Al ₂ O ₃ , MnS	3-40		up
Fe-alloys (FeTi, FeCr, FeNb...)		SiO ₂ , Al ₂ O ₃ , TiO _x , ...	OK	OK	1-30		up

* - Statistics of Extreme Value (applied for estimation of largest size of inclusions and clusters);

** up - unpublished results

Today, the electrolytic extraction method can successfully be used for 3D investigations of inclusions and clusters in different industrial steels and alloys. Some examples of the application of EE the method on different samples are given in Table 2. It can be seen that EE+SEM method can be used for analysis of composition, size distribution and morphology of different inclusions and clusters in steel samples taken from different stages of steelmaking for a wide spectrum of steel grades. Moreover, a combination of Statistics of Extreme Value (SEV) analysis and EE+SEM method can significantly improve the accuracy of estimating the maximum size of inclusions and clusters in steel samples.^[12,13]

The main characteristics of different analytical methods used in present study for on-line and off-line investigations of inclusions and clusters in steel samples are given in Table 3. It should be noted that each technique for investigation of inclusions and clusters in steel samples has advantages and disadvantages depending on the purposes and analysed steel grade. However, only the OES/PDA and LA-ICP-MS techniques can be used for rapid estimations of inclusions in steel for on-line correction during an industrial steel production. However, the accuracy of other analytical methods (such as EE+SEM and CS+SEM) is higher. Thus, these may be used for off-line determinations of the inclusion characteristics.

Table 3 Characteristics of different methods for investigation of inclusions and clusters in steel samples

Method of analysis	CS+SEM	EE+SEM	EE+SPOS	OES-PDA	LA-ICP-MS
Investigation	2D	3D	3D	2D (3D)	3D
Ref.		[11-13]	[17]		[6,7]
Depth of analysed metal layer (μm)	surface	20-500	20-500	≤ 11	10-70
Total analysed metal weight (g)		0.05-0.50	0.05-0.50	~ 0.0008	
Size range of analysed particles (μm)	> 0.1	0.015-300	0.5-400	2-6	0.5-100
Analysis of inclusion composition:					
- Composition of each phase (heterogeneous particles);	OK	OK (on surface)	No	No	No
- total composition	OK	OK ($< 2\mu\text{m}$)	No	OK	OK
Analysis of size distribution:					
- total PSD;	OK	OK	OK	limited	OK
- PSD as a function of inclusion composition	OK	OK	No	limited	OK
Number of measured particles for PSD	200-1000	200-5000	10^5 - 10^8		100-500
Special preparation of sample surface	polishing	needed	needed	grinding	grinding
Time consuming (h)	2-6	(2~12)+ (2~6)	(2~12)+1	0.1-0.3	0.2-0.5
	(Off-line)	(Off-line)	(Off-line)	(On-line)	(On-line)

5. Conclusions

The scientific results, which were obtained in recent 10 years by members of the Division of Applied Process Metallurgy in KTH in the field of testing and improvement of analytical techniques for investigation of inclusions and clusters in steel samples, can be summarized as follows:

1. For minimization of harmful effect of reoxidation, involving of slag inclusions and decreasing of collision and agglomeration of inclusions, the argon-protection during sampling of liquid steel is more effective in comparison with a conventional metal-cap-protection. Particularly it is important by investigation of inclusions and clusters in Lollipop samples with 6 and 12 mm thicknesses.
2. The PDA/OES and LA-ICP-MS techniques can be applied for rapid determination of inclusion characteristics (such as composition, size and number) in steel samples to obtain feedback for on-line correction of technological processes during ladle refining and other stages of steel production. The number of inclusions in the size ranges from 2 to 6 μm and from 1 to 40 μm obtained from PDA/OES and LA-ICP-MS techniques, respectively, agreed satisfactorily well with those from electrolytic extraction method (EE+SEM). However, the accuracy for the determination of inclusion characteristics in steel samples depends on the composition of steel and analysed elements.
3. The electrolytic extraction method in combination with other analytical techniques can be successfully applied as a reference method for 3D investigation of inclusions and cluster in steel samples taken on different

steelmaking stages of various industrial steel grades.

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