EVALUATION OF THE RESULTS OF STEEL PURITY ACHIEVED IN STEEL PRODUCTION USING SSE METHOD

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Abstract

The paper presents the results of measurements which test steel purity achieved using the Single Spark Evaluation (SSE) method. It describes the possibilities of using this method for the ways in which it is possible to carry out the correction of the current technology of steel making in the production line of an electric arc furnace, ladle furnace and caisson (EOP-LF-VD) in the steel works of Vítkovice Heavy Machinery a.s. Increasing steel purity and all mechanical properties of steel can be achieved by means of technologies which use secondary metallurgy equipment. Controlling the production of melts in the secondary equipment affects mainly the quantity, size and morphology of inclusions.

Keywords: purity

1. INTRODUCTION

The ever higher requirements for the attainment of the level of mechanical values as ductility, toughness or fatigue properties of steel increase the demands put on a manufacturer in seeking new technological procedures. One of the main potentialities on how to attain higher level of the mechanical properties of steel is improvement of the metallurgical purity. This can be attained by applying the technological procedures used in the secondary metallurgy that make it possible to influence the size, amount, and morphology of inclusions [1]. In the conditions of the Vítkovice Heavy Machinery, a.s. Steel Works, this is assured by the equipment for steel processed in pot furnace (LF) and the equipment for vacuum processing of steel in caisson (VD). The work was aimed at assessing the continuous metallographic purity as soon as when the steel is being produced in the electric gas furnace (EOP), in the follow-up pot furnace (LF) and - when vacuum processed in caisson - using the VD technology. It is also aimed at acquiring a general idea of the effect of the introduced detail technological procedures that are used to ensure metallographic purity.

The metal samples were taken from the steel production process to be processed on the optical emission spectrometer that evaluates the analytical signal with resolution of each single spark using the Single Spark Evaluation (SSE) method. A part of the analytical signal is proportional to the average element content in the taken sample (conventionally, during the steel production process) while the other signal part corresponds to the concentration of element in the inclusion. The Single Spark Evaluation (SSE) method evaluates the information on the inclusion amount, type, any composition. The great advantage of SSE method is the fact that preparation of a sample for analysis on a spectrometer is identical to the disk-pin sample preparation method. The analyses of a standard test sample of a chemical composition of steel were extended by 20 seconds only. The method took place in on-line manner. Depending on the chemical composition of produced steel, the emission optical spectrometer's software was set for the element analysis [2].

2. STEEL SAMPLING PROCEDURE AND METHOD

The samples were taken from the pot manually using the immersion samplers of the disk-pin. The first sample designated as LF1 represents the start of steel processing technology on a pot furnace as well as
the result of preliminary steel deoxidation in the pot after the slag-free tapping from the electric arc furnace. Once the start of sampling of each LF1 sample has been standardized, the attainment of 1580°C on LF was selected in this case. Given a specific time dwell associated with necessary heating of the tapped and deoxidated melt to this temperature there was a presupposition that a good concentration homogeneity of the melt is obtained together with a liquid, well mixed, newly generated alkaline slag. LF2 sample represents the condition of melt at the melt processing end in the pot furnace, the point where the main emphasis was put on the arrival at the slag melting mode. LP with a melt was prepared for consequent vacuuming of steel in the pot caisson using VD procedure. Detail technological guideline determined predominantly the steel temperature before vacuuming, chemical composition of steel, slag parameters, and oxygen activity. The VD1 and VD2 samples were taken at the start and at the end of vacuum processing of steel in the pot. Sampling of VD2 sample was followed by 15 minutes of homogenization of a porous shaped brick with argon. The samples were taken from the melts produced according to two technological procedures. The samples from melts no. K58494, K58500, K58507, K58508, K58509, K58555 were taken according to the TS 1 technological procedure while the samples of melts no. K58489, K58495 were taken according to the TS 2 technological procedure – melts with C of min. 0.5 of weight %.

Technological guidelines differ mainly in the preliminary steel deoxidation method in the teeming ladle after the tapping from the electric arc furnace. The other principles of melt control in the pot furnace and caisson are almost identical [2].

**TS 1**
- into the teeming ladle before tapping of 2kg/t CaC₂,
- make final alloying of FeSi to 0.15 hm. % (steel in furnace without Si after oxidation),
- in the production of higher carbon steels, e.g. C45, make final alloying with a calcinated anthracite
- in the first third of tapping a steel into the stream of Al 0.3 - 0.6 kg/t depending on the quality of produced steel and on the melted content of C
- after the admixture of Al, CaO + synthetic slag + FeMn (make final alloying to 2/3 of the final chemical composition)
- alternatively, FeCr (it was not present in case of the monitored melts)
- in the final third of the tapping time - 0.3 – 0.6 kg Al/t again

**TS 2**
- into the teeming ladle, given the production of high carbon steels, 3.0 kg/t CaC₂ + min. 3.0 kg/t of calcinated anthracite
- during the tapping of FeMn (to 2/3 of the final chemical composition) + CaO + synthetic slag before the end of tapping
- 0.5 - 0.7 kg/t Al depending on the melted content of C [2].

3. **STEEL SAMPLE ANALYSIS USING SSE METHOD**

Optical emission spectrometry represents certain analytical methods that are based on the measuring of ultraviolet radiation emitted by the sample. Individual methods may differ in their spectrum excitation procedures, e.g. by using an electric spark, arc, glow discharge or, alternatively, laser. Optical spectrometers with a spark discharge are usually completed with circuits to assure a high-energy pre-sparking for a perfect melting of a sample on the sample's surface at the point of sparking. Excitation is performed at the area of several tens of mm² and it penetrates into a depth of a up to several μm. The radiation that is emitted by the
sample is further processed by the optical and dispersion system depending on the wavelengths of individual elements. The wavelength is typical for each element and it represents a quality characteristic of radiation. We can read their intensities that are further converted into individual concentrations.

New special technique in the optical emission spectrometry, Single Spark Evaluation, makes use of a statistical evaluation of individual sparks to determine soluble and insoluble fractions of elements in the sample to be analysed. It provides information on in quantity and chemical composition of inclusions. Unlike the common analyses on the optical emission spectrometers where all the electric discharges are integrated. The Single Spark Evaluation technology processes individual spark discharges separately as depicted on Fig. 1 [3].

Fig. 1 Depiction of discharges using SSE technique (x-axis - impulses; y-axis - number of discharges)

Fig. 2 depicts homogeneous behaviour of the intensities of individual spark discharges. Individual spark discharges are illustrated on x-axis while the numbers of these discharges are displayed on y-axis. The distribution of acquired impulses can be approximated using Gaussian distribution. With a multiple of the standard mean-root-square error (3s, 7s) from the basic signal we will obtain a limit value for the quantification of impurities in a sample [3].

If the sample contains any inhomogeneity, higher intensity is measured than in the matrix element. This value will be demonstrated by formation of a high peak and the error from Gaussian distribution, that represents quantification of individual constituents, is detected. An example is given on Fig. 3 [3].
Fig. 2 Homogeneous representation of the results

Fig. 3 Homogeneous and inhomogeneous representation of the results

In case the high intensities of peaks are measured for more elements on the identical spark discharge that is illustrated on Fig. 4 we can say that specific spark discharge hit the area where an inclusion based on these elements is located. We can use a correlation analysis to identify which elements are present in the inclusion [3].

Fig. 4 Al- and Ca-based inclusions

4. ANALYSIS OF READINGS

The analysis of results that was carried out fails to correspond fully to the resultant composition of inclusions in the final product. This difference is caused by different rate of cooling of relatively small metal sample as compared to a multi-ton ingot. The analysis does not illustrate composition of inclusions and their size in the
final product but the actual inclusion types and steel purity that are analysed during the LF, VD technological step.

The sizes of analysed areas of small metal sample using SSE method were fully comparable to, for example, a micro-probe analysis. The total evaluated area for one sample is 80 fields that is equal to about 5 mm$^2$. The results obtained using SSE method are based on the analysis of 2000 sparks. If any particles with composition that differs from the basic metal matrix are present in specific point the peaks of present elements are detected and it is possible to analyse their bonds then.

The analyses are graphically represented on Fig. 5, 6, 7 and 8.

**Fig. 5** Average number of impulses for individual elements and melts LF1
Fig. 6 Average number of impulses for individual elements and melts LF2

Fig. 7 Average number of impulses for individual elements and melts VD1
Fig. 8 Average number of impulses for individual elements and melts VD2

Certain conclusions may be derived from the analyses and graphical representations of individual elements and melts from the technological steps of material's production flow:

- The mutual contexts in contents and the amount of inclusions among individual samples for a melt or for individual technological steps LF1, LF2, VD1 and VD2 appear to be hard to define.
- More frequent occurrence of AlO than AlCaO appears to be logical.
- SSE method disallows analysis of the occurrence of MnS bond.
- MnS is determined as MnS = S corr – CaS, where S corr is a summary content of the inclusions that contain sulphur.
- The occurrence of AlN and TiN appears to be minimal.
- Logical is CaS with its dominant bond of sulphur.

Fig. 9 depicts the development of a total number of detected impulses during individual technological operations for all the evaluated melts. In the total of detected impulses, these data correspond to the number of volume of inclusions in steel. The behaviour of detected impulses does not show a significant drop in the amount of inclusions as was expected in connection with the LF1 – VD2 steel processing procedure.
Fig. 9 Number of impulses with the LF1 – VD2 steel processing procedure

What results from the graphical representation on Fig. 10 to 19 are certain dependences of individual inclusions measured by using the SSE method for individual melts where the numbers of impulses were converted into the relative coordinates. We attribute the development of chart on Fig. 11 in the LF 1 technological operation to the processing error.

Fig. 10 Relative frequency for individual elements of K 58489 melt
Fig. 11 Relative frequency for individual elements of K 58494 melt

Fig. 12 Relative frequency for individual elements of K 58495 melt
Fig. 13 Relative frequency for individual elements of K 58507 melt

Fig. 14 Relative frequency for individual elements of K 58500 melt
Fig. 15 Relative frequency for individual elements of K 58508 melt

Fig. 16 Relative frequency for individual elements of K 58509 melt
Certain conclusions may be derived from individual analyses and graphical representations of elements and melts, the technological steps of material's production flow:

- The mutual contexts in contents and the amount of inclusions among individual samples for a melt or for individual technological steps LF1, LF2, VD1 and VD2 appear to be hard to define.
- More frequent occurrence of AIO than AlCaO appears to be logical.
- SSE method disallows analysis of the occurrence of MnS bond.
- The content of AIN and TiN appears to be minimal.
- Logical is CaS with its dominant bond of sulphur. The remainder of sulphur is not identified and it is presumed to be present with the occurrence of MnS.
- The occurrence of CaO has mostly the descending character.
- The total sulphur content has mostly the descending character.
- A special feature is relatively imbalanced content of SiO.

5. CONCLUSION

In the last decade of improvements to the technological and metallurgical processes, the content of non-metallic inclusions in the produced steel was reduced significantly, namely due to the application of the secondary metallurgy equipment. The LF – VD technology was employed in the conditions of our work. The performed work was aimed at testing the utilisation of SSE method in specific operating conditions.

Extraordinary economical profits can be expected on condition of a reliable system of monitoring the continuous quantity and composition of inclusions as early as during the melt control. Possible occurrences of poorer steel purity could be partially or completely removed by the on-line technological intervention into the melt control. This ability to monitor, interpret and the capacity to respond in convenient and timely
manner as early as when the steel processing is in progress should be allowed by the new Single Spark Evaluation technique employed in the optical emission spectrometry.

What accrues from the obtained results is the recommendation to distinguish composition of individual impulses and identify the correlations between individual elements in specific impulse. Also, what appears to be important is paying more attention to the differentiation of included peak. Further operating tests should be carried out for more significant number of melts.

REFERENCES