Combustion analysis of granular steel chips- The effect of the scale layer on the analysis of carbon and sulfur

Abstract

The new cylindrical cutter module of the HS-F 1000 milling machine allows the production of special chips from steel and iron samples. The chips show a smooth granular morphology and are particularly suited for thermal evolution methods. In the present study we examined the effect of the circumferential scale layer of production samples on the C and S content as determined by combustion analysis. For this purpose, we analyzed chips which were obtained from conical steel sample before and after removal of the scale layer. Statistical evaluation revealed no significant differences of the C and S content in chips with and without scale layer. The results demonstrate that the scale layer does not interfere significantly with the combustion analysis of production samples.

Key words

• **Combustion analysis** • **Chips** • **Milling** • **Scale layer** • **Steel**

Introduction

As an option for the HS-F 1000 milling machine, Herzog recently launched a new cylindrical milling unit that allows the production of chips which are particularly suitable for analysis by means of thermal evolution methods. The chips are characterized by an evenly granular shape and uniform size distribution. These morphological features make dosing and sample loading into the analytical instruments especially easy.

In the previous application note [1] we presented the analytical results for C, S and N by using granular chips. For all elements we found an excellent repeatability with values of the relative standard deviation that were in the same range as reported in previous studies. Furthermore, we did not notice any significant differences between analyses obtained from granular chips and from solid pieces cut out of the specimen. All test series were carried out with reference samples and bar material.

In the present study, we aim at investigating the analytical accuracy of chips obtained from production samples that were taken with immersion sampling probes. During solidification and cooling of the liquid steel usually a scale layer is formed at the surface of the production sample as a consequence of oxidation effects. The scale layer has an inhomogeneous

structure and consists of three different iron oxides, namely wuestite, magnetite and haematite. Moreover, there are indications that especially C might show an enhanced concentration in the scale layer compared to the bulk material [2].

In order to examine the impact of the scale layer on the combustion analysis we determined the C and S content of chips which were obtained from the same production samples before and after removal of the scale layer

Methods

All chips were produced in an automatic milling machine of the type HS-F 1000 with an integrated module for production of granular chips. All tests were carried out on conical steel samples. The samples were manually inserted into the machine by an operator.

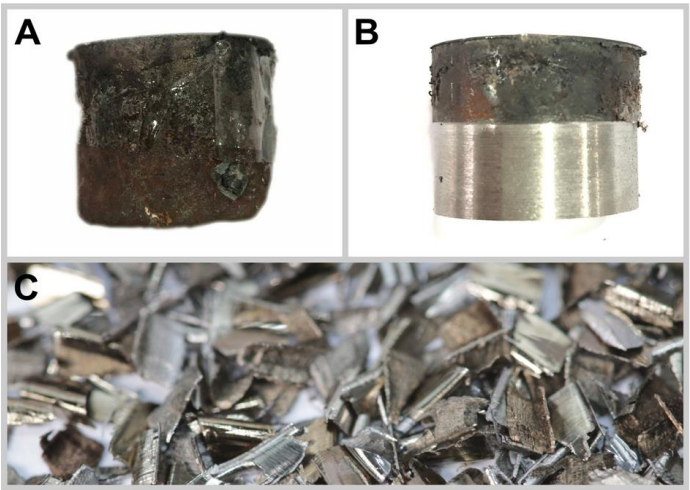

Figure 1: *(A) Conical steel production sample with scale layer at the sample circumference, (B) after removal of the scale layer by using a lathe. (C) Typical granular chips produced from the sample*

In condition 1 of the test procedure, the scale layer at the top of the sample was removed by a face-milling cutter. The scale layer in the region of the sample circumference remained (Figure 1, A). The support of the machine transported the sample to the chip production module. Here the sample was passed over the cylindrical milling cutter to produce the chips. The chips were collected in a cup which was taken out of the machine after each milling cycle for later analysis. Following thorough cleaning the cup was inserted again and the next milling cycle for the chip production was started. From each production sample we collected eight subsequent chip samples with preserved scale layer.

In condition 2 of the test procedure, we removed the scale layer from the circumference of the production sample by using a lathe (Figure 1, B). Subsequently, we produced a test series of eight chip samples without scale layer. Five different production samples were examined in this way resulting in a total of 80 chip samples.

For determination of C and S, we weighed approx. 1000 mg of the chips into a ceramic crucible. Subsequently, we added approx. 2.0 g tungsten as accelerator and introduced the crucible into the analyzer (Elementrac CS-i, Eltra, Haan Germany). In each production sample, we calculated the mean and standard deviation of the C and S content in chips with and without scale layer. In addition, we carried out a student's t-test to assess statistical differences in the concentration between the two types of chips. The respective p-values were reported in Tables 1 and 2, the significance level was set to 0.05.

Results

In the five samples examined, the mean C content in chips with scale layer was 0.1936 %, 0.2123 %, 0.5180 %, 0.4777 % and 0.1085 % (Figure 2, Table 1). The corresponding C values in chips without scale layer were 0.1915 %, 0.2201 %, 0.5253 %, 0.4845 % and 0.1092 %. For samples 2-5, the standard deviation (SD) for chips with and without scale layer was in the range between 0.0018 % and 0.0160 %. Only for sample 1, the SD both in chips with and without scale layer was higher with 0.0279 % and 0.0273 %. The underlying cause for the increased SD is the slight drop of the C content in analyses no. 7 and 8 of chips with scale layer and analyses no. 1, 2 and 3 in chips without scale layer (Figure 1). Since these measurements were carried out in directly adjacent layers, this finding suggests that the underlying cause is a local inhomogeneity of the C content within the sample.

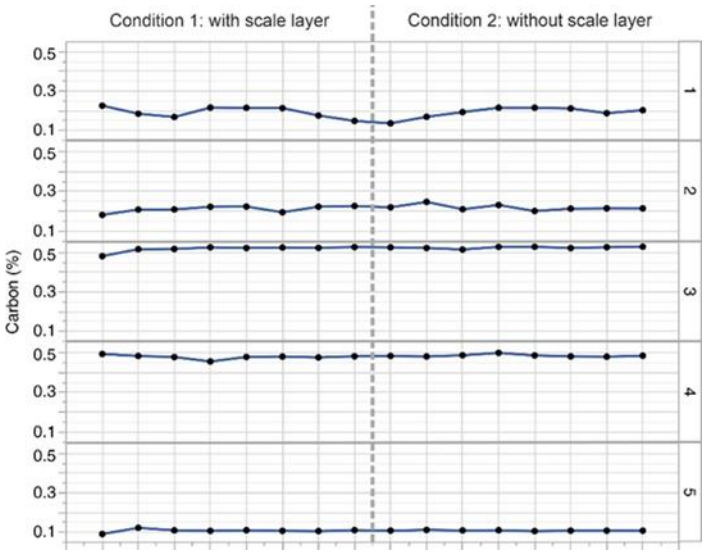

Figure 2: *Graphical display of the analytical results for C as determined by combustion analysis. In each of the five sample analyses were carried out with (condition 1) and without scale layer (condition 2).*

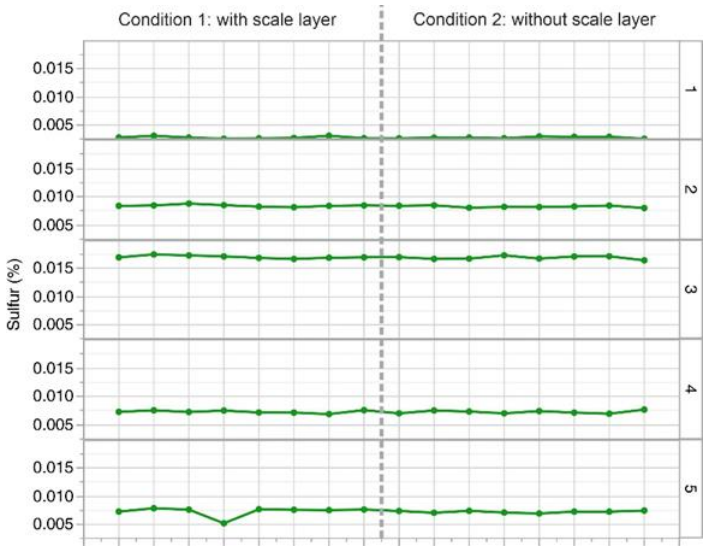

Figure 3: *Graphical display of the analytical results for S as determined by combustion analysis. In each of the five sample analyses were carried out with (condition 1) and without scale layer (condition 2).*

Sample	Condition 1: with scale layer		Condition 2: without scale layer		p-value
	Mean (%)	SD (%)	Mean (%)	SD (%)	
1	0.1936	0.0279	0.1915	0.0273	0.8828
2	0.2123	0.0160	0.2201	0.0141	0.3214
3	0.5180	0.0150	0.5253	0.0046	0.2218
4	0.4777	0.0106	0.4845	0.0061	0.1424
5	0.1085	0.0085	0.1092	0.0018	0.8339

Table 1: *Mean average and standard deviation (SD) of the C content for each of the five samples. The results for chips with (condition 1) and without scale layer (condition 2) are compared by a student's t-test. The p-value is listed. None of the differences was significant.*

Sample	Condition 1: with scale layer		Condition 2: without scale layer		p-value
	Mean (%)	SD (%)	Mean (%)	SD (%)	
1	0.0028	0.0002	0.0027	0.0001	0.8280
2	0.0084	0.0002	0.0083	0.0002	0.0920
3	0.0171	0.0003	0.0169	0.0003	0.3436
4	0.0073	0.0002	0.0073	0.0003	0.7765
5	0.0073	0.0009	0.0072	0.0002	0.8372

Table 2: *Mean average and standard deviation (SD) of the S content for each of the five samples. The results for chips with (condition 1) and without scale layer (condition 2) are compared by a student's t-test. The p-value is listed. None of the differences was significant.*

The mean S content in chips with scale layer was 0.0028 %, 0.0084 %, 0.0171 %, 0.0073 % and 0.0073 % whereas the values for chips without scale layer were 0.0027 %, 0.0083 %, 0.0169 %, 0.0073 % and 0.0072 %. The SD of the S content for chips with and without scale layer was comparable in the range between 0.0001 % and 0.0009 %.

Both for the C and S content, the t-test revealed no significant differences between chips with and without scale layer (Table 1 and 2).

Discussion

In the production samples evaluated for this study, we found no significant influence of the scale layer on the C and S content as measured by thermal evolution of granular chips. It should be emphasized that for all trials the scale layer at the top of the sample was removed with the help of a face-milling cutter. By contrast, in condition 1 of the test procedure, the scale layer in the sample circumference has been left in place. We recommend that the top scale layer

should always be removed before production of granular chip samples. The reason for this is that milling of the porous top scale layer would result in a high proportion of finest particles potentially resulting in contamination of machine surfaces.

The concentration of C in the examined samples was in the range of 0.5 % and less. For samples with a comparable chemical composition it is therefore unlikely that the scale layer interferes with the results of the combustion analysis. However, for samples from high-carbon or ultra-high-carbon steel we cannot exclude that there might be a local accumulation of C in the scale layer which could possibly have an effect on the C analysis. For these samples, a systematic influence of the scale layer should be investigated before routine chip analysis is implemented in the laboratory. If an influence is detected, the circumferential scale layer sample can be removed automatically by the deburring function of the HS-F 1000 before chip production.

The analysis of other light elements including N was not subject of this study and would have to be investigated separately. For C and S analysis, the present study demonstrates that production of granular chips from production samples ensures the optimal preparation for thermal evolution methods.

References

[1] Herzog Application Note no. xx

[2] Sturm, V., Vrengor, J., Noll, R. and M. Hemmerlin (2004): Bulk analysis of steel samples with surface layers by enhanced laser ablation and LIBS analysis of C, P, S, Al, Cr, Cu, Mn and Mo. J. Anal. At. Spectrom., 19: p. 451-456

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