Application note No. 50 | 2022

HERZOG

The Influence of Sample Preparation on the Combustion Analysis of Chips Obtained from Steel Samples

Abstract

The recently launched cylindrical cutter module of the HS-F 1000 milling machine enables the production of special “granular chips” from steel samples. The chips have a short, even and smooth morphology and are particularly suited for determination of the C, S, O, N and H content by using combustion analysis. We have previously shown that there were no significant differences in the analytical results obtained from granular chips and punched pieces. In the present study we investigated whether the analysis of granular chips was different from chips produced by face-milling cutter and chips produced by drilling. Six different CRM samples were used for the study. From each CRM sample we produced 10 aliquots of each of the three chip types (granular, face-milling, drilling) so that a total of 180 samples were examined by combustion analysis. The statistical analysis of the C and S results showed no significant differences between the different chip types (ANOVA). The findings of this study confirm that the mode of chip preparation has not any impact on the determination of the C and S content.

Key words

• **Combustion Analysis** • **Chips** • **Drilling** • **Milling** • **HS-F 1000**

Introduction

Combustion analysis is a frequently used analytical method in quality control laboratories of the steel industry. The technology is based on infrared absorption and thermal conductivity to measure combustion gases within metallic sample and to determine the content of carbon, sulfur, oxygen, nitrogen, or hydrogen. Typically, samples of about 500- 1500 mg in form of punched pieces, chips, slugs, pins, strips etc. are used for analysis. Herzog has recently developed a new technology to produce samples in form of chips that are especially suited for automatic combustion analysis. The so-called “granular chips” are characterized by an even shape and uniform size distribution which make exact dosing and handling particularly easy.

Herzog provides a fully integrated system for automatic sample preparation and combustion analysis of chips from steel samples. The granular chips are produced by a specially developed cylindrical cutter module which is built into the HS-F 1000 milling machine (Figure

1) . The chips are collected by an included trap and transported pneumatically to a cyclone. There, the chips are separated and forwarded to a dosing chute positioned in the CNSLab. Within the CNSLab, gravimetric dosing, addition of accelerators, input into the analytical instruments, analysis and handling of reference materials are performed fully automatically. The smooth and uniform shape of the chips (Figure 2) and the special design of all automation components minimize the potential cross-contamination from previous samples.

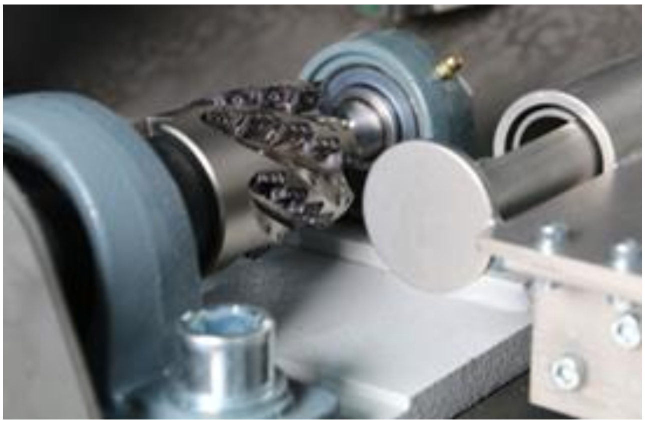

Figure 1: *Cylindrical cutter module within the HS-F 1000 for production of granular chips*

In previous studies, we have demonstrated the excellent repeatability for C, S and N analysis by using granular chips [1, 2, 3]. We have also shown that there are no significant analytical differences between analyses made from punched pieces and granular chips taken from the same sample [1]. In this application note, we aimed at investigating whether there are significant variations between granular chips, chips produced by face-milling cutter and chips produced by drilling.

Figure 2: *Typical aliquot of granular chips obtained from a steel sample showing the typical even, smooth and short morphology.*

Methods

The three different types of chips (granular, face-milling, drilling) were produced from six CRM samples (CRM A – CRM F) provided by a large international steel manufacturer. First, we produced the granular chips. All samples were manually inserted into the HS-F 1000 milling machine containing the cylindrical milling cutter module. After initial removal of the sample's top layer by the face-milling cutter, we produced 10 aliquots of 2000 mg from each of the six CRM samples. Each sample was automatically collected and pneumatically transported to a receiving magazine. Here, the samples were carefully removed and bagged into a plastic container.

Subsequently, we produced chips from each of the six CRM samples by means of the face-milling cutter in the HS-F 1000. Again, we produced 10 aliquots of 2000 mg per sample. Finally, each of the six samples was clamped on a drilling machine. By means of vertical drilling at different locations, ten aliquots of 2000 mg per sample were prepared from each CRM sample. Between different CRM samples, the drill head was thoroughly cleaned to avoid carryover between samples. Altogether 180 samples were produced for this analysis.

All samples were sent to a reference laboratories of the steel manufacturer. Sample dosing and analysis were done manually. For determination of C and S, 500- 1000 mg of the chips was weighed into a ceramic crucible. Subsequently, 1.5- 2.0 g tungsten were added as accelerator and then the crucible was introduced into the analyzer (LECO, model CS844, St. Joseph, MI).

From the ten analytical results for each CRM sample and each chip type we calculated the mean. We used the analysis of variance (ANOVA) to determine whether the analytical results of the three chip types obtained from each CRM sample were equal or different. The significance level was set at 0.05. All P values below that value would indicate a significant difference in the analytical results of the three chip types.

Results

The mean percentage carbon content in all six CRM samples was between 0.0020 % (CRM D) and 0.2760 % (CRM C). For S, the range was between 0.0016 % (CRM F) and 0.0080 % (CRM E). The detailed mean values are specified in Table 1 and displayed in Figure 3. For neither CRM sample we found a significant difference of the C or S mass content as assessed by the ANOVA test.

	CRM A				CRM B				CRM C			
	Granular chips	Face-milling	Drilling	*ANOVA P value*	Granular chips	Face-milling	Drilling	*ANOVA P value*	Granular chips	Face-milling	Drilling	*ANOVA P value*
C	0.1252	0.1244	0.1257	*0.3794*	0.0839	0.0839	0.0839	*0.9768*	0.2750	0.2776	0.2768	*0.2062*
S	0.0066	0.0065	0.0065	*0.8368*	0.0026	0.0026	0.0026	*0.8999*	0.0029	0.0028	0.0028	*0.2329*

	CRM D				CRM E				CRM F			
	Granular chips	Face-milling	Drilling	*ANOVA P value*	Granular chips	Face-milling	Drilling	*ANOVA P value*	Granular chips	Face-milling	Drilling	*ANOVA P value*
C	0.0022	0.0020	0.0019	*0.5360*	0.0048	0.0049	0.0049	*0.6954*	0.0483	0.0485	0.0480	*0.2286*
S	0.0048	0.0049	0.0049	*0.2329*	0.0079	0.0080	0.0080	*0.8186*	0.0015	0.0018	0.0017	*0.1994*

Table 1: *Mean percentage C and S mass content (%) of the different chip types obtained from various CRM samples. As assessed by ANOVA we did not observe any significant difference of the mass content between the different chip types. All ANOVA P values were higher than 0.05.*

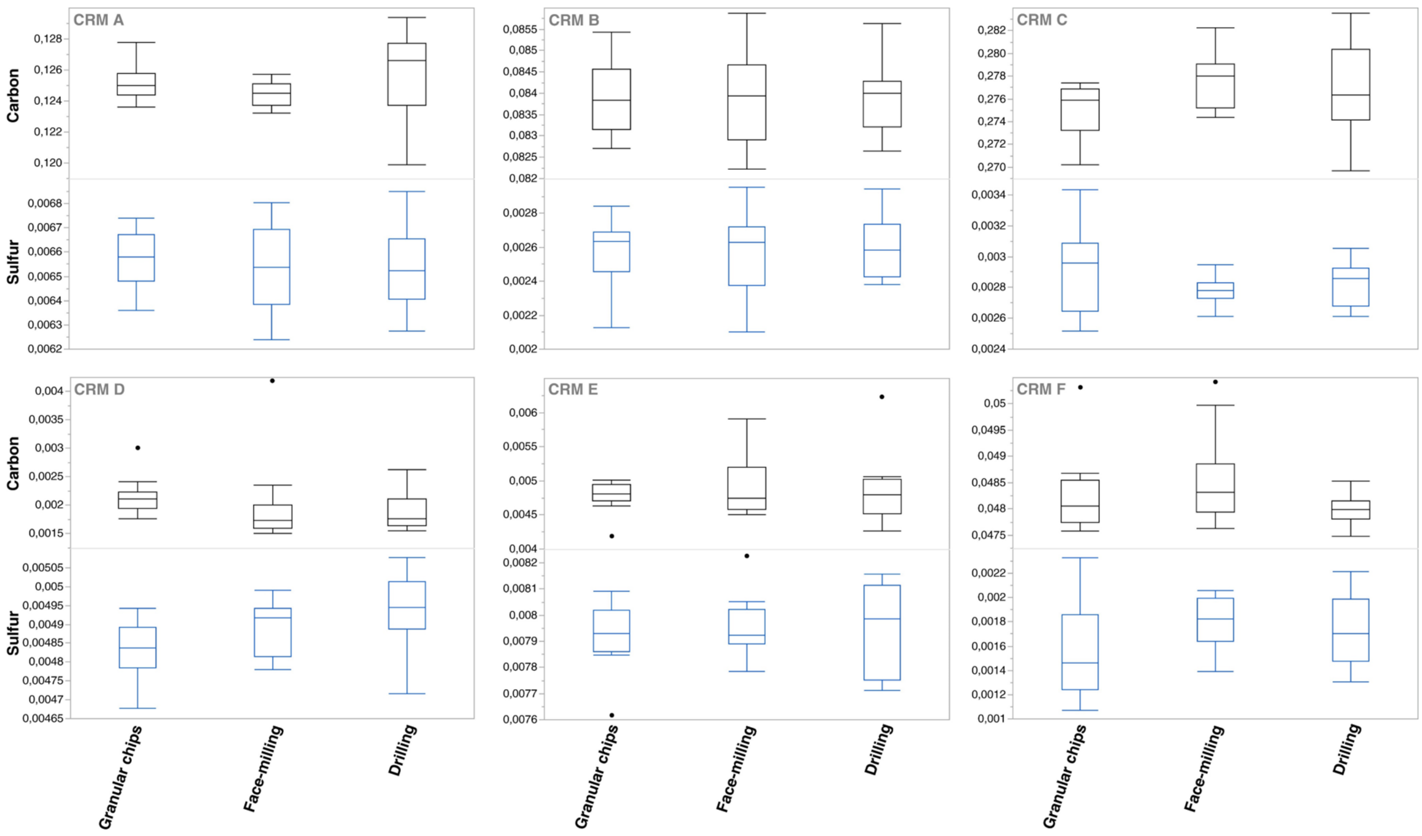

Figure 3: *Boxplot diagrams of the C and S mass content showing the locality, spread and skewness of the data obtained from analysis of the three different chip types in the six CRM samples.*

Discussion

In this study, we were able to demonstrate that combustion analysis of granular chips leads to the same analysis results as using chips produced by a face milling cutter or a drilling machine. For this study, we used CRM material to rule out any bias originating from sample inhomogeneities as a possible cause of differences.

The use of face milling cutters or drilling machines is a very common method for obtaining chips for combustion analysis. However, the disadvantage of these two methods is that they can hardly be automated. The chips produced by face milling cutters generally have a spiral shape. These chips tend to entangle with each other and thus form interlocking chains. Depending on the hardness and ductility of the sample material, the chip spirals may become very long. Precise weighing of the chips is hardly possible without manual intervention to sort out long chips and destroy entanglements. Therefore, automation of the dosing process is usually not an option.

Chips produced by use of a drilling machine usually have a short helix shape. This makes automatic dosing easier. However, the automated chip production and collection within a drilling machine is technically challenging. It requires a dedicated automated machine that needs to be seamlessly integrated into the sample preparation circuit. No automatic solution has yet been developed for the fully automated processing of drilling chips.

Chip production with the help of the cylindrical cutter module enables easy sample preparation within the HS-F 1000, contamination-free transport, and precise dosing within the Herzog CNSLab. This guarantees a fully automated combustion analysis that no longer requires the involvement of any laboratory personnel. The results of this study confirm that the mode of chip preparation has not any impact on the determination of the C and S content. Therefore, the use of granular chips makes laboratory processes more efficient and allows the laboratory staff to focus on more demanding tasks.

References

[1] HERZOG Application note 38/2021: A novel approach for thermal evolution analysis of steel samples by using chips with granular morphology.

[2] HERZOG Application note 40/2021: Combustion analysis of granular steel chips- The effect of the scale layer on the analysis of carbon and sulfur.

[3] HERZOG Application note 41/2021: Contamination-free sample preparation and transport of granular chips used for combustion analysis.

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