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Equipment for lustrous carbon determination

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Abstract

In this paper methods of determination of total pyrolytic carbon and its fraction: lustrous and amorphous carbon was shown. They are used in foundry industry as carbonaceous materials, i.e. in coal dust replacements and moulding sands. The principle of model analyser working, which uses NDIR module detection, and example results of such analysis were also presented.

Keywords: Cast quality management; Coal dust replacements; Foundry moulds; Pyrolytic carbon; Lustrous carbon fraction.

1. Introduction

Fast and accurate method of lustrous carbon contents determination in carboniferous materials (i.e. coal dust replacements and foundry moulds) is essential for industrial plants and research institutes connected with foundry and matter of coal dust replacements, which are used as additives to moulding sands. The importance of lustrous carbon in foundry processes is well-known [1-4]. It is formed from carbonaceous materials as a result of thermal influence of liquid metal introduced into foundry form. Gases, which are produced with limited access of oxygen, creates reductive atmosphere in area of contact of liquid alloy with mould. This atmosphere and catalytic reaction of various components of mould creates condition for production of lustrous carbon from gaseous phase.

It precipitates with a thin lamina on the surface of grains of mould, heated up to temperature of 650÷1200^{o}C, especially in chemical inactive places, and it creates concise and strongly sticky layer.

That layer prevents foundry alloy from penetrating into foundry mould, and assures that received casts has low roughness and corrugation of surface, highly accurate dimensions and shape, reduced casting defects.

It is assumed, that optimal addition of carbonaceous materials in foundry mould should produce in process of it's gassy content of lustrous carbon in amount from 0,3 to 0,6% [4].

Too small content of additives in mould does not prevent in sufficient degree from casting defects. On the other hand, too high amount could be the reason of worsen surface of the cast, excess carburization, higher harmful for environment and worsen process economics.

Determination of lustrous carbon content in added to foundry mould materials (coal dust and its replacements), that allows for batching of that additive to newly created foundry mould in optimal amount. It requires usage of appropriate method of measurement.

2. Methods of lustrous carbon (pyrolytic) determination – present state of knowledge

Methods discussed below, although that their titles suggests of lustrous carbon content determination, in fact they allow determination of total amount of lustrous (LC) and amorphous (AC) carbon. In reality they concern to determination of pyrolytic carbon. In process of forming the shape of cast the main role plays LC. That's why the issue of determination of LC is currently subject of intensive research [6-9]. It follows from above reports, that is defined in literature [1-5] as "lustrous carbon" is in fact a mixture of two different morphological components: lustrous and amorphous carbon. LC is structurally similar to graphite and it oxidizes in temperatures 650-850°C, while AC is structurally similar to soot (temperature of oxidization: 500-600°C). The quality of cast, as presented above, depends on amount of LC.

2.1. Bindernagel gravimetric method

In foundry industry commonly used method of determination of lustrous carbon content in carbonaceous materials (replacements of coal dust) is retort method worked out by J. Bindernagel [1]. It consists on precipitation of carbon on quartz wool and weighting of retort before and after the process of thermal treatment in temperature of 875°C ± 25°C.The common of usage of that method does not follow form its advantages, but from lack of another simple in use, repeatable way of determination. This method allows also only the total pyrolytic carbon determination in tested material, but lustrous carbon determination is not possible. Analysis made with this method are not only burdened with errors of determination of total content of lustrous and amorphous carbon fractions in pyrolytic carbon. Errors also flow from losses due to explosive freeing of gasses, as a result of leakiness that occurs between quartz pipe and crucible-pot with carbonaceous material. From time to time technical literature mentions about trials of modification of above method. They mainly focuse on changing of shape and size of analytical vessel, that in intention of authors should eliminate disadvantages of Bindernagel method. Despite that this is still most commonly used method of determination of lustrous carbon content. Also mandatory in Poland branch standard BN-88/4024-09 is based on this retort method [2].

2.2. IFG Düsseldorf method

The principles were worked out in IFG Düsseldorf prototype equipment for determination of lustrous carbon and were described in [3]. Tested sample is heated in condition of low vacuum without presence of oxygen. Pyrolytic gases thermaly decompose on incandescent wolfram wire [diameter 0,2mm, temperature (1300-2000°C)], that goes through the centre of measuring cell. Precipitated on wire lustrous carbon changes its electrical resistance and gives a measuring signal. Measuring cell has built-in window, through which temperature measurement of incandescent wire with optical pyrometer is possible.Until now this method was not implemented to industrial practice yet.

3. Model analyzer for determination of lustrous carbon content in pyrolytic carbon

Methods mentioned in § 2 in fact apply to pyrolytic carbon. In foundry industry determination of real content of lustrous carbon in coal dust replacements and moulding sands is essential. This was an inspiration for working out model analyzer, that makes possible separation of lustrous carbon from pyrolythic carbon and determination of its content [8, 9].
As a result of these activities a model of analyser was designed, as unique of the word scale was able to measure in tested sample the determination of pyrolytic carbon or its fraction (Amorphous carbon fraction and Lustrous carbon fraction) and semi coke (remains after carbon pyrolysys)
The method of model working was based on principle, where the sample was analyzed in turns in pyrolysys process, selective pyrolysys products burning and fumes analyse using NDIR detector for exhaled carbon oxides.The pyrolysis process of tested sample is executed inside the reactor placed in horizontal electric tubular furnace in temperature of 900-1100°C. Inside the reactor there is a controlled gas atmosphere (neutral gas during pyrolysis, oxygen during burning process). Gas had flown through the reactor and probe was driven with a gas pump. This flow allows to carbon pyrolysis product sedimentation on the quartz wool and further it allows to feed them to the NDIR detector. Operating service works was reduced to prepare testing sample, load and discharge it from the analyzer after test. Process of determination pyrolitic carbon, or its fraction of semi coke is full automated and controlled by computer, as well as all values of process parameters; input / output gas flow, temperature and process duration, what gives a high repeatability of measurements and reduction of staff mistakes, and gives full storage data of executed results. Presented analyser allows both determinations; relative and absolute content of carbon in analysed material. There is possible to determinate amount of pyrolytic carbon and its fractions in materials which are usually used as a raw material in foundry moulds production and being widely applicable lately, carbon dust replacements. Fig. 1 shows the model of analyser. Exemplary results achieved are presented in tables below:

Fig. 1. Analyzer for determination of the lustrous and amorphous carbon in materials used as additives to the foundry moulds

Table 1.
Results of the pyrolytic carbon determination in the standard sample (GIG 75,75%) used with application of executed analyzer model

Sample number	Weight [g]	Results of pyrolytic carbon contain		
		C in CO_2	C in CO	Σ C
		[%]	[%]	[%]
75-A	0.1275	70.02	3.94	73.96
76-A	0.1243	72.83	4.87	77.70
77-A	0.1406	69.43	5.02	74.45
78-A	0.1295	72.13	5.28	77.41

Table 2.
Comparison of results for amorphous carbon (AC, lustrous carbon (LC) and total pyrolitic carbon (∑ C – as a sum of amorphous carbon and lustrous carbon) in standard samples made of carbon black, graphite and inert material - SiO_2). Process temperature was 900°C

Sample number	Weight			Result of determination						
	∑ mass	AC	LC	∑ C	AC	LC	∑ C	AC relative error	LC Relative error	∑ C relative error
-	[g]	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]
B-43	0.2018	30.48	20.61	51.09	31.33	19.59	50.93	2.79	4.95	0.33
B-63	0.1020	91.64	8.34	100	85.74	8.67	96.39	6.44	3.96	3.61
B-64	0.09998	92.09	7.91	100	93.11	852	101.72	1.11	7.71	1.72
B-65	0.0816	92.03	7.97	100	93.86	8.08	102.02	1.70	1.38	2.02
B-66	0.0817	92.17	7.83	100	92.95	7.99	100.95	0.84	2.04	0.95

Table 3.
Results of the pyrolytic carbon content determination (fraction of the lustrous carbon and amorphous carbon) and semicoke in the tetraphenylsilane samples with c content – 80,37% used with application of executed analyzer model

Sample number	RESULTS					
	Pyrolysis ΣC* [%]	Amorphous carbon ΣC* [%]	Lustrous carbon ΣC* [%]	Pyrolythic carbon ΣC* [%]	Semicoke ΣC* [%]	Σ Carbon in sample ΣC* [%]
C-36	13.12	37.40	9.75	60.27	20.09	80.36
C-37	10.21	39.06	10.51	59.78	18.88	78.66
C-42	8.72	42.22	7.55	58.49	17.92	76.41
C-43	9.46	42.59	8.65	60.70	18.00	78,70

where:
ΣC* [%] – is a sum of carbon (C from CO_2 + C from CO) [%].
** % - is a content of coal in tetraphenylsilan sample, where main ingredient content was 96% there is 82.72% C.
The tetraphenylsilane sample where consist of moisture was 1,90% → content of %C was 80.37% C.

Fig 2 shows an exemplary process window in pyrolitic carbon determination (as a fraction of amorphous and lustrous carbon) and semicoke for the standard tetrafphenylsilane sample.
Results, presented above, achieved with this analyzer concern to materials where content of carbon was over 8 %. These materials are used in the casting industry as carbon dust replacements. Actually, under design is a model similar to the model presented above, desired for measuring pyrolytic carbon content of foundry moulds. Initial results of those efforts are shown below.

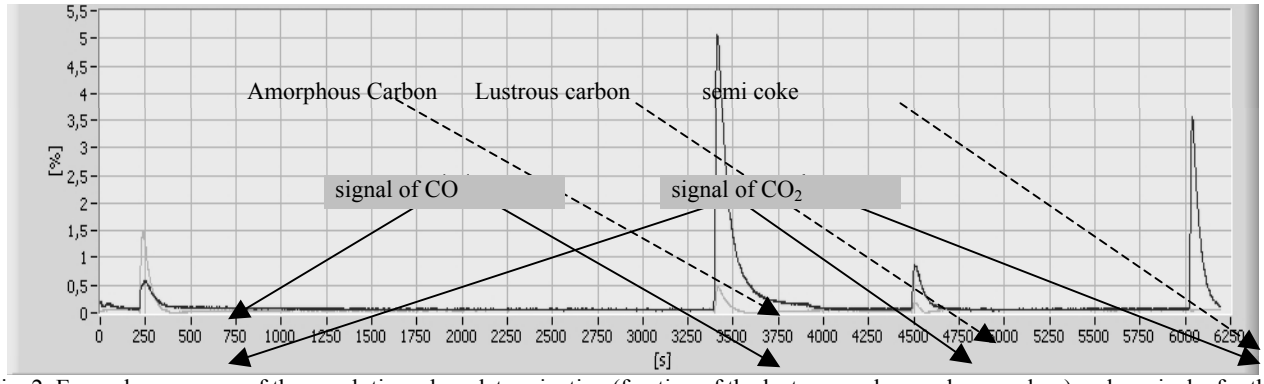

Fig. 2. Exemplary process of the pyrolytic carbon determination (fraction of the lustrous and amorphous carbon) and semicoke for the standard sample – tetraphenylsilane

Table 4.
Results for foundry moulds where carbon content was about 0.32%.

Sample number	Weight [g]	Pyrolysis ∑ C [%]	Burning ∑ C [%]	∑ C [%]
F-86	2.1429	0.20	0.07	0.28
F-88	2.934	0.22	0.06	0,28

4. Summary

Presented analyzer can be successfully used for relative and absolute measuring of carbon content in carbon materials where carbon content is on level 8÷100%. For this purpose there is possible measuring content of pyrolytic carbon and its fractions in materials which are traditionally used in foundry moulds technology, as in carbon dust and its replacement – in resins.
To have this technical knowledge and experience we design now a new model of analyzer for pyrolytic carbon and its fraction content measured in low carbon content materials < 1%, including foundry moulds. The originators of this analyzer thinks, that presented design will meet with demand form casting companies and research laboratories, who are busy with casting problems and foundry carbon dust replacements - as device what offer simple and repeatable method to lustrous carbon measuring. This equipment should be essential for units should be applicable for accreditation and foundries working with ISO 9000 system.
Presented model of analyzer, will be designed in two versions, for measuring of pyrolytic carbon content, and fractions - lustrous carbon and amorphous fraction, measured both in carbon dust replacements and in foundry moulds give current technology control with optimal raw material dosing (including recycled materials as well). In summary it gives a higher quality level, job reduction involved with cast cleaning and it will prevent for early wear and tear of foundry moulds – what make better economy of production.

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