

Rapid Quantitative Analysis of Silicon, Calcium, Chromium, Manganese and Iron in Ferrosilicon by Benchtop EDXRF

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Until June 2016, the total ferrosilicon (covert into 75%Si) product in China has reached 1.55 million ton. Ferrosilicon is the main raw material for steelmaking, magnesium industry, foundry industry and non-ferrous metal smelting, in which silicon is not only the main basis to evaluate the price of ferrosilicon, but also an indicator to adjust the steel-making process. The other four main elements including calcium, chromium, manganese and iron are important indicator to ferrosilicon alloy quality and other applications, so it is especially important to determinate these five main elements fast and accurately.

Gravimetric analysis (GA) is usually the first method to analyze silicon in ferrosilicon specimens, but it has some shortages such as complex process, time-consuming, professional and dangerous. It takes about 2~3 days to get results. XRF is a relatively simple and quick method to analyze ferrosilicon. However most of current determinations of ferrosilicon alloy are WDXRF with melting Method, which are expensive and mostly imported. There are fewer application reports on ferrosilicon with EDXRF before, because silicon element has lower fluorescence yield and high noise which cause a relatively low and instable intensity, else there is no good pretreatment and model.

We have done many experiments on Benchtop EDXRF and established a rapid accurate method to determinate silicon, calcium, chromium, manganese and iron in ferrosilicon. First, press ferrosilicon specimen powder into piece. To lower the scatter background and particle effect and increase the silicon intensity, ferrosilicon specimen is grinded to about 100 meshes, then to be tableted. While ferrosilicon has a poor cohesive force, adhesive is added at a certain proportion to increase the compression success rate. Through the experiments, the chose adhesive starch ratio to specimen is 1:5 in compression method condition. And after mixing 3 minutes in ball mill, 5 gram specimen is weighed and pressed at 40 MPa for 120 s. Second, to improve the accuracy of silicon for its lower transition probability, we adopt thin plastic sheet like polypropylene filter and PTFE collimator at 8kV 500 μ A, 100s. Finally, combined with fundamental parameter method, we select 10 to 20 reference sample including known content of all elements, to establish the curve through the normalization method. The linear regression coefficients of Si, Ca, Mn, Cr, Fe are between 0.96~0.99. The table below shows a testing stability result on the standard sample 25615.

11 times results	Si	Fe	Ca	Mn	Cr
AVG	71.213	26.527	1.037	0.359	0.261
SD	0.1318	0.1410	0.0127	0.0145	0.0070
RSD	0.18%	0.53%	1.23%	4.03%	2.69%
Reference	69.88%(GA)	26.29%	1.01%	0.29%	0.31%

From the data above, we know that EDXRF has already gotten a good performance and reach gravimetric analysis. It can take less than 10 minutes to get testing results, meanwhile with a lower price than WDXRF. Although there are some existing problems, we are still trying to improve and enhance the stability and accuracy.

Key words: Ferrosilicon, silicon, EDXRF, compression