Bulgarian Society for NDT
International Journal "NDT Days"
Volume IV, Issue 4, Year 2021
ISSN: 2603-4018
eISSN: 2603-4646

Phase Analysis of Graphite Powder Mixtures and Boron Carbide

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Abstract.
A methodology for determining the amount of graphite impurities in boron carbide powder has been developed. Based on the reference mixtures, a correlation dependence of the intensity ratio of 100% graphite-lines and carbide-lines and the amount of boron carbide was obtained. A linear regression analysis of the dependence was performed and the empirical dependence was obtained, connecting the quantitative parameters of the mixture.

Keywords: X-ray analysis, graphite, boron carbide, mixture, regression analysis

1. Introduction

The use of boron carbide powder in practice requires continuous monitoring of its composition in connection with the presence of graphite in it, as an inevitable technological impurity. To some extent, the content of graphite is a guarantee of the suitability and qualities of the sold or produced boron carbide. The aim of the work is to create a methodology for practical determination by X-ray diffraction analysis of the phase composition of powders – a mixture of graphite and boron carbide.

2. Material and methods

At the heart of X-ray phase analysis are several experimentally established principles, namely [1-5]:

- The X-ray diffraction pattern of a multiphase system is a superposition of the radiographs of each of the available phases.
- The qualitative identification of the phases is performed on the basis of comparison of the most intensive and characteristic for them lines of reflection of the diffraction pattern, with reference data. These lines are considered as benchmarks and are identified in each experiment.
- The quantities of the phases are proportional to the intensity of the lines of reflection and to the corresponding absorption coefficients.
- For each specific case there is a certain amount of a given phase, below which it cannot be established experimentally. Usually this values are different for different phases and ranges from 5-10% by weight.
- For quantitative calculations, the values of the most intensive reference lines of each phase are used, which are assumed to be 100% or 1.

In cases two phases have similar absorption coefficients, ie. close angles of the 100% lines of reflection and the use of a simple dependence are allowed to determine the percentage of each of them. This is the case for determining the amount of rest austenite in hardened steel [6-8].
For this purpose, we have prepared powder mixtures with a predetermined composition – 0, 5, 10, 15, 20 and 25% graphite, the rest is pine carbide – B4C. The diffraction lines in a wide range of reflection angles were X-ray diffracted on the reference mixtures. Standard equipment

"Philips" with Coα – radiation in mode – step 0.02o / min and speed 20 sec per step is used. The equipment is equipped with an interface that makes an automatic recording of the X-ray spectrum by means of a personal computer. For practical processing of the experimental data the possibilities of the program "Origin 5.0 Professional" were used.
Figures 1 and 2 show on an appropriate scale the 100% lines of graphite and boron carbide in standards with respective contents – 95% and 90% of B4C.

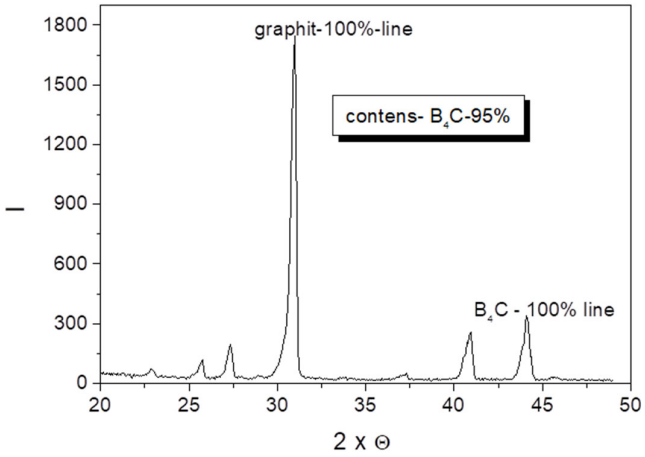

Fig. 1. The X-ray diffraction pattern of powder mixture with content of 5% graphite and 95% B4C

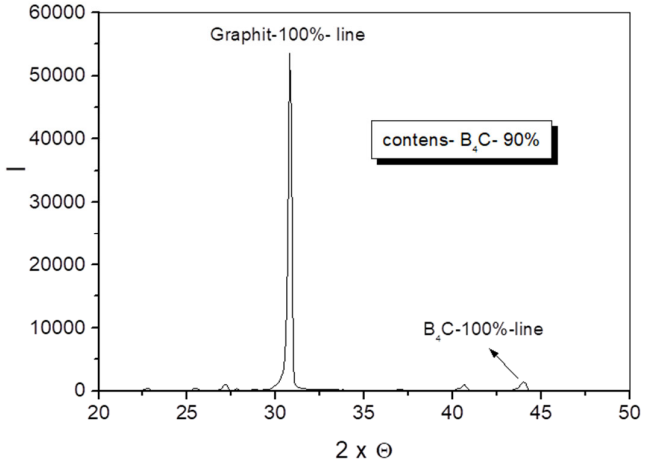

Fig.2. The X-ray diffraction pattern of a powder mixture containing 10% graphite and 90% B4C

Figure 3 shows the dependence of the coefficient "k", which is the ratio of the intensities of 100% lines of graphite and carbide, depending on the percentage of boron carbide in the reference mixtures. A linear regression analysis was performed and the results are shown in the figure.
The regression equation can be expressed by the empirical dependence:

$$[\%]\ B4C = 278.86 - 2.877.\ k,$$

where

k=Ic/I_{B4C},
Ic and IB4C – values of the intensities of the 100% lines of graphite and carbide.

The constructed reference dependence allows to study quantitatively and qualitatively similar powder mixtures with any composition.

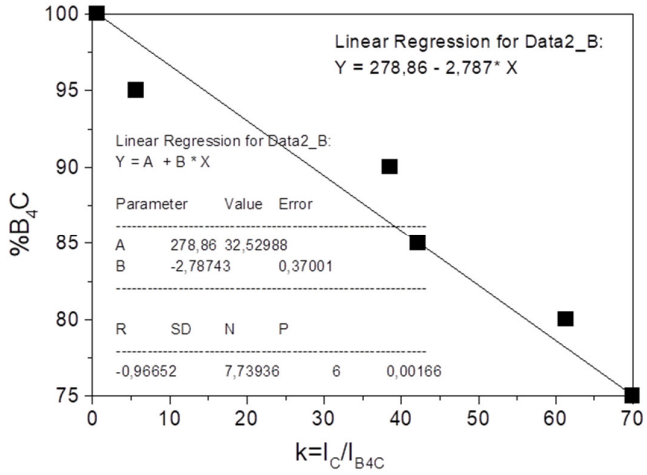

Fig.3. The dependence of the coefficient "k" on the percentage of boron carbide in the reference mixtures

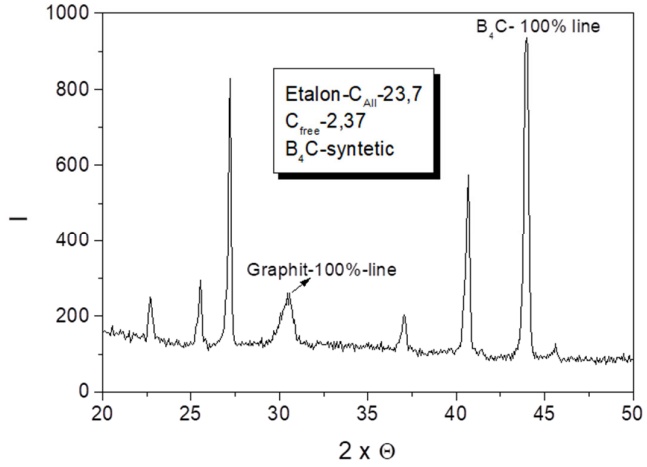

Fig.4 Quantitative calculation of the composition of a mixture of graphite and boron carbide using the above procedure.

3. Experimental verification

In FIG. 4 shows a quantitative calculation of the composition of a mixture of graphite and boron carbide using the above procedure. The content of graphite was determined chemically on a pre-prepared mixture – 2.37% (% by weight). Our calculations by the above X-ray diffraction

method showed the amount of graphite in the mixture – 2.27%, which value we consider as close as possible to that obtained by chemical analysis.

4. Conclusions

The developed methodology allows determine relatively accurately the amount of impurities from graphite in the powder of boron carbide. Achieving maximum accuracy in the examination requires X-ray equipment to be equipped with an interface device and a personal computer.

Acknowledgements
The author is grateful to the financial support of Bulgarian National Science Fund at the Ministry of Education and Science, Contract No DN17/17 /12.12.2017/.
All equipment and experimental units used in this work was funded by the European Regional Development Fund within the OP "Science and Education for Smart Growth 2014 – 2020", project CoE "National center of mechatronics and clean technologies", No BG05M2OP001-1.001-0008-C08.

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