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FERROUS METALLURGY INDUSTRY STANDARD
OF THE PEOPLE'S REPUBLIC OF CHINA

YB/T 5320-2006

(Adjusted from GB/T 5225-1985)

**Metal materials - Quantitative phase analysis - "Value
K" method of X-ray diffraction**

金属材料定量相分析 - X 射线衍射 K 值法

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$I_r^r(h_jk_jl_j)$ - The cumulative intensity of diffraction lines of the reference material from the crystal plane $(h_jk_jl_j)$, in the reference specimen;

W_r - The weight of the reference substance, after adding the powder to be tested into the reference substance;

W_o - The weight of the powder to be tested, when adding the powder to be tested into the reference substance;

W_r^r - The weight of the reference substance, in the reference specimen;

W_a^r - The weight of the powder to be tested, in the reference specimen.

The characteristic of this method is to calculate the K_r^a first AND finally determine the X_a .

Note: The reference specimen refers to the specimen, which is prepared when the K_r^a value is calculated.

2 Requirements and preparation of specimens

2.1 Sampling

2.1.1 Weighing

When weighing the sample, the relative deviation of the weight shall not exceed 0.1%.

2.1.2 Reference materials and reference specimens

2.1.2.1 Selection of reference materials

The principles for selecting reference materials are:

- a. During the test, its physical and chemical properties are stable AND not easy to deliquesce;
- b. The intensity of the diffraction line, which is used for measurement, shall be strong; its peak position is close to the measured diffraction line of the phase to be measured, without overlapping each other; meanwhile it is not interfered by other diffraction lines;
- c. The linear absorption coefficient and particle radius of the reference material shall be as close as possible to the phase to be measured. The

The overall stability of the diffractometer¹ is better than 1%.

3.2 Test conditions

3.2.1 Scanning speed

It should choose 0.5 degrees per minute or 0.25 degrees per minute.

3.2.2 Time constant

It should choose 2 s or 4 s accordingly.

3.3 Requirements for diffraction peak shape

The peak height shall be 4 times larger than the fluctuation range of the background;

The peak height should be about 4 times the half-height width.

4 Test procedure and result calculation

Before the quantitative determination, the intensity of the diffraction lines of the reference specimen and the mixed specimen shall first be calibrated with the JCPDS standard card, to check whether the reference material and the phase to be measured have a preferred orientation. If there is a preferred orientation, it shall prepare the specimen again, to improve or eliminate the preferred orientation, as much as possible. When the preferred orientation is not serious, follow Appendix B to make corrections.

4.1 Determination of K_r^a value of reference specimen

4.1.1 To calculate the pure phase to be measured of the K_r^a value, it shall first carry out qualitative analysis of the X-ray diffraction line, to check the purity; there shall be no impurity diffraction lines.

4.1.2 For the pure reference material selected according to 2.1.2, it shall check its purity according to 4.1.1.

4.1.3 Mix samples according to 2.1.1 and 2.1.2, respectively.

4.1.4 Prepare samples according to 2.1.4 and 2.2.

¹ The comprehensive stability of this standard instrument refers to the relative standard deviation σ_{-1} of the measured diffraction line intensity value within 8 hours, when measuring the diffraction intensity once every 10 min, on the same diffraction surface of the same specimen, under the same test conditions.

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