The Quantitative Estimation of Amorphous Content at the Zirconium-containing Chloraluminates by the XRD-background Level

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Abstract. The work is devoted to the method for quantity determination of the amorphous content based on the XRD-pattern of the sample. The traditional methods for estimation of the amorphous amount was unsuitable for use because of the some specific features of the analyzed samples. The method based on investigation of the model samples with known weight percent of the amorphous phase was applied. The experimentally obtained calibration curve made possible to estimate the amorphous content by the XRD-background level.

System: The powder samples, containing
Crystalline - Chloraluminate (80-90%),
+ small quantity of chlorides, oxychlorides of
Al, K, Zr.
Amorphous Al2O3 or ZrO2?


Method: Bragg-Brentano powder diffractometer using a copper X-ray source.

Problems:
1. Weight percent of a phase depends on the amorphous component amounts.
   \[ w_{\text{phase\_true}} = \frac{m_{\text{phase}}}{m_{\text{crystall}} \times m_{\text{amorph}}} \neq \frac{m_{\text{phase}}}{m_{\text{crystal}}} = w_{\text{phase\_XRD}} \]
2. Time limit for XRD-analyzing and preparing of the sample because of system features.
3. A lot of peaks at the diffractogram exclude the traditional methods for quantity amorphous estimation.
4. The quantity of Zr by XRD-analysis is in contradiction to data from the X-ray fluorescence analysis (XRF).

1. Experimental results

Fig.1 X-Ray diffractograms of the analyzed samples in increasing order of the Zr weight percent, obtained by XRF. 

Result 1  ZrO2 is in amorphous

2. Experimental results

Fig.2 X-Ray diffractograms of the human-made mixtures based on Up2-5 sample by adding 10% (weight percent) of amorphous Al2O3 and ZrO2.

Result 2  It is able to know the amorphous weight present of the any over samples

2. Чукин, Д.Г. Способ дифрактометрического определения степени кристалличности веществ/Д.Г. Чукин, А.В. Малюк, И.В. Тышкунова, Л.В. Майер, Е.В. Новожилов.
Кристаллография, том 61, №3, 2016, с. 375-379.

Features:
- High water absorption
- Texture
- High chemical activity
- Multiphase

Dependence of the XRD-background level (2θ≈33°) from the concentration of Zr (XRF)