The development of a new sample preparation procedure for the analysis of CuAgP solder by XRF spectrometry

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CuAgP hard solders are a family of materials containing 0.2%–15% Ag and 4%–8% P. These types of materials are the object of commercial analysis at the Department of Analytical Chemistry of the Institute of Non-Ferrous Metals. We have observed that typical sample preparation, i.e. surface grinding of the primary samples, could result in obtaining incorrect results in the XRF analysis, especially in the case of materials that contain more than 7% of P. As we have observed, this could be caused by the differences in the structural composition between the primary samples and the reference materials. Because the samples are collected in non-controlled conditions at the production site, their microstructures might have various size. This parameter could be essential for XRF analysis of such materials, especially in the case of phosphorus determination. The critical thickness \( t_c \), which is the depth of the sample that produces the fluorescence radiation registered by the spectrometer (beyond this limit this radiation is entirely absorbed by the sample matrix) for P in such a type of matrix, is about 2–3 µm. Because of this, the smallest possible size of the microstructures is preferred due to better averaging and representativeness of the analyzed specimen volume. As our studies show, it is impossible to obtain microstructures smaller than phosphorus \( t_c \), which allows to register phosphorus fluorescence from more than one layer of the microstructures. However, decreasing the microstructures size from 80 µm to 20 µm gives quite good results. A new procedure for sample preparation, based on re-melting of the primary sample with an automatic centrifugal cast, was developed. The prepared specimens were characterized by minimal possible microstructure size and homogeneity. The procedure also allows for homogenization and preparation of chip samples. An analytical application for the analysis of CuAgP alloys by a WD XRF spectrometer was developed and initially validated. The calibration materials were prepared in the same procedure, which ensured compatibility with the specimens.

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