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Mouad Essani, Emmanuelle Brackx, Philippe Jonnard, Emmanuel Excoffier. A new standardless approach in EPMA for quantitative analysis of uranium microparticles. EUROMAT 2019, European Congress and Exhibition on Advanced materials and Processes, Jan 2019, Stockholm, Sweden. hal-03932755

**HAL Id: hal-03932755**

**<https://hal.science/hal-03932755v1>**

Submitted on 13 Jan 2023

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# A new standardless approach in EPMA for quantitative analysis of uranium microparticles

Mouad Essani<sup>1,2</sup>, Emmanuelle Brackx<sup>1</sup>, Philippe Jonnard<sup>2</sup> and Emmanuel Excoffier<sup>1</sup>

<sup>1</sup>CEA Marcoule, Laboratoire de Métallographie et d'Analyse Chimique, BP 17171, 30207 Bagnols sur Cèze, France

<sup>2</sup>Sorbonne Université, CNRS UMR 7614, Laboratoire de Chimie Physique-Matière et Rayonnement, 4 place Jussieu, F-75252 Paris Cedex 05, France

Quantitative electron probe microanalysis (EPMA) is usually performed using analysis based on standards. The x-ray spectrum is acquired for an unknown sample and compared to the spectrum of a reference standard. Matrix effects are further corrected using conventional ZAF or  $\varphi(\rho z)$  [1][2] methods. However, ZAF and  $\varphi(\rho z)$  models were developed for bulk specimens with plane surfaces and involve many assumptions about the thickness and morphology of the sample. These models are no longer accurate when quantifying the composition of non-conventional samples such as microparticles. In fact, microparticles can have irregular shapes and exhibit high surface roughness and porosity. Thus, absorption path lengths and fluorescence inside the particle might be significantly different than those in a bulk polished sample. Moreover, quantitative models based on bulk standards do not take into account the loss of x-ray signal caused by electrons side scattering and/or transmission of electrons through the micro sized particles. Quantitative standardless procedures [3] can be used to overcome these limitations. A virtual x-ray spectrum is calculated in that regard and used as a reference standard to quantify the sample composition. Standardless models based on Monte Carlo (MC) simulation were shown to be the most viable approaches to deal with non-conventional samples such as porous media [4], thin films [5] and microparticles [6].

In this work, we present a new MC quantitative standardless approach. Weight fractions are computed iteratively by means of the deviation between simulated and experimental intensities. The measured x-ray spectra are corrected after introducing the detector response function and compared to simulated ones (figure 3). The virtual x-ray spectra are calculated using the general-purpose MC code PENELOPE [7] in which the morphological parameters of the sample can be described. PENELOPE uses Bote et al. ionization cross sections [8] which was shown to give accurate results when M subshells are used [9].

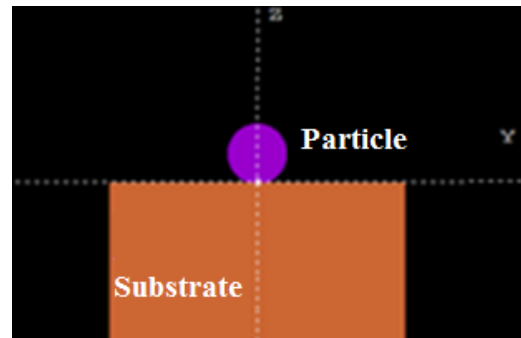
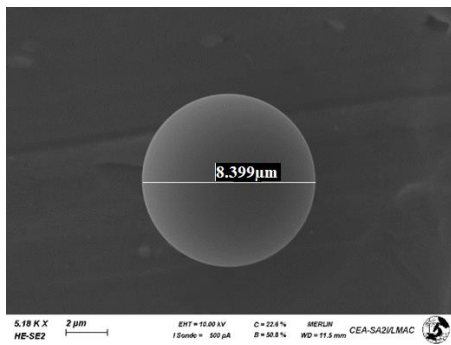
A preliminary study was performed on a porous bulk sample of  $\text{UO}_2$  and a spherical microparticle of K411 (figure 1) glass analyzed at different beam incidence angles. The correction of intensity loss due to porosity, geometric (figure 2) and incidence angle effects is achieved, which leads to promising quantitative results with relative errors  $\frac{|C_c - C_n|}{C_n} < 15\%$ . The approach was further applied on microparticles of  $\text{U}_3\text{O}_8$ ,  $\text{UF}_4$  and  $\text{UCeO}$ .

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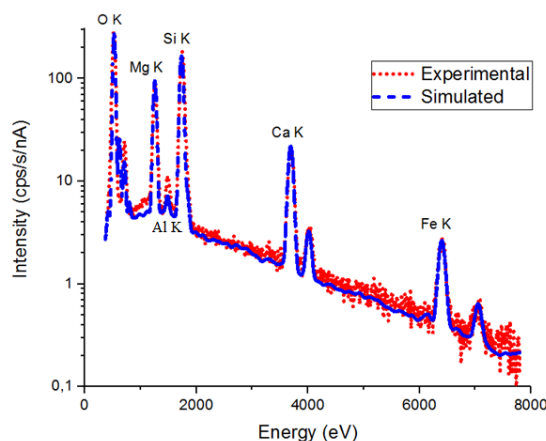
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**Figure 1** SEM image of a spherical particle deposited on a flat substrate



al particle on a flat substrate  
the geometry package of

**Figure 3** Comparison between measured and simulated spectrum for spherical particle of K411 glass. The deviation between intensities do not exceed 9%