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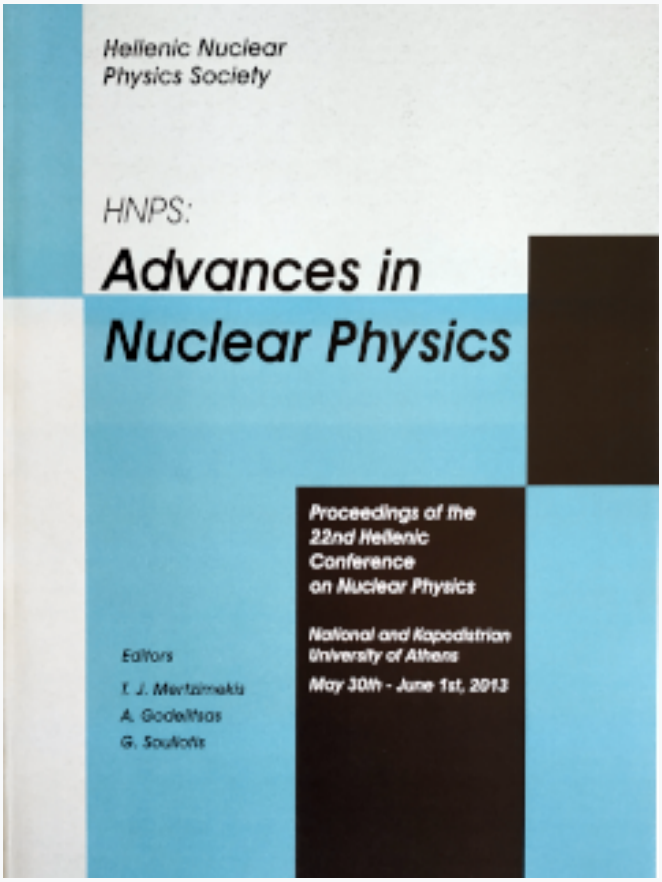

A Study On The Heterogeneity of Reference Metal Alloys Using A Micro-XRF Spectrometer

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A STUDY ON THE HETEROGENEITY OF REFERENCE METAL ALLOYS USING A MICRO-XRF SPECTROMETER

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Introduction

The introduction of highly performance x-ray focusing devices in XRF spectrometers lead to spatially resolved elemental analysis of surface details by desktop or portable scanning x-ray microprobes [1]. Heterogeneities having the same size or being even larger than the exciting x-ray beam spot size are expected to produce misleading quantitative results when single spot or comparable size area analysis is performed. This paper proposes an analytical methodology and criteria to define an analyzed area or mass that will result to representative results of the metal alloy bulk composition. Further on, the XRF analytical data are explored and evaluated with respect to the characterization of the heterogeneity components and features.

Experimental

The IAEA-NSAL scanning micro-XRF spectrometer [2] is consisted of a 3 kW Mo-anode diffraction X-ray tube, a polycapillary x-ray lens (nominal FWHM = 24 μm at 15-20 keV) for focusing the exciting x-ray beam, a computer-controlled fouraxis sample holder stage, a Si(Li) detector (170 eV at MnK), whereas the inspection and documentation of the analysis area is provided by an optical microscope coupled to a CMOS camera. To avoid the presence of interfering diffraction lines, a combined filter composed by 50 μm Ni and 12.5 μm Mo was used in the exciting x-ray beam path. The tube was operated at 45 kV/30 mA. The scanning area was selected to be about 5×5 mm^2 with a step size of 50 μm and live time 10-30 s/spot. Certified copper, silver and gold alloys resembling the composition of ancient or historical metal alloys were selected for analysis.

Results

The mean values of characteristic x-ray intensities and of their respective percentage relative standard deviations (RSD), are calculated by summing each time an increasing number of neighboring not-overlapping spot-measurements. For each accumulation of spot- measurements, the respective analyzed mass calculated by taking into account the information depth. The homogeneity level is defined at the corresponding mass where the overall RSD becomes comparable with the one generated due to the instrumental and statistical precision only. Another approach investigated, incorporates the lacunarity concept, originally developed to describe fractals but it is also used as a scale dependant measure of an object heterogeneity [3]. In Fig. 1a the variation of PbL intensities is shown for the quaternary BCR-691-A certified copper alloy within the scanned area (6.5mm × 6.5mm). The percentage RSD of all single spot measurements is more than 100%, but summing an increasing number of neighboring not overlapping spots, the percentage RSD drops down to about 3.5% for a total measured area of about 7.3 mm^2.

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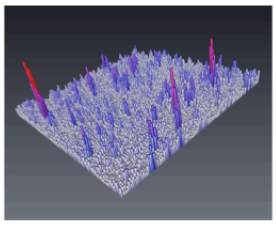

Fig. 1a: Variation of PbLβ intensities for the BCR-691A certified copper alloy within a 6.5mm x 6.5mm scanned area (step= 50 um)

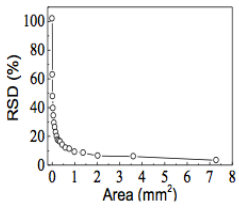

Fig. 1b: The variation of PbLβ STD(%) calculated from an increasing number of neighboring not-overlapping spot-measurements versus area measured.

Conclusions

This study reveals the problem of micro-XRF analysis of heterogeneous metal alloys and proposes an analytical methodology to ensure meaningful quantitative results.

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