doi:10.1017/S1431927617003312
Microsc. Microanal. 23 (Suppl 1), 2017

Microprobe Analysis of Pu-Ga Standards

Angelique D. Wall[1], Joseph P. Romero[1] and Daniel Schwartz[1]

[1.] Los Alamos National Laboratory, MST-16, Los Alamos, NM, USA

In order to obtain quantitative analysis using an Electron Scanning Microprobe it is essential to have a standard of known composition. Most elemental and multi-elemental standards can be easily obtained from places like Elemental Scientific or other standards organizations that are NIST (National Institute of Standards and Technology) traceable. It is, however, more challenging to find standards for plutonium. Past work performed in our group has typically involved using the plutonium sample to be analysed as its own standard as long as all other known components of the sample have standards to be compared to [1,2,3]. This method works well enough, but this experiment was performed in order to develop a more reliable standard for plutonium using five samples of known chemistry of a plutonium gallium mix that could then be used as the main plutonium and gallium standards for future experiments.

Typical samples analysed contain approximately 0.6wt% gallium in a plutonium matrix and are analysed for gallium content. The five samples in this experiment used to create the standards ranged from 0.2 to 0.6wt% gallium and were sent for chemical analysis prior to preparation for micro probe analysis. The plutonium samples were subsequently cut and mounted in thermosetting epoxy. The mounted section was ground and polished to the final polish using one micron diamond paste. The metallographic mount was then carbon coated for charge compensation in preparation for electron microprobe analysis with the JEOL JXA 8200. Elemental maps were collected for each element at a 256x256 micron area with a spatial resolution of 1 micron at an accelerating voltage of 18kV and approximately 100ηA probe current in order to have a qualitative view of the sample. The relative precision for this analysis is 10% at an expected concentration level of 0.5 wt. % gallium. Quantitative gallium horizontal and vertical line scans were obtained of the mapped region. Figure 1 is an example of one of the elemental maps taken and the qualitative analysis results for the gallium in the area can be seen in Figures 2 and 3. The relative precision for the line scan analysis is 1.0 percent at an expected concentration level of 0.5 wt. % gallium. The standards were analyzed quantitatively twice in order to confirm that the data was reproducible and the results were subsequently compared to chemical analysis data.

Results indicated that the data was reproducible and was relatively close to the chemical analysis data within error margin. Further experiments will be performed to ensure statistical accuracy before the standards are officially added to the standard block for future analysis of customers' samples.

References:

[1] AD Neuman *et al*, Microsc. Microanal., **17** (2011), p. 1044-1045.
[2] AD Neuman *et al*, Materials Science & Technology Conference (2011).
[3] FJ Freibert *et al*, MRS Proceedings, **1215** (2009), p. 1215-V10-06.

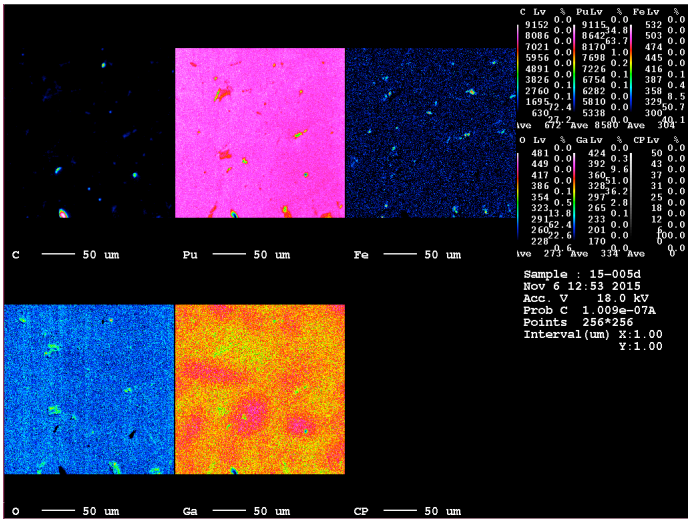

Figure 1. Qualitative x-ray elemental maps of carbon, plutonium, iron, oxygen, and gallium at 256x256 mm area with a spatial resolution of 1 mm.

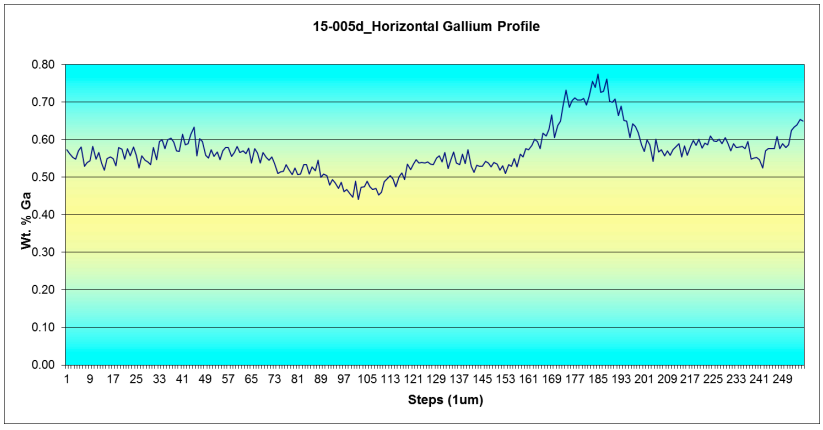

Figure 2. Vertical quantitative gallium line scan. Average gallium distribution 0.569 +/- 0.061.

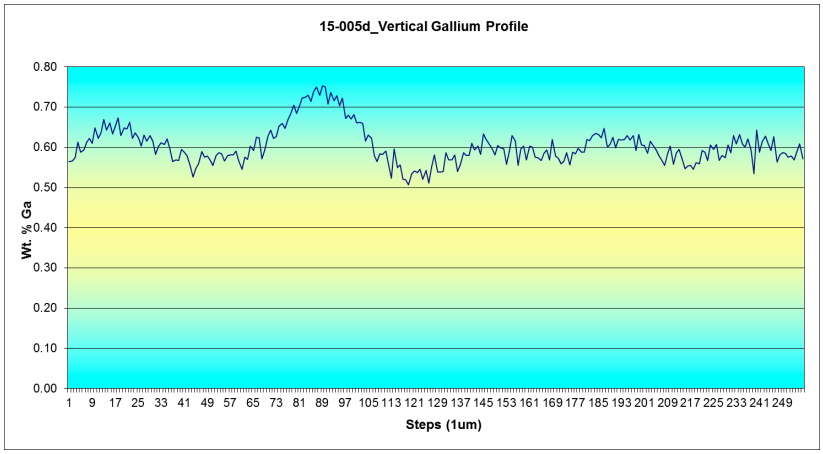

Figure 3. Vertical quantitative gallium line scan. Average gallium distribution 0.604 +/- 0.047.

https://doi.org/10.1017/S1431927617003312 Published online by Cambridge University Press