An assessment of the potential benefits of ion implants as trace-element reference material for electron probe X-ray microanalysis: The case of invisible gold

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ABSTRACT

The reliability of trace element concentrations obtained by EPMA can be significantly improved with the use of high-quality secondary standards. In the case of Au residing in sulfides, such standards are lacking. Natural materials have heterogeneous Au distribution, whereas synthesis is very difficult. The benefits of using ion implants as trace-element reference material for EPMA were assessed by characterizing grains of magnetite, pyrite and galena implanted with \( 1 \times 10^{14} \) to \( 5 \times 10^{14} \) Au atoms/cm\(^2\) at energies from 1 to 3 MeV. The first interesting observation is the excellent lateral micrometer-scale homogeneity of the Au levels across the implants. The ratio of analytical to statistical standard deviations never exceeds 1.7. Additionally, the Au X-ray intensities measured by EPMA show excellent correlation with those predicted for multilayered structures used to model the continuous Au concentration profile for the three implants investigated. Small discrepancies arise only at low accelerating voltage. In these situations, the predicted Au X-ray intensities become sensitive to uncertainties in the determination of the location of the Au concentration profile because of insufficient excitation of the bottom of the Au layer. Fortunately, by varying the implantation energy, optimal implants yielding X-ray intensities that are insensitive to uncertainties on the Au depth profile can be obtained for a wide range of accelerating voltages. These results suggest that ion implants may represent excellent EPMA reference material, especially in cases where natural and synthetic standards are unavailable. Interesting materials presenting specific analytical challenges can be engineered due to the excellent control of the implantation parameters.

Keywords: EPMA, ion implants, standards, trace element, Au

INTRODUCTION

To optimize gold ore processing strategies, it is critical to evaluate the levels and distribution of refractory “invisible” Au that can reside either as discrete nanometer-sized inclusions or in solution within common sulfides or their oxidation products (e.g., Cabri et al. 1989; Pratt and Duke 2003; Chouinard et al. 2005; Paktunc et al. 2006; Reich et al. 2006). In conjunction with other analytical techniques such as secondary ion mass spectrometry (SIMS) and laser ablation inductively coupled plasma mass spectrometry (LA-ICPMS), the electron probe X-ray microanalyzer (EPMA) remains an instrument of choice to quantitatively correlate trace concentrations of economically important elements such as Au with chemical and morphological features within a sulfide or oxide grain at optimal spatial resolution. This becomes more relevant with the current performance of wavelength-dispersive diffracting crystals that allows detection limits of a few parts per million and the recent introduction of commercial field-emission EPMA capable of sub-micrometer resolution at low accelerating voltage.

A major obstacle in evaluating the consistency and reliability of trace Au concentrations obtained by EPMA is the lack of high-quality secondary standards that are essential to test the ability to correctly extract peak intensity from the background at minimal theoretical detection limits. Natural sulfides are known to have very heterogeneous distribution of Au and consequently are poor candidates. Moreover, because gold has restricted solubility in most common sulfides and the substitution mechanisms are poorly understood, synthesis routes to produce homogeneous grains with optimal Au concentrations are very limited. In this context, ion implants, typically used as standards for SIMS analyses, were evaluated as trace-element reference material for EPMA. During implantation, the distribution and the total concentration of the ion in a target can be independently and precisely controlled by choosing specific energy and dose respectively. However, the narrow depth distribution of the implanted ions relative to typical electron interaction volumes confers to these materials interesting properties. For example, if treated as a simple bulk homogeneous material when analyzed by EPMA, the X-ray intensities obtained for a specific implant would yield “apparent” Au concentrations that vary as a function of accelerating voltage (Fig. 1). Consequently, the material needs to be considered as a multilayered structure and, as such, requires appropriate correction procedures (e.g., Pouchou and Pichoir 1991; Pouchou 2002).

ANALYTICAL AND CALCULATION METHODS

Au implantation procedure

Centimeter-sized polished massive grains of Au-free (<1 ppm) magnetite, pyrite and galena were implanted with Au ions using a 1.7 MV Tandetron accelerator at the Interface Science Western research center. For magnetite, the dose was 5.0