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X-ray K-ratio Derived Using Extreme Overvoltage Conditions.

Ililochi Onwuzu[1], John J. Donovan[2]

[1.] Department of Physics, University of Oregon, Eugene OR USA
[2.] Micro Analytical Facility CAMCOR Labs, University of Oregon, Eugene OR USA

Electron Probe MicroAnalysis (EPMA) is a method of material analysis used to calculate elemental concentration from x-ray intensities. A focused high energy electron beam is used to excite characteristic x-rays from the specimen of interest. The threshold energy required to excite these characteristic x-rays should be greater than the critical excitation energy of the x-rays. If E_0 just equals the E_c, the overvoltage (E_0/E_c) equals one and no x-rays are excited. For high spatial resolution analysis, a low overvoltage is one approach to minimizing the interaction volume. However, at such incident energies, the x-ray count is very low thus degrading precision. On the other side of the coin, where high overvoltage is needed for bulk analysis, measurements are increasingly inaccurate due to large absorption effects. Mathematical models using both Monte-Carlo and analytical expressions have been developed to simulate EPMA experiments in order to derive these matrix correction factors. These models work well with an accuracy of approximately 2% for typical overvoltage situations. However, for low and high overvoltages, the calculations are less accurate. We will compare measured elemental K-ratios ($K_{measured}$) obtained from some metal alloys to those predicted by the Monte-Carlo program PENEPMA [1] to see how it performs at low and high overvoltages. Elemental K-ratios are the intensity ratio of unknown to a pure standard so they are normalized for differences in instrumental efficiencies. We will also use the $K_{measured}$ to investigate the validity of $K_{analytical}$ values derived from analytical models such as Philibert/Duncumb-Reed, Armstrong/Love-Scott and Pouchou et Pichoir. We propose that a database to complement our mathematical models should be created containing measured K-ratios of standard materials at low and high overvoltages. The measurements presented here will mark the first entry to the XTREME (X-ray Table of Ratios with Exceptional Matrix Effects) database and we urge the scientific community to contribute. The database will act as: (1) A direct source for accurate and carefully measured K-ratios (2) A reference for physicists in the field of electron-solid modeling to use in modifying analytical expressions to allow for improved accuracy at these extreme overvoltages.

The measurements presented here were performed with two gold-copper alloys of composition 80:20 and 60:40 respectively. A 12keV focused electron beam at 30nA beam current was directed at the specimens. The PENEPMA data was also run for these specimens at 12keV. The intensities were obtained from the K-α line (L3-K transition) for Cu and the M-α line (N7-M5 transition) was used for Au. The Cu K-α transition gives an overvoltage of about 1.5 so we will use it to investigate accuracies at low overvoltage. The Au M-α transition results in an overvoltage of about 5.5 and so we will use this to look at the high overvoltage region. In the Table 2 (60:40), we notice a much higher % error for the Cu K-α low overvoltage case. For the Cu K-α low overvoltage case, in going from a 20% to 40% composition, there is only a 0.5% increase in relative error. On the other hand, there is about a 2% decrease in going from an 80% to 60% composition.

Ultimately, the K-ratios of alloys, carefully measured at low and high overvoltage conditions will be entered into the XTREME database. In the future, we plan to experiment with varying overvoltages and then verify the agreement at medium overvoltages. We encourage other experimenters to submit entries for this database in order to improve physics models to perform better at the extremes of overvoltage.

References:

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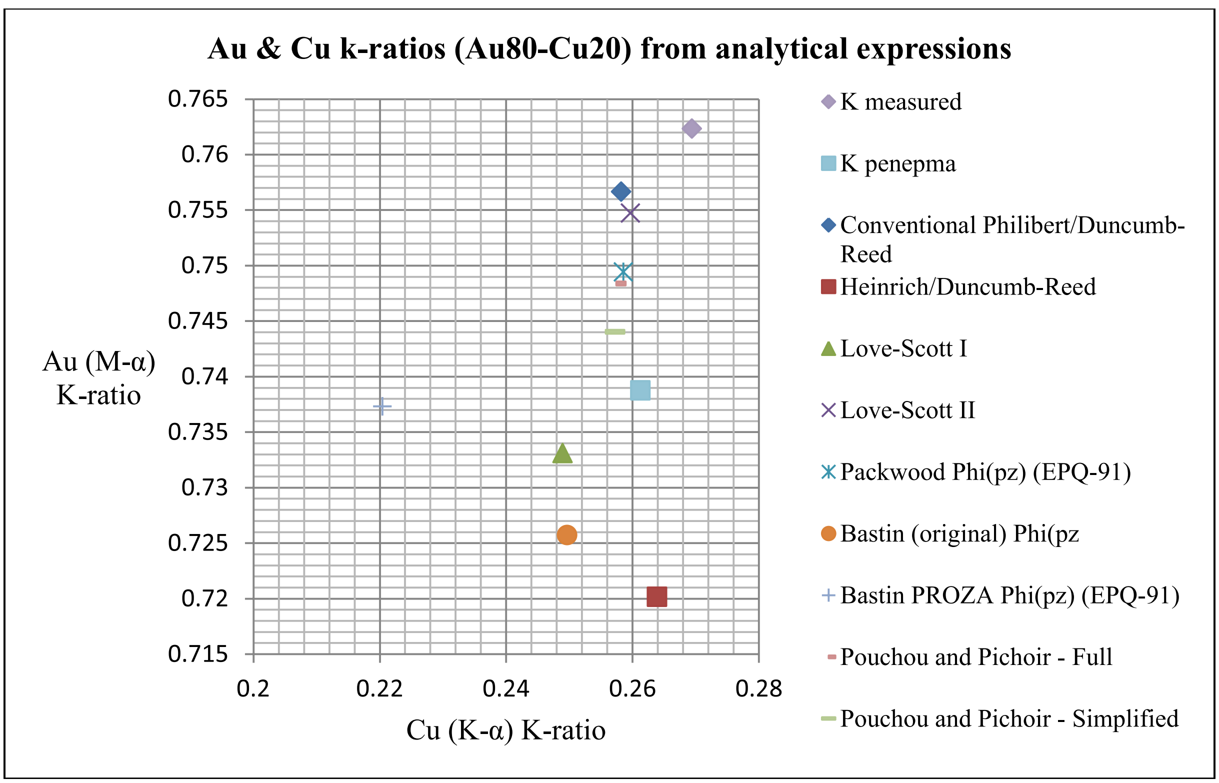
Au 80 – Cu 20 ALLOY					
Element	$K_{measured}$	Uncertainty (%)	$K_{penepma}$	Uncertainty (%)	Rel. % error
Au (M-α)	0.76232	± 0.59030	0.73874	± 0.00003	3.192
Cu (K-α)	0.26945	± 0.49731	0.26130	± 0.00001	3.119

Table 1: $K_{measured}$ and $K_{penepma}$, their uncertainties and relative % error between the two K-ratios

Au 60 – Cu 40 ALLOY					
Element	$K_{measured}$	Uncertainty (%)	$K_{penepma}$	Uncertainty (%)	Rel. % error
Au (M-α)	0.52339	± 0.25793	0.51843	± 0.00003	0.957
Cu (K-α)	0.50098	± 0.22556	0.48289	± 0.00001	3.746

Table 2: $K_{measured}$ and $K_{penepma}$, their uncertainties and relative % error between the two K-ratios



Graph 1: Au and Cu K-ratios: $K_{measured}$, $K_{penepma}$ plotted alongside $K_{analytical}$ for nine analytical models

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