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X-Ray Calibration Copper Foil on Aluminium Grid

AGS149

Many transmission electron microscopes and scanning transmission electron microscopes are interfaced to energy dispersive X-ray (EDX) detectors and are used routinely to carry out quantitative and qualitative microanalysis.

Qualitative analysis is straightforward and requires that the energies of the X-rays incident upon the X-ray detector are measured with the necessary accuracy - typically this can be 1eV. The energy calibration required is carried out differently on the various EDX facilities which are available commercially. If a sample is available which generates two well separated K-lines then this can be used to check the calibration and in some cases it can be used for the calibration procedure itself. A suitable type of sample which is readily available from Agar Scientific consists of a pure metal foil on an aluminium grid. Suitable pure metals which cover the useful energy range are copper and silver; a molybdenum trioxide crystal will provide an X-ray line between these; the aluminium grid will generate the low energy line (~1.49 keV), copper will generate a line centred at ~8.04 at ~17.44 keV and silver at ~22.10 keV. In the case of the molybdenum and silver the generation of $K\alpha_1$ *and* $K\alpha_2$ *peaks leads to complex X-ray peaks and calibration has to be carried out with due care. In fact, copper, together with aluminium is generally sufficient for most purposes.*

Quantitative analysis can be performed using either commercially available in software or software developed by individual users. Effectively, all software make use of "efficiency" factors which relate measured relative X-ray intensities to composition; the "efficiency" factors cover both generation and detection efficiencies and may be based on standardless programs or programs that use pure metal or alloy standards. Whichever approach is adopted, an important parameter built into all programs is called the "detector efficiency", and once it has been modelled and input to the computer software, it is essential that it does not change before the next check. Unfortunately there is a risk that the efficiency will change with time since very often contamination builds up on the Be window, thus absorbing more X-rays and making the detector assembly less efficient. A simple way of monitoring the detector efficiency is to use a very thin copper sample and to measure the ratio of Cu K/Cu L X-ray intensities.

With this in mind Agar Scientific now have available uniformly thin (~600°A) Cu films which are carbon-coated on both sides mounted on Al grids: they give consistent K:L ratios over the whole specimen.

Typical data taken from different specimen regions is shown in the table for a specific EDX system. Cu $K\alpha$ *and Cu* $L\alpha$ *intensities and their ratios are given, and it can be seen that the ratio is sensibly constant. The spectra can be obtained without fully focusing the electron probe so the risk of contaminating the sample is small. The carbon coating limits the rate of oxidation of the samples and they can be kept for many months in a vacuum desiccator. The ratio of Cu K:Cu L has also been measured on specimens made at very different times and the values lie within the range shown in the table. The absolute value will of course be different for different EDX systems.*

The calculated influence on the Cu K/Cu L ratio, of contamination (taken to be carbon) on the Be window, is modelled, together with the change in detector efficiency for the Cu K and Cu L, which is given for a particular EDX system. The calculated change in the Cu K:Cu L ratio originates totally from absorption of the Cu L in the C. It can be seen from the figure that the ratio increases very significantly if a few hundred nanometres of C are deposited on the window. While the Cu K detection efficiency is constant, the Cu L detection efficiency falls away. This reflects the manner in which the softer (lower energy) signal is absorbed.

By actually measuring the Cu K and Cu L peaks using the Agar test specimen, data similar to that shown in the table can be obtained to monitor the detector performance.

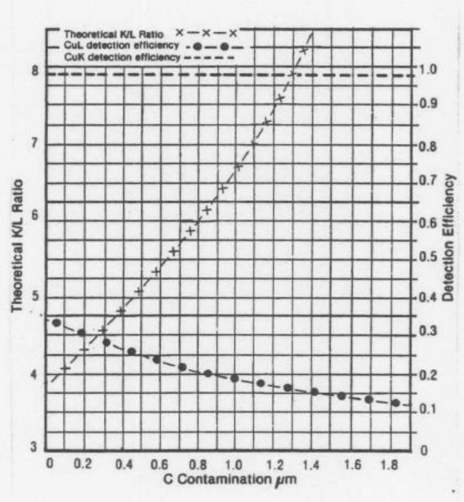

Table showing X-ray Data obtained from Thin Cu Films

	Integrated X-ray Counts		*Ratio of CuKα /CuLα*
Sample 1	*CuKα*	*CuLα*	
Area 1	*216,831*	*53,912*	*4.022*
Area 2	*239,006*	*59,678*	*4.005*
Area 3	*251,683*	*63,691*	*3.952*
Area 4	*258,388*	*65,299*	*3.957*
Sample 2			
Area 1	*248,226*	*61,736*	*4.021*

Cu K(α) energy = 8.04 keV

Cu L(α) energy = 0.93 keV