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X-ray Emission From Thin Films on a Substrate – Experiments and Simulation

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A method of x-ray microanalysis based on Monte Carlo simulation has been developed to determine simultaneously both the composition and thickness of multilayer materials [1]. CASINO, a well-developed software based on Monte Carlo method is widely used to simulate the interaction of incident beam and sample to generate all of the recorded signals (x-rays and backscattered electrons) in an SEM [2,3]. The reliability and accuracy of using CASINO to determine composition and thickness of multilayer materials need to be validated with experimental results.

Thin Cr layers with the thickness of 10, 50, 200, and 500 nm were deposited on Si substrates by sputtering (as shown in Fig. 1 a)). The Si substrate was a Si wafer with a flat surface. The intensity of emitted x-rays were measured using an SEM (Hitachi SU8000) at different accelerating voltage and then corrected for the variation of probe current, which were measured using Faraday cup. A polished specimen of pure Cr and a Si wafer were used as standard sample to calculate the experimental k-ratio values (ratio of the measured characteristic x-ray intensities of one element in the sample and the intensity in the pure standard sample) of Cr K_α line and Si K_α line. These results show that a cold field emission electron microscope can be used to measure k-ratio value.

The k-ratio experimental data and simulation results from CASINO v2 were compared for different film thickness. Note that the standard deviation of experimental data have been calculated and they are all less than 0.01. An example of the comparison is shown in Fig. 1 b) for 10 kV accelerating voltage. The results show that the agreement between the measured and simulated k-ratio values is relatively good at low thickness both for Cr K_α line and Si K_α line, while larger discrepancies appeared when the layer is relatively thick (thickness between 200 and 500 nm) for Cr K_α line. The difference could be explained by the fluorescence effect of the bremsstrahlung, which is not simulated with CASINO v2. In the standard sample, the total intensity of Cr K_α line increase by fluorescence from the bremsstrahlung. On the other hand, the presence of Si substrate decrease the contribution of the bremsstrahlung fluorescence on intensity of Cr layer [4]. Thus, the experimental values of k-ratio for Cr K_α line is lower than that of simulation result. This difference between experimental data and simulation results is more important at larger thickness as observed with variation of k-ratio versus accelerating voltage for different thickness (as shown in Fig. 2).

To confirm the bremsstrahlung fluorescence effect hypothesis, more samples with different thickness of Cr film and with different film composition and k-ratio measurement are needed. These results provide a database to test the accuracy of simulation results using CASINO v2 and also to develop a fluorescence correction model.

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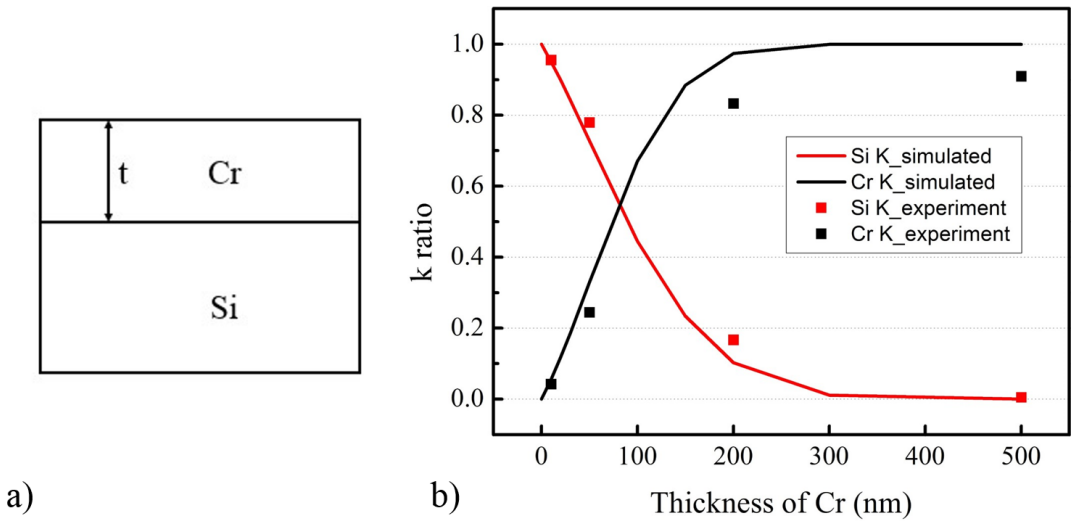

Figure 1. a) shown the geometry of Cr-Si multilayer sample: Si substrate with Cr coating with thickness t; **b)** shown the variation of k-ratio versus thickness of Cr for Cr K_α line and Si K_α line at 10 kV and comparing experimental results and simulation results using CASINO v2.

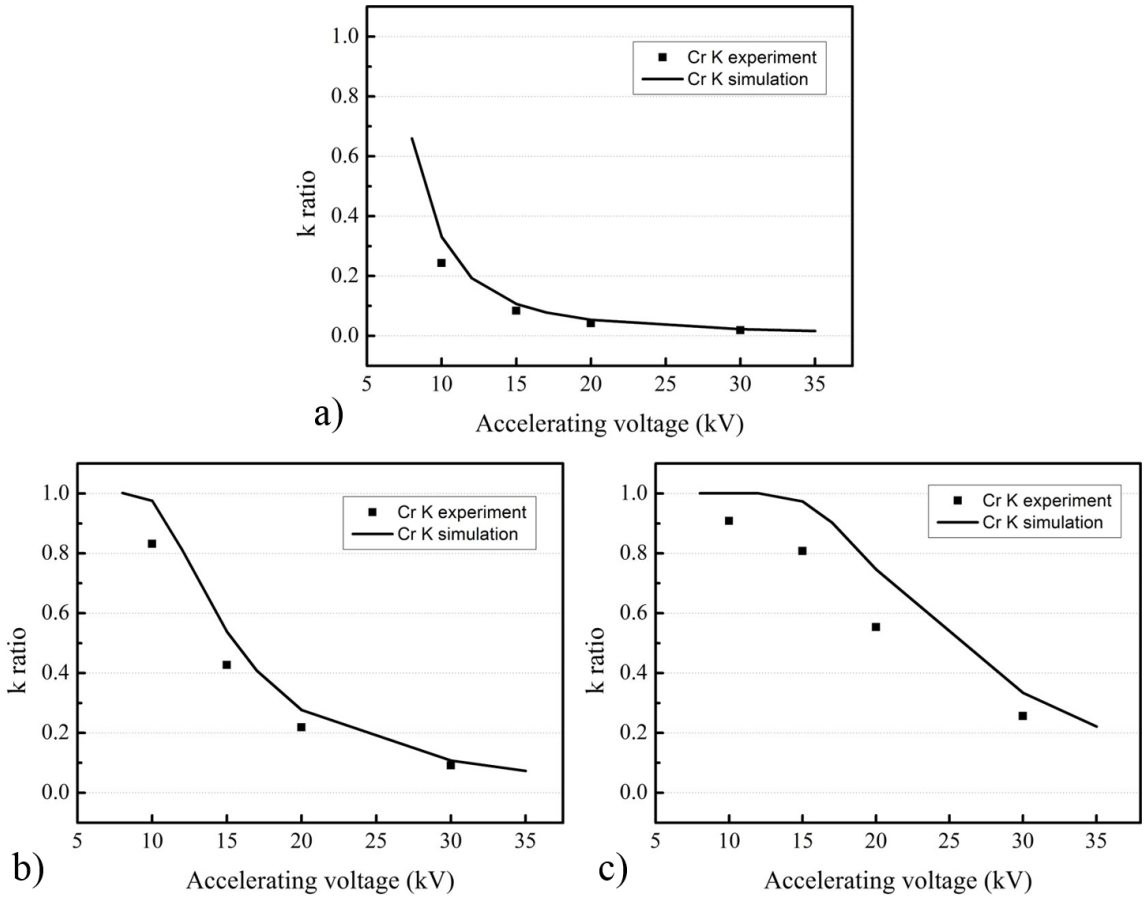

Figure 2. Comparison of experimental results and simulation results (using CASINO v2) of the variation of k-ratio versus the accelerating voltage of Cr K_α line with different thickness of Cr. The thickness of Cr is: **a)** 50 nm, **b)** 200 nm, and **c)** 500 nm.

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