

Electron Probe Microanalysis of Cr Films on Semiconducting and Insulating Substrates

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The determination of composition and thickness of thin films on thick substrates is a technologically important problem. Nondestructive methods are particularly desirable in the semiconductor industry since a processed Si wafer or a lithographic mask can cost thousands of dollars and destructive analysis is not a desirable option. The current study was undertaken to determine if in the analysis of thin films the calibration curve of K ratio versus film thickness is influenced by the presence of an insulating substrate. The K ratio is defined as the background and deadtime corrected intensity measured from a given film thickness to that of a similarly corrected intensity obtained from a bulk specimen. Specifically, measurements were made on chromium films varying in thickness from 20 nm to 400 nm deposited by e-beam evaporation onto a range of substrates that included pure silicon, pure silicon dioxide and silicon with oxide layers of 100, 500 and 2000 nm. This system is of great interest because chromium on glass is used extensively for lithography masks. K ratio versus film thickness plots were obtained for all of these systems over a range of beam energies from 7 keV to 20 keV.

Over the past four decades there have been surprisingly few published studies that present experimental K ratio plots as a function of film thickness (or film mass per unit area) for either pure elements or alloy films [1, 2]. In addition, several models have been proposed to generate calibration curves that could be used to determine thickness and in the case of multicomponent films, composition [3, 4]. However, with the general lack of data, they have not been tested extensively.

The previously mentioned substrates were divided into ten sets and a film of Cr was deposited simultaneously on all substrates of each set by electron beam evaporation. After the completion of all depositions a piece of each substrate was selected for film thickness measurements. The thickness measurement samples were coated by sputtering with approximately 200 nm of AuPd which would serve as a protective layer during polishing, which was performed with particular care not to distort the measured thickness. Measurements were made with a Carl Zeiss 1550 Schottky emitter SEM calibrated with a MOXTEK magnification standard, certified at 292 nm. High resolution SEM cross-sectional images like the ones shown in Figure 1, provided film thickness measurements from at least ten different points on each sample with error bars less than 4 nm (2 sigma). The remaining samples were then analyzed in a JEOL 8600 electron probe microanalyzer equipped with both wavelength dispersive and energy dispersive spectrometers with take off angles of 40°. The EDS spectrometer was used to look for the presence of any impurities in the film and also to measure accurately the beam landing energy by use of the Duane-Hunt limit. No impurities were detected although oxygen is a difficult element to measure in the presence of Cr, and hydrogen requires other methods of analysis that are presently under consideration.

For the set of samples with no intermediate SiO₂ layer excess electrons are conducted through the substrate directly to the ground but with an oxide present the situation may differ. One should expect differences in the chromium X-ray emission intensity between the Cr/Si, Cr/SiO₂ and Cr/SiO₂/Si samples because of the varying backscattered electron contribution of the substrate and possible charging effects since there is no clear path for all the electrons to ground. Figure 2(a) shows plots of K-ratios versus films thickness for Cr/Si, Cr/500 nm SiO₂/Si, and Cr/Quartz at 10, 15 and 20 keV. Within the error bars, less than 2% (2 sigma) of the measured K-ratio, the combination of backscattered electrons and charging effects, if they exist, do not appear to appreciably change the measured K-ratios. For completeness, Figure 2(b) shows the Si K-ratios as a function of Cr film thickness. However, for the cases where the SiO₂ intermediate layer is present, the signal can come from both the oxide and the underlying Si substrate making data more difficult to interpret. Work is also underway to evaluate the $\phi(\rho z)$ and Monte Carlo models in view of these new results.

References

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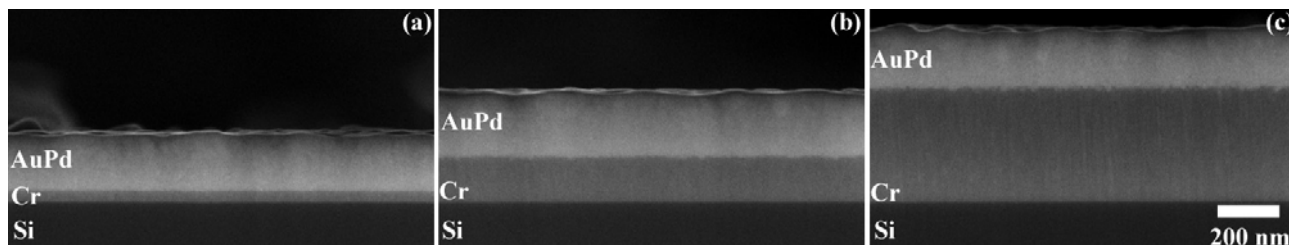


FIG. 1. Cross-sectional SEM images of (a) 35 ± 2.1 nm, (b) 150 ± 2.2 nm and (c) 370 ± 3.9 nm Cr films on Si substrates coated with 200 nm of AuPd.

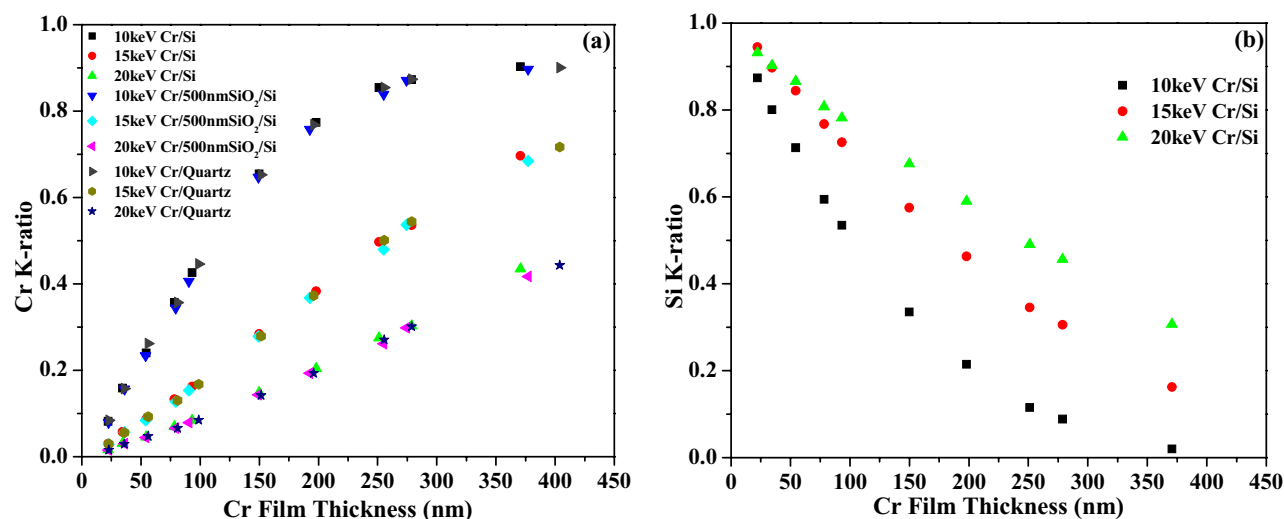


FIG. 2. (a) Cr K-ratio vs Cr film thickness at 10, 15 and 20 keV on Si, 500 nm SiO₂/Si and Quartz substrates. (b) Si K-ratio vs Cr film thickness at 10, 15 and 20 keV for Cr/Si.