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D8 ADVANCE

CR-Kα GÖBEL MIRRORS FOR IMPROVED RESOLUTION IN X-RAY REFLECTOMETRY INVESTIGATIONS

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X-ray reflectometry is a powerful, non-destructive tool for the characterization of surfaces, thin films and layer stackings [1,2]. The physical properties that can be determined from specular reflectivity measurements are thicknesses and density or chemistry of the layers, as well as surface and interface roughnesses perpendicular to the sample surface. X-ray reflectometry is the technique able to determine layer thicknesses with an accuracy of some 0.01 nanometers. Even layer sequences with a complicated architecture which are build up from many different layers can be analyzed.
Furthermore, the root mean square roughness of a surface or an interface can be determined in a range between about 20 Å and 0.2 Å. Densities can be determined down to 1% accuracy. In addition to the specular reflection, the off-specular reflectivity can be used to determine lateral features such as the lateral roughness coherence and the interfacial jagginess [2,3]. However, the off-specular reflectivity is usually weak and requires a powerful X-ray source.

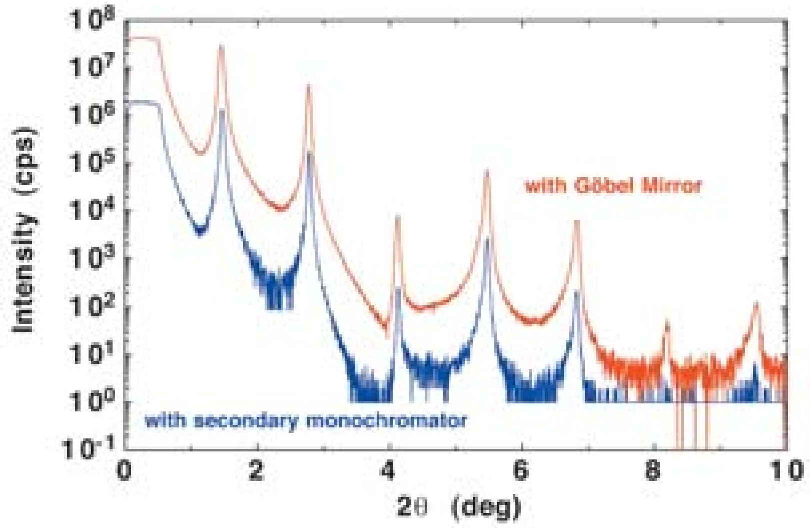

Fig. 1 – Reflection curves of a 6.5 nm-period V-C multilayer film with 40 periods, measured with Cu radiation and an incident beam Göbel Mirror and with a secondary monochromator, respectively.

Under this point of view, the development of Göbel Mirrors for laboratory X-ray sources was a significant step towards enhanced performance in X-ray reflectometry. For this application the Göbel Mirrors give a remarkable intensity gain [4-6]. To illustrate this intensity gain, Fig. 1 shows reflection curves of a Vanadium-Carbon multilayer film measured with Cu-Kα radiation. The measurements were executed with a D8 ADVANCE diffractometer (fig. 2) equipped with the reflectometry stage [1] with Knife Edge Collimator (fig. 3). The upper reflection curve was measured with an incident beam Göbel Mirror for Cu-Kα radiation, the lower without a Göbel Mirror but with a Graphite diffracted beam monochromator to suppress the influence of Cu-Kß radiation. The distance between knife edge (fig. 3) and sample surface was 10 µm. All other measurement conditions were identical. The measurements are plotted in figure 1 in units counts per second without normalizing. Therefore the difference demonstrates the more than one order of magnitude higher primary intensity provided by the Göbel Mirror for this application.

Similar intensity gains can be expected for Co-Kα radiation. However, if Cu- Kα radiation (λ = 0.1542 nm) is used, intensity oscillations (Kiessig fringes [7]) caused by layer thicknesses bigger than about 200 nm can not be resolved with a conventional X-ray diffractometer because the typical instrumental line broadening is of the order of 0.006°. To overcome this thickness limitation, we have used Cr-Kα radiation (λ = 0.229 nm) in combination with our recently developed Göbel Mirrors optimized for this wavelength.

To compare the resolution of Cr-Kα radiation to Cu-Kα radiation figure 4 shows measurement results obtained on a thick boron film. For easier comparison the measured intensities are displayed versus a wavelength-independent $\sin\theta/\lambda = 1/(2d)$ x-scale. It can be seen that the Kiessig fringes are well resolved with Cr-Kα radiation, but only poorly resolved with Cu-Kα radiation. An evaluation of the Cr-Kα measurement shows that the boron film has a thickness of 290.7 nm, a density of 1.7 g/cm^3, and an interface and a surface roughness of 0.4 nm. The resolution of the Cu-Kα measurement is insufficient to deduce this result unambiguously. It is also worth to note that the regime of total external reflection can better be measured with the use of Cr-Kα radiation than with Cu-Kα radiation due to the higher critical angle (the critical angle of total external reflection scales with the selected wavelength λ). This is advantageous in cases where the samples are relatively small and have low densities.

Fig. 2 - D8 ADVANCE goniometer with incident beam Göbel Mirror, reflectometry sample stage, fixed slit assembly with automated absorber and scintillation counter.

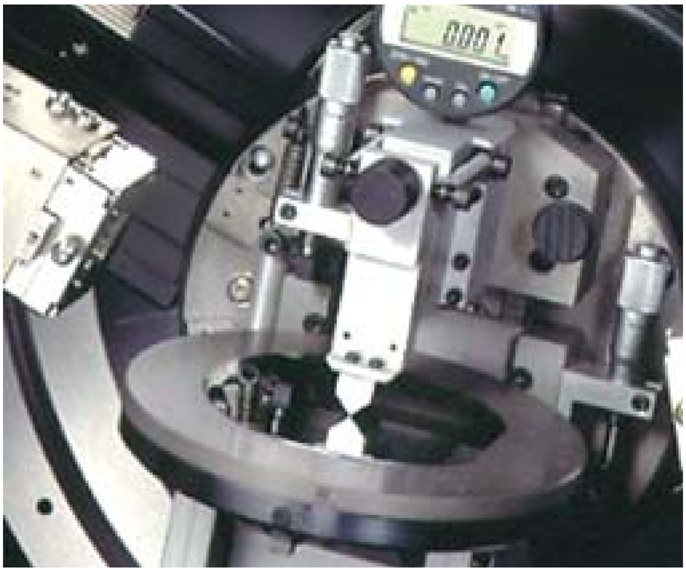

Fig. 3 – Reflectometry sample stage with Knife Edge Collimator. The dial indicates a distance of 1 µm between sample surface and knife edge.

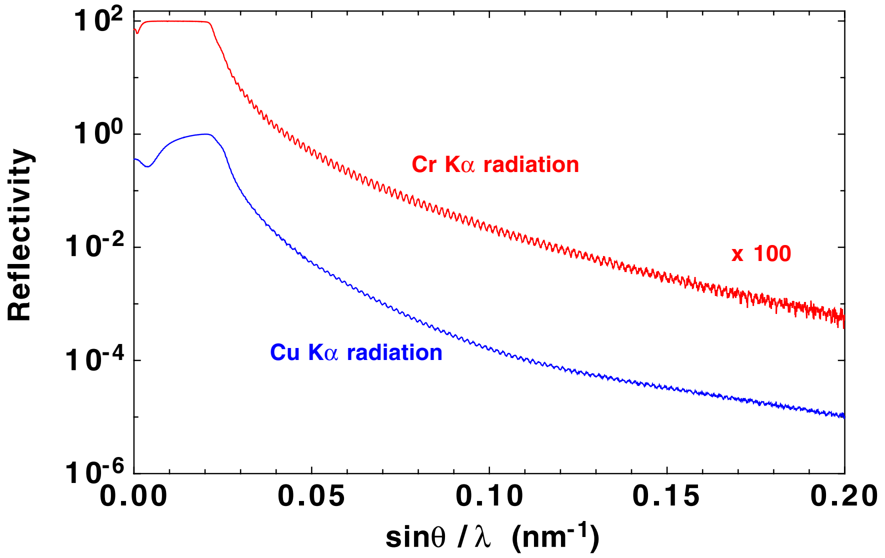

Fig. 4 – Reflection curves of a 290.7 nm thick boron film sputtered onto a silicon wafer, measured with Cr-Kα (35kV 35mA) and Cu-Kα (40kV 40mA) radiation, respectively, and using Göbel Mirrors optimized for the selected wavelengths. The 2θ step size was 0.002°, the scan speed was 1 step/sec, and the total measurement time was 41 min 40 sec.

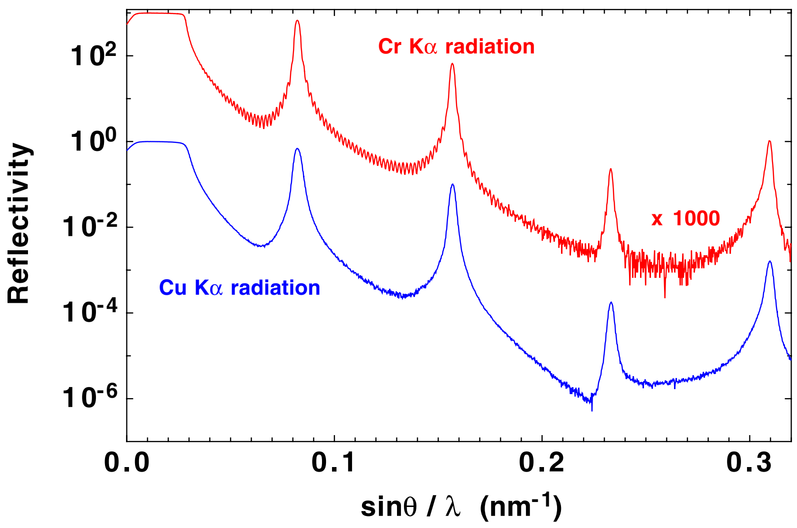

Fig. 5 - Reflectivity measurements of the V-C multilayer film of Fig. 1, obtained with Cu-Kα (40kV 40mA) and Cr-Kα (35kV 35mA) radiation, respectively, and using Göbel Mirrors optimized for these wavelengths. The 2θ step size was 0.004°, the scan speed was 1 step/sec, and the total measurement time was 35 min 25 sec each.

Figure 5 shows a second example for the improved resolution obtained with Cr-Kα radiation. Again the measurement results obtained on the V-C multilayer are shown, but in figure 5 the reflection curves of Cu-Kα radiation and Cr-Kα are compared. For each measurement a Göbel Mirror optimized for the selected wavelength was used. The measurement with Cr-Kα radiation resolves the Kiessig fringes between the main order reflections of the multilayer. They are due to the total film thickness of 260 nm. These Kiessig fringes are not resolved with Cu-Kα radiation. With this additional information it was possible to determine the number (N=40) of double layers from the number of fringes (N-2) measured with Cr-Kα.

It is worth noting that the spectral purity of both wavelength-optimized Göbel Mirrors is high, with Kβ/Kα ratios $< 5 \times 10^{-4}$, as determined by measurements on a silicon wafer. This is sufficient to eliminate any Kβ contributions even in reflectivity measurement where usually many orders of magnitude in intensity are covered. The 35 minutes lasting measurements shown in Fig. 5 cover about 6 orders of magnitude in intensity for Cr-Kα radiation, and about 7 orders of magnitude for Cu-Kα radiation. This difference is mainly due to the stronger air absorption and scattering of Cr-Kα radiation. As a result, the onset of background in the low-intensity regions is more pronounced in the Cr–Kα measurement.

These two examples demonstrate the higher resolution that can be obtained with Cr-Kα radiation for X-ray reflectometry applications. They also demonstrate the high quality, particularly the high spectral purity that is nowadays routinely available with the latest development of Göbel Mirrors. With the improved brilliance provided by Göbel Mirrors, which typically provide a 1 mm wide beam of 3×10^8 cps Cr-Kα respectively 3×10^9 cps Cu-Kα at a source-to-detector distance of 435 mm, even off-specular reflectivity measurements can be performed in conventional sealed-tube diffractometers, as demonstrated in Ref. [8].

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