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In-situ X-Ray Diffraction Measurements during Low Energy Ion Beam Etching

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In-situ X-ray diffraction measurements following the details of ion nitriding of stainless steel has been developed during the last 5 years. Nevertheless, the amount of local information is limited as diffusion and relaxation processes due to the elevated temperature during the process will lead to dynamic processes difficult to be resolved as a function of depth. At the same time, samples produced by plasma nitriding or PIII cannot be analyzed. Here, we propose to use low energy ion beam etching coupled with in-situ XRD to obtain detailed, depth-resolved data. Limiting the ion energy to 1 keV or less will lead to minor modifications of material, avoiding potential plastic or elastic deformation during mechanical removal, restricted to the immediate surface zone of only up to 10 nm. At the same time, the XRD information depth is between 2 and 200 µm, conditional on the specific materials system. With a current density near 100 µA/cm^2, a depth resolution of 15 – 25 nm per spectrum can be realized. The analysis of the results can be performed using the intensity of reflections from the layer system as well from an underlying substrate. As the experimental setup is constrained to Bragg-Brentano geometry, surface roughening may result in a continuously degrading depth sensitivity. As one example, the ion beam sputtering of expanded austenite formed on austenite using PIII is compared to samples formed by low energy ion nitriding. Several interesting points have been identified here: (i) orientation dependent sputtering (but not orientation dependent diffusion), (ii) the XRD peak shape and position does not follow the nitrogen depth profiles of the expanded phase as obtained by SIMS, (iii) PIII samples show consistently broader peaks not confined to the surface zone affected by the ion implantation, and (iv) by adjusting the ion energy, beam induced surface roughening can be minimized. A further experiment to demonstrate the versatility of the system was performed using a thin magnesium foil fixed on a substrate for a determination of the relative sputter yield for the surface oxide and the bulk foil.

Keywords
in-situ XRD
depth profiling
ion etching