Reduction of iron oxides as indirect hydrogen detection method in XPS

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Challenges of analysing hydrogen must be addressed to keep up with increasing interest of hydrogen effects in the field of material science. In contrast to other elements, most characterization methods are not able to detect hydrogen directly which necessitates alternative detection techniques. X-Ray photoelectron spectroscopy XPS can be used to indirectly prove hydrogen diffusion through bulk steel samples. To prove this concept, we electrochemically charged high strength dual-phase steel DP1000 on one side (hydrogen entry side) with hydrogen and monitoring the reduction of iron in oxide layers on the opposite side (hydrogen exit side) due to diffused hydrogen. This reaction is resolvable in the high-resolution Fe2p$_{3/2}$ peak (see Fig. 1) and was shown for native and artificially grown oxide layers. A shift in the order of few eV towards lower binding energies as well as the position of the shake-up satellites are used to resolve the reduction of oxide layers. The amount of iron transitioned from the third to the second oxidation state was estimated through an appropriate fitting procedure. Additionally, a layer model of the most common iron oxide species is presented for the reduction processes and re-oxidation experiments that quantitatively represents the volume ratios of the species.

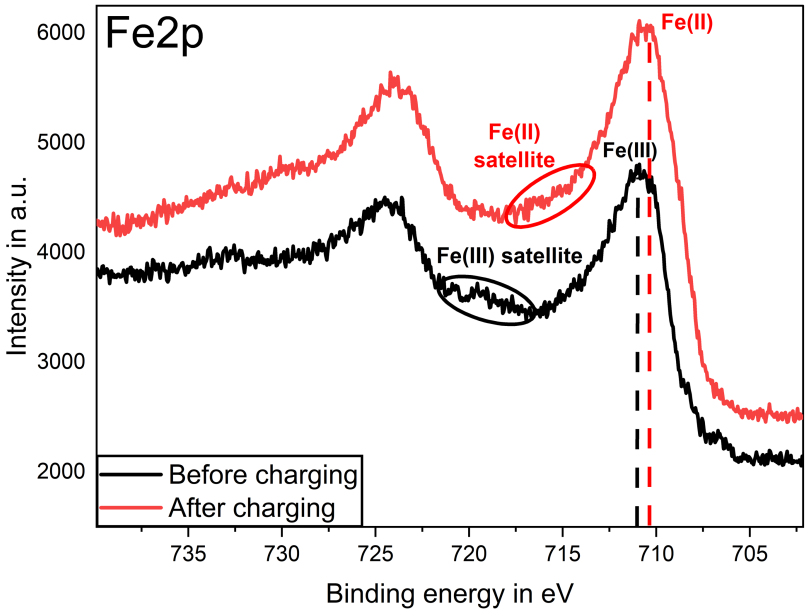

Figure 1. High-resolution Fe2p$_{3/2}$ peak indicating the reduction of the iron oxide layer.