

Calibration of the XRD and TDS Equipment for Investigation of Hydrogen Induced Near-Surface Effects in Fusion Relevant Materials

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For calibration of XRD measurements to perform the depth-controllable analysis, we have studied the steel and iron samples with a thin (submicron) copper and tungsten coatings. The thickness of the coatings was estimated from the iron substrate's diffraction lines intensity decrease. Using asymmetric (glancing-incidence) measurements, the conditions for the disappearance of lines from the Fe-substrate were established, finally allowing us to estimate the thickness of the steel layer (underneath the coating) participating in the formation of the diffraction pattern. For copper plated steel (510 nm), $\text{CoK}\alpha$ penetration depth is approximately 6 to 15 μm , for $\text{CoK}\beta$ it is approximately 1-2 μm . This makes it possible to obtain simultaneously the structural information from the upper ("interfacial") and more in-depth (conditionally bulk) regions of the steel substrate using a single (cobalt) X-ray tube and constant geometrical conditions of data acquisition. Thus, the method of the depth-differentiated estimation of the structural characteristics of the "interface" and conditionally "bulk" regions of the α -Fe substrate using polychromatic cobalt radiation was tested and discussed. This approach is suitable for the phase analysis and determination of the lattice constant of the near-surface regions of steel and iron samples.

In addition, we performed the comparative XRD analysis of the stressed state of tungsten thin ribbon and magnetron sputtered tungsten coatings. As known, the residual stresses in the surface may strongly influence the sustainability of plasma-facing materials to hydrogen permeation. To measure the residual stresses we applied so called $\sin 2\Psi$ method: the measurement of angle-dependent lattice strains by tilting the sample in an axis that is perpendicular to the beam detector axis. Significant compressive macrostresses in magnetron sputtered tungsten coatings were found (seven times higher than those on the surface of the tungsten thin ribbon). Also, some structural and textural characteristics of tungsten layers have been studied. This experience will be useful for the depth controllable analysis of near-surface structural alterations induced by hydrogen saturation.

For calibration of the thermal desorption mass spectrometry (TDMS) setup, in order to obtain quantitative results, the powdered reference material of $\text{TiH}_{1.924}$ were used. The powder was examined by XRD in the initial state and after annealing at 800 °C to confirm the phase purity of supplied titanium hydride and to be sure that after such heat treatment the hydrogen was removed completely. At the calibration procedure, the small sample masses (from 0.02 to 10 mg) were used at a range of the heating rates from 5 to 15 K/min. The linear response of the mass spectral signal to introducing different amounts of $\text{TiH}_{1.924}$, i.e. hydrogen, into the system has been demonstrated, assuming that the area under the desorption curve is proportional to the total number of hydrogen atoms released. As established from the calibration, the setup allows to study the evolved gas flow 10^{14} - 10^{17} mol·c⁻¹ at the detection limit for H_2 of ~ 50 appm.

We have recorded several TDMS spectra from electrolytically hydrogenated samples of steel. Owing to the performed calibration, we quantified the total amount of hydrogen released, and at the decomposition of a wide desorption band (overlapping peaks) into relatively narrow desorption peaks, the same can be done for each of these components. This allows us to obtain the information on relative amount of hydrogen trapped into different specific internal sites of metallic materials. For evaluating a specific activation or binding energy of each trapping site (a_i), we apply the calculation method based on a single TDMS spectrum. This approach does not pretend to be a method for determining the absolute values of a_i , but allows us to identify and to compare the same forms of hydrogen in different samples of similar materials.

Primary authors: DANILCHENKO, Sergii (National Academy of Sciences of Ukraine); KALINKEVYCH, Oleksii (National Academy of Sciences of Ukraine)

Presenter: DANILCHENKO, Sergii (National Academy of Sciences of Ukraine)

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