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Development of the X-Ray Diffraction and Thermal Desorption Spectroscopy Techniques for Investigation of Proton (Deuterium/Helium) Induced Near-Surface Effects in Fusion Relevant Materials

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Experimental simulations of ITER-like events in which hydrogen, helium and heavy ion irradiation causes ion-induced radiation damages in the near-surface region of a metal target is a common method; and XRD is widely used for assessment of the structural alterations in the targets after such irradiation. To get the reliable data, the damage depth (irradiated layer) and penetration depth of X-rays in the XRD analysis (the layers from which the information is collected, the "information depth") should be of the same order of magnitude. The reactor wall materials have quite different X-ray absorption rates: tungsten in the conventional analysis in reflective mode may be probed in depth of few (0.5-2.5) micrometres, while beryllium is almost transparent for X-rays of any commonly used tubes (the penetration depth in it is up to several thousand micrometres). For alloys based on Fe (like RAFM steels) or based on Ni, Cr and Fe (like Inconel), the information depth of XRD analysis () depends on their composition, ranging from few micrometres to dozen micrometres. To reduce X-ray penetration depth inside the material down to few hundreds or even tens of nanometres, we are planning to apply the **grazing incidence XRD** (GIXRD), where depends on the grazing incidence angle (). Additional advantage of GIXRD is that in this case is almost independent from the diffraction angle 2 (in contrast to the conventional mode), that allows the microstructure analysis (crystallite sizes, microstrain, dislocation density, etc.), since the volume of the material participating in the formation of total diffraction pattern does not change for reflections (peaks) with different *hkl* indices.
We performed the calculation of for several materials of interest (W, Be, Inconel690, F82H steel, Li2TiO3) at a number of incidence angles , and for several selected X-ray sources (tubes). The appropriate graphical material is given in the presentation. These evaluations show that XRD experiment can be adapted to certain materials and conditions of their ion irradiation. The use of GIXRD may provide selective analysis of the near-surface regions in order to measure the structural features of affected layers, eliminating the contribution of the non-irradiated volume to the measured diffraction pattern. As depends not only on the material's properties (density and absorption coefficient), but also on instrumental factors (mainly detection system), the calibration should be done to estimate the part of total X-ray radiation, which comes from the subsurface layer at the depth (I0/I). For this the model samples with the coating of known thickness (two-layer systems) will be examined.
In the project GIXRD method for structural study of thin irradiated layer will be combined with thermal desorption spectrometry (TDS) to study hydrogen (helium) trapping and retention in these layers. For this we have constructed and tested two laboratory setups allowing to examine gaseous species (including hydrogen and helium) released from metallic materials under programmed heating. First one uses a mass spectrometer as the detector (TD-MS setup), and the second is based on a gas chromatograph (TD-GC setup). From our own experience and the available literature, it is seems that the quantitative assessment of hydrogen content by TD-GC is more realistic than by TD-MS. Moreover, TD-GC technique has some other advantages compared to TD-MS, such as: lesser discrimination effects in quantitative analyses of gas mixtures; the ability to detect separately 4He and D2 in contrast to conventional MS; TD-GC technique does not need high vacuum equipment and is easier in calibration than TD-MS. At the same time, TD-GC has several limitations, some of which are mentioned in the presentation and can be partially compensated by applying TD-MS. The general restrictions for any TDS approach should be taken into account aware as well. Nevertheless, in our opinion, the combined application of TD-MS and TD-GC may give certain benefits in investigation of gas impurities in fusion materials.

The work plan describing the near-future work (1-2 years).
- GIXRD calibration using model samples with coatings with the known thickness (two-layer system) to get information about the dependence of effective penetration depth vs. the grazing incidence angle for metals of interest;
- to study the possibility to apply unfiltered X-ray radiation (containing K and K components) exploiting the K-absorption edge effect for simultaneous structural examination of surface and bulk by using the same X-ray tube; e.g., for steels the CoK radiation can provide "bulk" information due to low absorption, while the CoK radiation may represent "surface" information owing to rather big absorption;

- to perform calculations and preliminary experiments to evaluate the possibility of depth controlled GIXRD structural analysis of samples with the coatings, e.g., copper-coated, tungsten-coated and beryllium-coated RAFM steel; the aim is the structural study of near-interface layers of the substrate;
- further development of the TDS methods (equipment and protocols) to determine the total content of the gas-forming impurities (hydrogen, helium etc.) in irradiated metals (alloys), getting the information on the relative amount of hydrogen trapped into different internal sites of the materials, and for evaluation of the kinetic parameters (the activation energy and frequency factor) associated to each detrapping event;
- calibration of the TDS setups (the mass spectrometric system and the gas chromatography system) using reference materials (some hydrides, gaseous mixtures).

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