Measurement of hydrogen isotopes and other light elements in thin films of materials of interest in fusion-reactor technology

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The capability of detecting and depth profiling hydrogen and its isotopes (deuterium, tritium) in the near surface region of solid materials is of great importance in different areas of materials science, one of them being the fusion-reactor technology [1]. Such as, in the investigation of the plasma-wall interaction in fusion research the total content as well as the depth profiles of hydrogen isotopes implanted and accumulated in the surface layers of the wessel walls have to be measured. Ion beam analysis (IBA) techniques are especially valuable since the quantification of low mass elements is straightforward and sensitive. Among several ion-beam technique for detection and depth profiling of hydrogen isotopes, Elastic Recoil Detection Analysis (ERDA) technique using a low energy ^{4}He beam proposed by Doyle and Peercy [2] is particularly advantageous; all hydrogen isotopes can be profiled simultaneously with a sensitivity as high 0.1 at.%, the measurements can be performed using a relatively low energy accelerator and the samples undergoes less damage as compared with the use of high-Z analysis [2,3].

The large recoil cross sections and the possibility of placing detectors at forward (ERDA) and backward (RBS) angles was leading to a rapid development of the technique. So, the helium-induced hydrogen forward-recoil method has been finding increasing use in different laboratories.

The materials which will be investigated are of high interest for fusion technology (in actual ITER and future DEMO fusion reactors): mixtures of W and C containing H or D, and also SiC. These will be produced by a novel deposition technique in plasma (sequential deposition [6], developed in the last two years by one of the project participants). The material samples will be investigated as regarding their H and D content (for W/C mixtures) and their ability to act as diffusion barrier for H and D (for SiC implanted with He). The IBA measurements results (as regarding the H and D content in the W/C samples) will allow achieving one of the project partial objectives: definition of the sequential deposition process parameters for producing samples with predefined elemental composition (W, C, H, D). Simultaneously with IBA material investigations, the mentioned materials will be also investigated by SIMS, a technique less time and resources consuming in comparison with IBA. The elemental IBA profiles (obtained with high accuracy) will allow calibration of the SIMS device for higher accuracy in the range of low atomic masses, for future use in low cost investigation of samples containing H and/or D (second partial objective).

The material samples which will be investigated in this project by previously described IBA techniques will be produced using a novel deposited technique in plasma (the sequential deposition method) developed recently by the INFLPR partner and used to the moment for deposition of W/C composite and multilayered thin films [4]. The deposition procedure and the preliminary results are in brief presented bellow. Two plasma sources (a magnetron for tungsten deposition in Ar and a Plasma Enhanced Chemical Vapor Deposition (PECVD) plasma source for carbon deposition from Ar, and C_2H_2) are orthogonally mounted on a vacuum chamber. The film is grown by cyclic exposing the substrate to the plasma sources for predefined time intervals. The substrate (which is grounded) is transported between the plasma sources by means of a stepper motor. During the substrate movement the plasma sources are not energized. One cycle of the sequential deposition process consist in: metal deposition step in magnetron discharge (lasting t_W seconds), substrate transport in front of PECVD plasma source (lasting t_{TR} seconds), carbon deposition step (lasting t_C seconds) and backward transport of the substrate in the front of magnetron (lasting t_{TR} seconds). For samples preparation, the single deposition cycle, which last for a time interval equal to $t_{CYC} = t_W + 2t_{TR} + t_C$, is repeated N_{CYC} times, depending on the desired film thickness. For small plasma exposures times (t_W and t_C ~ seconds) composite films are grown while for much longer values (t_W and t_C ~ tens of seconds) there are deposited multilayer films. The elemental composition in the deposited composite layers is adjusted by modifying the ratio between the plasma exposure durations during deposition cycles; such as, there were deposited W/C composite layers (similar with the codeposited layers present in ITER) with W/(W+C) ratio in between 10% (for t_W=2 s, t_C=11s) and 50% (for t_W=10 s, t_C=3s). Increasing the plasma exposure duration up to tens of seconds (t_W=20 s, t_C=40s) there were deposited periodic (20nm) W/C multilayered structures.

While the substrate is grounded, the carbon deposited during the PECVD step is an amorphous hydrogenated carbon (a-C:H), with high content of hydrogen (up to 50%). Composite layers containing Deuterium are obtained using a mixture of D, C_2H_2 and Ar during the PECVD discharge steps. The H and D content of the deposited samples is possible to be varied by applying a voltage bias on the substrate during deposition; preliminary qualitative measurements (performed by Secondary ions mass spectrometry (SIMS) depth profiling) proved this hypothesis. Still, for an accurate quantitative evaluation of the H and D incorporated in the deposited layers it is necessary to perform their characterization by means of advanced nuclear techniques, as the present project proposes. The sequential deposition process offer a good repeatability of the deposited samples as regarding their composition (tested for over one year).

Beam request:

4 days at the 3 MV Tandetron

4 days at the 9 MV Tandem

References

[1] H Nakamura, J Dietz, P Ladd, Wall conditioning in ITER, Vacuum, 47, 6-8, 653-

655, (1996)

[2] B. L. Doyle and P. S. Peercy, Appl. Phys. Lett. 34 (1979)811

[3] B. L. Doyle and P. S. Peercy, Proc. Of Workshop on the Analysis of Hydrogen in

Solids, Albuquerque, NM (Jan. 23-25, 1979)

[4] T. Acsente, E.R. Ionita, D. Colceag, A. Moldovan, C. Luculescu, R. Birjega, G. Dinescu, Properties of composite a-C:H/metal layers deposited by combined RF PECVD/magnetron sputtering techniques, Thin Solid Films, 519, 12, 4054-4058, (2011)