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A small and compact AMS facility for tritium depth profiling

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Abstract. Depth profiling measurements of tritium in carbon samples have been performed during the past seven years at the AMS facility installed at the Rossendorf 3 MV Tandetron. The samples have been cut from the inner walls of the fusion experiments ASDEX-upgrade/Garching and JET/Culham. The tritium content of the samples from JET required a dedicated AMS facility to prevent any contamination of the versatile 3 MV Tandetron. On the basis of an air-insulated 100 kV tandem accelerator equipped with a gas stripper an AMS facility exclusively devoted to tritium depth profiling was installed, tested and used for routine measurements. After additional successful tests employing diamond-like carbon (DLC) stripper foils at this accelerator, another small and compact 100 kV tandem accelerator with SF_6 insulation and a DLC stripper has been installed at the AMS facility. Results obtained with the different tandem accelerators are presented.

Keywords. Mass spectrometry; tritium; fusion; electrostatic accelerators.

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1. Introduction

Detailed measurements of the concentration and depth profiles of hydrogen and other light elements have been performed in the Forschungszentrum Rossendorf by nuclear reaction analysis (NRA), elastic recoil detection analysis (ERDA) and accelerator mass spectrometry (AMS) [1–3]. For the detection of very small concentration of tritium in the vessel wall of fusion experiments only the sensitivity of AMS is sufficient. This method is a combination of the conventional secondary negative ion mass spectrometry (SIMS) and mostly an electrostatic tandem accelerator with nuclear physics detection techniques for isotope separation [4]. The disturbing isobar molecules are disintegrated in the stripper of a tandem accelerator. Some remaining molecules can be separated by special particle detectors or by use of a stopping foil in front of the detector. Suppression factors for molecular ions of more than 10^{13} are achievable by AMS.

For the separation of different isotopes tandem accelerators with final energies of some MeV are usually applied. In the last years the development of AMS facilities is moving towards low-energy accelerators [5]. The high relative mass difference $\Delta M/M$ in the case of tritium detection has initiated the application of a tandem accelerator with final energy of some hundred keV.

2. Experimental and results

2.1 *Tritium AMS at the 3 MV Tandetron*

From 1994 a dedicated facility for tritium depth profiling by AMS was built up at the 30° beam line of the Rossendorf 3 MV Tandetron [3,6]. The negative ions are generated in a commercial Cs sputtering ion source HVEE 860-C and accelerated to 23 keV. The T^-, H_3^- and HD^- ions are selected with an 82.5° deflection magnet and are accelerated using the Tandetron at a terminal voltage of 1.5 MV. No disturbing negative ^{3}He ions are generated inside a Cs sputtering ion source. During stripping in the nitrogen gas stripper canal most of the HD and H_3 ions are split into atomic ions. The positive ions are again accelerated back to the ground potential to obtain a final energy of about 3.0 MeV. After analysis with the switching magnet the tritium ions are counted with a surface barrier detector. The remaining HD^+, H_3^+ and some heavier ions generated by charge exchange processes in the residual gas are separated in a 17 μm-thick Al foil in front of the detector. The 1 MeV H^+ ions from cracked HD^+ and H_3^+ ions are fully stopped in this foil, while the 2 MeV D^+ ions pass the foil with an energy of about 1.1 MeV. They can be separated in the detector from the T^+ ions passing the foil with 2.1 MeV. The 2 MeV D^+ ions can be also completely suppressed using two 17 μm-thick Al foils. Due to the wide range of measured tritium inventories the ion beam can be scanned across an aperture in front of the detector by two pairs of electrostatic deflecting plates to prevent overloading of the detector.

For depth profiling, the sample must be uniformly sputtered. Therefore, the sample is mechanically scanned by two off-axis disks rotating with different frequencies in connection with a modified target rod [7]. The sample has a diameter of 3 mm and a thickness of 1–4 mm. Using this arrangement a cylindrical sputter crater has been obtained instead of the common bell-shaped crater [3]. Finally electronic gating is used to collect signals only when the primary beam passes the centre of the crater. Due to this gating the ions sputtered from the side wall of the crater are not detected. All ions of interest except tritium, such as H^-, D^-, C^-, Be^- and other interesting elements can by measured with a retractable Faraday cup between the injection magnet and the entrance of the accelerator (SIMS mode).

From deuterium implanted carbon samples an ionization yield of 0.92% for negative deuterium ions was measured. The transmission of the accelerator for hydrogen ions at 1.5 MV terminal voltage was about 30%, depending mainly on the used stripper gas pressure.

With the calibrated AMS and SIMS facility tritium, deuterium and beryllium depth profiles were measured simultaneously for plasma facing samples of the Garching fusion experiment ASDEX-Upgrade and from JET Culham/UK. Results of these measurements are published in refs [3,7–11].

The measured maximum tritium concentrations in samples from ASDEX-Upgrade were in the range of 10^{14}–5×10^{15} atoms/cm^3, the resulting amounts were about 10^{11}–10^{12}

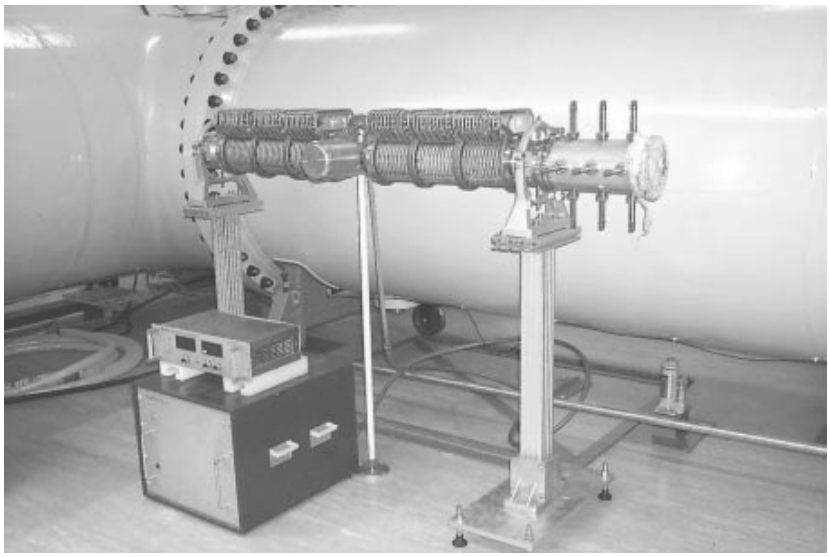

Figure 1. Air-insulated 100 kV tandem accelerator in comparison with the 3 MV Tandetron.

atoms/cm^2. For deuterium, values of 10^{19}–10^{22} atoms/cm^3 for the maximum concentration close to the surface and 10^{16}–10^{18} atoms/cm^2 for the resulting amount were obtained. The mean background of the ion source after some measurements is about 20 counts/s corresponding to a tritium concentration in the carbon samples of about 5.0×10^{10} atoms/cm^3.

Profiles of hydrogen cannot be obtained due to the hydrogen ion current from the residual gas, which is of the same order of magnitude as the expected beam current from the samples.

2.2 *Tritium AMS using a 100 kV tandem accelerator*

At the European experimental fusion facility JET Culham, which has been operated partly with a D-T plasma, a much higher tritium inventory (up to five orders of magnitude in comparison with ASDEX-Upgrade) is stored inside the vessel wall. These high activities required a dedicated AMS facility for depth profiling to prevent any contamination of the versatile 3 MV Tandetron accelerator and to obtain the necessary handling and working permissions for the high-dose tritium samples.

The installation of a new AMS facility was also initiated by the idea of a low-energy tandem accelerator. In the first step an air-insulated 100 kV tandem accelerator (figure 1) was built up using 2×3 acceleration tube modules from the switched off NEC tandem accelerator at the HMI Berlin. The injector of this facility has been nearly identical with that of the 3 MV Tandetron. This has given rise to the possibility to use all calibration parameters obtained at the Tandetron. Because no foil strippers were available in the Forschungszentrum Rossendorf at this time, the first version of this small tandem accelerator was equipped with a gas stripper. The stripper pressure was generated by air inlet. At both sides of the

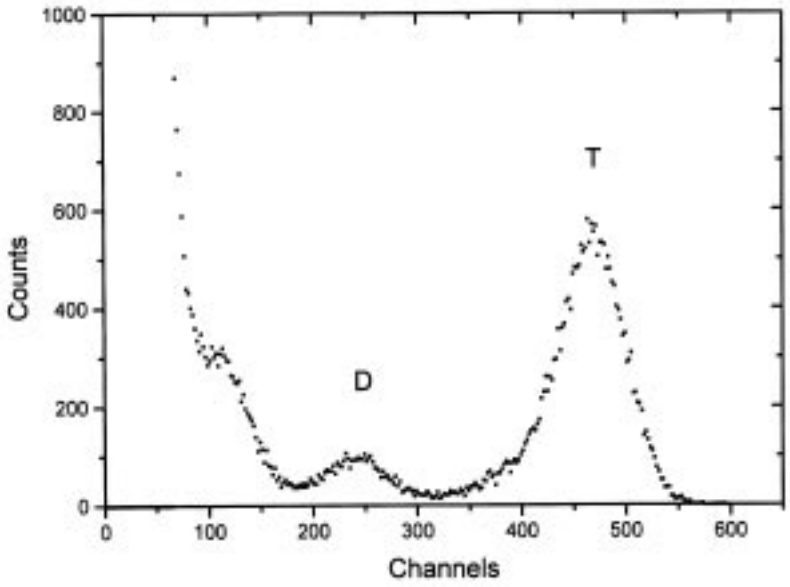

Figure 2. Measured energy spectrum of tritium and HD ions.

stripper the gas was pumped out by an 80 l/s turbo-molecular pump. After acceleration the positive ions were analyzed by a 15° deflecting magnet. The transmission of hydrogen ions through the accelerator up to the detector was about 40% as measured with a 4 cm diameter Faraday cup and 5% for the detector area of 1 cm^2.

In front of the detector the tritium ions were separated by a stopping foil. For the stopping foil a 75 $\mu g/cm^2$ Formvar foil evaporated with 40 $\mu g/cm^2$ Au was tested. The particle energy spectrum measured with the surface barrier detector has given an acceptable separation of the tritium ions from the H and D ions (figure 2). With this facility the depth profiles of high-dose samples from JET were investigated for about one year.

In cooperation with the Kurchatov Institute Moscow diamond-like carbon (DLC) foils [12] were tested as stripper foils at the 100 kV tandem. In figure 3 the transmission of the accelerator for 200 keV hydrogen ions is shown as a function of the foil stripper density. During the test measurements with low hydrogen beam currents no limitation for the life time of the foils has been found. Due to the higher transmission 1 $\mu g/cm^2$ carbon foils have been applied for the following measurements. The foil stripper has given the possibility to replace all vacuum equipments, power supplies and the gas inlet from the terminal. Because the insulation property of SF_6 gas under atmospheric pressure is three times higher than for air the remaining simple acceleration structure could be reduced to 2×1 NEC tube modules and installed inside an ISO NW-500 vacuum chamber filled with SF_6 gas (figure 4). During test measurements some disturbing counts appeared in the spectrum of tritium ions. They were generated by the stronger angular scattering of the foil stripper in connection with the small resolution of the 15° analyzing magnet. Therefore, this magnet was replaced by a double-focusing 90° analyzing magnet. The transmission for this accelerating and analyzing system was 80% as measured with a 4 cm diameter Faraday cup and 20% for the 1 cm^2 detector area.

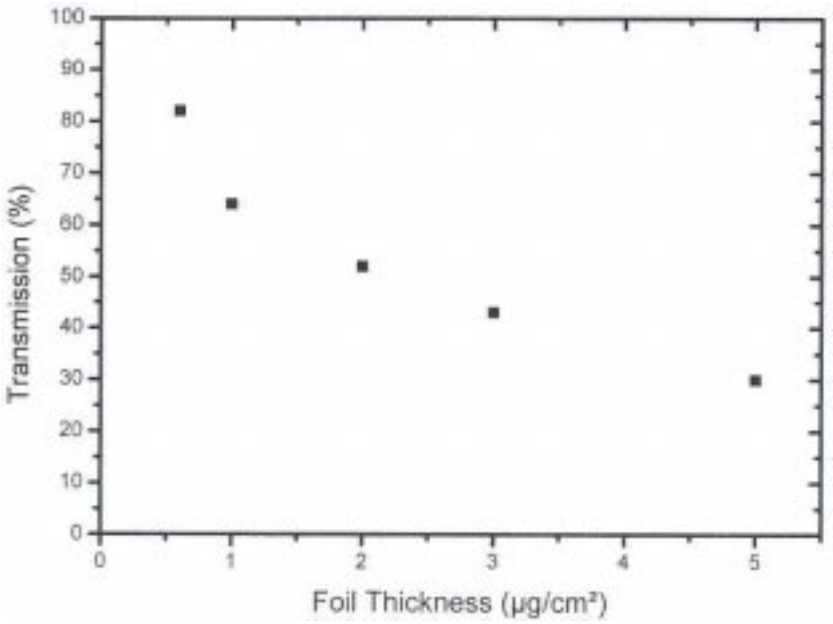

Figure 3. Transmission of the AMS facility as a function of the thickness of diamond-like carbon stripper foils.

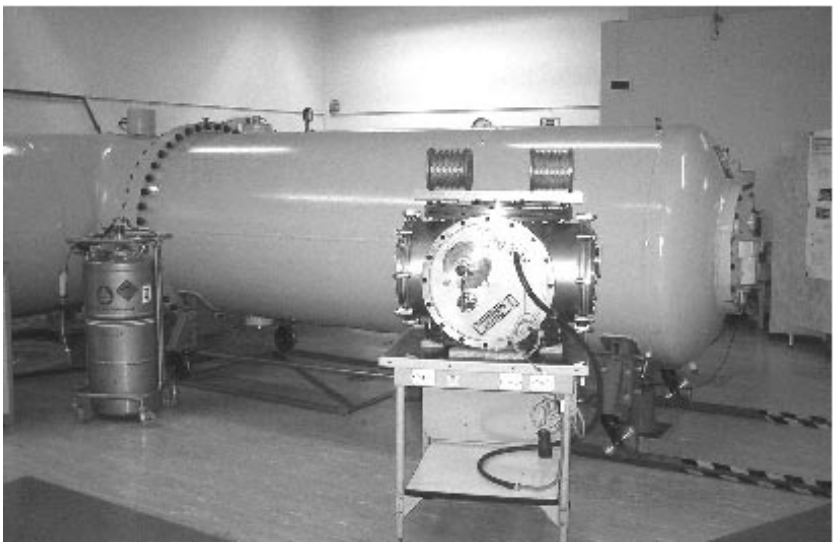

Figure 4. Compact SF_6-insulated 100 kV tandem accelerator. On the top of the chamber the installed two-acceleration tube modules are shown for comparison. The length of a tube module is 170 mm.

Test measurements at this compact AMS facility have been performed with standard samples from the LLNL Livermore/USA (TiH_2) and from the Tritium Laboratory of the IFIN-Horia Hulubei Bucharest/Romania (tritium in carbon). They have confirmed our former results at the 3 MV Tandetron and at the air-insulated 100 kV tandem.

On the other hand, a difference of a few orders of magnitude has been observed between samples measured by AMS and by the full combustion method. Full combustion coupled to liquid scintillation counting (LSC) is well-established and a well-known technique to give absolute values for tritium content in a carbon sample [13]. It seems that this difference is much smaller when the total amount of tritium contained in the samples is higher than 10^{15} T/cm^2. The difference may also be generated by tritium, outgassing from the small AMS samples during storage, sample preparation, or sampling. Special investigations changing the conditions during sample preparation and sampling by using a stronger sample cooling inside the sputter source are in preparation.

Beside tritium depth profiling by AMS an increasing part of the measurements are directed to simultaneous depth profiling of D, Li, Be, C and other light elements using the Faraday cup at the entrance of the accelerator (SIMS mode). Some parts of the inner wall of the discharge chamber at JET are coated with a Be layer. Using simultaneously depth profiling of T, C and Be the measured thickness of the Be layer and the variations in the Be and C concentrations allows a more accurate evaluation of the plasma–surface interaction. The deposition- or erosion-dominated regions appear as an increase or decrease of the layer thickness [10].

The tritium ion current at samples from JET with high tritium inventory can also be partly measured in the SIMS mode under consideration of the isobar molecules. The contribution of the molecular ion current is about 2.3×10^{-5} of the atomic ions [14] and can be neglected, if the tritium concentration is higher than about 10^{-3} of the hydrogen and deuterium concentration, respectively. At lower tritium concentrations the depth profiles must be measured by AMS.

3. Conclusions

AMS has been successfully applied to depth profiling of tritium in graphite samples from the wall material of the fusion experiments ASDEX-Upgrade and JET. The detection limit of this method is about 5.0×10^{10} atoms/cm^3 and a depth range larger than 50 μm can be analyzed. The depth profiles of other light elements can be measured simultaneously in a SIMS mode without acceleration of the considered ions.

The installation of a dedicated compact AMS facility based on a SF_6-insulated 100 kV tandem accelerator has given rise to the possibility of depth profiling measurements at samples with high tritium inventory.

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