

Surface hydrogen isotopes detection by low angle ion scattering spectroscopy

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Introduction

Hydrogen isotopes retention in thermonuclear fusion reactors is limited due to safety regulations and should be well controlled. Surface conditions can strongly affect accumulation rates of hydrogen isotopes in the bulk of plasma facing materials. Therefore, in vacuo methods of surface composition control may help to investigate mechanisms of this effects.

Low- and medium-energy ion scattering (LEIS/MEIS) and direct recoil spectroscopy (DRS) are widely used techniques for surface analysis[1-4]. These methods have extreme surface sensitivity and even can analyze only the first atomic layer of adsorbate without any signal from the background [5]. In addition, **DRS is sensitive** even for hydrogen isotopes adsorption [6]. Thus, capabilities of the LEIS+DRS combination for the estimation of hydrogen isotopes concentrations in the very first surface layers are discussed.



Ne⁺ scattering from hydrogen and deuterium gases

Intensities of protium and deuterium recoils on the gas pressure in interaction chamber









possible to scatter

ions on gas targets

and pressures.

for

different gases

- Operating pressure 10⁻⁷ Torr
- Diagnostic ion beam– H^+ , H_2^+ , H_3^+ He⁺, Ne⁺, Ar⁺
- Ion beam energy 1-25 keV
- Ion beam current density in the range 1-1000 nA/cm²
- Scattering angle $\theta = 32^{\circ}$
- Energy resolution ~1%
- Scattered and recoil ions detection
- Cryopanel for reduction of partial pressure of water vapor and hydrocarbons
- Sputtering Ar⁺ ion gun with 2 keV energy and current

Gas targets allow to find a relationship between the scattering/recoil peak intensity and the number of particles in the "gas target" that is proportional to the pressure. The gas target body can be roughly estimated as cylinder with $\sim 2 \text{ cm}^3$ volume. The number of particles in it is N = pV/kT, that can be varied from $3 \cdot 10^{11}$ to $3 \cdot 10^{13}$ for the pressure range from $3 \cdot 10^{-5}$ to $3 \cdot 10^{-3}$ Torr.

- The peak intensity of the recoils was divided on primary ion beam current, scattering cross-section and the peak energy.
- The intensity has non-linear dependence with the pressure for 3 energies of the primary beam. This can be explained by reduction of primary ion beam intensity before interaction with "gas target" due to scattering on the gas.
- The sensitivity of the method is sufficient to detect 10^{12} particles that is 1000 times lower the number of atoms in the first layer of tungsten for analyzed surface area.
- The DRS method estimate roughly the same number of particles for pressures lower 3.10-4 Torr for 3 different energies and primary beam current. For 10keV energy the sensitivity for H and D is comparable.



Ar + scattering on W with D₂O puffing



Hydrogen recoil can be seen in spectrum of Ar⁺ from W surface. From the comparison of the normalized intensities of H and W peaks the ratio between H and W atomic concentration the on surface appeared to be about 26%.

density up to 10 mkA/cm²



To restore the energy distribution, it is necessary to divide the spectrum by the energy.

Scattering from water vapor allows to compare H and O peak intensities knowing the actual atomic concentrations ratio. Unlike Ne⁺, Ar⁺ can not scatter from oxygen at large angles, so only recoils can be detected. The ratios between H and O were calculated and were slightly higher than 1. **Do you know why?**

D₂O vapor was injected in vacuum chamber to check whether H will be replaced with D. It was possible to achieve the ratio H/D ~1.4 for D₂O vapor pressure $3 \cdot 10^{-6}$ Torr. However D⁺ recoil disappeared shortly after the end of the leakage.

Ne⁺ scattering from tungsten target with H₂O and D₂O puffing



LEIS calculator

The scattering cross section was calculated using the "magic formula" [7] for 3 types of potentials: Ziegler-Birzak-Littmark, Thomas-Fermi-Molière and krypton-carbon potential. However, the choice of potential can change the calculated cross section by no more than 2%. The program is intended not for obtaining the absolute values of the cross sections, but for the correct estimate of the ratio of atoms of different sorts on the target surface by dividing the experimental peak intensity by the calculated cross section. The program is able to obtain cross sections either for directly scattered ions or for recoils, has a user-friendly interface and is patented.

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- Initially the tungsten surface is covered by H_2O and H concentration can be up to several tens atomic percent. Sputtering by Ar⁺ gun was used for cleaning the surface to study the dynamic of H⁺ peak just after the end of sputtering. The concentration of 50% of initial amplitude was achieved after 1 minute after switching the gun off.
- The experiment with D_2O was performed to investigate how fast specially placed D_2O on the surface will be replaced by H_2O . Unfortunately it was impossible to measure D⁺ peak dynamic since it was in the same place with W⁺ recoil. However, a rise of H⁺ peak right after switching off sputtering gun was not observed with D_2O puffing. Even after switching off D_2O source H⁺ peak achieve initial intensity after 100 minutes while in previous case it take only 1 minute.

Summary

- Protium and deuterium isotopes concentrations on the tungsten surface were investigated using direct recoil spectroscopy.
- Experiments with gas targets allow to check experimentally the sensitivity of the method for different elements and make quantity calibration.
- Experiments with D_2O puffing demonstrates that under 5.10⁻⁷ Torr partial pressure of H_2O protium replace deuterium on the surface approximately after 100 minutes of exposure.