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Elastic recoil cross section determination of deuterium by helium-4 ions at 30° with the energy range of 2.6–7.4 MeV

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ABSTRACT

The elastic recoil cross section for D(^{4}He, D) ^{4}He was determined at a recoil angle of 30° over an incident helium energy range from 2.6 to 7.4 MeV. A thin solid target Ta/TiD$_x$/Si used for cross section measurement was prepared by direct current magnetron sputtering, and it was so stable to ion beam bombardment that nearly no deuterium loss (less than 0.2%) exists over the whole experiment. A relative determination method is adopted in this measurement. It can avoid the error from the beam dose and the solid angle of the detectors and it is also free to direct measurement of D content in the film. The total uncertainty in the cross section determination is less than 5%.

1. Introduction

Deuterium, the isotope of hydrogen, and its storage in materials have an important application in nuclear industry, especially in the fusion field [1]. Hence the accurate measurement of the deuterium depth profiling information in materials will be an integral part in nuclear technology. There are several ways to determine the deuterium profiling depth information in materials such as micro combustion analysis, secondary ion mass spectroscopy (SIMS), nuclear magnetic resonance (NMR), nuclear reaction analysis (NRA) and elastic recoil detection analysis (ERDA). Among those methods, the elastic recoil detection analysis is usually used to achieve the determination of hydrogen isotopes benefiting from its high resolution and non-destruction in practical application [2]. Therefore, the exact alpha-deuterium elastic scattering cross sections are quite needed. In previous works, many elastic scattering cross-sections for the interaction of helium with deuterium in certain energy ranges have been measured [3–8], but the energy ranges in their works were either lower than 3.0 MeV or higher than 9.0 MeV. In order to determine the profiling information in a deeper depth in materials, the cross-section in energy range of 3.0–9.0 MeV is much needed.

In this work, the cross-section of the interaction of deuterium with helium at a recoil angle of 30° over the energy range from 2.6 to 7.4 MeV based on our accelerator was measured. The main error source of the accurate determination of the cross sections is the value of the deuterium in the sample during the whole measurement because of the deuterium loss caused by the ion bombardment to the sample. The content of deuterium in the sample could be measured by using the ^{16}O–D Rutherford cross section at a certain energy in our measurement. The sample of Ta/TiD$_x$/Si, prepared by magnetron sputtering in the atmosphere of Ar and D$_2$ mixture gas, is stable to the ion bombardment and almost suffers no loss of deuterium in the experiment. Therefore, the total number of the incident ion and the error caused by the solid angle can be avoided in this measurement method.

2. Experimental

2.1. Experimental system

The measurement was carried out in a high vacuum chamber with pressure $<1 \times 10^{-4}$ Pa. The incident ^{4}He ion beams in these cross-section measurements, in the energy range from 2.6 to 7.4 MeV, were provided by NEC 9SDH-2 2 × 3MV tandem accelerator at Fudan University. The beam energy was calibrated using nuclear resonance reactions of ^{27}Al(p,γ)^{28}Si at 992 keV and ^{19}F(p,γ)^{16}O at 872 keV. After calibration, the beam energy had a precision better than ±6 keV and energy spread of around 1 keV. This measurement was done by employing two detectors simultaneously, Au/Si surface barrier detector was placed at the laboratory angle of 165° and subtended a solid angle of 1.83×10^{-3} sr, defined by a 3 × 4 mm slit. The ERDA detector was fixed at the laboratory

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recoil angle of 30° and it subtended a solid angle of 1.12×10^{-3} sr. The angular resolution of both detectors was <1°. The beam was incident at 15° from the sample surface. In order to stop all the scattered He ions, Mylar foils with different thickness between 13 and 38 μm were selectively placed before the elastic recoil detection analysis (ERDA) detector. The energy resolution of this detection system was about 18 keV. The beam current should be kept sufficiently low to ensure that the dead time of the detector system was minimized.

In this measurement, an energy step in the helium ion beam energy of 100 keV was taken in the range of 2.6–7.4 MeV. The accumulated charge per energy interval was usually ~20 μC. The content of the D in the sample was stable under the helium bombardment and its loss was less than 0.2% compared to the D in the sample.

2.2. Sample preparation

The sample Ta/TiD_x/Si used in this experiment was prepared by a method named DC magnetron sputtering. Firstly, a uniform film of TiD_x with 2.8×10^{17} atoms/cm^2 Ti atoms and 4.7×10^{17} atoms/cm^2 D atoms was deposited on Si substrate by reactive sputtering in an Ar/D gas mixture. The deuterium content in the Ti film can be controlled by adjusting the ratio of the deuterium and argon fluxes i.e. Q_D/Q_{Ar} in sputtering. The D/Ar ratio of the sputter gas was $Q_D/Q_{Ar} = 8/2$ (sccm) and the pressure was 0.6 Pa. The deposition was performed near room temperature, and the maximum elevated temperature for the holder is lower than 70 °C. During the deposition, the sputtering current was about 0.24 A and the concurrent discharge voltage was 380 V. Following this, a Ta overlayer of 2.6×10^{16} atoms/cm^2 (about 5 nm in thickness) was deposited on the TiD_x film. The thin Ta layer was used to avoid the oxidation of TiD_x film and served as an internal ion dose reference in the cross section calculation. It can also prevent D atoms from coming out of the sample during the ion bombardment [9].

The concentration of deuterium in the sample was measured by ERD using a beam of 5.6 MeV ^{16}O ions (after subtracting the energy loss of the O ions in the Ta and TiD_x films). With this energy, Rutherford cross-section of $^{16}O(D,D)^{16}O$ can be used to determine the concentration of deuterium in the sample [10]. The recoil angle φ_1 and incident energy $E_{1.1}$ are relate to the scatting angle θ_2 and incident energy $E_{1.2}$ in the inversion by Eqs. (1) and (2) as below [11,12]:

$$\tan\theta_2 = \frac{\sin 2\varphi_1}{\frac{M_2}{M_1} - \cos 2\varphi_1} \tag{1}$$

$$E_{1.2} = \frac{M_2}{M_1} E_{1.1} \tag{2}$$

where the $E_{1.2}$ is the energy of incident ions and θ_2 is the scatting angle in $^{16}O(D,D)^{16}O$ case. The atomic ratio of D to Ti in the film is above 160%. There is also 2 at.% H in the TiD_x sample by residual gas in the process of deposition. Fig. 1 shows a typical RBS spectrum of the sample and SIMNRA program [13] can determine the areal density of the TiD_x and Ta layer. And Fig. 2 is an ERD spectra of the sample by incident particle of 2.6 MeV ^4He. The visible peak in Fig. 2 represents 8 at.% H in the surface which is considered as the absorption of H_2O on the surface.

3. Measurement and analysis

In this measurement, 5.6 MeV ^{16}O was used to determine the areal density of deuterium in the sample. It was done by two detectors simultaneously with the recoiling angle of 30°. As the indication about Rutherford scattering in [14], the closest approach of

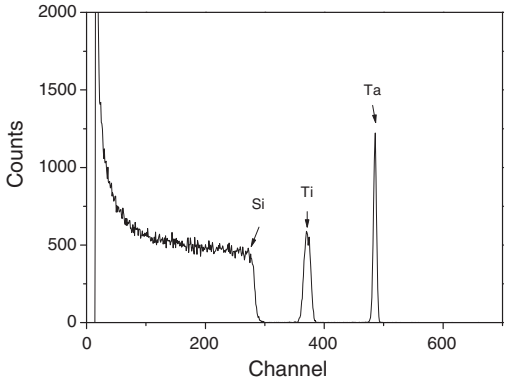

Fig. 1. The RBS spectrum of the Ta/TiD_x/Si sample by 2.0 MeV ^4He ions.

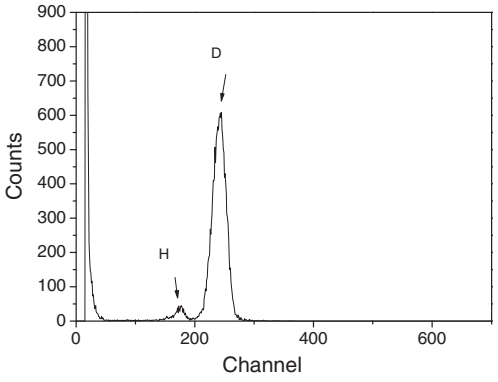

Fig. 2. The ERD spectrum of the sample by 2.6 MeV ^4He ions.

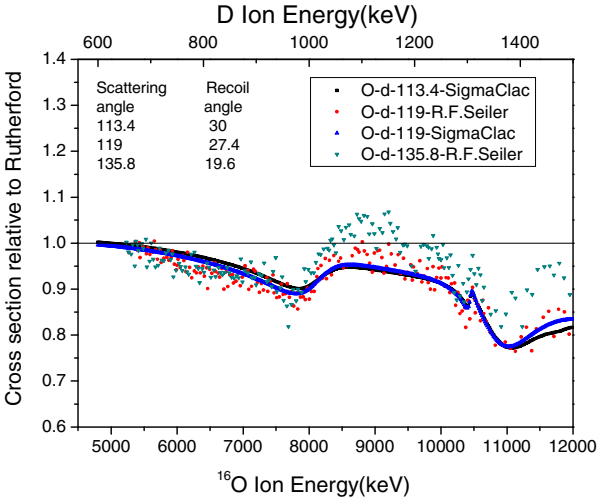

Fig. 3. Measured cross-section divided by the Rutherford cross-section at different scattering angles, obtained from previous data.

two atoms is larger than 3–4 times the sum $D_0/2$ of the nuclear radii. Basing on the formula in [15], the energy of the 5.6 MeV ^{16}O utilized for the ERD analysis was quite below that of the non-Rutherford threshold theoretically. In order to find the energy region for the Rutherford scattering in experiment, the literature values of σ/σ_{Ruth} (the ratio of the scattering cross section to the Rutherford cross section) for deuterium ions scattering from the oxygen at the angles of 113–136°, are shown in Fig. 3. The corresponding recoil angles of 30–19° were also calculated out by Eqs.