

Tritium and helium analyses in thin films by enhanced proton backscattering*

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Abstract: In order to study how to reliably perform quantitative tritium and helium analyses in thin film samples using enhanced proton backscattering (EPBS), several EPBS spectra for some samples consisting of non-RBS light elements (i.e., T, ^4He , ^{12}C , ^{16}O , $^{\text{nat}}\text{Si}$), medium and heavy elements have been measured and analyzed using analytical SIMNRA and Monte Carlo-based CORTEO codes. The non-RBS cross sections needed in the CORTEO code are taken from the ENDF/B-VII.1 database and the calculations of SigmaCalc code and are incorporated into the CORTEO code. All non-RBS cross section data over the entire proton incident energy-scattering angle plane are obtained by interpolation. It is quantitatively observed that in EPBS analysis the multiple and plural scattering effects have little impact on the energy spectra for light elements and the RBS cross sections of light elements can be used in the SIMNRA code for dual scattering calculations. It is also observed that the results given by the CORTEO code are higher than the results of the SIMNRA code in the low energy part of EPBS spectra, and are in better agreement with the experimental data. Tritium and helium analyses in thin film samples using EPBS can be performed reliably when the multiple and plural scattering contributions are completely accounted.

Key words: EPBS, Monte Carlo method, tritium analysis, multiple scattering, plural scattering

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1 Introduction

Measurements of tritium and helium in materials play an important role in nuclear energy research and in applications of nuclear technology, for example, in the analyses of the first wall materials used in fusion reactors [1] and of the tritium-containing targets used in neutron generators [2]. Among the various analysis techniques developed for measuring tritium and helium in materials, the ion beam analysis (IBA) techniques, including nuclear reaction analysis (NRA) [3, 4], elastic recoil detection analysis (ERDA) [5, 6] and enhanced proton backscattering (EPBS) [7, 8] have been developed for many years and can provide information on tritium and helium concentration and depth distribution in materials in an almost nondestructive manner. In recent years, based on the work of Matsuyama, et al. [9], we have tried to develop the β -decay induced X-ray spectroscopy (BIXS) into a routine, accurate and in situ tritium analysis method for tritium-containing films by incorporat-

ing Monte Carlo simulation and Tikhonov regularization for dealing with the ill-posed inverse problems involved in the BIXS method [10–15]. We have previously employed the BIXS method to analyze tritium concentrations and depth distributions in tritium-containing Ti films with Mo substrate, and found that the total tritium concentrations obtained by the BIXS method were in good agreement with the results given by the PVT method [13]. Meanwhile, we also carried out the EPBS analyses for tritium-containing Ti film samples and intended to examine whether the tritium depth distributions and concentrations given by the EPBS analysis are consistent with those given by the BIXS method. However, we found that, in the low-energy part where the signals from tritium appear, the EPBS experimental spectra for tritium-containing Ti film samples with Mo substrate cannot be fitted well using the SIMNRA code [16], even including multiple and dual scattering in the fitting. A similar situation also occurred in EPBS analyses for helium-containing Ti film samples with Mo

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substrate. We examined several possible reasons for this situation [17], including 1) the energy deposits in the Au(Si) surface barrier detector (used to detect backscattered protons) due to the impact from tritium β -decay electrons, 2) the energy deposits in the Au(Si) detector due to the impact from neutrons which are produced in the reaction $T(p,n)^3\text{He}$ when the incident energy of the proton is larger than the threshold energy, 1.02 MeV, and 3) possible inaccuracy of non-Rutherford backscattering (non-RBS) cross sections of $T(p, p)T$. We observed that these possible reasons can not explain the disagreement between the EPBS experimental spectra and the fitting spectra given by SIMNRA code at the low-energy part for tritium analysis. Moreover, we noticed that the SIMNRA code utilizes Rutherford backscattering (RBS) cross sections, instead of non-RBS cross sections which are usually one to three orders of magnitude larger than the RBS cross sections (in particular for tritium, the ratio of non-RBS cross sections to RBS cross sections can be ~ 1000 at ~ 3.5 MeV [7]), to calculate the dual scattering contributions for non-RBS light elements (e.g., T, ^4He , ^{12}C , ^{14}N , ^{16}O , $^{\text{nat}}\text{Si}$ and so on). For small angle scattering, non-RBS cross sections tend to be equal to RBS cross sections, therefore whether non-RBS cross sections or RBS cross sections are used is not important in this case. However, for large angle plural scattering, non-RBS cross sections possibly play an important role, and the SIMNRA code (Version 6.06, the newest version now) also warns users that inaccurate results will possibly be given when EPBS spectra for non-RBS light elements are analyzed by using RBS cross sections for calculating the dual scattering contributions. Therefore, whether or not RBS cross sections, rather than non-RBS cross sections, can be used to calculate the large angle plural scattering contributions for non-RBS light elements still needs quantitative verification. On the other hand, the SIMNRA code also neglects the higher order large angle scattering contributions with more than two scattering events. Barradas [18] also pointed out some causes in the RBS analytical model (as opposed to Monte Carlo method) for the disagreement between experiments and calculation results from analytical models at the low-energy part of RBS spectra, and developed an approximate analytical method to deal with this issue. Because Monte Carlo methods can result in a very realistic simulation of the RBS spectrum and because we need quantitative analyses for tritium and helium in thin films, we therefore, in this paper, employ a Monte Carlo method for EPBS spectrum analysis (i.e., the CORTEO code [19]), based on the non-RBS cross sections of proton backscattering from tritium and helium, to examine whether the disagreement between the EPBS experimental spectra and the fitting spectra given by SIMNRA code at the low-energy part can be quanti-

tatively and accurately explained. In addition, in order to further investigate the effect of RBS cross sections being used instead of non-RBS cross sections to calculate the dual scattering contributions by the SIMNRA code when non-RBS elements exist in samples, we also analyze some other EPBS experimental spectra of samples which consist of non-RBS elements, e.g., SiO_2 and SiC . Moreover, tungsten is the most important candidate for the first wall materials of fusion reactors. Retention of tritium and helium will possibly happen when tungsten is used as the first wall material in a fusion reactor and this needs to be analyzed, so we also analyze the EPBS experimental spectrum of tungsten in this paper.

This paper is organized as follows. Section 2 describes the sample preparation and the EPBS experiment. Section 3 introduces the codes used in this paper and code modifications. Section 4 shows the results and discussion. The conclusions are given in Section 5.

2 Experiment

2.1 Sample preparation

The tritium-containing Ti film sample was prepared by first evaporating Ti onto a smooth Mo substrate, and then placing it in a tritium gas to absorb tritium [13]. The tritium content absorbed in the Ti film sample was determined by the pressure change of tritium gas based on the equation of state of an ideal gas, i.e., the PVT method. The thickness of Ti film was about $5\ \mu\text{m}$, the thickness of Mo substrate was about 1 mm, and the T/Ti ratio in the Ti film measured by the PVT method was about 1.51. The tritium depth distribution in this sample prepared by the processes described above should usually be uniform, or else the tritium concentration will decrease as the depth increases. The helium-containing Ti film sample was fabricated by depositing Ti onto smooth Mo or Si substrate using magnetron co-sputtering in a gas mixture of argon and helium. The details for sample fabrication are given in Ref. [20]. The thickness of Ti film for this sample was about $1.5\ \mu\text{m}$, the thickness of Mo or Si substrate was about 1 mm, and the He/Ti ratio in the Ti film measured by the EPBS method was about 0.60. The helium depth distribution in this sample should usually be uniform [20]. SiO_2 , SiC and W samples were of high purity ($>99.9\%$) and about 1 mm thick, and the sample surfaces were polished.

2.2 EPBS experiments

The EPBS experiments were performed at the 2.5 MeV Van de Graaff accelerator at the Institute of Nuclear Science and Technology of Sichuan University. The incident proton energy was about 2 MeV, the beam spot size was about 2 mm in diameter and the direction of incident proton beam was vertical to the sample surf-

ace. A semiconductor Au(Si) surface barrier detector with a depletion depth of 100 μm was placed in the target chamber at an angle of 160° or 165° with respect to the incident proton beam direction to record the backscattered protons. The intensity of the incident proton beam was adjusted to keep the dead time correction less than 2%.

3 Codes and modifications

Several software packages, analytical or Monte Carlo-based, have been developed for many years to perform NRA, ERDA, RBS and EPBS analyses. The statuses of these codes are reviewed in Ref. [21]. Comparisons between some codes have already been extensively made with respect to many aspects [22], and their advantages and weaknesses have been discussed [23]. In this paper, analytical SIMNRA code and Monte Carlo-based CORTEO code are utilized.

3.1 The SIMNRA code

The SIMNRA code [16] is a widely used Microsoft Windows program with full graphical user interface for the simulation of back or forward scattering spectra for IBA techniques, e.g., NRA, ERDA, RBS and non-RBS. About several hundred different non-Rutherford and nuclear reaction cross sections for incident protons, deuterons and He-ions are included. New cross section data can be added by users in R33 file format, for example, from the IBANDL database [24] or from theoretical calculations by SigmaCalc code developed by Gurbich [25, 26]. Correction factors by L'Ecuyer or by Andersen can be applied in SIMNRA code due to partial screening of nuclear charges by the electron shells surrounding nuclei. SIMNRA code can use several different sets of stopping power data for the stopping of light and heavy ions in all elements, e.g., Andersen-Ziegler stopping, Ziegler-Biersack stopping, KKK stopping and SRIM stopping, and it can also use stopping power data that users have defined. Bohr's model, Chu's model or Yang's model for energy loss straggling can be used in the SIMNRA code. This code can treat the surface roughness of a sample for two cases, i.e., rough film on a smooth substrate and smooth film on a rough substrate. The SIMNRA code can also take into account multiple (small angle) scattering and dual (large angle) scattering as an approximate calculation of plural scattering. However, the SIMNRA code uses Rutherford scattering cross sections for the dual scattering calculation when non-RBS elements exist in samples; this may result in inaccurate results.

3.2 The CORTEO code

The CORTEO code is a Monte Carlo-based program that simulates ion beam analysis spectra, i.e., RBS and

ERDA, and is freely available with its source code under the terms of the GNU General Public License. By simulating the trajectory of each ion, it can take into account more naturally and accurately some effects such as multiple and plural scattering. Some improvements have been made so that the simulations can be achieved with sufficient statistics on a personal computer in a reasonable amount of time [19]. Correction factors by Andersen for RBS cross sections due to partial screening of nuclear charges by the electron shells can be used in the CORTEO code. Stopping power data are obtained from SRIM's SRModule. Bohr's model, Chu's model or Yang's model for energy loss straggling can be used in this code.

Monte Carlo analysis for EPBS spectra needs non-RBS cross sections of proton scattering over the entire proton incident energy-scattering angle plane in question. Therefore, for the purpose of this paper, we have modified the CORTEO code and incorporated some non-RBS cross sections of proton scattering from T, ^4He , ^{12}C , ^{14}N , ^{16}O and $^{\text{nat}}\text{Si}$ elements into this code. The cross sections of EPBS for tritium are taken from the ENDF/B-VII.1 database, which are based on *R*-matrix analysis [27]. The cross sections of EPBS for ^4He , ^{12}C , ^{14}N , ^{16}O and $^{\text{nat}}\text{Si}$ elements are taken from the calculations of SigmaCalc code [25, 26]. These cross sections are taken at certain grids of incident proton energies and scattering angles. All cross sections needed in the Monte Carlo analysis are obtained by interpolation. In particular, the ratios of these non-RBS to corresponding RBS cross sections at the scattering angle of 0° or below the energies determined by Bozoian's formulae [28] are set to be one. The cross section data at 165° for tritium from the ENDF/B-VII.1 database are also compared with the available experimental data [7], and they are in good agreement [17].

4 Results and discussion

In this section, we utilize SIMNRA and modified CORTEO codes to analyze the EPBS experimental spectra we obtained. For comparability, the calculations based on these two codes are performed under the same conditions, i.e., the experimental setup and target structure used in these two codes are the same, and the correction factor by Andersen, SRIM stopping power and Yang's model for energy loss straggling are used in these two codes, as well as the EPBS cross sections described in Section 3.2. The cone angles used in the CORTEO code are determined according to the method given in its users' manual [19]. In addition, the simulated spectra given by the SIMNRA code in the following discussion are the results calculated with multiple and dual scattering and the default cutoff energy (i.e., 10 keV). The SIMNRA code can only use Rutherford backscattering cross

sections, instead of non-RBS cross sections, to calculate the dual scattering contributions for non-RBS light elements.

Figures 1 and 2 show the measured and simulated spectra for 2 MeV protons backscattered from SiO_2 and SiC samples respectively at a scattering angle of 160° . We have compared the simulated spectra given by the SIMNRA code with and without multiple and dual scattering, although the dual scattering contributions are calculated with RBS cross sections instead of non-RBS cross sections, and found that they are almost the same. As can be seen for the SiO_2 sample in Fig. 1, the result calculated with the SIMNRA code is, overall, in good agreement with the experimental data and also in good agreement with the result given by the CORTEO code except in the very low energy part, where the result given by the CORTEO code is higher than the result of the SIMNRA code and is closer to the experimental data. For the SiC sample, in Fig. 2 the result calculated with the SIMNRA code is also in good agreement overall with the experimental data as well as the result given by the CORTEO code except around the carbon resonance peak where the results given by the SIMNRA and CORTEO codes are higher than the experimental data. We think that this may be caused by the simulation not considering the surface roughness and the possible inaccuracy of cross sections used here around the carbon resonance. On the other hand, the results given by these two codes are also somewhat different around the carbon resonance, this difference may be due to the different treatments of the cross section and straggling, which have been discussed in detail in Section 3.4 of Ref. [22]. The difference

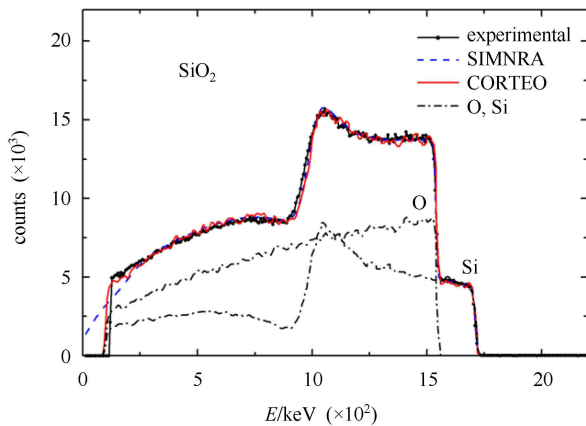


Fig. 1. (color online) Comparison of the experimental and simulated energy spectra with the SIMNRA and CORTEO codes for 2 MeV protons backscattered from thick SiO_2 sample at a scattering angle of 160° . The simulated spectrum given by the SIMNRA code is calculated with multiple and dual scattering. Individual elemental spectra calculated from the CORTEO code are also shown.

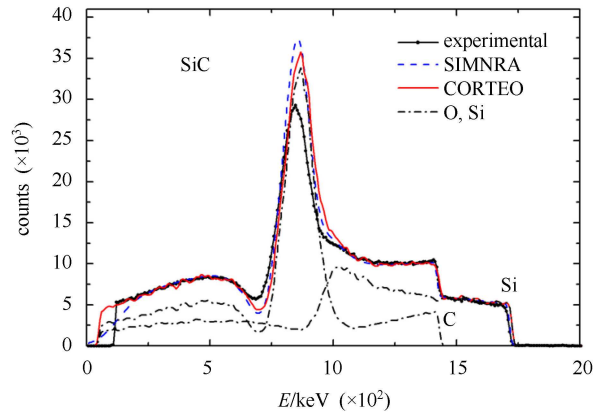


Fig. 2. (color online) The same as Fig. 1 but for 2 MeV protons backscattered from the thick SiC sample.

between the experiment and the simulations at the very low energy part may be due to the fact that the SIMNRA code neglects the higher order large angle scattering contributions with more than two scattering events, whereas the CORTEO code takes into account all scattering events.

The above results also indicate that the multiple and plural scattering effects have little impact on the energy spectrum for light elements like C, O and Si except for the very low energy part. Therefore RBS cross sections for EPBS analysis of light elements, instead of non-RBS cross sections, are acceptable in the SIMNRA code for dual scattering calculations. This result can be approximately understood as follows: when a proton collides with a light nucleus, from the two-body kinematics we know that the incident proton will transfer a larger energy to the target nucleus in comparison with the case of a proton colliding with a heavier nucleus. Therefore, the energy of the backscattered proton decreases rapidly when a proton collides with a light nucleus. Moreover, we also know that the non-RBS cross sections will approach to the RBS cross sections when a lower energy proton collides with a light nucleus because the lower energy proton cannot yet penetrate the Coulomb potential barrier of the light nucleus. The RBS cross sections are proportional to Z^2 (Z is the atomic number of the target atom) and hence the RBS cross sections are very small for the collision of a proton with a light nucleus. Therefore, due to the fact that the higher order (≥ 2) large angle scattering when a proton collides with a light nucleus may be a Rutherford scattering with a very small scattering cross section, we can know that the probability for plural scattering will become very small for light elements even when the non-RBS cross sections are large.

Figures 3 and 4 show the measured and simulated spectra for 2 MeV protons backscattered from the helium-containing Ti film samples with smooth Si and Mo substrates respectively at a scattering angle of 160° .

The RBS cross sections for He and Si elements are used in the SIMNRA code for dual scattering calculations. The helium depth distributions can be reasonably assumed to be uniform. From Fig. 3 and Fig. 4, we can see that the results obtained from the SIMNRA code are, overall, in good agreement with both the experimental data and the results given by the CORTEO code, except for the low energy part, where the results given by the CORTEO code are higher than the results of the SIMNRA code and are closer to the experimental data. We notice that the differences among the results of the SIMNRA and CORTEO codes and experimental data originate from heavier substrate elements, e.g., Mo, and the heavier the substrate elements are, the larger the differences are. This difference may also be due to the fact that the CORTEO code takes into account all scattering events. However, for these two samples, the helium analyses have not yet

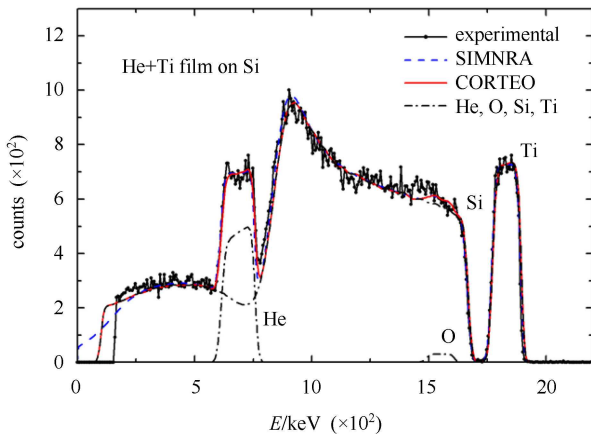


Fig. 3. (color online) The same as Fig. 1 but for 2 MeV protons backscattered from the He-containing Ti film sample with smooth thick Si substrate.

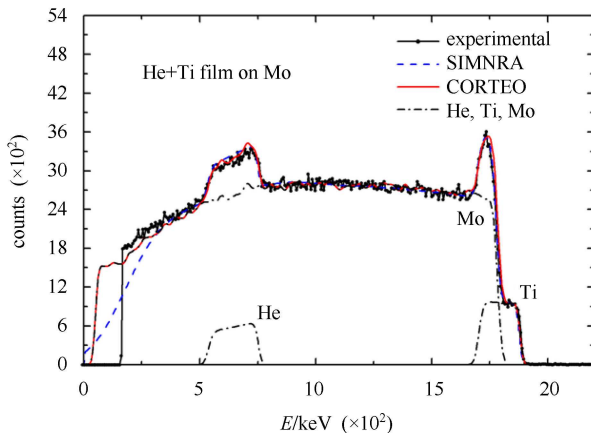


Fig. 4. (color online) The same as Fig. 1 but for 2 MeV protons backscattered from the He-containing Ti film sample with smooth thick Mo substrate.

been affected. In addition, the results shown in Fig. 3 and Fig. 4 also indicate, as in Fig. 1 and Fig. 2, that the RBS cross sections for EPBS analysis of light elements, instead of the non-RBS cross sections, can be used in the SIMNRA code for dual scattering calculations.

In Fig. 5 the measured and simulated spectra for 2 MeV protons backscattered from the tritium-containing Ti film sample with smooth Mo substrate at a scattering angle of 165° are presented. The RBS cross sections for tritium are used in the SIMNRA code for dual scattering calculations. The tritium depth distribution is reasonably assumed to be uniform. From Fig. 5, we can observe that the result obtained from the SIMNRA code overall is in good agreement with the experimental data and also in good agreement with the result given by the CORTEO code, except around the low energy spectrum where the signals from tritium appear. At this low energy part the result given by the CORTEO code is higher than the result of the SIMNRA code and is closer to the experimental data. This comparison indicates that the difference between the result obtained from the SIMNRA code and the experimental data should not be solved simply by adjusting the tritium depth distribution, or else inaccurate results for tritium analysis may be obtained. In Fig. 5, we also show the comparison of individual spectra of tritium from the CORTEO code and from the SIMNRA code with single scattering model, we can see that they are almost the same. This further indicates that the multiple and plural scattering contributions from light elements, e.g., tritium, are not important even when the non-RBS cross sections are much larger than the corresponding RBS cross sections and the differences between the results of the SIMNRA and CORTEO codes and the experimental data are mainly due to the multiple and plural scattering contributions from the heavier substrate element, e.g., Mo.

Finally, the measured and simulated spectra for 2 MeV protons backscattered from W are shown in Fig. 6. We can see that at the low energy part of the spectrum, neither the CORTEO nor the SIMNRA code can give satisfactory agreement with the experimental result. In fact, in Fig. 5, although the result from the CORTEO code is better than that from the SIMNRA code in the low energy part when comparing with the experimental data, the result from the CORTEO code also needs to be improved. This disagreement may be caused by approximate algorithms in the codes. For example, although in the CORTEO code some improvements have been made in order that the computing time can be decreased by several orders of magnitude, at the same time these improvements also introduce some problems, which have been pointed out in Ref. [23]. Therefore, in Figs. 5 and 6, we also show the results of full Monte Carlo RBS

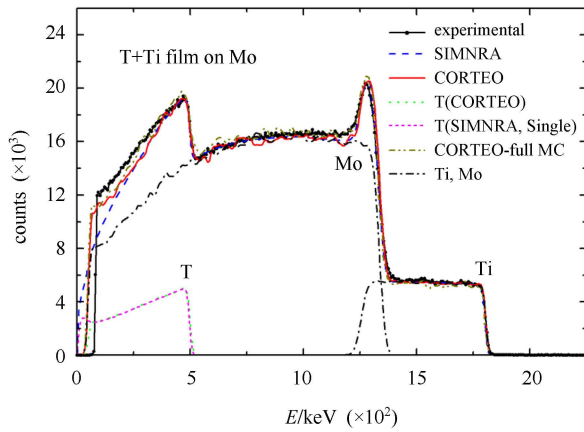


Fig. 5. (color online) Comparison of the experimental and simulated energy spectra with the SIMNRA and CORTEO codes for 2 MeV protons backscattered from the T-containing Ti film sample with smooth thick Mo substrate at a scattering angle of 165° . The total simulated spectrum given by the SIMNRA code is calculated with multiple and dual scattering, the individual spectra of tritium from the CORTEO code and from the SIMNRA code with single scattering model are shown, and other individual elemental spectra calculated from the CORTEO code are also shown. A full Monte Carlo calculation is also shown, which is the sum of the full RBS calculations for Ti and Mo and the RBS calculation for T using the CORTEO code.

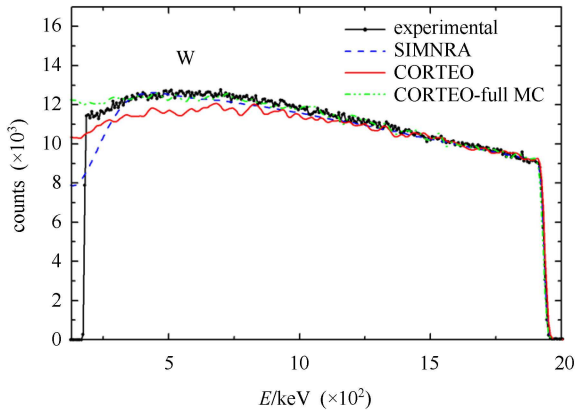


Fig. 6. (color online) The same as Fig. 1 but for 2 MeV protons backscattered from smooth thick W sample. A full Monte Carlo calculation using the CORTEO code is also shown.

calculations (by using the CORTEO code on a computer cluster) for Ti, Mo and W. We can see that the full Monte Carlo results improve the agreement with the experimental spectra in the low energy part, and hence under these circumstances where the multiple and plural scattering

contributions are sufficiently accounted for, the quantitative tritium and helium analyses in thin film samples can be performed reliably by using enhanced proton backscattering. Although a full Monte Carlo calculation requires long computing time for practical applications, an analytical model simulation can be first performed and then followed by a full Monte Carlo calculation when necessary, for example, for our cases of quantitative tritium and helium analyses. In addition, some other causes, for example, slit scattering, low-energy component in the beam, inaccurate physical data (cross section, stopping power and so on) or unaccounted physical phenomenon, may also contribute to the disagreement between experiments and calculations in the low energy part of RBS spectra, some of which have been discussed in Refs. [22, 23, 29, 30]. For our cases, we observe that different stopping powers, provided by the SIMNRA code, can lead to apparent differences for a thick W target at the low energy part of the EPBS spectrum, while for a thick Mo target the calculation results are relatively stable and the differences are smaller for different stopping powers.

5 Conclusions

EPBS spectra for several samples consisting of non-RBS light elements, medium and heavy elements have been measured and analyzed using analytical SIMNRA and Monte Carlo-based CORTEO codes. The CORTEO code is modified and some non-RBS cross sections of proton scattering from T, ^4He , ^{12}C , ^{14}N , ^{16}O and $^{\text{nat}}\text{Si}$ elements are incorporated. We quantitatively observe that the multiple and plural scattering effects have little impact on the energy spectra for light elements like T, He, C, O and Si, and the RBS cross sections of light elements, instead of the non-RBS cross sections, can be used in the SIMNRA code for dual scattering calculations for EPBS analysis even when the non-RBS cross sections are much larger than the corresponding RBS cross sections. We also observe that in the low energy part, the results given by the CORTEO code are higher than the results of the SIMNRA code and are closer to the experimental data, especially when heavier elements exist in samples. This may be due to the fact that the SIMNRA code neglects the higher order large angle scattering contributions with more than two scattering events whereas the CORTEO code takes into account all scattering events. For tritium analysis, the tritium depth distributions should not simply be adjusted to fit the experimental spectra when the multiple and plural scattering contributions are not completely accounted for, or else inaccurate results may be obtained. For medium and heavy matrix elements, when full Monte

Carlo RBS calculations are used in the CORTEO code, the results from the CORTEO code are in good agreement with the experimental results in the low energy part of the EPBS spectra. Now, quantitative tritium and helium analyses in thin film sample can be performed reliably using EPBS. For practical applications, an analytical model simulation can first be performed

and then followed by a (full) Monte Carlo calculation when necessary.

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