ELASTIC RECOIL DETECTION ANALYSIS OF LIGHT ELEMENTS IN THIN FILMS USING 35 MeV $^{35}Cl^{6+}$ BEAM

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ABSTRACT

In this paper, an elastic recoil detection analysis method is described using 35 MeV ^{35}Cl as incident ions. This method can determine and profile simultaneously H, D, He, C and O or in the other case, H, C, N and O. The depth resolution for the elements heavier than He is better than 20 nm. It has been applied to study the Co/Si and TiN thin films, and the depth profiles of He implanted in monocrystal silicon.

Keywords: Elastic recoil detection analysis Depth Resolution Mass resolution He implantation profile Co/Si and TiN thin films

1 INTRODUCTION

Elastic recoil detection analysis (ERDA), suggested by L'Ecuyer *et al*[1] fifteen years ago, is an ion beam analysis technique, in which an energetic heavy ion beam is used and the recoil light atoms are detected in the forward direction. By unfolding the energy spectra of these recoil atoms, the sample's atomic composition and its impurity concentration profiles in the near-surface region can be obtained. ERDA is one of the most suitable methods to determine simultaneously the depth profiles of several light elements in thin films[2]. This method is very important in material researches because of the dramatic effects of light elements, such as hydrogen, helium, carbon, notrogen, oxygen *etc.*, on physical, mechanical, chemical and electronic properties of materials.[3-6] Although the use of ERDA to profile H and He is becoming increasingly common[7-10], there are still some remainder problems which must be experimentally investigated in order to put this method to practical use, especially when the element analyzed is heavier than He.

In this work, 35 MeV $^{35}Cl^{6+}$ was used as incident ion beam. By a careful choice of the absorber thickness, in one case, we can determine simultaneously the depth profiles of H, D, He, C and O; and in the other case, those of H, C, N and O. In this paper, we discuss the influence of experimental conditions on depth resolution and

mass resolution of ERDA with 35 MeV ^{35}Cl beam and some applications of this method in semiconductors. We also developed a data analysis method for ERDA.

2 EXPERIMENTAL AND DATA ANALYSIS

2.1 Experimental arrangement

The ERDA experiment was carried out with 35 MeV $^{35}Cl^{6+}$ ion beams from EN-18 2×6 MV Tandem at the Institute of Heavy Ion Physics of Peking University. The ^{35}Cl incident beam has an angle of 15° with respect to the target surface. The recoil atoms were detected at 30° in the forward direction by a Si(Au) detector with a resolution of 16 keV for 5 MeV α. A Mylar foil of 12.94 μm in thickness was mounted in front of this detector to absorb the ^{35}Cl ions scattered by target, but the light elements up to F will pass through this film without big loss of their energy. The typical beam current of $^{35}Cl^{6+}$ was, measured on target, about 60 nA. The time of measurement for each sample is about 20 min.

2.2 Sample preparation

Commercial polished and etched monocrystal silicon slices were used as substrates. Two types of targets were used:

2.2.1 *Standard sample* 10 keV deuterium, 20 keV oxygen and 30 keV helium were implanted into a monocrystal silicon slice at the same dose of 2×10^{16} atoms/cm^2. This sample was used to scale the energy of ERD spectra and to estimate the mass resolution of our experimental system.

2.2.2 *Samples to be analyzed* Three kinds of samples were analyzed: (a) a film of 56.8 nm Co was deposited on Si wafer by dc-magnetron sputtering at a rate of 4 nm/min. The base pressure was about 6.67×10^{-4} Pa. (b) a TiN thin film prepared by chemical reaction on titanium film deposited on c-Si in nitrogen gas at high temperature. (c) two implanted samples, in which, helium of 4×10^{16} atoms/cm^2 was implanted into monocrystal Si with two different ion energies: 30 keV and 40 keV.

2.3 Data analysis

The scattering process of ERD is similar to that of RBS. However because the recoils will transmit through several μm absorber foil, the data analysis in ERD is more difficult than that in RBS. Considering the general geometry for ERDA shown in Fig.1, If $R_a(E)$ is the range of the recoils of energy E in the absorber foil, $E(x)$ is the energy of the detected recoiling particle, then the energy before the absorber foil is

$$E_2 = R_a^{-1}\,(R_a(E(x)) + t) \qquad (1)$$

where R_a^{-1} is the reverse function of $R_a(E)$, t is the absorber thickness. In Fig.2, a graphic method is illustrated to solve the Eq.(1). So, after this transition, we have the energy spectrum before the absorber foil. To unfold this energy spectrum into depth spectrum, a same method is used as applied in RBS[15], except that the kinematic factor

k_R and the cross section $\sigma(E, \theta)$ are different.

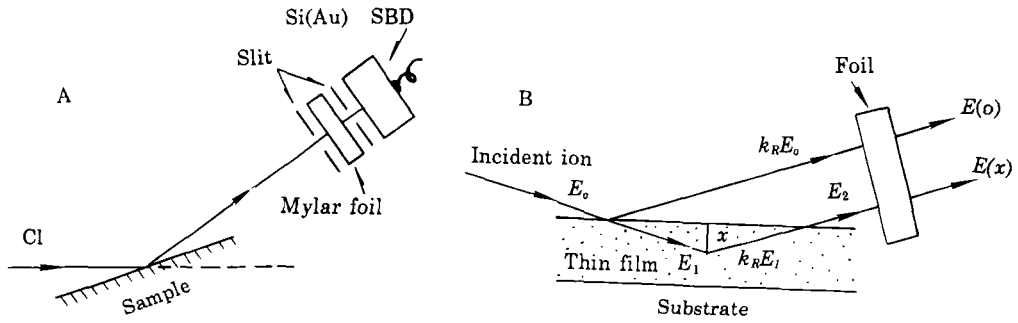


Fig.1 (A) Schematic presentation of the general geometry and (B) the data analysis of ERDA

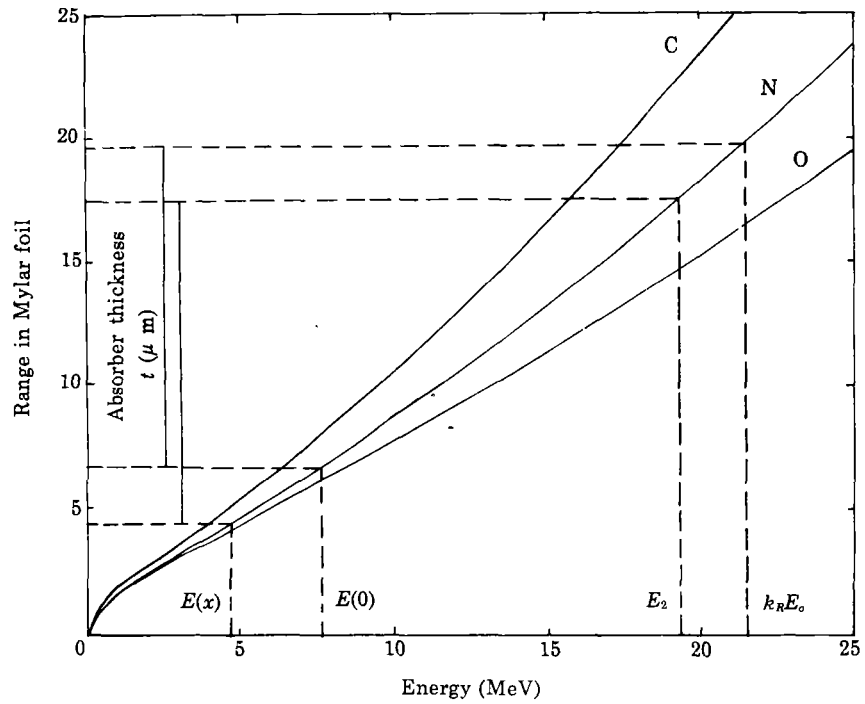


Fig.2 Graphic method to transform the detected energy spectrum into the energy spectrum before absorber foil

3 RESULTS AND DISCUSSION

The energy spectrum of the standard sample is shown in Fig.3. The contributions from the recoiled H, D, He, C and O are separated completely. The separation between oxygen and carbon is about 5 MeV. This means that this experimental system has a good mass resolution and can be used to analyze simultaneously H, D, He, C and O in one sample. The hydrogen and carbon are from the surface contamination of sample.

These two peaks should decrease or disappear if the experiments were carried out in higher vacuum chamber.

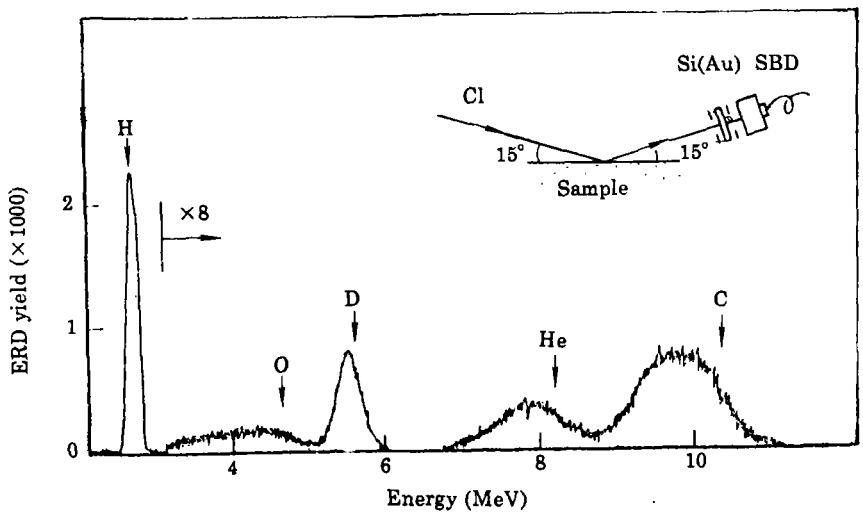


Fig.3 ERDA spectrum of a standard sample (a—Si) containing H and C (on surface), D(10 keV implanted), He(30 keV implanted) and O(20 keV implanted) by using 35 MeV ^{35}Cl

The Mylar absorber thickness is 12.94 μm

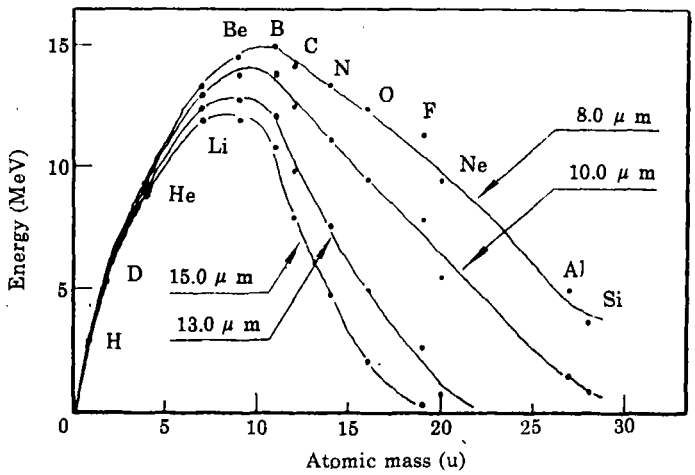


Fig.4 Variation of the energy of the different light elements recoiled by 35 MeV ^{35}Cl at 30° on sample surface as function of the thickness of the Mylar absorber

Mass resolution and depth resolution are the two key factors for the choice of ERDA experimental conditions. A good mass resolution means that each element in a ERDA spectrum is well separated from others. The mass resolution depends on the following factors: (1) mass of incident ions and that of the recoils; (2) beam energy; (3) detection geometry; (4) detector energy resolution and (5) thickness of the absorber foil. Among these, the contribution of the absorber foil is the most important one.

Fig.4 shows the energy of different light elements on surface recoiled by 35 MeV ^{35}Cl at 30° scattering angle after passing through a Mylar foil of different thickness. The energy of recoil elements, except H, D and He, is seriously influenced by the absorber thickness. Thus we must choose this thickness carefully in order to obtain good mass resolution. In our experimental arrangement, a Mylar foil of about 12.9 μm is the best choice. Because the recoil energies of H, D and He are not sensitive to the absorber thickness, it is favorable for us to scale the energy spectrum of ERDA by the surface signals of H, D or He.

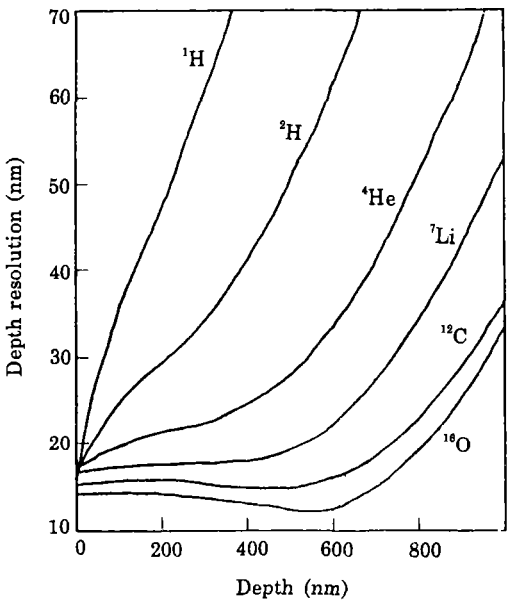


Fig.5 Depth resolution of different recoil elements as function of the analyzed depth in our experimental conditions

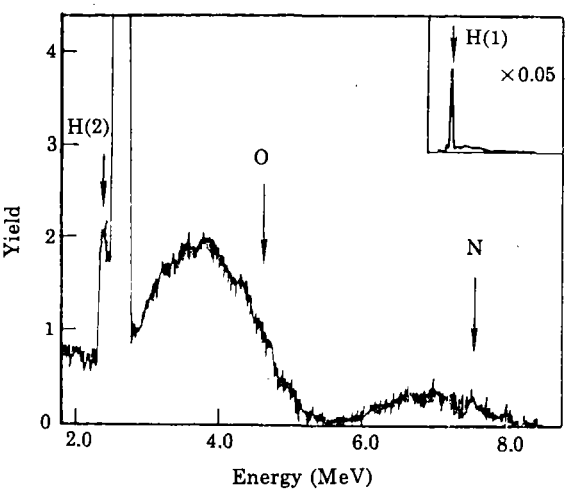


Fig.6 Energy spectrum of the Co/Si sample, showing clearly the existence of a large amount of nitrogen

H(1)—surface hydrogen; H(2)—interfacial hydrogen

The depth resolution of the ERDA experiment has been discussed in detail by many papers.[11,12] It contains the following contributions: (1) energy width of the incident beam; (2) detector energy resolution; (3) geometrical bordening due to the finite detector acceptance angle which causes the kinematic factor variation and the path length difference; (4) energy straggling in target and absorber foil; (5) multiple scattering in target and absorber foil; (6) surface roughness. Fig.5 gives the depth resolution of different recoil elements in Si substrate in our experimental conditions. The depth resolution is better than 20 nm for almost all light elements on surface. However, for H and D, this resolution increases dramatically with increase of depth. But for the light elements heavier than He, it changes slowly and is better than 20 nm in a large range of thickness (>500nm). This means that the ERDA of 35 MeV ^{35}Cl in

our experimental arrangement is good for depth profiling of light elements heavier than He, but not so suitable for H and D.

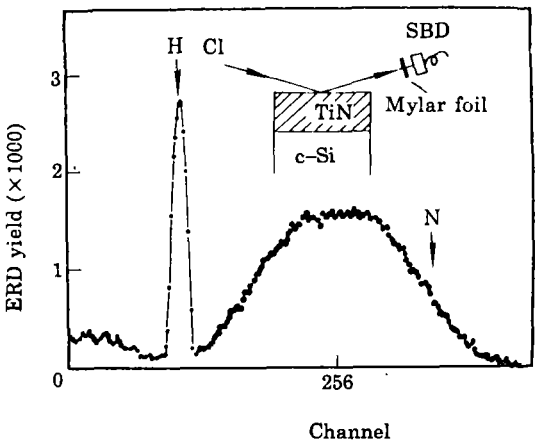
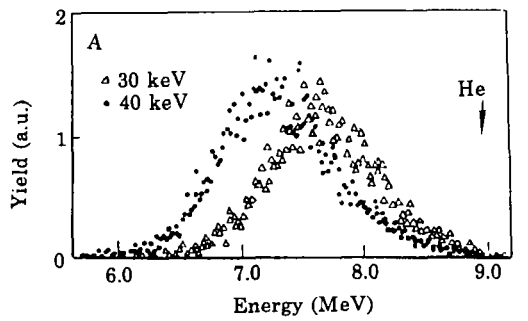


Fig.7 Energy spectrum of TiN/Si thin film

Fig.6 illustrates the recoil spectrum of the Co/Si sample. As expected, it contains a large amount of oxygen and hydrogen. But its nitrogen content is as large as 2.5 $\times 10^{14}$ at/cm^2. It is not clear why so great amount of nitrogen exists in the cobalt film. This result will be discussed in an other paper. As another application of this method, we have studied the TiN/Si sample. It is to note that the nitrogen in TiN is difficult to be determined by Auger electron spectrometry, because of the overlap of N(KLL) and Ti(LMM) transitions[13]. But our results, shown in Fig.7, demonstrated clearly the effectiveness of our method in sudying such film.

The ERDA spectra of 30 keV or 40 keV He implanted into monocrystal Si are illustrated in Fig.8(A). Fig.8(B) displays the depth profiles of He in Si obtained



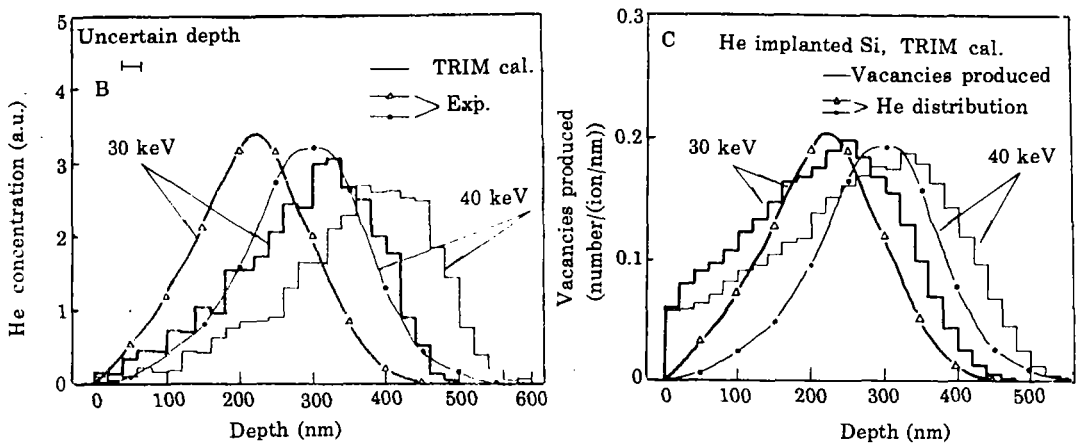


Fig.8 (A) Helium recoil spectra of 30 keV and 40 keV implanted in c—Si, (B) Comparison of the measured profiles with those of TRIM simulations, (C) Comparison of the measured profiles with the TRIM simulated vacancy distributions produced during implantation

from our experiments and those from the TRIM simulation. Fig.8(C) shows the TRIM

calculation of the vacancy distributions produced during He implantations. It is interesting to note that the depth profiles of He obtained from our experiments are about 120 nm shallower than those from TRIM simulation, but they are similar to those of vacancy distributions. It is probably caused by a migration process of He during or after implantation. As we know, helium is a smallest inert element and is practically insoluble in defect-free crystals, its migration energy is very low. So, the interstitial helium is very easy to be trapped by any open-volume defects, especially by vacancy-type defects, or to be released at the external surface[14]. In our sample preparation, the He implantation was carried out in the room temperature and no temperature control system was used. Thus the helium could diffuse towards the vacancies produced by itself. This may explain why the helium depth profiles observed are similar to the vacancy distributions.

4 CONCLUSION

In this paper, we have described an ERDA method using 35 MeV ^{35}Cl as incident ions. With a surface barrier silicon detector, we can analyze simultaneously H, D, He, C and O or in other case, H, C, N and O on target surface and near-surface. The depth resolution for the elements heavier than He is better than 20 nm. As applications, the Co/Si and TiN/Si thin films have been studied. The experimental results clearly show the effectiveness of this method in studying these samples. The depth profiles of He implanted in c-Si measured by this method have a remarkable difference with the TRIM simulation. This is probably due to He migration towards vacancies produced during the He implantation.

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