# EXPERIMENTS OF MeV ION BEAM INDUCED ATOMIC MIXING\*

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(Received November 1989)

#### **ABSTRACT**

An experimental apparatus for studies of MeV ion beam modification of materials has been established on a 3 MV tandem accelerator at Fudan university. A system of X-Y electrostatic scanning implantation of MeV heavy ions and in situ Rutherford-backscattering analysis was included in it. The uniformity of scanning implantation was checked by the RBS measurement of a Si wafer implanted with 1 MeV Au<sup>+</sup> ions. MeV ion beam mixing of Au/Si, Au/Ge and Ag/Si systems was preliminarily studied. The samples were irradiated by certain fluences of 1 MeV Ag<sup>+</sup> ions at room temperature. The mixed layers were analyzed in situ using the glancing RBS technique with 2 MeV <sup>4</sup>He<sup>+</sup> ions. For Au/Si system, a uniformly mixed layer with a defined composition is obtained, and the intermixing is much less for Ag/Si system than for Au/Si system.

Key words: MeV Ag tions Atomic mixing Au/Si, Au/Ge and Ag/Si systems

### 1. INTRODUCTION

Atomic mixing between a film and its substrate, induced by energetic heavy ions, has been widely studied for the interest of the understanding the interaction of ions and solids and the practical applications of surface modification of materials. The most studies of ion beam mixing (IBM) for metal/metal and metal/semiconductor systems up to now were carried out by using a few tens to hundreds of keV heavy ions, especially inert gas ions<sup>[1-3]</sup>. The film thickness of only a few tens of nm was used in most ion beam mixing experiments because of the limitation of ion energies.

Recently, MeV ion beam modification of materials has been developed for technological applications. Some investigations of MeV ion beam mixing have been reported<sup>[4-5]</sup>. The main advantage of MeV ion beam is the larger penetrating power, which allows intermixing at the interface after passing through thicker layers into greater depth in substrates and production of a uniform damage region near the interface. MeV ion beam mixing extends the region of investigations and will provide more information for the understanding the basic phenomena of atomic mixing.

The Project Supported by National Natural Science Foundation of China

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Information for the understanding the basic phenomena of atomic mixing.

Considering the experimental condition of various MeV ions supplied by the new 3 MV tandem accelerator (NEC 9 SDH-2) installed at Fudan university, we set up an apparatus of X-Y scanning implantation of MeV heavy ions and in situ RBS analysis with  ${}^4\mathrm{He}^+$  ions, and start to investigate the MeV ion beam induced atomic mixing. Au/Si, Au/Ge and Ag/Si systems  ${}^{[3.6-8]}$  are chosen in our study. In addition to atomic mixing, adhesion enhancement and high energy implantation are also being studied using this apparatus.

The experimental setup is described and preliminary results of 1 MeV Ag<sup>+</sup> ion beam induced atomic mixing for three systems at room temperature are presented in this paper.

#### II. EXPERIMENTAL

An experimental apparatus for MeV ion irradiation and in situ RBS analysis is schematically shown in Fig.1. Heavy ion beam from  $15^{\circ}$  exit of a magnetic analyzer with a mass energy product 300 was focused by quadruple magnets, and passed through a central collimator  $C_1$  with 6 mm in diameter. The ion beam was swept by an

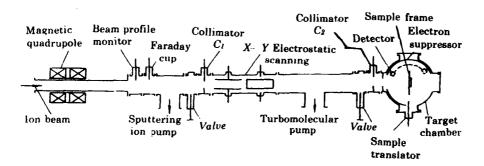


Fig.1 A schematical diagram of the experimental apparatus of MeV ion implantation and in situ RBS analysis

X— Y electrostatic scanning system. The length of deflecting plates was 30 cm and the gap between two plates was 2 cm. Maximum sweeping voltages were  $\pm$  5 kV, and frequencies for X— Y sweeping were 208 and 1723 Hz, respectively. A maximum irradiated spot of about 4 cm  $\times$  4 cm on the sample at a distance of 2 m from the sweeping plates could be obtained for charge state  $q=1^+$  and energy E=3 MeV ions.

Ion irradiation and RBS analysis were performed in the same target chamber with a variable collimator  $C_2$  in front of it. A sample frame was electrically and thermally isolated from the chamber. Samples could be heated to  $300^{\circ}$ C, or cooled by a LN<sub>2</sub> trap located at the top flage of the chamber. The sample was removable and could be tilted to meet the need of glancing RBS analysis. A secondary electron suppression plate

was located around the sample frame. A surface barrier detector with a  $\varphi=3$ mm collimator could be rotated. The detector was hiden behind the sample frame during irradiations and relocated at  $165^{\circ}$  position during RBS analysis. The base pressure in the chamber was better than  $266.66\times10^{-6}$  Pa.

Thin films of Au and Ag were evaporated on Si<111> and Ge<111> single crystal substrates ( $\varphi=25-40$ mm). The Si and Ge substrates were pretreated by cleaning in alcohol and etching in A diluted HNO<sup>3</sup>:HF mixture solution. Film thicknesses of 30-40nm were used in order to compare the results with the experiments already performed by the 100-400 keV ion beam mixing<sup>[3.6-8]</sup>. a thin layer of about 5 nm C was deposited on the surface of the films, to reduce sputtering induced by heavy ion bombardment.

Samples at room temperature were bombarded with 1 MeV Ag<sup>+</sup> ion beam of a current density of  $0.1-0.2\mu$  A/cm<sup>2</sup> over an irradiation spot of about 1 cm<sup>2</sup>. Fluences of  $1.6\times10^{15}$ ,  $3.2\times10^{15}$ ,  $5.2\times10^{15}$  and  $8\times10^{15}$ /cm<sup>2</sup> were used. Then the samples were in situ analyzed by RBS technique with 2 MeV <sup>4</sup>He ions at an incident angle of  $70^{\circ}$  or  $40^{\circ}$  with respect to the normal of the sample.

## III. RESULTS AND DISCUSSION

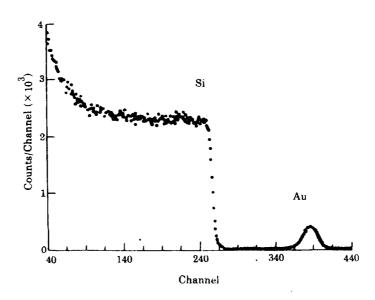


Fig.2 RBS spectrum of <sup>4</sup>He ions backscattered from a Si(100) wafer implanted with 1 MeV Au<sup>+</sup> ions

Prior to ion beam mixing experiments, the uniformity of the ion distribution over the bombarded area was checked along the diagonal of the irradiated square ( $4 \times 4$  cm<sup>2</sup>), using the RBS measurement of Si(100) wafer implanted with 1 MeV Au<sup>+</sup> ions. Fig.2 is a RBS spectrum of <sup>4</sup>He ions backscattered from this implanted sample. The

obtained uniformity over the whole diagonal is within the statistical error of 2% of the Au backscattering counts.

Fig.3a shows the RBS spectra of a Au/Si sample before and after irradiation with 1 MeV  $Ag^+$  ions at a fluence of  $1.6 \times 10^{15}$  ions/cm<sup>2</sup>. and Fig.3b is a part of this RBS spectrum. The thickness of the Au films without  $Ag^+$  irradiation is determined to be 32nm. From the Au peak area of RBS spectra, there is no obvious evidence of any loss of Au atoms during irradiations. This is attributable to the C protection layer for sputtering and the low sputtering rate of MeV ions. The signal of He ions backscattered from implanted Ag ions is located in the region around channel 296 of RBS spectrum, according to the  $Ag^+$  ion range calculated by TRIM<sup>[9]</sup>.

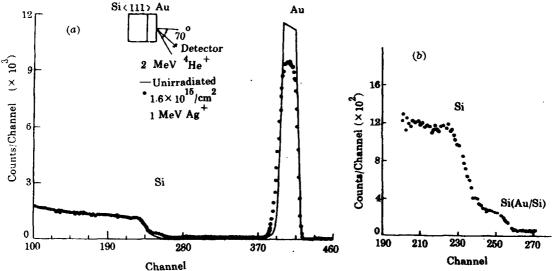


Fig.3 RBS spectra of a Au/Si sample before and after irradiation with 1 MeV  $Ag^+$  ions at room temperature (a) and part of this spectrum (b)

-Unirradiated  $1.6 \times 10^{15}$ /cm<sup>2</sup> 1 MeV  $Ag^+$ 

From Fig.3 (a) and (b), a plateau can been seen in the RBS spectrum under  $1.6 \times 10^{15}$  Ag  $^+$ /cm $^2$  irradiation, which indicates that an intermixing layer was formed near the initial interface between the Au and Si substrate. The RBS spectrum of the sample irradiated by  $3.2 \times 10^{15}$  Ag  $^+$ /cm $^2$  (not shown in Fig.3) is near the same as the spectrum for  $1.6 \times 10^{15}$  Ag  $^+$ /cm $^2$  irradiation. A composition Au<sub>20</sub>Si<sub>31</sub> of the uniformly intermixed layer formed by  $1.6 \times 10^{15}$  Ag  $^+$ /cm $^2$  irradiation can be deduced from the heights of the Au and Si signals in the RBS spectrum [10]. This composition is nearly the same as the Au<sub>71</sub>Si<sub>29</sub> obtained from the ion beam mixing by 300keV Xe  $^+$  of  $9 \times 10^{14}$ /cm $^2$  at room temperature [3]. Because the damage density induced by 1 MeV Ag  $^+$  ions in sample is lower than that induced by Xe ions, it requires a higher fluence to form the completely intermixed layer.

The RBS spectra of a Au/Ge sample before and after irradiations with 1 MeV Ag+

ions of fluences of  $1.6 \times 10^{15}$  and  $3.2 \times 10^{15}$  /cm<sup>2</sup> at room temperature are shown in Fig.4. In measurements of this sample, a glancing angle of 40°, instead of 70°, was used in order to avoid the overlap of Au and Ge signals. After an irradiation with a fluence of  $1.6 \times 10^{15} \text{Ag}^+$  /cm<sup>2</sup>, an intermixing at the interface can be clearly seen. A composition of Au<sub>48</sub>Ge<sub>52</sub> is formed after  $3.2 \times 10^{15}$  Ag<sup>+</sup>/cm<sup>2</sup> irradiation. This composition is close to that obtained in Ref.[3. 6].

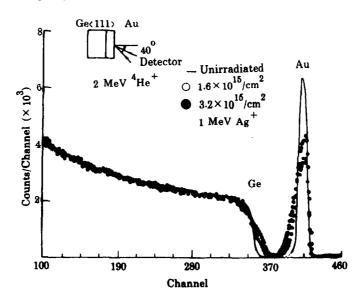


Fig.4 RBS spectra of a Au/Ge sample before and after irradiation with 1 MeV

Ag<sup>+</sup> ions at room temperature

Fig.5 Shows the RBS spectra of a Ag/Si sample before and after irradiations with 1 MeV Ag<sup>+</sup> ions at room temperature. Only a small amount of intermixing occurs at the interface after irradiation of  $3.2 \times 10^{16}$  Ag<sup>+</sup>/cm<sup>2</sup>. There is no apparent change in the width and hight of Ag peak even after irradiation of  $8 \times 10^{16}$  Ag<sup>+</sup>/cm<sup>2</sup>. Previous data<sup>[4]</sup> on Ag/Si mixing at room temperature with 1 MeV Xe<sup>+</sup> ions at a fluence of  $5 \times 10^{16}$  /cm<sup>2</sup> had shown an intermixed layer throughout the entire 40 mm Ag film. However, our Ag/Si data at room temperature show only limited interfacial mixing. This is due to less energy deposited in nuclear collisions for 1 MeV Ag irradiation than for 1 MeV Xe. Although both the Au/Si and Ag/Si belong to the eutectic systems, the mixing for Ag/Si system is more difficult. Chemical driving forces probably play an important role in mixing process<sup>[8]</sup>.

In conclusion, our apparatus of MeV ion implantation with X-Y electrostatic scanning and in situ RBS analysis system now can be supplied to perform the experimental study of MeV ion beam modification of materials. Preliminary measurements of 1 MeV Ag<sup>+</sup> ion beam induced atomic mixing in Au/Si, Au/Ge and Ag/Si systems indicate that for the Au/Si a uniformly mixed layer with a defined

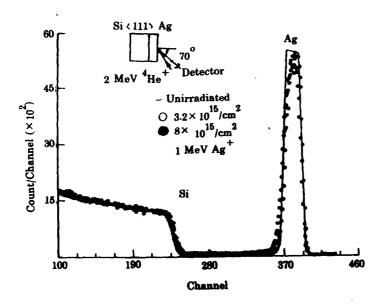


Fig.5 RBS spectra of a Ag/Si sample before and after irradiation with 1 MeV

Ag<sup>+</sup> ions at room temperature

composition is obtained, and the intermixing is much less for the Ag/Si system than for the Au/Si system. In order to provide more information on the dependence of mixing upon the ion species, fluence and temperature, more measurements are necessitated.

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