ChinaRxiv 中X

AI translation · View original & related papers at
chinarxiv.org/items/chinaxiv-202306.00329

Instrumentation and Application of the Ion Beam Analysis Line of the In Situ Ion Beam System (Postprint)

Authors: HUANG Zhi-Hong, ZHANG Zao-Di, WANG Ze-Song, WANG Lang-Ping, De-Jun Fu

Date: 2023-06-18T00:00:00+00:00

Abstract

An ion beam analysis system was established on a 1.7 MV tandem accelerator, enabling Rutherford backscattering (RBS), elastic recoil detection (ERD), nuclear reaction analysis (NRA) and channeling measurements. The system was tested by performing qualitative and quantitative analysis of Si, Ni/Si, BiFeO3:La/Si, MoC/Mo/Si and TiBN/Si samples. RBS of a BiFeO3:La film was used as system calibration. Tested by ion beam channeling, a Si(100) is of good crystallinity (min = 3.01%). For thin film samples, the measured thickness agrees well with simulation results by SIMNRA. In particular, composition of a MoC/Mo/Si and TiBN film samples were analyzed by RBS and non-Rutherford elastic backscattering.

Full Text

Preamble

Instrumentation and Application of the Ion Beam Analysis Line of the In Situ Ion Beam System

Zhi-Hong Huang, Zao-Di Zhang, Ze-Song Wang, Lang-Ping Wang, and De-Jun Fu

1Accelerator Laboratory, Key Laboratory of Artificial Micro- and Nano-Materials of Ministry of Education of China, School of Physics and Technology, Wuhan University, Wuhan 430072, China

2State Key Laboratory of Advanced Welding and Joining, Harbin Institute of Technology, Harbin 150001, China

(Received March 12, 2014; accepted in revised form April 9, 2014; published online February 2, 2015)

An ion beam analysis system was established on a 1.7 MV tandem accelerator, enabling Rutherford backscattering (RBS), elastic recoil detection (ERD), nuclear reaction analysis (NRA), and channeling measurements. The system was tested by performing qualitative and quantitative analysis of Si, Ni/Si, $BiFeO_3$:La/Si, MoC/Mo/Si, and TiBN/Si samples. RBS of a $BiFeO_3$:La film was used as system calibration. Tested by ion beam channeling, a Si(100) wafer exhibits good crystallinity (= 3.01%). For thin film samples, the measured thickness agrees well with simulation results from SIMNRA. In particular, composition of MoC/Mo/Si and TiBN film samples were analyzed by RBS and non-Rutherford elastic backscattering.

Keywords: Ion beam analysis, Channeling, Rutherford backscattering, Non-Rutherford elastic backscattering

DOI: 10.13538/j.1001-8042/nst.26.010202

Introduction

Based on interactions between energetic ions and target materials, ion beam analysis techniques are widely used for characterizing thin films and multilayer structures. These methods provide quantitative information on elemental composition of solids from several atomic layers to micrometer depths beneath the surface, as well as depth profiling of impurities. Consequently, these techniques have attracted considerable attention in the ion beam community, leading to the development and upgrading of numerous ion beam systems. These include conventional ion beam analysis systems, microbeams [?], and combined ion beam facilities such as the 5 MV tandem accelerator interfaced with a 200 MeV proton synchrotron at the Wakasa Wan Energy Research Center [?] and the multi-ion irradiation platform JANNUS (Joint Accelerators for Nano Sciences and Nuclear Simulation) at Saclay, where the first triple beam irradiation using Fe, He, and H ion beams was carried out in 2010 [?].

Ion beam analysis methods are frequently employed due to their efficiency in providing a broad spectrum of qualitative and quantitative information on elemental composition and crystal structure of solids. While Rutherford backscattering (RBS) provides composition and depth profiles of heavy elements in a light matrix, elastic recoil detection analysis (ERDA) uniquely profiles light elements (C, B, Li, H) in a heavy matrix. Nuclear reaction analysis (NRA) provides specific elemental profiling capable of resolving isotopes. Proton induced X-ray emission (PIXE) enables simultaneous detection of elements with intermediate atomic numbers. Ion beam channeling, compared with random ion beam scattering spectroscopy, enables high-precision measurement of crystallinity in single-crystalline wafers or epitaxial layers [?]. Ion beam analysis methods share the common advantage of non-destructiveness, as the corresponding ion-solid interactions do not consume target material since the beam current is at analyt-

ical level (i.e., a few nA). Damage may occur only for sensitive samples through ionization or beam heating, sputtering or nuclear stopping, and in channeling studies of single crystals, through defect production that induces dechanneling [**?**].

The accelerator system at Wuhan University is multifunctional equipment: the 1.7 MV tandem accelerator is interfaced with a 200 kV ion implanter and a transmission electron microscope (TEM). This configuration extends the capability of ion beam techniques to emerging fields of physics and materials science. In the present work, we have designed an RBS/PIXE/C system aimed at characterizing solids with a broad range of elements in the same chamber and enabling in situ measurement of implanted samples in a dual beam chamber. The advantage of low-energy RBS lies in its capability for quantitative analysis of major and minor constituents residing in the first 0.5–2.0 μm of a material, depending on sample structure and composition. Detection limits for some light elements may be further improved using resonant scattering. This paper reports tests of basic system functions based on RBS and channeling, and demonstrates quantitative characterization of elemental profiling, interface analysis, and crystal quality of wafers and thin film materials.

II. System Design

[Figure 1: see original paper] shows a schematic diagram of the in situ ion beam system. Ion beams extracted from the negative ion source by cesium sputtering (SNICS) are transported either to the low-energy cluster ion beam chamber for low-energy ion implantation or to the RBS/PIXE/C chamber after passing through the tandem accelerator transport lines. The dual beam chamber is used for in situ implantation with ions from the tandetron or 200 kV implanter. It will also be possible to conduct in situ ion beam analysis of implanted samples following establishment of the RBS/PIXE/C system in the dual beam chamber. The ion beam analysis line thus consists of the SNICS source, tandetron accelerator, and analysis chamber.

[Figure 2: see original paper] shows a schematic diagram of the measurement system of the ion beam analysis line. Conventional ion beam scattering, recoil detection, and nuclear reaction analysis are performed with Si:Au surface barrier detectors (SBD) connected through a charge-sensitive preamplifier, an amplifier, and a multichannel analyzer (MCA). The total ion dose is recorded by a charge integrator. For channeling measurements, a single channel analyzer (SCA) and linear rate-meter are used. For PIXE measurements, we employ an Ortec SLP-1080 detector with liquid nitrogen cooling and a miniature XR-100CR detector with thermoelectric cooling. The SLP-1080 detector is inserted into the chamber through a cylindrical port, while the XR-100CR detector is placed inside the chamber, and the electronics for RBS measurement can be used for signal amplification and recording.

Ion beams extracted from the SNICS source are focused and deflected

before being accelerated to 1-3 MeV, depending on the energy requirements for different measurements. The accelerated ion beams are focused again by an electrostatic quadrupole lens and deflected into the analysis chamber by a magnetic switcher. Samples are mounted on a $ 70mmcopperholderattachedtoafour − axisgoniometer, whichiscomputer − controlledviaasteppingmotorcontroller.Duringmeasurement, thesteppingmotorcontrollergovernsgoniometer axis).Thez − axisisalignedwiththeincidentionbeamdirection.They − axisisdefinedorthogonaltobooththex − andz − axes, completingaright − handtriad.Sampleorientationisspecifiedbythreeangles— , η, andζ—correspondingtorotationsaboutthex−, y- axes, respectively.Thegoniometerenablessamplestotranslatealongthey − axisorrotateaboutthex−, y−, andz−axes.Thespatialrelationshipbetweenthesampleandincidentionbeamisdes $(\eta^{2} + $^2)^1/^2$ and the azimuthal angle .

In RBS analysis, the ion beam is collimated to 0.3 mm by an aperture slot. The SBD, with an effective area of 50 mm^2, is positioned 10–15 cm from the sample at a backscattering angle of 150°–170° relative to the beam incidence. Signals from the preamplifier and spectroscopic amplifier are sent to the MCA installed in the computer. The current integrator records the charge collected during the experiment.

The RBS/C measurement involves three additional steps. First, signals from the amplifier are sent to an MCA gate interface through a single channel analyzer (SCA), while simultaneously selecting an energy window through coincidence by adjusting the upper and lower level discriminators of the SCA. Next, the gate interface is disconnected and signals from the selected energy range from the SCA are sent to linear rate-meters. Finally, tilt or rotation of the crystal sample relative to the beam incidence is controlled by the stepping motor controller, and the reduction in scattering yield is displayed on the linear rate-meters to determine the channeling effect of the incident beams [?]. Detailed information on determining crystal channels through computerized control programs was described in the literature [?].

III. Analysis and Discussion

A. System Calibration and Determination of Crystal Quality

The RBS system was calibrated by measuring the Au peak position and Si edge of an Au-deposited Si sample. RBS measurement was performed on a $BiFeO_3$:La layer sample. $BiFeO_3$ is a typical multiferroic material with ferroelectric and antiferromagnetic properties, offering broad application prospects in spintronics and multibit memory devices. Doping with rare earth elements may significantly improve its ferroelectric and ferromagnetic properties, making accurate measurement of each element important. A La-doped $BiFeO_3$ thin film was prepared by sol-gel method on a Si(100) substrate. Figure 3: see original paper shows the RBS spectrum of the sample using 2.9 MeV $^7Li^{2+}$ beams with a backscattering angle of 160°. The leading edge of Si is at channel 147 (at half-height), and the Bi, La, Fe, and O peak positions are at channel numbers

of 386, 362, 265, and 62, respectively. These correspond to kinematic factors of 0.8778 for Bi, 0.8219 for La, 0.6126 for Fe, 0.3713 for Si, and 0.1607 for O. This indicates that Li ion beams provide excellent mass resolution for many elements in film analysis. Assuming the deposited La-$BiFeO_3$ thin layer is uniformly distributed on the substrate, the film thickness is estimated at 135 nm. Compared with other methods, RBS analysis is very accurate, with an error of just 2%.

Figure 3: see original paper shows RBS/C measurement results for a single-crystal Si(100) sample using 2.75 MeV Li ion beams. Compared with the random spectrum, the backscattering yield is greatly reduced in the aligned spectrum, whose flat shape demonstrates that incident ions penetrate deeply into crystal channels without dechanneling. At the high-energy edge, a small peak was observed, revealing an amorphous surface layer resulting from cutting during sample preparation. The minimum yield, , defined as the ratio of backscattered yield in the aligned spectrum to that in the random spectrum in the same channel region (i.e., same depth), represents the structural quality of the entire lattice of a monatomic crystal. For a perfect crystal, could be 1-2% [?]. The measured for the Si(100) sample is 3.01%, indicating a high-quality crystal structure.

B. Quantitative Analysis of Thin Films

The thickness of a Ni film prepared by electron beam evaporation on an SiO_2:Si substrate was measured by RBS with 2.9 MeV $^7Li^{2+}$ at normal incidence and a backscattering angle of 160° ([Figure 4: see original paper]). The yield of backscattered Li ions was detected by a Si:Au surface barrier detector with an effective detection area of 50 mm^2, positioned 112.6 mm from the sample, corresponding to a solid angle of $\Omega = 3.94$ msr. The measured spectrum overlaps with the theoretical spectrum simulated using the iterative analytical code SIMNRA. The simulated areal density of the Ni film is 2.21×10^{18} atoms/cm^2, corresponding to a thickness of 242 nm. From the measured spectrum, the Ni peak area is A = 510,000 cts, and the areal density N $= 2.20 \times 10^{18}$ atoms/cm^2, corresponding to a thickness of 240 nm, using the equation N $= A/\sigma\Omega Q$, where the scattering cross section of Ni is $\sigma = 1.25$ barn from $d\sigma/d\Omega = [Z_1Z_2e^2/(4E)]^2(4/\sin^2\ )$, and the number of incident ions is $Q = 4.7 \times 10^{13}$ (7.5 µC).

C. Composition Analysis of Multi-Element and Multilayer Samples

A MoC thin film was prepared by multi-arc ion plating on a Si substrate with a Mo film as a transition layer. The Mo and C contents, and thickness of the MoC film, were determined by RBS analysis using a 1.75 MeV proton beam at a backscattering angle of $= 170°$. The RBS spectra are shown in Figure 5: see original paper together with a fit using the SIMNRA code. The Mo signal appears approximately as a rectangular peak with a sharply rising front edge, indicating that molybdenum in the MoC layer is quite uniformly distributed, while the hump at the back edge represents the Mo signal from the transition layer. The carbon signal appears as a sharp peak rather than a hillock because

non-Rutherford elastic backscattering occurred for carbon, with a cross section 60 times larger than that of Rutherford backscattering at 1.74 MeV [?]. With 1.75 MeV protons, non-Rutherford elastic backscattering of carbon occurred near the surface. A raised Si edge can also be observed due to the enhanced elastic scattering cross-section for Si atoms at 1.75 MeV proton energy. According to the simulation result, the Mo content is 0.71 and the C content is 0.29 in the compound film layer, with an areal density of 1.52×10^{19} atoms/cm^2. The Mo transition layer thickness is 296.4 nm. Figure 5: see original paper shows RBS spectra of the sample measured at energies of 1.75–1.79 MeV. The spectrum broadens and the C peaks shift to lower energy regions with increasing incident beam energy, as the occurrence of non-Rutherford elastic backscattering of carbon deepens with proton energy, and energy resolution worsens with increasing proton energy.

D. Detection and Analysis of Light Elements in Compound Films

It is quite difficult to detect films containing light elements on substrates of higher atomic mass by backscattering techniques, as the substrate yield is too high to distinguish signals from the light elements. In such cases, resonant scattering can be employed, which has a greater scattering cross section than Rutherford scattering. Appropriate higher energy should be applied so that resonant nuclear reaction occurs for certain elements.

A TiBN film sample deposited on a Si substrate by multi-arc ion plating was analyzed using 2 MeV protons at a backscattering angle of = 170°. [Figure 6: see original paper] shows the energy spectra of the sample, with the fitting line and spectrum of the TiBN film after subtraction of the Si substrate. The strong non-Rutherford resonances in ^{1}H-^{14}N and ^{1}H-^{11}B cross sections lead to sufficient enhancement of the N and B signals, allowing the areal density of the film to be calculated from the peak areas. The net integrated counts of the Ti, N, and B peaks in the blue spectrum are A = 186,921 cts, A_N = 118,432 cts, and A_B = 47,248 cts, respectively. Large uncertainty may arise from subtraction of the substantial Si substrate signal. The accuracy of the N and B yields is determined by the error of intercept and slope of the straight regression line $y = y_0 + kx$, where y approximates the yield Y in channel x, y_0 is the intercept, and k is the slope, best fitted to the experimental data. With 2 MeV protons, the Ti cross section is Rutherford while the N and B cross sections are non-Rutherford. The ^{1}H-^{14}N cross-section enhancement factor is $(\sigma/\sigma$_R)_N = 5 and the ^{1}H-^{11}B factor is $(\sigma/\sigma$_B)_N = 7 [?]. The Rutherford backscattering cross sections for Ti, B, and N can be calculated as σ_R,Ti(E_0, 170°) = (0.6359/22) × 10^{-24} = 1.590 × 10^{-25} cm^2/sr, σ_R,N(E_0, 170°) = (0.0638/22) × 10^{-24} = 1.595 × 10^{-26} cm^2/sr, σ_R,B(E_0, 170°) = (0.0323/22) × 10^{-24} = 8.078 × 10^{-27} cm^2/sr.

The number of backscattered particles detected using the surface energy approximation for normal beam incidence can be expressed as $A_0 = \sigma(E_0)\Omega QNt$, where $\sigma(E_0)$ is the scattering cross-section at incident proton energy E_0, Ω is the solid angle, Q is the incident proton fluence, N is the atomic number density, and

t is the film thickness. Then, for Ti:N and Ti:B ratios from the backscattered spectrum, we have:

N /N_N = (A /A_N)σ_R,N(E_0, 170°)/σ_R,Ti(E_0, 170°)_N

N /N_B = (A /A_B)σ_R,B(E_0, 170°)/σ_R,Ti(E_0, 170°)_B

The average stoichiometric ratio for this film was calculated as Ti:N:B = 1.4:1.75:1.

IV. Conclusion

Measurements were carried out using the ion beam analysis facility on the 1.7 MV tandem accelerator at Wuhan University. RBS spectrum calibration using a $BiFeO_3$:La/Si sample indicates the system functions properly. Crystal quality of Si(100) was characterized by RBS/C, yielding = 3.01%. A Ni film thickness was measured at 240 nm. A MoC film on Si substrate with a Mo transition layer was analyzed, revealing a film content ratio of Mo:C = 0.71:0.29, with the Mo transition layer being 296.4 nm thick. Light element analysis of a TiBN film was performed by non-Rutherford elastic backscattering, yielding a stoichiometric ratio of Ti/N/B = 1.4:1.75:1.

[1] Jeynes C, Barradas N P, Blewett M J, et al. Improved ion beam analysis facilities at the University of Surrey. Nucl Instrum Meth B, 1998, 136-138: 1229–1234. DOI: 10.1016/S0168-583X(97)00818-5

[2] Lopez J G, Ager F J, Rank M B, et al. CNA: The first accelerator-based IBA facility in Spain. Nucl Instrum Meth B, 2000, 161-163: 1137–1142. DOI: 10.1016/S0168-583X(99)00702-8

[3] Uhrmacher M, Hofsass H. Ion accelerator facilities at the University of Gottingen. Nucl Instrum Meth B, 2005, 240: 48–54. DOI: 10.1016/j.nimb.2005.06.087

[4] Roumie M, Nsouli B, Zahraman K, et al. First accelerator based ion beam analysis facility in Lebanon: development and applications. Nucl Instrum Meth B, 2004, 219-220: 389–393. DOI: 10.1016/j.nimb.2004.01.088

[5] Hatori S, Kurita T, Hayashi Y, et al. Developments and applications of accelerator system at the Wakasa Wan Energy Research Center. Nucl Instrum Meth B, 2005, 241: 862–869. DOI: 10.1016/j.nimb.2005.07.176

[6] Pellegrino S, Trocellier P, Miro S, et al. The JANNUS Saclay facility: A new platform for materials irradiation, implantation and ion beam analysis. Nucl Instrum Meth B, 2012, 273: 213–217. DOI: 10.1016/j.nimb.2011.07.078

[7] Wang Y Q and Nastasi M A. Handbook of modern ion beam materials analysis, Material Research Society, 2009.

[8] Amsel G, Battistig G. The impact on materials science of ion beam analysis with electrostatic accelerators. Nucl Instrum Meth B, 2005, 240: 1–12. DOI: 10.1016/j.nimb.2005.06.078

[9] Chu W K, Mayer J W, Nicolet M A. Backscattering spectrometry, New York: Academic Press, 1978.

[10] He J, Lee J C, Li M, et al. Computerized control and operation of rutherford backscattering/channeling for an in situ ion beam system and its application for measurement of Si(001) and ZnO(001). Chin Phys Lett, 2011, 28: 012901. DOI: 10.1088/0256-307X/28/1/012901

Note: Figure translations are in progress. See original paper for figures.

Source: ChinaXiv —Machine translation. Verify with original.