THE SCANNING NUCLEAR MICROPROBE AT SINR*

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ABSTRACT

This paper describes the scanning nuclear microprobe facilities established at the Shanghai Institute of Nuclear Research. The Russion quadruplet constructed with four magnetic quadrupolets is used for microbeam formation. The long focus of the lens makes the working distance long enough to accommodate the scanning coils and the detectors for PIXE, RBS, RFS, NRA and SE experiments. A 5 μ m focussed beam is scanned continously by a pair of coils. A multiparameter multidetector data system for the nuclear microprobe is based on the idea of Total quantitative scanning analysis (TQSA) suggested by Melbourne University. A digital graphic displayer is a good substitute for an ordinary storage oscilloscope for on- line scanned area monitoring. The new microprobe meets high demands of biologists and geologists on applications.

Keywords: Nuclear microprobe Amoeba proleus Deep- sea manganese nodules

I . INTRODUCTION

Since the first nuclear microprobe was reported in 1970[1], it has been proved that nuclear and atomic techniques can be successfully applied to microscopic analysis of trace elements. Due to the low bremsstrahlung background when protons or heavier ions interact with a specimen, the sensitivity of PIXE is approximately three orders of magnitude higher than that of electron induced X- ray emission[2]. This fact, coupled with the relative ease of analysis involved, has made PIXE the technique most commonly used with nuclear microprobes. Some light elements, such as hydrogen, carbon, nitrogen and oxygen etc., are essential to material science and biology. NRA is ideally suited to such investigations[3]. When a scanning microprobe utilizes depth sensitive ion beam analysis techniques, such as RBS, three dimensional profiling can be performed[4]. Nuclear microprobes will become even more powerful if data can be collected simultaneously with different analysis techniques during a single experiment run.

Based on the considerations above, a new scanning nuclear microprobe was built at Shanghai Institute of Nuclear Research (SINR) in Nov. 1989. It was a result of collaborative efforts of this institute and the Melbourne group. It employs an NEC 4 MV Pelletron accelerator as an ion beam injector. In primary test experiments this microprobe manifested its potentialities with a spatial resolution of 5 μ m with 400 pA

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current of 3 MeV proton beams.

In this paper we outline the microprobe facilities, discuss the principles of the multiparameter and multidetector data system and finally reveal some new projects we are going to undertake in order to explore some new applications of the nuclear microprobe.

II. THE CONSTRUCTION OF THE SINR NUCLEAR MICROPROBE

The nuclear microprobe is built on the 30° beam line of the accelerator with a RF ion source which delivers a positive ion beam of several μ A. The ion beam is prefocussed to a spot of several mm in diameter at the entrance of the microprobe formation system. The core of the focussed accelerator beam is selected with a diaphragm which acts as an object to be demagnified by the probeforming lens. The size of the object diaphragm can be selected from 10 to 100μ m in diameter. A Faraday cup, which is called as a monitor cup, with a small hole on it is placed in front of the object diaphragm to monitor the working condition of the accelerator for maximum beam current. The monitor cup, which stops most of the beam current, refrains the object diaphragm from being overheated by the beam. The lens is a Russian quadruplet constructed with four magnetic quadrupoles. The focal length of the magnetic lens is greater than 40 cm and the overall length of the microprobe line is about 9 m, in order to achieve a demagnification of 20. The overall length is comparable with that of the accelerator itself. The admittance of the unfocussed beam is further restricted by a second diaphragm which is placed at the middle way from the object diaphragm to the lens. It controls the aperture of the ion beam and hence the aberrations of the lens. The size of the aperture diaphragm can be selected from 0.2 to 2 mm in diameter. A double slit with four insulated edges is situated in front of the aperture diaphragm and provides a means to find the position of the beam line when it is disaligned with the axis line of the two diaphragms. A pair of deflection coils located near the object diaphragm can steer the beam to improve the alignment of the two lines.

The magnetic quadrupoles are mounted on four trolleys. Each of them can be gently moved along the beam axis. Slight rotation, tilt and displacements in both vertical and horizontal directions can be made independently for the quadrupoles in order to minimize the parasitic aberrations of the lens. Before the focussed beam gets into the target chamber, it is deflected by a set of coils, which can scan the focussed beam over an area up to about 0.5mm by 0.5mm on the sample surface.

The target chamber is an octagonal construction. A sample holder can be moved in three directions with an accuracy of 1μ m. A retractable 80 mm^2 Si(Li) detector covered with a thin beryllium window is mounted at 135° to the beam direction. Two surface barrier detectors are mounted on separate rings, which can be rotated by

drivers outside the chamber in order to put the detectors at appropriate directions for RBS, RFS and STIM measurements. A concave mirror used to illuminate the sample surface is mounted on another rotateable ring. There are several auxiliary ports on the chamber available for other detectors and new devices. At the top of the chamber there is a large piece of glass which gives a good view of the details inside the chamber. A binocular microscope with a highest magnification of 200x can be used to view the target from its back. It helps while adjusting the magnetic lens to get the finest focussed beam and locating the scanned beam within an interesting area on the sample. The objective lens and a deflection prism are put inside the vaccum chamber to get a close look at the sample. A 40x stereozoom microscope sits above the viewing glass for front viewing of thick or opaque samples. In order to have a perpendicular view of the sample, a 45° mirror is put in front of it. At the centre of the mirror there is a canal allowing the ion beam to go through the mirror.

The complete beam line and target chamber are rigidly mounted on a girder supported by two pedestals, which are situated in two pits and surrounded with vibration insulation materials. It is a clean stainless steel vacuum system with metal seals and all bellows movements. There are two ion pumps with turbomolecular pump for roughing. One is for beamline and the other for target chamber.

III. THE MULTIPARAMETER DATA SYSTEM FOR NUCLEAR MICROPROBE

The main tasks of the control and data handling system for a nuclear microprobe can be classified mainly into three parts: scanning control, data acquisition and one or two dimensional spectra reconstruction and analysis. There are several different ways to control the scanning. A straight for ward way is to move samples relative to a focused beam vertically and horizontally by micrometers driven by digitally controlled step motors. The obvious disadvantage of the approach is that it can not scan at a high speed, although its dynamic scanning distance can be up to many centimeters. An alternative way, which is used on our system, is to deflect a focused ion beam in vertical (Y) and horizontal (X) directions with a pair of scanning coils. The scanning frequency usually ranges from 1 Hz to 5 kHz[6]. There are basically two major modes of scanning operation: slow raster and fast continuous. In raster mode[6] the beam is located at a pixel and an energy spectrum collected from it for either a fixed time or a fixed amount of accumulated charge. After that the beam is moved to the next pixel. In continuous mode the beam is moving continuously across pixels on a sample. Whenever a quantum emitted from a pixel which is visited by a beam is detected, the information of the quantum energy E and coordinates X and Y of this pixel is collected as an event. The event by event data collection mode was promoted to the idea of total quantitative scanning analysis (TQSA) by Melbourne university[1]. The

data system described in this paragraph is based on the strategy of TQSA.

According to the TQSA all the energy information of a quantum detected by any one of the detectors, accompanied by the position information associated with the emitted quantum, should be recorded without any loss due to energy window presetting or position restriction. The principal structure of the whole data system can be seen in Fig 1.

The system is divided into two separate parts. The front one is located in the experiment hall and the other in the computer room. The information of quantum energy, pixel position and time of an event are transferred to the computer via cables of 20 meters length. The power supplies for quadrupoles and scanning coils are digitally controlled by an IBM compatible PC computer. A graphic displayer is also digitally controlled. They pave the way to automatic focusing of the beam spot and tracing the scanned area in further developments. A multiparameter multichannel analyzer ND- 76 is employed for event- by- event data collection. The ND- 76 is working in quadruplex list mode. It means that there are four parameters in an event being listed. Three of them represent the values of energy E and pixel coordinates X and Y. The fourth parameter is reserved for another use.

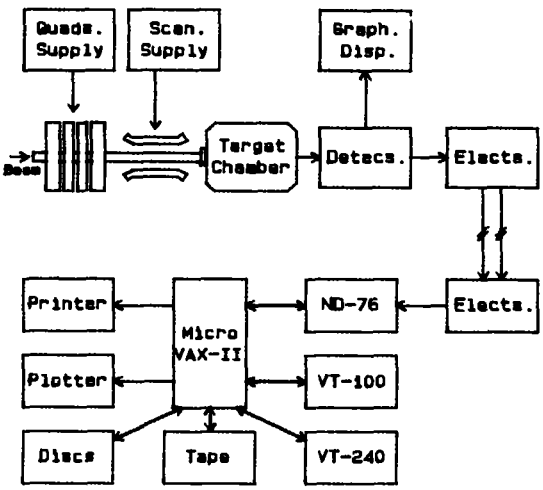

Fig. 1 The principal structure of multiparameter data system for SINR nuclear microprobe

At the completion of an experiment the multiparameter data block is stored on the tape. It is essentially a sequence of events, of which the energy E and coordinates X and Y are arranged at random. Before any further processing the data must be sorted by their energy or coordinates. There are two major objects in this process. One is to sort the data for an energy spectrum from the events, of which the coordinates are restricted within an interesting area. The other is to sort the data for a map of the events, of which the energy falls into an interesting interval. In the case of PIXE analysis, for example, if the energy interval corresponds with the characteristic energy of a certain element a distribution map of this element will be created. A sorting program DOSPT was written for the creation of energy spectra and another sorting program DOMAP for that of element maps. Each of the two programs can preset up to 64 boundaries of interesting areas or interesting energy intervals to create up to 64 different energy spectra or element maps in a single operation. These spectra from different areas can be summed to get a new spectrum from a larger area

of grotesque shapes.

The spectrum is then fed to programs, by which some routine manipulations for PIXE, RBS and NRA are carried out. They are energy calibration, background fitting, interference elimination and peak searching, peak area integration etc. For reference or publication, the spectrum can be plotted and a report of the peaks with all their parameters can be printed. A sophisticated graphic program was built in to display element maps in the form of three dimensional isometric and two- or three- dimensional contour maps, of which an example can be seen in Fig.2. These

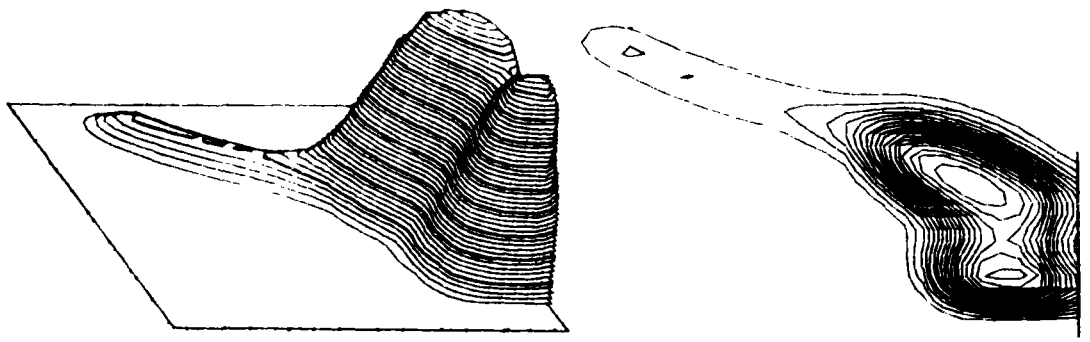

Fig. 2 Distribution of calcium in a pollen tube by the nuclear microprobe

maps show the distribution of calcium within a lily pollen tube and part of its body. The relatively high calcium concentration within the tube tip confirms the result found at Oxford[8]. The contour maps are more favourable than other maps because from them one can get both qualitative information on distribution profile and quantitative information on elemental intensity and localization[9].

IV. SOME FORESEEN APPLICATIONS

The establishment of the scanning nuclear microprobe has interested groups of people in research institutions and industries as well. Some research projects have been arranged for its applications. Two of them are described below as examples.

It has been well known that trace elements play an important part in biological organisms[10] at concentration levels out of reach of electron microprobes. The nuclear microprobe, however, may play a fundamental role in many areas of medical and biological research. We chose the Amoeba proteus as the sample for feasibility test of our microprobe. The Amoeba proteus is one of the largest unicellular organisms. It has the size of about 300μ m in diameter and the nucleus

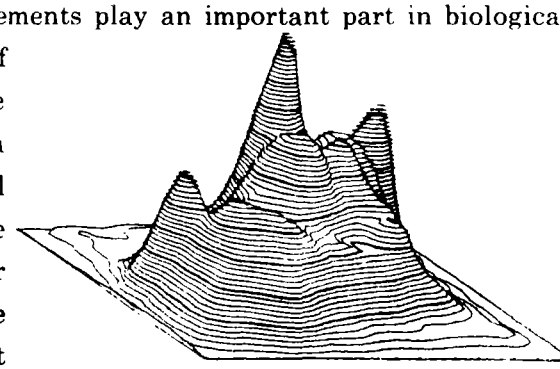

Fig. 3 Elemental distribution within an Amoeba cell

of its subcellular can be easily identified. From Fig.3 we can see the shape of the elemental distribution in a single Amoeba cell.

The trace element zinc is now known to play a very important role in the growth of the Amoeba proteus[11]. We are going to correlate the zinc concentration and its distribution within the Amoeba proteus to its pathological consequences. Element zinc is also one of the vitals for DNA synthesis. Certain amount of zinc is necessary at each step of its growth. The pathological effects of excessive zinc in culture medium have been demonstrated[11]. The question we are interested in is what will happen if the distribution of zinc is not even within a cell at either normal or excess condition. The mechanism of and the response to the uneven distribution of zinc within a cell are not clear at present. It is certainly worthwhile investigating the distribution and variation of zinc concentration within the Amoeba proteus after introducing an recombinant DNA which can overexpress a metallothionein product known to bind zinc and so negate the pathological effects of excess zinc in the cell.

The application of the nuclear microprobe as a tool for trace element microanalysis in the geosciences has brought anticipated benefits in an ever increasing number of areas[12]. Geological materials are often complex heterogeneous assemblages of minerals containing many elements. Many of them contain inclusions or individual mineral grains of sizes varying down to micron dimensions. By studying the composition and distribution of trace elements in a micron scale of the mineral ore, we can obtain clues to the genesis and the physical and chemical history of the mine and relate this to the overall history of the geological region.

The deep- sea manganese nodules have aroused great interests in recent years because of their economic importance. Investigation of the origin and local distribution of the manganese nodules and their growth processes by which they selectively concentrate iron, manganese,copper, nickel and other metals has been done under some international collaborative programs[13]. The nodules and their principle constituents, todorokites, are formed in a layered structure around a nucleus. The recognizable X- ray diffraction patterns show that they are composed of cryptocrystalline minerals with three major manganese phases[14]. X- ray patterns have not been adequate for structure determination. It is believed that the nodule composition most typical of growth under oxic diagenesis is the composition of the nodule bottom which takes trace metales from a siliceous sediment[15]. The radiometric dating techniques employing isotopes with different origin and half- lives are common means used to measure the growth rate of the nodules[16,17]. Many attentions have been drawn to the relationship between the growth rate and the chemical composition of the manganese nodules[18,19]. From the information available on the accretion rates and the bulk chemical composition of the nodules, an equation that predicts the nodule growth rates from their chemical compositions has been derived[20]. It has been

known[21] that microscale chemical inhomogeneities exist in nodules, which represent either short- term growth rate variation or small scale, close- system diagenetic reorganization. Attempts have been made to study the spatial variability of the growth rate on mm scales by scraping a thin layer of the nodule with a drill and analysing it[22,23]. Because the growth rate of the nodule is around a few mm in a million years, it will be interesting to find a means to measure the spatial variation of the composition more accurately. By means of the nuclear microprobe we can get the chemical compositions and consequently the spatial distributions of the accretion rate of the nodules in microscales. Comparing them with the layered structure which can be seen with an optical microscope, we can have a better understanding of the growth processes of the nodules.

V. CONCLUSION

A new scanning nuclear microprobe has been built up at SINR. It has four features: (1) The quadruplet lens has a long focus, which makes the working distance long enough to accommodate the scanning coils and different detectors for PIXE, RBS, RFS, STIM, Channelling, SE and NRA. (2) Above the target chamber there are two binocular microscopes viewing sample surfaces forwards and backwards. They make it easy to search and to aim precisely at interesting regions for scanning. (3) The power supplies for quadrupoles and scanning coils are digitally controlled by a computer. They are easy to use and good for stability. A digital scanning graphical displayer replaces the usual storage oscilloscope. The digital controls of these devices make it possible to focus the beam spot and to trace the scanned area automatically from the view of further development. (4) The continuous operation of beam scanning moderates the problems of sample volatilization during bombardment with ions of high energy and high current density and guarantees the uniformity of data collection from different positions of scanned area even if an experiment lasts hours.

The correlation of zinc concentration and its distribution within an Amoeba proteus to the pathological consequences of the cell and the spatial distribution of accretion rates of the deep- sea manganese nodules will be investigated with the scanning nuclear microprobe.

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