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# Development of neutron depth profiling at CMRR

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ABSTRACT

A neutron depth profiling (NDP) system has been developed at China Mianyang Research Reactor (CMRR) at Institute of Nuclear Physics and Chemistry (INPC), CAEP. The INPC-NDP system utilizes cold neutrons which are transported along the C1 neutron guide from the cold neutron source. It consists of a beam entrance, a target chamber, a beam stopper, and data acquisition electronics for charged particle pulse-height analysis. A 90 cm in diameter stainless steel target chamber was designed to control the positions of the sample and detector. The neutron beam intensity of $2.1 \times 10^8$ n cm$^{-2}$ s$^{-1}$ was calibrated by the Au foil activation method at the sample position. The INPC-NDP system was tested by using a Standard Reference Materials SRM-2137. The measured results agreed well with the reference values.



## 1. Introduction

Neutron Depth Profiling (NDP) is a non-destructive technique for measuring the depth profiles of some light elements (e.g., $^3$He, $^{10}$B, $^6$Li, etc.) in the near-surface region of a sample. The sample is illuminated by a thermal or cold neutron beam, and the concentration profile of the isotope of interest is inferred from the residual energy of ions produced by neutron reactions with the isotope of interest. The NDP technique was first proposed by Ziegler et al. [1]. The capabilities of NDP have been thoroughly investigated by Fink et al. Because of the simplicity and high sensitivity of the NDP technique, it has been further developed and extensively applied in surface analysis, solid state research, lithium ion batteries, and other material sciences [2–10]. Downing from NIST builds a web site to collect and introduce the information of NDP facilities and literature around the world [11]. Nowadays, there are several NDP facilities and most of them are in the U.S.A and Europe. A new Cold NDP was developed at HANARO in 2014 [10].

This paper presents a new neutron depth profiling facility developed at China Mianyang Research Reactor (CMRR) at Institute of Nuclear Physics and Chemistry (INPC), CAEP. The SRM-2137 sample was measured to validate the functionality of the INPC-NDP system.

## 2. INPC-NDP description

The NDP facility is built at a cold neutron beam port of CMRR, which is shared with a cold neutron radiograph system. The beam size is a square up to $4 \times 4$ cm. However, it is able to be adjusted. The eigen-wavelength of the neutron beam is 2 Å, and the neutron intensity at the sample surface is about $2.1 \times 10^8$ n cm$^{-2}$ s$^{-1}$ (thermal neutron equivalent), which is calibrated by using Au foils.

The INPC-NDP consists of a beam entrance of 1.5 cm in diameter in a boron carbide block, a target chamber fixed on a supporting bracket, a beam stopper, and three separate charged particle detector systems with various distances and angles to the sample. The structure is illustrated in Fig. 1.

### 2.1. Target chamber

The target chamber is composed of a body and a hemisphere top cover both made of 6 mm thick stainless steel. There is a plexiglass viewing window on the top cover, which will be covered during operation due to the fact that the visible light saturates the detector signals. The body and top cover are joined by an O-ring to provide vacuum seals. On the side of the chamber, there are two 1 mm thickness Aluminum windows for neutron beam in and out, and the diameters are respectively 1.5 cm and 8 cm. A small door is constructed to replace beam collimator and samples without opening the top cover. Three flanges are designed for the molecular vacuum pump and gauges. The air pressure is less than $1.0 \times 10^{-3}$ Pa during operation. Two flanges are designed for detector cables. The chamber has additional flanges available for possible future applications.

Photographic views of inside the chamber and of several individual components are shown in Fig. 2. The inner diameter of the chamber is 90 cm and the height is 60 cm. Three brackets are respectively for detectors, neutron beam collimator, and sample mounting. There are three separate detector systems, which enable them to independently acquire energy spectrum

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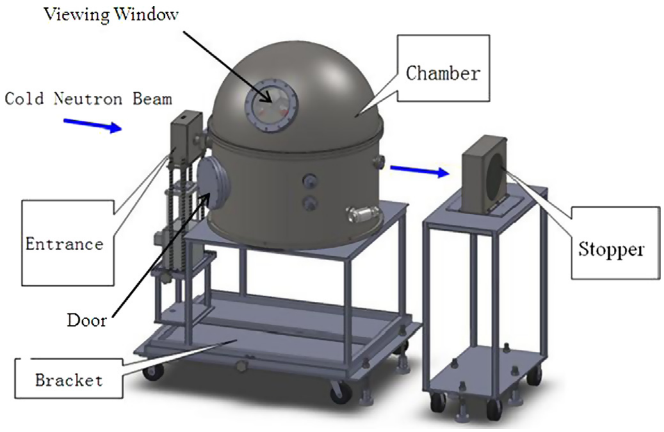


**Fig. 1.** Layout of the INPC-NDP structure.

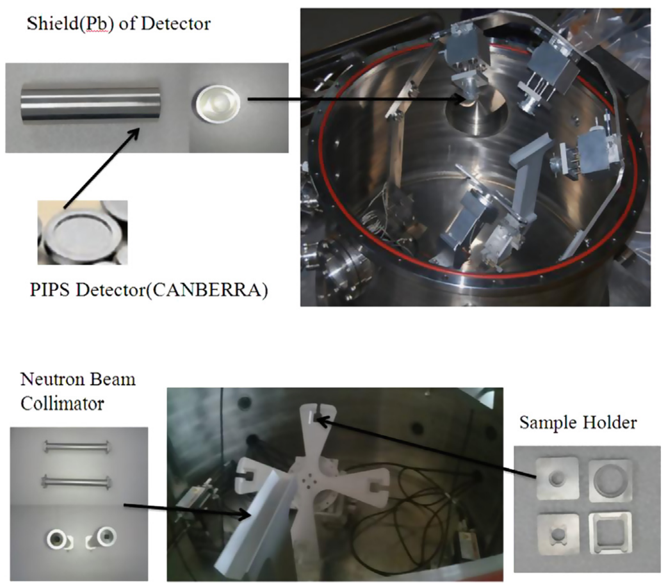


**Fig. 2.** Inside of the target chamber.

data at the same time. Detector assembles are positioned into 5 mm thick Pb shield to attenuate γ-ray or other background particles and then mounted onto the detector bracket. The distance between the detectors and sample can be adjusted from 7 cm to 20 cm and the detector angles varied relative to the sample surface. The neutron beam approaches the sample at an angle of 45 degrees from the sample normal. A 30 cm long beam collimator is positioned at the beam inlet inside the chamber to define the size of the beam and decrease the neutron scattering in the chamber. It's composed of boron carbide and lead. The boron carbide collimator is cyclindrical and has a tapered bore aligned with the sample. The collimator is wrapped with 5 mm of lead sheet to attenuate γ and ions produced by nuclear reactions. There are several collimators with various outlet diameters, 0.5 cm, 0.75 cm, and 1 cm. The sample bracket of 18 cm in diameter is made of 3.5 mm thick Aluminum, and apertures are designed to decrease the background. It is rotated by a stepper motor, and has four positions for sample mountings, which means that four samples can be mounted at the same time. Mountings of various shapes and sizes are designed for different samples. Thus rectangular or circular shaped samples varying in the size from 5 mm to 20 mm can be mounted. In addition, it's able to perform a scan measurement for some larger samples by rotating the bracket. As shown in Fig. 3, the inside of the installed chamber can be viewed via the window on the top cover.

**Fig. 3.** Photograph of the inside of the installed chamber.

### 2.2. Data acquisition electronics

The electronics for the INPC-NDP consist of three detecting modules for the charged particle pulse-height analysis. Fig. 4 shows a block diagram of the data acquisition and analysis electronics. The CANBERRA Passivated Implanted Planar Silicon (PIPS) detectors are used to measure the energy spectrum of charged particles. The detectors employ a 150 mm$^2$ or 300 mm$^2$ active area with a depletion layer thickness of 100 μm. The dead layer thickness of the Si detector is less than 50 nm. The energy shifting of detectors is tested by measuring an $^{241}$Am $\alpha$ source. The $\alpha$ source is measured every half an hour, and the channel shifting of the energy peak is less than 0.05% for all detectors in 8 h, which indicates that the detectors have acceptable reliabilities and stabilities. The recommended bias for detectors is from 20 V to 60 V, whereas, the bias is often set to 20 V to decrease the depletion layer thickness, which further decreases the background, and the depletion layer is still thick enough to fully collect the energy of the charged particles.

## 3. Validation of the INPC-NDP

### 3.1. Spectra of boron samples

Two boron samples were measured by INPC-NDP. One was manufactured by depositing 10 nm thick natural boron on 2 × 2 cm square and 0.5 mm thick silicon substrate, which was used for the channel-energy calibration. The other was a standard reference material 2137 (SRM-2137) from NIST, which was manufactured by implanting $^{10}$B ions into a 1 × 1 cm square and 1 mm thick silicon substrate. When $^{10}$B absorbs a thermal or cold neutron, two distinct energies are produced for each of the $^4$He and $^7$Li ions since the $^7$Li nucleus is produced either in the ground state or in an excited state as shown below.

$$ {}_0^1\text{n} + {}_5^{10}\text{B} \rightarrow \begin{cases} {}_3^7\text{Li}^* + {}_2^4\text{He}(93.7\%) \\ {}_3^7\text{Li} + {}_2^4\text{He}(6.3\%). \end{cases} $$