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3.2 Status of Neutron Scattering in Thailand

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Abstract

In 1992, the Office of Atomic Energy for Peace had established a spectrometer upgrade program, the main objective was to improve the performance of the old double-axis spectrometer which used to be situated at TRR1. At present, installation and alignment of the upgraded diffractometer have been accomplished. This report gives brief description about the current status of the facility.

In addition, the Office of Atomic Energy is in process of constructing a 10 MW nuclear research reactor at the Ongkharak Nuclear Research Center site. The reactor will be equipped with a High Resolution Powder Diffractometer which is anticipated to be in operation soon after the commission of the reactor. Feature of the HRPD including its potential utilization are presented in this report.

1. Introduction

The Thai Research Reactor TRR1 achieved its first criticality on October 27, 1962. It was a multipurpose 1 MW pool type using plate type HEU fuel. In 1968, the first double-axis neutron spectrometer in Thailand was installed at one of its beam tubes. It was about a decade that this diffractometer had served a number of magnetic materials researches. In 1977, the reactor core was changed into TRIGA MARK III type with nominal power of 2 MW at steady state and pulsing capacity up to 2000 MW. It was designated as TRR-1/M1. At present the reactor is normally operated at 1.2 MW for 3 days a week, about 12 hours a day to meet 40 MW-hours per week as required by radioisotope production.

During the modification of TRR1, the diffractometer was dismantled. Since then neutron scattering activities had ceased for many years. As the Office of Atomic Energy for Peace recognized the important role of neutron scattering technique in broad areas of research particularly in materials science, therefore in 1992, a spectrometer upgrade program has been established. The purpose was to improve the performance of the obsolete existing diffractometer. Implementation of the program included modification of the old monochromator shielding, construction of a new in-pile collimator and a specimen table as well as purchasing a new detection system. At present time, installation of the diffractometer is completed and performance test is being carried on.

2. Description of the Upgraded Neutron Powder Diffractometer

The upgraded diffractometer was aimed to have medium resolution. Optimization of resolution and intensity was conducted through selection of 60° takeoff angle in combination with 30', 30' and 20' for the first, the second and the third collimations, respectively. In order to minimize the background, heavy concrete blocks more than 50 cm. thick were added around the old monochromator shielding. The new detector shielding has been designed to accommodate four He-3 detectors setting at 6° apart. This detector bank was surrounded by a curved fixed-shielding made of 5% borated paraffin. At this moment, because of high background intensity, only a single He-3 detector is in use to provide more room for additional shielding. Automated data acquisition is accomplished by incorporating the motor controller and the counter with a personal computer through the GPIB communication controller system. Detail of the upgraded diffractometer is indicated in Table 1 and its layout is illustrated in Fig. 1.

Table 1 Characteristics of the upgraded neutron diffractometer

Beam size	20 mm.x 100 mm.
First collimation	30'
Second collimation	30'
Third collimation	20'
Monochromator :	
Take-off	59°
Wavelength : Cu(111)	2.0557 Å
PG(004)	1.6524 Å
Neutron detectors	4 x He-3 (4 atm.)
Neutron source to monochromator	400 cm.
Monochromator to specimen	180 cm.
Specimen to detector	70 cm.
Thermal neutron flux at specimen position	2×10^4 n.cm^{-2}.sec^{-1}

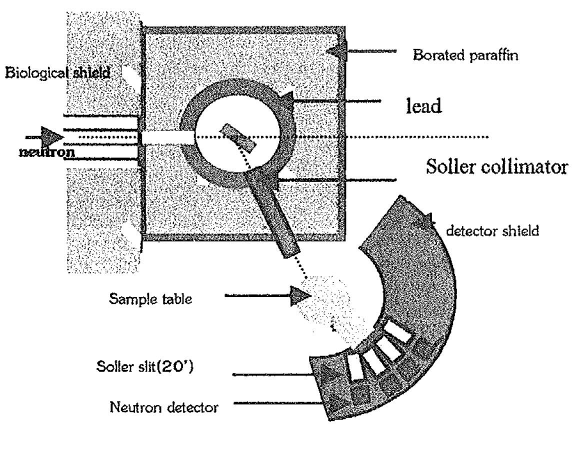

Fig. 1 Layout of the upgraded neutron powder diffractometer

3. Diffractometer Performance

Performance test has been carried out by running a standard silicon powder (NIST Standard Reference Material 640b)and a high purity corundum powder samples (99.999% Al_2O_3, Aldrich Chemical Company) on the diffractometer. The silicon sample was contained in small vanadium tubes, 10 mm. in diameter and 50 mm. high. In an attempt to gain higher peak intensity, the sample size of corundum was increased to 13 mm. in diameter and 60 mm high. Data were collected from 8° to 105° with 2° step size. The counting time was 5 minutes per step. A 75mm.x75 mm.x1 mm. pyrolytic graphite monochromator was employed to provide thermal neutron flux greater than 2 x 10^4 $n.cm^{-2}.sec^{-1}$ at the sample position.

The recorded data were refined by Rietveld method. The Voigt profile function was assigned to fit the diffraction peak shapes. Since examination of the diffraction patterns from silicon and corundum samples revealed severe contamination from 2λ , 2/3λ and ½λ, thus in the refinement both samples were treated as four-phase compounds. It was found that data obtained beyond 100° were ambiguous, therefore they were not taken into account in the analysis. The wavelength using in the refinement was 1.6537 Å

4. Results of Analysis from Silicon and Corundum Diffraction Pattern

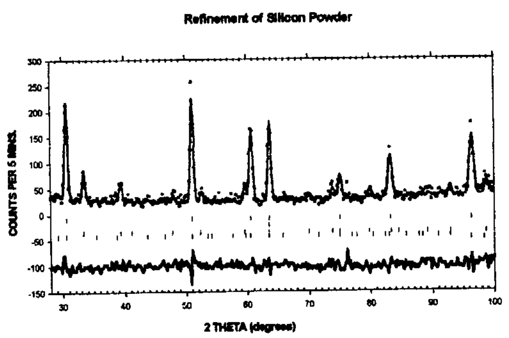

Fig.2 Rietveld refinement of silicon powder sample showing contamination from higher order beams.

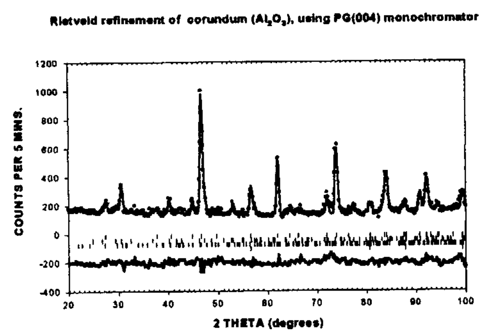

Fig.3 Rietveld refinement of corundum showing contamination from higher order beams.

Table 2 Results of Rietveld Analysis of silicon and corundum samples, data from OAEP ND

Parameters	Silicon				Corundum			
	λ	2λ	2/3λ	1/2λ	λ	2λ	2/3λ	1/2λ
Scale factor	0.32(1)	0.96(8)	0.035(3)	0.003(1)	0.165(3)	0.41(11)	0.019(3)	.0001(0)
Scale factor ratio	1	2.94	0.11	.009	1	2.48	0.11	.006
Cell dimension(Å)	a=5.4300(4)				a=4.759(2) c=12.997(3)			
R_B (%)	2.34	0.91	6.85	7.28	3.45	2.93	3.42	4.73
R_p (%)	13.07				7.60			
R_{wp} (%)	16.91				9.52			
R_{exp} (%)	15.28				7.14			
GOF (%)	1.22				1.78			
U	.4986				0.8712			
V	-.2034				-0.9201			
W	.2957				0.5069			

Fig.2 indicates the results of Rietveld refinement from silicon powder diffraction pattern. The resulting reliability factors shown in Table 2 imply that the observed data do not fit well the calculated values. Nevertheless, its observed cell dimension which is a=5.4300(4) Å agrees closely with the expecting 5.4309Å.

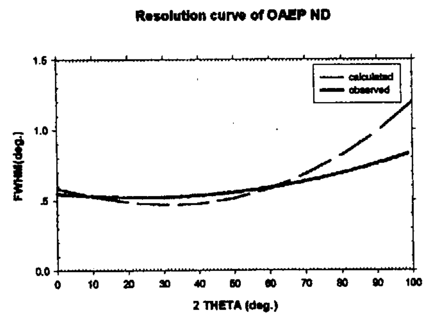

Fig.4 Comparison of the observed and calculated peak widths

The peak widths of silicon derived from the Rietveld analysis were plotted in comparison with the calculated values as indicated in Fig.4. It is obvious that the observed and calculated peak resolutions are consistent for scattering angles below (2θ) 65°. At higher angles, the observed peaks widths are comparable small.

Results of Rietveld refinement from corundum diffraction data are shown in Fig.3 and Table2. Although more than three hundred peaks produced from 2λ , 2/3λ and ½λ. are observed, there is a good agreement between the observed and calculated profiles. The reliability factors obtained from the analysis are included in Table2.

5. Future development of ND and its applications

From the scale factor ratio of the contaminants λ:2λ:2/3λ:½λ. indicated in Table2, interference from the half lamda was less than 1% and could be neglected. Contribution from 2λ was more than 250% but could be reduced by using small unit cell sample. It seemed that contribution from 2/3λ which was about 10% had significant effect on the analytical results particularly on the reliability factors.

Besides the interference from the higher order beams, other significant problems are due to low neutron intensity and high background. It is noted that the peak to background ratio were about 9 at peak(111) of silicon and 7 at peak (113) of corundum.

To improve the neutron intensity at specimen position, the existing monochromator may be replaced with a vertical focusing or a bent focusing crystals. The data acquisition rate could be enhanced by adding more detectors. Meanwhile, better shielding for detectors is needed. Alternatively, the diffractometer could be modified to serve residual stress measurement, which requires smaller data range. However, at the present time the facility could be employed for investigation of small unit cell samples and training university students in the field of neutron scattering.

6. High Resolution Powder Diffractometer at the Ongkharak Nuclear Research Center

The Office of Atomic Energy for Peace is presently in process of constructing a 10 MW TRIGA nuclear research reactor at the Ongkharak Nuclear Research Center (ONRC) site which is about 60 km. from Bangkok. The reactor is a multipurpose, pool type, cooled and moderated by light water using low enriched uranium fuel. Reflectors are heavy water and beryllium. The expecting maximum thermal neutron flux is $2x10^{14}$ $n.cm^{-2}.sec^{-1}$. Six horizontal beam tubes are available for beam experiment, four of them are allocated for experiments in neutron scattering, neutron radiography, prompt gamma neutron activation analysis and BNCT. The rest are reserved for the future neutron guide tube and unforeseen experiment. Configuration of the reactor core is illustrated in Fig. 5 [1]. Provision of a High Resolution Powder Diffractometer has also been included as part of the ONRC project contract. Installation of the HRPD shall be completed before the commission of the reactor.

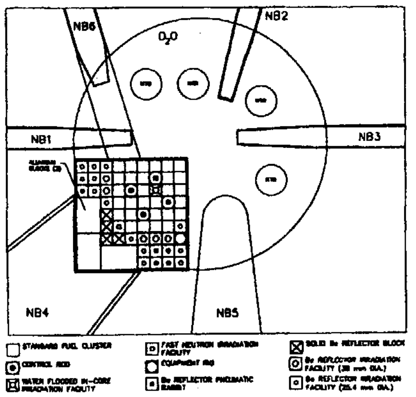

Fig.5 Configuration of the reactor core at ONRC

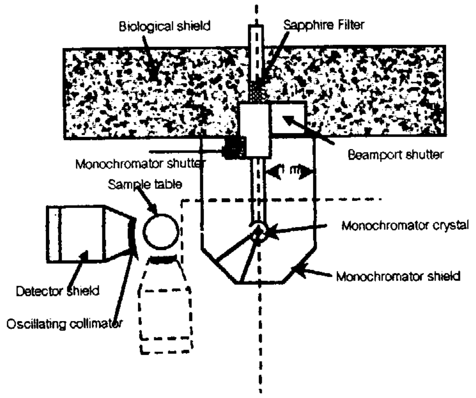

Fig.6 Layout of the HRPD at ONRC

Layout of the HRPD at ONRC is shown in Fig.6. The design basis of the proposed HRPD involves the matching of a bent focusing monochromator, small sample and position sensitive detector[2]. No collimators are required in this system. The monochromator is a doubly focusing bent silicon perfect crystal, which is at 95° takeoff, could be adjusted to provide four wavelengths, that is 2.41 Å, 1.84 Å, 1.54 Å and 1.22 Å, from diffraction planes (311), (331), (511) and (533) respectively. To achieve high peak resolution, the sample diameter needs to be less than 3 mm. The detector bank is consisted of 7 He-3 position sensitive detectors assembled together with 20° horizontal and 6.27° vertical spanning at 160 cm. distance. The detector electronics provide position resolution better than 3 mm. An oscillation collimator is positioned between the sample and the detector bank to reduce background from the environment. The monochromator shielding is designed to provide takeoff angles at 95°, 60 ° and 30°.

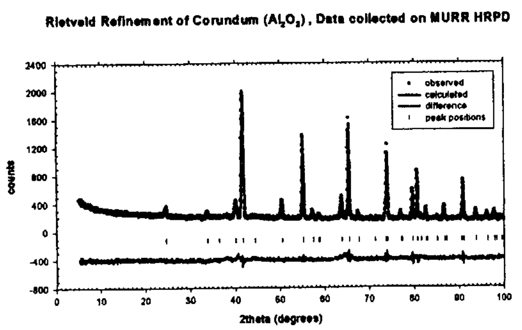

Fig.7 Rietveld refinement of alumina sample showing good agreement between the observed and calculated profiles.

Performance of the proposed facility has been investigated by measuring the diffraction pattern of a standard alumina (Al_2O_3) sample with the High Resolution Neutron Powder Diffractometer at the Missouri University Research Reactor, which is a similar type diffractometer. It took several hours to collect more than 2000 counts at peak (113) of alumina with the peak to background ratio of 10. The result of Rietveld refinement, which is illustrated in Fig.7 shows good agreement between the observed and calculated profiles. The unit cell parameters obtained from the analysis were a= 4.7589(4) Å, c=12.9941(7) Å with the reliability factors R_{wp} = 6.89%, R_{exp} = 6.36%, R_B=1.56% and Goodness of Fit =1.17%.

The High Resolution Powder Diffractometer at the ONRC site would be dedicated for broad range research areas. Potential applications would be in the area of advanced materials research e.g. high temperature superconducting materials, PZT, magnetic materials, structural transitions and training.

7. Neutron Scattering Research Program

At present, our Nuclear Material Science Group has a national joint research project on gemstone enhancement with the Department of Physics of Kasetsart University and the Department of Mineral Resources. We are taking parts in gemstone characterization and coloration by irradiation. Two collaborative projects with Mahidol university in the investigation on the atomic positions in RE-doped PZT and the study of the morphology of elastomer under strain are being planned.

Besides the above projects, an international research program on the investigation of high temperature superconductors is being proceeded in collaboration with BATAN, Indonesia and Vietnam.

Acknowledgement

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References

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2. W.B. Yelon, B. Berliner and M. Popovici, **Physica, B241-243**, 237-239 (1998).