



## 1.22 Ten year's activity in the field of neutron scattering workshop

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### Abstract

'Neutron scattering' is in the frame of the 'Utilization of Research Reactors', which held the workshop from FY 1992. This report is a summary of the results and activities of neutron scattering workshop and sub-workshop started from FY 1992.

### 1. Historical review

'Neutron scattering' is in the frame of the 'Utilization of Research Reactors', which held the workshop from FY 1992. We would like to summarize the results and activities of neutron scattering workshop and sub-workshop started from FY 1992.

FY 1992: Report on the current status of neutron scattering in each participating country, and the discussion on the future program were presented in Jakarta, Indonesia.

FY 1993: Operated the On-the-Job training using high resolution powder diffractometer (HRPD) installed RSG-GAS research reactor in Serpong, Indonesia, reported on the current status of neutron scattering in each participating country and discussed on the future program in Jakarta, Indonesia.

FY 1994: Conducted the real experiments and data analysis of several samples using the improved HRPD in Serpong as the sub-workshop. Workshop was held in Jakarta, Indonesia. In the workshop three cooperative research programs were proposed and decided.

FY 1995: Discussed on the results of three cooperative research programs at the sub-workshop in Serpong. In the workshop held in Jakarta, Indonesia, additional discussion and comments were made.

FY 1996: In the workshop held in Bandung, Indonesia, three cooperative research programs were summarized and decided to continue as the name of 'structural research of functional materials'. New program 'polymer research using small angle neutron scattering (SANS)' was proposed and decided. Several projects on the planning of new neutron source were reported from each country.

FY 1997: In the workshop held in Yogyakarta, Indonesia, the status of neutron scattering in each participating country and the results of cooperative research program were reported. After the workshop sub-workshop on the demonstration of measurement and data analysis using SANS instrument installed in Serpong was continued.

FY 1999: Sub-workshop on the utilization of SANS instrument was held in Tokai, Japan. Not only standard polymer sample, but also many kinds of sample provided from the participating countries have been measured and analyzed. In the workshop held in Mito, Japan, the plan of future collaboration of polymer study using SANS was discussed.

FY 2000: During the workshop, many kinds of sample including the standard polymer sample provided from the participating country were measured using the SANS instrument installed in HANARO. The results of analyzed data were discussed. Next candidate of cooperative research program was also discussed. In the round table discussion, the

philosophy and target of FNCA project were explained from the Japanese coordinator. In the field of neutron scattering, it was indicated that target of cooperation must be focused to satisfy the socio-economical benefit. Some discussion was held during the lunch time, and it should be discussed in the next workshop.

## 2 Comment

### (1) Cooperative research program of structural analysis of functional materials using HRPD

Neutron diffraction is useful technique to understand the crystallographic and magnetic structure of materials, which is related to physical and chemical properties of materials. In the stage of starting the workshop (FY 1993), the instruments, which can be used for the study, were that installed in JRR-3M in JAERI, HIFER in Australia and RSG-GAS in Indonesia. We decided to use HRPD installed in RSG-GAS reactor for the cooperative research, and started the 'On-the Job' training for becoming familiar with neutron diffraction to the researcher of Asian area. At that time the capability of Indonesian HRPD was not enough, but improved too much and enough to use for the cooperative research. From FY 1994 three cooperative research programs have been started in the period of three years. The three cooperative research programs were magnetic materials, super-ionic conductor and high T<sub>c</sub> superconductor.

In the field of magnetic materials, cooperation between Indonesia and China worked well. Several papers have been published in international journals as shown in the list of publications. The cooperation between neutron group in China Institute of Atomic Energy (CIAE) and magnetic materials group in China Academy of Science played an important role in the success of this program. Usually the neutron scattering group in an atomic energy institute has not so much activity on the sample preparation and measurement of physical properties of sample. Magnetic materials group in China academy of science has enough power to produce many kinds of ferromagnetic materials, which have been provided to the neutron scattering group in Serpong through CIAE. Neutron scattering group has measured the magnetic structure using HRPD, and mutual communication and discussion on the relation between magnetic structure and physical properties were made to obtain the reasonable results. For the research of the neutron scattering interdisciplinary cooperation is essential to the success of research, since neutron scattering is one of the measuring technique of physical and/or chemical property.

In the field of super-ionic conductor, the Philippines group made the sample of beta-alumina and Indonesian group measured the neutron diffraction pattern at room temperature. We regret to say that the neutron diffraction measurement at higher temperature could not be made due to the problem of furnace, and terminated. Another samples of super-ionic conductor were tried to produce by the Indonesian group, but no neutron data were obtained.

In the field of high T<sub>c</sub> superconductor, the neutron diffraction data have been obtained for the standard superconducting materials. Thailand and Vietnam group produced many kinds of new sample and measured their temperature dependence of electrical resistivity. But no neutron diffraction pattern we have. This field is advanced scientific and engineering one. Many works have been made in many advanced countries, but for the Asian region it seems to

be difficult to follow the advance.

For the analysis of neutron diffraction pattern to the determination of crystallographic and magnetic structure, full pattern fitting software 'RIETAN' of Macintosh and IBM-PC versions were provided to all participating countries from Dr. Izumi in Japan. It worked well in Serpong on the cooperative research works.

## (2) Polymer study using SANS

Small angle neutron scattering (SANS) is another useful technique of neutron scattering. Small angle scattering of light, X-ray and neutrons can obtain the information of semi-macro structure in materials in their own measuring scale. One of the typical examples is age-hardening alloy. The hardness and/or strength of age-hardening alloy depend on the size and inter-particle distance of precipitated particle in the alloy during the heat treatment. The information of which can be determined using SANX or SANS. Due to the deep penetration depth of neutrons, SANS can be applied to the materials including heavy atoms such as iron alloys.

In the case of polymer materials the sub-macro structure of molecules influence to the character of polymer, especially in polymer blend and block copolymer. SANS is one of the tools to study the semi-macro structure of polymer materials, which can use the special measuring technique of 'Contrast Variation Method' or replacement of some hydrogen atoms with deuterium.

The cooperative research on SANS has been started from FY 1997. In the sub-workshop held in Serpong all participants obtained the experience of SANS measurement and data analysis. The first stage of cooperative research is to make the inter-laboratory comparison of data taken in each SANS instrument. The sample of silver-behenate, provided from ANSTO was selected as the standard sample of q-calibration. The measurements have been made using the instruments installed in ANSTO, JAERI and BATAN. All data indicate excellent agreement. The silver-behenate sample was provided to KAERI and the data also indicate good agreement. It means that four SANS instruments in Asian area can be used for the future study. For the inter-laboratory calibration of measured intensity PSH/PSD (polystyrene-h/polystyrene-d) was selected as standard sample, which was circularized to Indonesia.

In the sub-workshop held in JAERI and KAERI many samples provided from participating countries have been measured and analyzed. Some of them are commercial products and the materials related to commercial problem.

## 3 Conclusion

During the last ten years, much progress of neutron scattering research activity in each participating country has been made. In Indonesia, HRPD and SANS instruments are working well and are obtaining the data from many samples. The obtained data can be analyzed by their own hands. In Thailand the reconstruction of double axis neutron diffractometer installed in Mark III is in progress and will be completed near future. The design and construction of HRPD, which will be installed in new research reactor is also in progress. The improvement

of SANS instrument in Malaysia is continued year by year. In China the plan of construction of new research reactor CARR seems to be going well. In Korea HRPD and SANS instruments installed in research reactor HANARO are now operating well.

The workshop on neutron scattering contributed too much to the exchange of scientific and technical information. In the sub-workshop many participants from developing countries obtained the much amount of useful experience on the utilization of hardware and software provided from the developed countries.

Neutron scattering is one of the techniques to obtain the static and dynamic structure of materials. During past 60 years the measuring technique and instrumentation of neutron scattering progress very much according to the construction of high flux research reactors and strong pulse neutron sources. The driven force of progress is mainly due to the request of the researcher of solid state physics, chemistry, biophysics and the originality of neutron scattering specialist. It means the study of neutron scattering is mainly concentrated to the academic research. Recently the request to apply the technique of neutron scattering for the industrial problem become severe even in the developed countries. The cost of neutron and maintenance of instruments are no little expensive. It means that in the Asian area we must consider and enhance the application of neutron scattering technique to industrial uses. One of the good examples is the research program in ANSTO, Australia. For the application of neutron scattering to industrial problem, we must keep tight network system in Asian region.

In the operation of workshop and sub-workshop we appreciate too much to the contribution of Australian and Japanese experts on the guidance of advanced technique of neutron scattering, and transmission of scientific and engineering information to the participants.

#### 4 List of publications

1. High-Resolution Neutron Powder Diffraction Study of  $\text{Dy}_2\text{Fe}_9\text{Al}_8$  at 65 K  
S.Ridwan, H.Mujamilah, M.Gunawan, P.Marsongkahadi, Q.W.Yan,  
P.L.Zhang, X.D.Sun, Z.H.Cheng, N.Minakawa and Y.Hamaguchi  
J. Phys. Soc. Jpn. 65 No.2 (1996) 348-350
2. Crystallographic and Magnetic Structure of  $\text{Er}_2\text{Fe}_{14}\text{Mn}_3\text{C}$  Studied by High-Resolution Powder Neutron Diffraction  
F.W.Wang, Q.W.Yan, P.L.Zhang, X.D.Sun, Y.M.Hao, Ridwan, Mujamilah,  
Gunawan, Marsongkahadi, C.Gou and D.F.Chen  
J. Phys.: Condens. Matter, 8 (1996) 741-744
3. A High-Resolution Neutron Study of  $\text{Y}_2\text{Fe}_{17-x}\text{Ga}_x$  ( $x=5,7$ )  
Q.W.Yan, P.L.Zhang, X.D.Sun, B.G.Chen, Z.H.Cheng, C.Gou, D.F.Cheng, Ridwan,  
Mujamilah, Gunawan, and Marsongkahadi  
J. Phys.: Condens. Matter, 8 (1996) 1485-1489
4. High-Resolution Neutron Study of  $\text{Y}_2\text{Fe}_{15}\text{Cr}_2$  at 77K and Magnetic Properties

- Y.M.Hao, P.L.Zhang, J.X.Zhang, X.D.Sun, Q.W.Yan, Ridwan, Mujamilah, Gunawan, and Marsongkahadi  
*J. Phys.: Condens. Matter*, **8** (1996) 1321-1324
5. High Resolution Neutron Powder Diffraction Study on  $\text{Ho}_2\text{Fe}_{17-x}\text{Si}_x$   
 Ridwan, Mujamilah, Gunawan, Marsongkohadi, Q.W.Yan, P.L.Zhang, X.D.Sun, X.M.Hao, N.Minakawa, Y.Hamaguchi,  
*Proc.of the 5th ASIAN Symposium on Research Reactor, Taejon, Korea;*  
 May 29-31, (1996), 573
  6. High Resolution Neutron Powder Diffraction of  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$  high Tc superconductor with lithium doping  
 W.Prasuad, S.Pongkasem and E.Sukirman :  
*Proc.of the 5th ASIAN Symposium on Research Reactor, Taejon, Korea;*  
 May 29-31, (1996), 507
  7. High resolution Neutron Diffraction Investigation on the Structure of  $\text{K}_2\text{O} \cdot 5.33(\text{Fe}_2\text{O}_3)$  Beta Alumina Type Superionic Conductors  
 A.K.Jahja, Gunawan, Marsongkohadi, V.S.Calix, N.R.Guillermo  
*Proc.of the 5th ASIAN Symposium on Research Reactor, Taejon, Korea;*  
 May 29-31, (1996), 566
  8. Neutron Structure analysis and magnetic properties of  $\text{Ho}_2\text{Fe}_{17-x}\text{Ga}_x$  ( $x=5,8$ )  
 Q. W. Yan, X. D. Sun, P. L. Zhang, B. G. Shen, F. W. Wang, S. Ridwan, H. Mujamilah, M. Ganawan , and P. Marsongkohadi  
*J. Phys.: Condensed matter* **9** (1997) 8553 - 8559
  9. Neutron Diffraction Studies of  $\text{Ho}_2\text{Fe}_9\text{Ga}_{8-x}\text{Al}_x$  ( $x=2,4$ ) at 50K and 300K  
 D.F.Chen, C.Gou, K.Sun, Ridwan, Mujamilah, Marsongkohadi, Q.W.Yan, P.L.Zhang, B.G.Shen, H.Y.Goug  
*J. Phys.: Condensed Matter* **10**, (1998), 255 - 260
  10. High concentration substitution of Ga and Al in  $\text{R}_2\text{Fe}_{17}$  (R=Ho or Y) :  
 A neutron study of crystallographic and magnetic structure  
 D.F.Chen, C.Gou, K.Sun, Q.W.Yan, P.L.Zhang, B.G.Shen, H.Y.Gong, Z.H.Cheng, Ridwan, Mujamilah and Marsongkohadi  
*Physica B*: **241 –243**,(1998), 640 – 642

## Comments on the "Ten Year's activity in the Field of Neutron Scattering"

### • The Philippines

The regional cooperative activities in the field of neutron scattering had contributed to the implementation of the Philippine's Program on R & D in high technology materials development:

1. The capability for structural analyses of material was enhanced through the training in the use of Rietan for data obtained by HRPD. Experience gained is also very useful for structural studies using x-rays.
2. The cooperative study on the superionic conductor with Indonesia had allowed Philippine researchers to conduct neutron diffraction measurement. The data obtained had contributed to the understanding of the material under study, although the planned experiments above room temperature is not materialized due to technical problems.
3. During this 10 years, a "bonding" among the region's researchers in neutron scattering was developed. In addition to the scientific and technical information exchange related to the 3 areas of cooperation, awareness and deeper understanding of the condition in the different countries were known enabling a better understanding between each other.

### • Korea

When this program was started in 1992, HANARO research reactor was under constitution, and neutron scattering group was planned to develop some instruments such as HRPD, FCD, NRF, SANS, etc. Korea got fruitful information of instrument design and fabrication, data analysis and application though this workshop. Especially during the commissioning of HANARO SANS, as shearing and/or comparing SANS data of some standard samples such as silver behenate, PS-PEP, Poly Ball etc., with SANS data of JAERI, ANSTO and BATAN as well as receiving a raw data treatment program and a IGOR software for data analysis from JAERI, the HANARO SANS was early stabilized to operate. SANS and HRPD in HANARO are operating well.

KOREA would like to have continuously collaboration with member states in this region on neutron beam application on nuclear and industrial subjects, instrument design and fabrication, and development of components for neutron beam instruments through the FNCA's support and bilateral collaboration, too.

### • Malaysia

This framework has benefited our neutron technology very much. Malaysian scientists participation almost in every workshop has made the subject matter a national agenda for the

last several years. Some projects in relation to this field have been funded by the government. The improvement of facility (MySANS) is one of the impact from this information dissemination and exchange obtained from the workshop and subworkshop technical discussion. The resolution of the workshop is another effective tool to create awareness for the department and ministry to comprehend and support this neutron technology related field. The field of materials science and technology research is indirectly enhanced through the unique capability of SANS in characterize structural information down to meso/nano-scale unit. This such small limit of probing power has made SANS as one of the candidates for nanomaterial study.

#### • Thailand

The benefit and improvement gain from 10-years workshop NS group at OEAP, Thailand has gained a lot of benefit and improvement from “Ten years activity in field of neutron scattering workshop” since 1992. We have already finished the installation of double axis neutron diffractometer in TRR-1/M1 (Triga Mark III) by receiving assistance from Japanese scientists. The diffractometer now can operate and get more improve we will have Mr. Minakawa who will have 2-week mission for installation of a new goniometer in the beginning of December 2001. For the improvement of the junior staff, we had sent them to Japan to train and get more experience in Japan, Indonesia, Korea and China.

For the publication, one Thai staff (Ms. Somchai Pongkaem) had named in Proc. Of 5<sup>th</sup> Asian Symposium on Research Reactor, May 29-31, (1996), p.502, that been held in Taejon Korea. The name of article is “High Resolution neutron Powder Diffraction of  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$  high Tc superconductor with lithium doping”.

#### • Vietnam

In fact, Vietnam was launched to ICNCA (FNCA now) program in 1995 and Vietnam neutron beam application group participated in the 1996 FNCA workshop in Jakarta, Indonesia as the workshop. Because there was no facility for neutron scattering study at their research reactor, at the 1996 workshop the participant from Vietnam engaged to participate in the production of and in study on high Tc superconductivity.

During last 4 years, Vietnam neutron diffraction group has studied and produced some kinds of high Tc superconductors, mainly of Pb and Sb doped BSCCO systems and did measurement of electrical resistivity depending on their temperature, as well as X-ray diffraction test. The obtained measurement data showed that they have got good results on their activity. Unfortunately, they have no neutron diffraction pattern yet.

Participating in the FNCA program, the staff of neutron beam application group in Vietnam has got good chances for exchanging information with other scientists of

participating countries and got experiences and knowledge in the field of neutron beam researches.

Based on the obtained experiences, in the forecoming time, neutron beam group on Vietnam could collaborate with other group, who carry out activities on condensed matter study on order to get more valuable results in the field of high Tc conductivity.

#### • China

During the ten-year cooperation in neutron scattering, Chinese scientists had take part in every workshop and made some researches on structure of magnetic materials. Especially, magnetic materials group in China Academy of Science has enough power to produce many kinds of ferromagnetic materials, which has been provided to the neutron scattering group in Serpong.

Through these cooperation they established the relationship between physical property and the structure of magnetic and promoted in formation exchange between Chinese scientists and other country's scientists. We also get the benefit from ten years workshop, particularly in cooperator SANS through practice operation we learn the method of collect data and treatment data.

#### • INDONESIA

##### NEUTRON SCATTERING 1992

The neutron scattering laboratory had just been inaugurated by the President in August 1992. It consisted of seven neutron beam instruments including 3 diffractometers, 3 spectrometers and a neutron radiography facility. These brand new instruments were built by the Japanese Consortium of Sumitomo. At that time there were very limited activities involving these instruments. The activities were mostly for characterization and calibration of these brand new instruments which were still under guarantees of the supplier. The man power at that time were very young in terms of their involvement with neutron scattering activities. They were fresh university's graduates with little experience in neutron scattering area but very eager to learn and to be exposed to neutron scattering activities using the brand instruments.

##### In the period of 1992-1999

The first seven workshops were held in Indonesia. They have given big opportunities to the young and fresh graduates in learning, practice and running neutron scattering instruments and research activities. On the job training for HRPD has improved the utilization of the instrument and capabilities of the man power in diffraction area, especially the ones related to functional materials such as magnetic, superconductor and superionic materials. Sub-workshop on SANS-Polymer has opened the avenue for other group in Serpong (out side

the neutron scattering group), especially the polymer group. This activities have increased the utilization and extended the capabilities of the SANS instrument towards the application in industrial areas which will have impact socially and economically.

#### NEUTRON SCATTERING 2001

Even though not all the seven neutron beam instruments are working right now, the ones, which have capabilities in the application to industry and have potential impact to the society are running well. They are neutron radiography, diffractometer for residual stress measurement and SANS instrument. The manpower are very much improved now academically, scientifically and have bulk of experiences in neutron scattering instruments and research activities. They have the capabilities of not only running the instruments but also maintaining, repairing and even developing and modifying the instruments towards better states. They also have the capabilities of performing research in the area of functional materials (magnetic, superconductor and superionic materials) and polymer science, especially the ones related to NR-TPE together with other polymer institutions in Indonesia.

#### CONCLUSION

There are many obvious benefits of having ten workshops in the field of neutron scattering. This foundation has been built by high amount of investments (fund, time, people, energy) and we cannot just let it disappeared and gone. We need to have and we will continue the activities in the region so the fruitful benefits will be prolonged and can be shared with the whole society economically.