RANS2-HUNS2
International Symposium
17 July-20 July 2018
At RIKEN and Hokkaido Univ.

RANS2

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RIKEN, Wako-shi, Saitama,

HUNS2

Dr. M.Ohnuma; ohnuma.masato@eng.hokudai.ac.jp
Hokkaido University, Sapporo,

(RIKEN symposium)
RANS2-HUNS2 Symposium Program
1st. day July 17th Tue. RIKEN Wako-campus Cooperation Center (S41) room W319,321

9:30～9:40 Opening remark
Dr. Yoshie Otake
Neutron Beam Technology Team, RIKEN

9:40～10:25 RIKEN Accelerator-driven compact neutron sources RANS, RANS2 and applications
Dr. Yoshie Otake
Neutron Beam Technology Team, RIKEN

10:25～11:10 Present Status and Future plan of HUNS-II
Dr. Masato Ohnuma
Hokkaido University

11:10～11:35 Development of cold neutron source using methyl-benzene derivatives for compact neutron source
Dr. Shin Takeda
Ultrahigh Precision Optics Technology Team, RIKEN

11:35～12:40 Lunch

12:40～13:10 RANS technical tour (at S22 Neutron Application Facilities)

13:10～13:45 X-ray and neutron sources for materials science - competitive or complementary?
Dr. Gernot Kostorz
ETH Zürich
13:45〜14:10  Development of transportable neutron source prototype RANS-II
Dr. Tomohiro Kobayashi
Neutron Beam Technology Team, RIKEN

14:10〜14:30  Development of nondestructive technique for salt distribution measurement in structural concrete by PGA at RANS
Dr. Yasuo Wakabayashi
Neutron Beam Technology Team, RIKEN

14:30〜14:50  Estimation of concrete deterioration observed with neutron imaging of water penetration at RANS
Mr. Yuichi Yoshimura
Neutron Beam Technology Team, RIKEN

14:50〜15:10  Coffee Break

15:10〜15:45  Development of the neutron source in Korea
Dr. Eun Je Lee
KAERI

15:45〜16:20  NOVA ERA - A compact neutron source for universities
Dr. Thomas Gutberlet
Jülich Centre for Neutron Science JCNS

16:20〜16:30  Closing remark
Dr. Yoshie Otake
Neutron Beam Technology Team, RIKEN

16:30〜17:20  RANS2 technical tour (at C21 Engineering Bldg.)
18:00～  Get-together party (at S22 Neutron Application Facilities)

2nd. day July 18th Wed. Move to Sapporo, Hokkaido
3rd. day July 19th Thu.  Hokkaido University, Faculty of Engineering, A1-17, "Quantum Beam for Materials Science"

10:00～10:05  Opening remark  
Prof. Yoshiaki Kiyanagi  
Professor emeritus of HU

10:05～10:45  Microstructure characterisation in industry  
- Synergy between scattering and microscopy -  
Dr. Kaoru Sato  
JFE Techno-Research Corporation

10:45～11:25  Combined scattering techniques in the study of metallic materials  
Prof. Gernot Kostorz  
ETH Zurich

11:25～12:00  Neutron Source for Materials Science  
Prof. Michihiro Furusaka  
AIST & Hokkaido University

11:55～13:15  Lunch

13:15～13:55  Nanoscale characterization of modern materials by microscopy  
- possibilities and limitations  
Prof. Malgorzata Lewandowska  
Warsaw University of Technology

13:55～14:30  Present status of Multi-Quantum Beam HVEM and its challenge to operand observation in liquids  
Prof. Tamaki Shibayama  
Hokkaido University
14:30~14:45  Coffee Break

14:45~15:20  Phase and inner structure of submicrometer spherical particles fabricated by pulsed laser melting
Prof. Naoto Koshizaki
Hokkaido University

15:20~16:00  Recent progress in the characterization of industrial alloys by SANS at ORNL
Dr. Ken Littrell
Oak Ridge National Laboratory

16:00~16:30  History of HUNS
Prof. Yoshiaki Kiyanagi
Nagoya University (Emeritus Prof. Hokkaido University)

16:30~17:30  HUNS-II Technical Tour

17:30~  Get-together party (Faculty house Trillium "Restaurant Elm")

4th. day  July 20th Fri.  Hokkaido University, Faculty of Engineering, A1-17

9:30~10:10  Further Application of Small-Angle Scattering
Prof. Masato Ohnuma
Hokkaido University

10:10~10:45  Rheology and function of foods with extremely complicated nano-structure
Prof. Isamu Kaneda
Rakunou Gakuen University
10:45～11:00  Coffee Break

11:00～11:35  Nano-scale microstructure in metal-based materials to be investigated
Prof. Seiji Miura
Hokkaido University

11:35～12:10  Investigation on the mechanism of magnetization reversal during the demagnetization process for NdFeB sintered magnets
Prof. Kozo Osamura
Research Institute for Applied Science

12:10～13:20  Lunch

13:20～13:50  Neutron Bragg imaging at HUNS
Dr. Hirotaka Sato
Hokkaido University

13:50～14:20  Research for neutron application using compact accelerator source
Prof. Takashi Kamiyama
Hokkaido University

14:20～14:35  Wrap-up
The Tokyo Metro subway system offers through trains to Kawagoe-shi station on the TOBU Tojo line, and Hanno station on the SEIBU Ikebukuro line. Please make sure to take a train leaving for Wako-shi & TOBU Tojo line.
RIKEN Wako campus, conference site S41 bldg

Contact Information: Tomohiro Kobayashi (RIKEN) 090-1661-0885, t-koba@riken.jp
Sapporo site: faculty of Engineering (red arrow), 1st floor A1-17
RIKEN Accelerator-driven compact neutron sources RANS, RANS2 and the applications

Yoshie OTAKE


Neutron Beam Technology Team, RIKEN Center for Advanced Photonics, RIKEN

2-1 Hirosawa, Wako-shi, Saitama, 351-0198, Japan

A compact neutron source by using a particle accelerator is a promising tool for actual material analysis, infrastructural diagnostics, nuclear detection, and medical treatment. We have been operating the neutron source RANS (RIKEN Accelerator-driven compact Neutron Source) with 7 MV proton LINAC with a beryllium long-life target for 6 years [1,2,3]. There are two major goals of RANS research and development. One is to establish a new compact low energy neutron system of floor-standing type for industrial use. Another goal is to invent a novel transportable compact neutron system for the preventive maintenance of large scale construction such as bridges and airports using a higher energy neutron beam of about 500 keV [1]. In recent years, imaging with fast and thermal neutrons [4], engineering diffraction for texture evolution estimation and austeninte volume fraction estimation for iron and steel [4], prompt-gamma neutron analysis (PGA) are the major activities [1]. The research and development results with RANS fast neutron imaging methods for large scale concrete structures revels that the requirement for the neutron yield is less than $10^{11}$ neutrons s$^{-1}$ especially for out-door type compact neutron source. Based on it, RANS2 has been designed and developed with 2.49MeV proton linac [5]. The potential of accelerator-driven compact neutron source will also be discussed.


Present Status and Future Plan of HUNS-II

M. Ohnuma, T. Kamiyama, H. Sato, M. Furusaka
Hokkaido University
E-mail: ohnuma.masato@eng.hokudai.ac.jp

After 40 years use, electron LINAC for Hokkaido University Neutron Source (HUNS)-I has shut down in last October. New LINAC is funded by Innovative Structural Materials Association (ISMA) and New Energy and Industrial Technology Development Organization (NEDO) in the project for developing innovative structural materials. It is probably the first neutron source that is funded for the industrial use as the first priority.

Our main targets for the project are characterization of microstructures with small-angle neutron source (SANS) and Bragg Edge Measurements. We will show the results obtained by both techniques till last year in this talk. About SANS, I will show the example that strongly indicates the merit for using neutron to steel research. New LINAC is now under commissioning and we will show the future plan to use this HUNS-II for industrial applications.
The development of a methyl benzene based cold neutron source at RIKEN

S. Takeda¹, Y. Yamagata¹, T. Hosobata¹, T. Kawai¹, M. Takeda¹, Y. Otake¹, T. Kobayashi¹, A. Taketani¹, Y. Wakabayashi¹, S. Uno²

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The RIKEN accelerator-driven neutron source (RANS) is a compact neutron source which aims to widely spread the use of neutron beams in the industrial field. To provide a wider variety of neutrons for applications such as small angle scattering, reflectometry and Bragg-edge measurements, a cold neutron source is being developed for RANS using methyl benzene derivatives. As the first trial, we used mesitylene as the moderator material for its non-explosive characteristics and rather long maintenance cycle. The mesitylene was sealed in an aluminum cavity with and cooled to 20K. The dimensions of the mesitylene inside the cavity were 100 mm x 100 mm x 2.5 mm. Calculations were done using “PHITS”, a beam transporting simulation software based on Monte Carlo code. The scattering kernel was provided by Dr. Granada. The performance estimation, pre-moderator and moderator thickness optimization, and pulse characteristics of different types of moderating systems were calculated. An installation experiment was operated using a prototype moderator unit at RANS and the wavelength spectrum, pulse width was measured. Figure 1 shows the TOF spectrum of the mesitylene moderator measured 5 m apart from the moderator surface. A neutron focusing test using an ellipsoidal mirror and a Bragg edge measurement test was also operated and showed rather promising results. Figure 2 shows the transmission of a steel sample measured using the mesitylene moderator.

![Figure 1: TOF spectrum of the mesitylene moderator at 5 m from the moderator surface.](image1)

![Figure 2: The transmission of a steel sample measured using the cold source at RIKEN.](image2)
The use of X-rays to study the structure and microstructure of materials has a long history. In fact, X-ray methods constitute one of the major pillars on which Materials Science was built as a discipline. Thermal neutrons, initially the domain of condensed matter physicists who used neutron beams at nuclear reactors, gained major interest for materials science issues only after large facilities had opened their gates to external users and user programs were established around the 1970s. For some time, neutron and X-ray methods were seen as competing with each other, but their complementarity was soon realized and intelligently used.

Since then, X-rays at dedicated synchrotron radiation sources have provided an ever-increasing reservoir of new applications in materials research, but the complementarity of X-rays and neutrons is, of course, a basic physical fact and remains a useful and productive asset. This has become particularly evident at places where both synchrotron radiation and neutron sources are operational in close neighborhood and may in part rely on joint facilities or even committees. Examples are the Grenoble (France) institutions ILL and ESRF or the Swiss facilities at the Paul Scherrer Institute (PSI) in Villigen (SLS and SINQ). Apart from the possibilities offered by the availability of both types of radiation for research, scientists meeting around these sources find it interesting and inspiring to exchange their views and to extend their knowledge by interacting with colleagues from various domains.

The talk will illustrate these points.
Development of transportable neutron source prototype RANS-II

Tomohiro KOBAYASHI 1, Yoshie OTAKE1, Yujiro IKEDA1, and Noriyosu HAYASHIZAKI2
1) RIKEN Center for Advanced Photonics, Japan
2) Laboratory for Advanced Nuclear Energy, Tokyo Institute of Technology, Japan
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RANS-II (RIKEN Accelerator Compact Neutron Source -II) project has started to develop a prototype of a transportable compact accelerator neutron source, which is primarily used for diagnostics of aged infrastructures. RANS-II aims to confirm the performance of accelerator, target, shielding, and instrumentation for measurement. Lithium was chosen as a target material for neutron production via the $^7\text{Li}(p,n)^7\text{Be}$ reaction, and the incident proton energy was determined to be 2.49 MeV. The specifications of RANS and RANS2 are shown in Table 1. Angular dependent neutron spectra at the Li target was calculated by the PHITS Monte Carlo particle transport code with ENDF/B-VII.0. According to the calculation, the neutron emission rises rapidly at forward angles, which exhibits a neutron flux peak in the energy from 500 keV to 700 keV with a maximum energy of about 800 keV (Fig.1). The total neutron flux (100 µA of incident proton) at 1 m distance from the target is estimated to be 1.65 x 10^5 cm^-2 sec^-1. This number suggests that the neutron transmission or reflection imaging technique could be applicable for up to 300 mm thickness in concrete. The pulsed 2.45 GHz microwave ECR ion source and the RFQ-LINAC are in operation tests for confirmation of design parameters. Power injection test for RFQ, which should be equivalent to the 2.49 MeV design value, at a low-duty was completed. The proton current at a pulse peak is 1.2 mA. The target station shielding, the lithium target with a cooling system, neutron extraction hole, and HEBT are in conceptual and engineering design phases. From the compactness point of view, we have obtained preliminary results for RANS-II, which indicate significant reductions in terms of both weight and size in comparison with those of RANS.

Table 1: Specification of RANS and RANS2

<table>
<thead>
<tr>
<th></th>
<th>RANS</th>
<th>RANS2</th>
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</thead>
<tbody>
<tr>
<td>Particle</td>
<td>Proton</td>
<td>Proton</td>
</tr>
<tr>
<td>Energy</td>
<td>7 MeV</td>
<td>2.49 MeV</td>
</tr>
<tr>
<td>Current</td>
<td>100 µA</td>
<td>100 µA</td>
</tr>
<tr>
<td>Reaction</td>
<td>$^9\text{Be}(p, n)^9\text{B}$</td>
<td>$^7\text{Li}(p, n)^7\text{Be}$</td>
</tr>
<tr>
<td>Accelerator</td>
<td>RFQ + DTL</td>
<td>RFQ</td>
</tr>
<tr>
<td>Weight(Accelerator)</td>
<td>5 t</td>
<td>3 t</td>
</tr>
<tr>
<td>Weight(Target&amp;Shield)</td>
<td>20 t</td>
<td>&lt; 0.7 t</td>
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<tr>
<td>Length</td>
<td>15 m</td>
<td>&lt; 5 m</td>
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Figure 1: Energy distribution of neutrons of $^7\text{Li}(p,n)^7\text{Be}$ with 2.49MeV proton at a distance of 10 cm calculated using PHITS code.
Development of nondestructive technique for salt distribution measurement in structural concrete by PGA at RANS

Yasuo WAKABAYASHI 1, Yuichi YOSHIMURA 1,2, Maki MIZUTA 1, Yoshimasa IKEDA 1, Takao HASHIGUCHI 1, Atsushi TAKETANI 1, Tomohiro KOBAYASHI 1, Makoto Goto 1, Hideyuki SUNAGA 1, Yujiro IKEDA 1,3, and Yoshie OTAKE 1

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2) Tokyo Institute of Technology, Japan
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Many concrete structures such as bridges and roads are exposed to seawater and deicing agents spreading in cold and mountain areas, and salt damage due to chloride ions in concrete is a serious issue. Steel corrosion starts when the chloride ion concentration on the surface of the steel bar in the concrete exceeds the marginal concentration of 1.2-2.5kg/m³ depending on the type of cement and the water cement ratio [1]. Therefore, in view of the maintenance for concrete structures, it is essential to measure the salt distribution towards the depth direction from the surface of the structure and to estimate the progress of steel corrosion.

Although there are some reliable and accurate conventional methods for salt distribution measurement, these methods need core sampling from structures, they take time and cost for pre-measurement processing and the locations for core sampling are limited. In view of efficient maintenance to solve such issues, nondestructive testing technique is required. Then, we started research and development aiming for practical use of nondestructive testing technique of salt distribution measurement in concrete structures using Neutron-capture Prompt Gamma-ray Analysis (NPGA).

As the first step of the development, the RIKEN Accelerator-driven compact Neutron Source, RANS [2, 3] is used to evaluate the detection limit of salt concentration under our current conditions by measuring a sample of mortar with an adjustment of its salt concentration. The result of the measurement identifies a salt concentration of 1 kg/m³ or less, which is lower than the limit of the concentration to involve steel corrosion. A technical examination is also performed to evaluate a method of detecting salt distribution in the depth direction.

In this conference, we will report the applicability of NPGA to the nondestructive testing technique.

References
Estimation of concrete deterioration observed with neutron imaging of water penetration at RANS

Yuichi Yoshimura 1,2,3, Maki Mizuta1, Hideyuki Sunaga1, Yoshie Otake1, and Noriyosu Hayashizaki2
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Deterioration of concrete mainly relies on water. Since the water content or water distribution in concrete may affect the deterioration process of the concrete, it is possible that the measurement of water in concrete will be useful for evaluating the durability of the concrete.

Concerning the measurement of water content of concrete, embedded-type moisture sensors have been used. This method is easily chosen, whereas it is semi-destructive one. Therefore, the continuous change cannot be followed by less information and it is difficult to confirm the reproducibility of the experimental results.

Considering the reasons mentioned above, we adopted neutron beam with high transmissivity for concrete and sensitivity for hydrogen as a probe of measuring the water content of concrete. RIKEN accelerator-driven compact neutron source, RANS was used as a neutron generator and the water penetration of concrete was visualized by neutron imaging. In this research, the water absorption tests of concrete were conducted and the water contents were measured by neutron transmission imaging at fixed intervals.

In this symposium, we report the results obtained from evaluation for eight days by neutron imaging with RANS about water penetration in a deteriorated concrete specimen.

(1) We performed neutron imaging on 5cm-thick concrete, and the successful relationship between neutron transmissivity and water content for quantification was confirmed.

(2) The progress of water content and water penetration varied by the degree of concrete expansion. These results suggest that water penetrability is influenced by the degree of deterioration.

References
Development of neutron sources in Korea

Eun Je Lee, Min Goo Hur, Chang Hee Lee

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Abstract

Neutrons have been used in various applications such as neutron radiography, boron neutron capture therapy (BNCT), small angle neutron scattering (SANS), and so on because of their unique physical property and interaction behavior. Neutrons extracted from research reactors have been conventionally utilized for these purposes. In addition, R&D of compact accelerator-driven neutron sources, as a relatively inexpensive and alternative neutron sources, has been intensively performed.

In this presentation, we show research activities relating to the development of neutron sources in Korea. Research about neutron sources based on cyclotron, proton LINAC, and electron LINAC is mainly being carried out in national research institutes including KAERI, IBS, KBSI, and so on. Furthermore, an attempt to establish KCANS network among related researchers is ongoing.
Neutron scattering has proven to be one of the most powerful methods for the investigation of structure and dynamics of condensed matter on atomic length and time scales. A severe drawback in using neutrons is the limited possibilities to access neutrons offered via nuclear research reactors or accelerator driven spallation sources, which are costly to build and to operate. To offer neutrons more easy accessible for science, training and industrial use is a challenge. The concept of a compact accelerator based neutron source is a new approach to tackle this challenge with the aim to bring neutrons to the users on demand and cost effective.

Compact accelerator based neutron sources (CANS) produce neutrons by the nuclear reaction between a low energy proton beam and light elements as beryllium or lithium. Depending on the power of the accelerator and the number of target stations and instruments such a source can be equivalent to small and medium flux reactor or spallation based neutron sources. With the aim to design CANS to be operated at universities, research institutes or industry laboratories a conceptual design report has been developed at JCNS for a small neutron source named NOVA ERA (Neutrons Obtained Via Accelerator for Education and Research Activities). Such a neutron source can be built at low cost with low maintenance efforts and without nuclear licencing procedure as small accelerator facility. Main features of this new concept will be presented and discussed.
Microstructure characterisation in industry
—Synergy between scattering and microscopy—

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We have been using microbeam analysis in industry to understand property/microstructure relationships\(^1\). Both transmission electron microscopes (TEM) and scanning electron microscopes (SEM) enable direct visualisation of microstructure. Thus, they play a key role in materials characterization.

However, they lack representativeness due to their limited observation volume. Thus X-rays and neutrons are utilised complementarily. Nanometer-scale information from a "bulk" specimen can be obtained using analysis using neutrons due to their strong penetration.

The small-angle neutron scattering (SANS) method allows a determination of nanometer-sized precipitates including TiC\(^2\) that are utilised to strengthen steels. The volume of analysis for SANS was about 10\(^8\) times larger than that used for ordinary TEM observation. This non-destructive technique is particularly useful when chemical extraction methods are difficult to apply due to a chemical instability of precipitates. Apart from SANS, we are using neutron diffraction for the analysis of residual stress and retained austenite.

Neutron-based analysis using compact sources is being pursued in Japan\(^3\). We expect that compact neutron sources will allow easy access to neutrons thus contributing to better understanding of materials’ microstructure. They will be useful for prolonged in-situ measurements or preliminary small-scale measurements before experiments at a large-scale facility such as J-PARC. We are indeed entering the days when a combined use of particle beams and high resolution direct-imaging techniques will lead material design by giving precise and quantitative structural information\(^4\).

References
2   H. Yasuhara et al, Tetsu to Hagane (In Japanese), 96(2010), 545.
3   e.g. Riken YouTube, Neutrons Transforming Japanese Manufacturing.
Combined scattering techniques in the study of metallic materials

Gernot Kostorz
ETH Zurich, Department of Physics, Zurich, Switzerland

Materials science has often to deal with inhomogeneous, i.e., nano- and micro-structured condensed matter. Thus, imaging techniques to resolve the local arrangement of atoms, molecules and larger agglomerates are required on all conceivable length and time scales. Apart from visible light, X-rays, neutrons and electrons are suitable probes. X-rays have been helpful since their discovery, especially for the study of metallic materials, but their relatively high energy with respect to solid-state dynamics and their high absorption impose some limitations even at today’s synchrotron radiation sources. Thermal neutrons may often have a higher penetration power and better energy resolution in scattering experiments, but the spatial resolution is limited, especially owing to the low power of neutron sources. Taking advantage of the special features of both types of radiation and their basically different interaction with condensed matter is often useful if not required to obtain a more complete view of materials structures, microstructures, properties and processes. Some examples will be discussed where issues of local order, clustering, precipitation upon diffusion controlled phase separation and structural phase transformation have benefitted from the combination of scattering techniques using X-rays and neutrons, where feasible supported by electron microscopy.
Neutron Instruments Tailored to Compact Accelerator Driven Neutron Source for Material Science

Michihiro Furusaka
AIST (National Institute of Advanced Industrial Science and Technology)
and Faculty of Engineering, Hokkaido University
Nanoscale characterization of modern materials by electron microscopy - possibilities and limitations

Malgorzata Lewandowska
Warsaw University of Technology, Faculty of Materials Science and Engineering
Woloska 141, 02-507 Warsaw, POLAND

Nanoscale materials play an increasing role in materials science and engineering, as they are enabler for high-tech products. The improved understanding of structure-property relationships of new materials are essential for their applications in many branches. Basic research is needed to investigate structure and properties of advanced materials on scales from product dimensions down to the atomic level. Nowadays, nanoanalysis is more and more needed for process and materials characterization during manufacturing of nanostructured systems and devices as well as for the understanding of nanoscale microstructure in materials.

In this context, modern electron microscopy techniques emerged as powerful and versatile methods enabled for in-depth characterization of materials’ microstructure at different length scales in imaging, diffraction and spectroscopic modes. However, electron microscopy techniques gives very local information. To have more global picture, the combination with other techniques such as X-ray and neutron scattering is required.

In this communication, the possibilities and limitations of electron microscopy techniques will be discussed for nanostructured materials (e.g. nanolayers and composites with embedded nanoparticles). The advantages of the combination of techniques for better understanding materials properties and performance will also be presented.
Present status of Multi-Quantum Beam HVEM and its challenge to operand observation in liquids

Tamaki Shibayama¹,² and Seiichi Watanabe¹,²
¹Faculty of Engineering, Hokkaido University,
²HVEM laboratory, Hokkaido University

As increasing the needs of high resolution studies from the academic and industrial societies, 3rd generation High Voltage transmission Electron Microscope as hereafter defined HVEM (ARM-1300, JEOL Ltd.) with two ion accelerators was started to construct from 1995 and finally successfully installed in March, 1998. It was called as Multi-Beam HVEM and used for quite broad research field in the world. Its point-to-point resolution is 0.117nm. Multi-Beam HVEM enables in situ observations at atomic scale under ion irradiation with two different ion species. After installation of the Multi-Beam HVEM, various microscopes and specimen preparation equipment have been running in Laboratory of High-Voltage Electron Microscope, Hokkaido University. As one of joint use facilities in Hokkaido University, our HVEMs contribute to cutting edge research activities to researchers for not only inside Hokkaido University but also outside Hokkaido University including private companies. In addition, our HVEMs play a roll of share use with supporting by the Minister of Education, Culture, Sports, Science and Technology (MEXT) and our HVEM laboratory is providing research opportunities for not only domestic but also international researchers. After 10 years operation, we started to renovate the Multi-Beam HVEM by adding one more laser beam line. This idea was extremely unique and it could be realize to observe photo induced phenomena by using liquid cells. Finally, we successfully renovated the 3rd generation HVEM to the 3.5th HVEM. As a result of this renovation, we can do in-situ experiment to observe microstructure evolution under controlling three kinds of quantum beam such as high energy electron beam, ion beam and laser beam at once. Representative of all elementary particle systems, namely photon, electron and ions will become one set for spectroscopic analysis of materials.

We are now challenging to operand observation in liquids. Recently, we successfully evaluated photo induced phenomena by in-situ observation of ZnO nano rods with CW He-Cd laser in liquid environments. [1] Our preliminary study revealed in-situ direct observation of photo corrosion in ZnO crystals in ionic liquid during ultraviolet irradiation in atomic scale by MQB-HVEM for the first time. This study also revealed that Zn and O atoms at the edge of the ZnO nanorods intended to dissolve into the ionic liquid. It was found that the polarity and facet of the nanorods were strongly related to photo corrosion and crystal growth [1].

The above studies were partly supported by the “Nanotechnology Platform” Program of MEXT, Japan. The authors would like to thank Dr. J. Ishioka, Dr. R. Yu, Mr. K. Ohkubo, Mr. T. Tanioka and Mr. R. Oota for their helpful discussions and cooperation.

Phase and Inner Structure of Submicrometer Spherical Particles Fabricated by Pulsed Laser Melting in Liquid

Naoto Koshizaki
Division of Quantum Science and Engineering
Hokkaido University

Our group has been intensively working on pulsed laser melting in liquid (PLML) for crystalline submicrometer spherical particle fabrication for a decade. These particles are quite interesting since the particles having submicrometer size are usually shapeless and have broad size distribution, except for some amorphous particles such as polymer and glass particles commercially available. In this presentation, we focus on the formation mechanism of submicrometer spherical particles and their inner structure analysis by various characterization techniques. Mechanical property of these particles is also described [1].

Recent progress in the characterization of industrial alloys by SANS at ORNL

Ken Littrell
Oak Ridge National Laboratory

In many alloy systems, the precipitation and growth of minority phases in the matrix at nanometer length scales play a major role in mediating the ductile-to-brittle transition. Thus, the size and distribution of these minority phases and how this changes in response to heat treatment or environmental service conditions is strongly related to the performance characteristics of these materials. Small-angle neutron scattering (SANS) is a powerful technique for probing the bulk, averaged structural properties of alloys on length scales ranging from less than one to hundreds of nanometers on a quantitative basis quickly and non-destructively; this capability is enhanced by the high transparency of many structural materials to neutrons, allowing the experiments to be performed in-situ under extreme conditions. For iron-based alloys, the neutron’s magnetic scattering cross-section provides additional information via this additional contrast variable. This presentation summarizes recent work from the ORNL neutron scattering user program using the CG2 GP-SANS at HFIR to characterize the response of the microstructure of ferrous and non-ferrous alloys to radiation damage, thermal aging, and in-situ heat treatment both for hardening and annealing of defects.

This research used resources at the High Flux Isotope Reactor, a DOE Office of Science User Facility operated by the Oak Ridge National Laboratory.
History of HUNS

Yoshiaki Kiyanagi
Emeritus Prof. Hokkaido University
Designated Prof. Nagoya University

The Hokkaido University electron linac was competed in 1973 after three years construction period. Before completion, namely at low energy acceleration stage, the linac was already used for measurements of neutron spectra from cold neutron moderators. After completion, this was used for pulse radiolysis, irradiation, neutron and other applications. Here, I would like to briefly introduce activities about neutrons.

At the beginning, cold moderator development was performed. First, we developed a methane cold moderator(1), which showed the best performance as a cold moderator at a pulsed neutron source. After then improvement of the neutronic performance and development of a new moderator for J-PARC were performed. From these study we measured many kinds of moderator materials and developed a coupled moderator(2). In parallel, we constructed a quasi-elastic spectrometer(3), which was transferred to KENS neutron source at KEK. Neutron focusing using a magnetic device was succeeded at the first time in the world(4) and a highest counting rate imaging detector was developed(5). Furthermore, pulsed neutron imaging has started at HUNS. The method is effective to obtain quantitative 2D information on crystal structure, magnetic field, elements, temperature(6). Based on this development, an imaging instrument RADEN was constructed at J-PARC.

About industrial applications, the world first soft error acceleration test using a compact neutron source was performed at HUNS(7), and now world standardization of this kind of test for telecommunication system is under progress.

After the upgrade of the linac, various researches are expected to be more active.

Further Application of Small-Angle Scattering

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One of the most important requirements for small-angle scattering (SAS) measurements in the field of metallurgy is high penetration rate of the beam through the samples in transmission geometry. For this reason, Synchrotron Radiation facilities or neutron facilities are used in the most of the recent SAS experiments. They are, of course, very powerful and give us high quality SAS profiles with wide q-region by short measurement time. However, obtaining beam time is very competitive because SAS can use in many different fields such as protein research etc. This is the barrier for challenging new topics and increasing the number of SAS users in our fields.

In addition, the performance of SAS instrument is often evaluated by "low-q limit". It is important in the field of soft-matter but it is not always the case in the metallurgy where we can use transmission and scanning electron microscope (TEM and SEM). Evaluation of a few ten nm is relatively easy using them. On the other hand, the nanostructure with single nanometer size embedded in matrix is often difficult especially in the case they have coherent structure with the matrix.

From these points of view, we have promoted to use laboratory SAXS with Mo since 1997 [1] and focused to characterizing the hetero-structure with single nanometer size. We found a certain number of the examples that show formations of nanostructures before obtaining clear image in TEM. These phenomena are very interesting but they bring us new task; How can we get the information of the phase only by SAS? One of the possible solutions is combination of neutron and X-ray [2]. For using the combination frequently, we need "laboratory scale small-angle neutron scattering (SANS)". To my surprise, laboratory SANS is now almost realized in Hokkaido University [3]. It may bring us new world such as "nano-characterization without special sample preparation". It is now easier to measure SANS than observing optical microscope for us.

Once we have two source, X-ray and neutron, we can always choose optimum one. For metals and alloys, SANS is better especially preventing double diffraction problem. But some applications with including water, SAXS is more suitable for observing nanostructure. In such case, Mo-SAXS is very powerful because we can use flat cell for liquid samples, meaning that we can keep same geometry for both liquid and solid in our labo-SAXS. This opens new applications, "solidification process" in many system, such as foods, cements, and more. Structures with single nanometer exist everywhere! They are waiting for characterizations...

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Rheology and function of foods with extremely complicated nano-structure

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Most foods can be considered as condensed colloid systems. For example, cheese is consists of 3D-network structure made with casein, and fat particles, and mayonnaise is o/w emulsions that have very high inner phase ratio. Physical characteristics of the foods particularly the rheology properties are strongly related to texture, and the evaluation of foods texture is important issue for understanding consumer’s demand. However, it is quite difficult to analyze the rheological properties of such condensed colloid systems due to several reasons. If we fortunately obtain a suitable constitution equation for such condensed colloid systems and are able to describe the properties in a minimum characteristic parameters, we have to try to demonstrate the molecular level evidence for the rheological properties. In other words, we always consider a correlation of rheological properties and micro or nano-structure of foods. We can employ two ways to observe the micro or nano-structure of foods, namely they are real-space and reciprocal-space observation. One of the typical methods of the real-space observation is electron micro scope (EMS). However, this method have some issues. Because foods generally contain large amount of water, the specimen of EMS have to be dry up therefore it is impossible to observe “wet state of foods” with EMS. On the other hand, scattering methods have big advantages for observation of the micro or nano-structure of foods. In particular, we can observe even opaque sample using SAXS. In this talk, as examples, the rheological properties of cheese and mayonnaise are introduced. There are vast interfaces in these foods and the flow behavior is strongly affected by the properties of the interfaces. It expected that if we can observe the nano-structure of these interfaces, we can understand reasons for the complicated flow behavior of Foods. We should discuss the need of SAXS for foods soft matter studies.
Nano-scale microstructure in metal-based materials to be investigated

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Between TEM and SEM observations, there is a broad size range of microstructure to be investigated in materials science. For the strengthening of Al-based and Fe-based materials, nano-meter-scale structures such as clusters and meta-stable carbides play important roles, while the details of their structure and compositions are not fully understood yet. Newly proposed alloys such as high entropy alloys also may have some unknown nano-scale structures, although it is thought to be an homogeneous solid solution, together with multi-element intermetallic compounds in which complicated site-occupancy tendency is expected. LPSO phase in Mg-Zn-Y alloy provides an improved strength to Mg-based alloy, however, the allying behavior in the phase is not fully investigated.

From this point of view, we would like to introduce some examples related to the nano-scale structures to be investigated for a further development of alloys and our understanding on materials physics and chemistry.
In order to explain the excellent magnetic property of NdFeB sintered magnets, historically two mechanisms have been proposed. One of them is called as a nucleation model proposed by Kronmuller. Another is a magnetic domain wall motion model proposed by Givord. On the other hand, with respect to the coercive force, when it is assumed that the magnetic reversal takes place independently in each grain according to the $1/\cos\theta$ dependence with magnetic field, the calculated alignment dependence of coercive force does not agree with the experimental result. This suggests that the magnetic reversal takes place cooperatively together with several neighboring grains. The present report explains this situation. At first, the sample is fully magnetized under the positive magnetic force field ($0<H$), where each grain is magnetized around the easy magnetization direction. At a perfect demagnetization ($H=H_{cJ}$), the negative and positive magnetic domains cancel magnetically each other and the magnetization becomes zero. So, the main purpose is to explain how the magnetization reversal takes place in the intermediate reverse magnetic force region ($H_{cJ} < H < 0$).
Neutron Bragg imaging at HUNS

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Neutron imaging using Bragg-edge transmission (polycrystal) analysis and Bragg-dip transmission (single crystal or large crystalline grains) analysis is a very powerful quantitative analysis and visualization tool for crystalline phases, crystallographic textures, crystallite sizes, macro/micro-strains and grain orientations in a material [1]. Because this method can be well performed at accelerator-driven pulsed neutron sources, various experimental and analysis methods for this method were effectively developed at the Hokkaido University Neutron Source (HUNS) which is a compact electron LINAC driven pulsed neutron source. Then, these new technologies were exported to the J-PARC MLF neutron facility which is a world-largest pulsed spallation neutron source driven by 1 MW proton accelerator. In addition, recently, industrial applications of HUNS are gradually important for promotion of utilization of neutron experiments. About both sides (neutron technology developments and industrial applications at a compact-accelerator driven neutron source), recent activities of neutron Bragg-edge/dip imaging at HUNS will be presented. The topics are:

- Development of the Rietveld-type Bragg-edge analysis software, and the first demonstration of quantitative imaging of crystallographic texture and crystallite size (welded/rolled steel) [2]
- Development of a new neutron time-of-flight (wavelength analysis) imaging detector using a high-speed camera, and high spatial-resolution and large field-of-view crystalline phase imaging (steel knife)
- Development of the high wavelength-resolution neutron beam-line, and martensite ($d$-spacing shift and broadening) imaging and grain orientation imaging (quenched ferritic steel and electromagnetic steel) [3]
- Imaging of position dependence of crystal lattice plane spacing (electric charge level) of graphite anode in a lithium-ion battery [4]

Research for neutron application using compact accelerator source

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Compact accelerator neutron sources are useful for developing new techniques of neutron applications. The Hokkaido University Neutron Source (HUNS) is one of the pioneers for such role, and gains a lot of achievements. In this presentation, some new topics at HUNS are introduced.

The first is the remote temperature measurement of mechanical part under operation by prompt gamma-ray type neutron resonance absorption spectroscopy (PG-NRAS). Though the shape of the resonance absorption peaks are affected by the influence of atomic thermal vibration, the temperature of the nuclide can be analyzed by PG-NRAS remotely and nondestructively [1]. Applying this technique, we set a resonance nuclide foil on a rotor inside an electric motor, and carried on the PG-NRAS thermometry to the moving motor. The analyzed result of the resonance peak showed the reasonable coincidence with the thermo-label measurement within several kelvins.

The second topic is the improvement of neutron color image intensifier detector (CNII). For neutron radiography under low flux of the compact accelerator neutron source, it is necessary to apply the long exposure time or accumulate the many number of shot frames. In such case, the long-time and high-sensitivity noises have to be noticed in addition to the radiation noise. Therefore, we developed a compact CNII detector system with the long-time stability under less noise in addition that it can be used for mobile use.

The third is the synergy imaging which obtains a nuclide distribution image with higher spatial resolution using the differences between cross sections of neutron and X-ray [2]. The concept of the synergy imaging is developed from the image alignment technique using the mutual information (MI). For making a 3D volume model, we assumed the use of X-ray computer tomography (CT) technique, and the neutron radiograms which were taken along only orthogonal three directions. This procedure has a great advantage for the 3D model construction with neutron, because the number of the neutron radiogram measurements is reduced greatly.