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CURRENT STATUS OF TAKUMI THE ENGINEERING MATERIALS DIFFRACTOMETER OF J-PARC

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ABSTRACT

A neutron diffractometer named TAKUMI that is dedicated for engineering applications has been constructed at MLF of J-PARC, and was officially completed on March 2009. While commissioning is continuing, user programs including general uses have been conducted. An instrumental use including commissioning, two JAEA internal project uses and 16 general uses have been conducted during 2009 FY. Since commissioning is still in progress, beam time allocation for instrumental use is still dominant.

1. Introduction

The Engineering Materials Diffractometer "TAKUMI" is a time of flight (TOF) neutron diffractometer dedicated to promote scientific and industrial studies in areas of mainly materials science and engineering and mechanical engineering. TAKUMI is designed to cover: 1) evaluation of strains or stresses inside engineering components, 2) evaluation of microstructural evolutions during deformations and/or thermal processes, during manufacturing and/or during service, 3) crystallographic investigation of small regions in engineering materials, 4) texture analysis, and so on. TAKUMI was officially completed on March 2009, while commissioning has been started from September 2008 being parallel with the final stage of the construction. From commissioning, performances of TAKUMI are confirmed as follows. The best instrumental resolution $\Delta d/d$ is less than 0.2 %, the d range at standard (25 Hz chopper rotation) operation is 0.05 nm to 0.27 nm in which it can be expanded to be 0.05 nm to 0.47 nm by lowering chopper rotation speed to be 12.5 Hz [1]. TAKUMI adopts an event recording method for the data collection, and the software is available to manipulate various kinds of histograms from the event data flexibly and speedy [2,3].

User programs has been started at TAKUMI since January 2009 with succeeded results obtained and published elsewhere.[4,5] In the same time, the development of several sample environments are now in progress based on requirements from user community. This paper reports mainly the current status of user programs running at

TAKUMI and the development status of sample environments, while the present instrument status [6] has been reported separately in this meeting.

2. User Programs in TAKUMI

2.1. Instrumentation Use

An instrumentation use with the title of "Residual Stress Study at BL19" is running to continue commissioning, and to keep maintenances and of course upgrading of instrument. In this beam time, confirmation of instrumental resolution, confirmation radial collimator's viewing width, reducing instrumental background and several demonstrated experiments including strain mapping, in situ measurement during loading and heating were performed as commissioning. Measurements of several standard samples for calibrations were performed as instrumental maintenances to provide high reliabilities to users. As upgrading, rietveld refinement application to data analysis which will be extended to be a tool for data analysis of texture measurement is in progress using data of standard powder samples, and an optimization of analysis software to evaluate microstructural information from peak broadening will be conducted. Commissioning of sample environmental devices that are under development will be the next target to be performed additionally in this beam time.

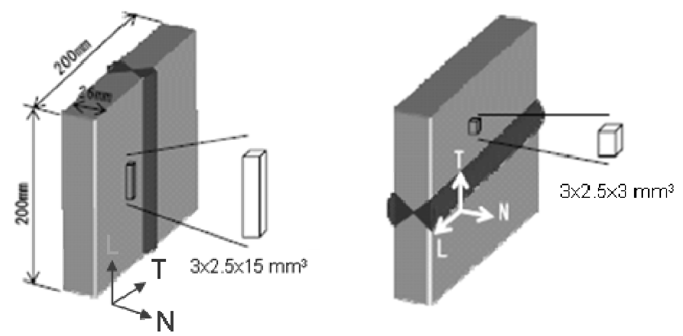

Fig. 1 Setup illustration of strain mapping measurement in welded sample

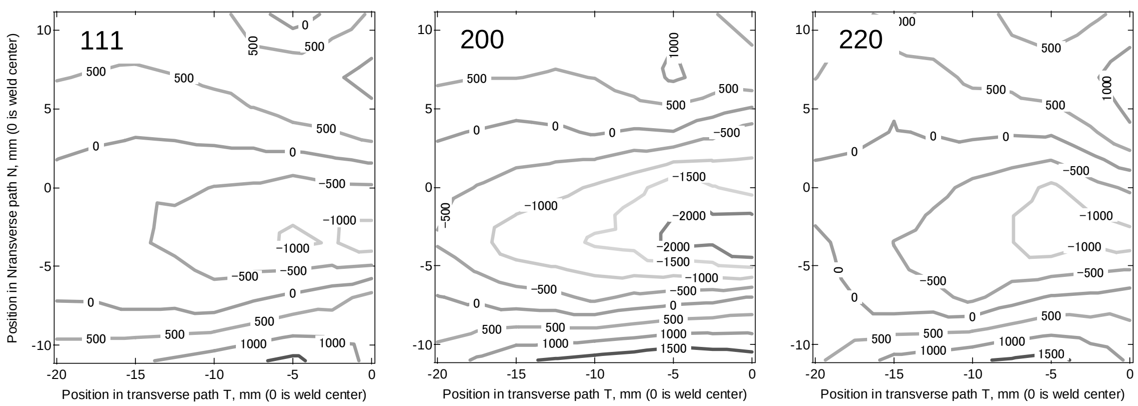

Fig. 2 Distributions of residual strains for T direction measured in various *hkl* peaks

For instance, strain mapping in a welded sample is introduced briefly. This work is performed under collaboration with the neutron source group of the MLF for development of welding method of future target vessels. As the first experiment, a welded sample with

26 mm thick was used to clarify data reliabilities measured in TAKUMI. The same sample was measured previously at RESA [7], a neutron diffractometer for residual stress analysis installed at JRR-3 of JAEA. Figure 1 shows experimental setup of the mapping measurement. Figure 2 shows distributions of residual strains for T direction (see description of strain direction in Fig. 1) measured at various *hkl* peaks. Position of 0 mm in N path is the center of thickness and position of 0 mm in T path is the weld center. These distributions were found to be in good agreement with those measured at RESA, in which the residual strains were compressive in the welding region and they changed to be tensile in the regions far from the welding part. The strain values measured at 200 peak show the largest value being consistent with the elastic stiffness of 200 grains that shows the lowest value.

2.2. Project Use 1: Stress/Strain Studies in Superconducting Composites

This research project aimed to clarify internal strains behaviors in industrial superconducting composites generated due to their processes and/or during uses (at high magnetic field, high current density), and to understand the relation between the internal strains and their superconducting properties. This project was a collaboration research among scientists from several universities and institutes, who work on industrial superconducting composites including Nb_3Sn, Nb_3Al, $Bi_2Sr_2Ca_2Cu_3O_{10}$ (BSCCO) and $YBa_2Cu_3O_7$ (YBCO) wires or tapes. Results from this project have been reported briefly as a review in Ref. [8].

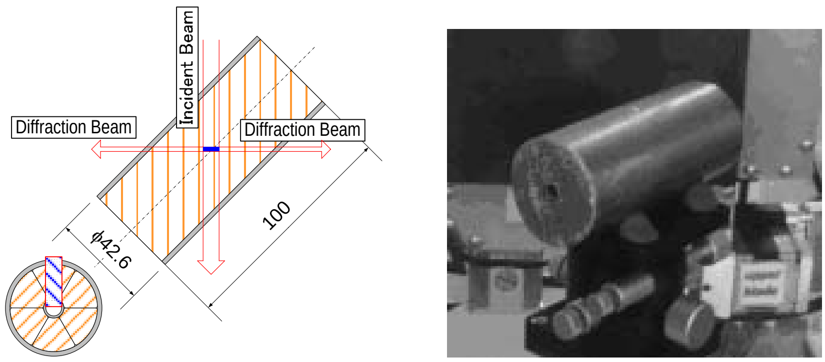

Fig. 3 Setup illustration of strain measurement in ITER TF conductor

Figure 3 shows measurement procedure to obtain residual strains in a Toroidal Field (TF) coil of International Thermonuclear Experimental Reactor (ITER) [9]. The gauge volume used in the measurement was $7\times2\times15$ mm^3 and the measured position is shown in Fig. 4 (left). As can be shown, the total neutron path is approximately 60 mm. Since the volume fraction of Nb_3Sn wire in TF coil is only about 6 % and no texture is found in Nb_3Sn wire, diffraction peaks from Nb_3Sn phase is predicted very weak. When a diffraction pattern of the TF coil obtained at TAKUMI was analyzed, the peak height of the strongest peak from Nb_3Sn phase was only 1.4 % in comparison to the 111 peak from Cu phase in TF coil. These may be reasons of the difficulties in measuring residual strains in Nb_3Sn wires in TF coil. These difficulties can be overcome by using TAKUMI due to its high intensity and low background. Peaks from Nb_3Sn phase were subsequently fitted to determine peak positions, and then reliable residual strains could be evaluated when Nb_3Sn filament dissolved from the TF coils was used as the d_0 sample. Data from neutron diffraction experiments herewith can be anticipated to help clarifying strain states in coils for 100 kA class fusion reactors.

2.3. Project Use 2: High Pressure Neutron Powder Diffraction Studies

This research project is intended to develop the high-pressure devices suitable for in situ neutron powder diffraction experiments under high-pressure. Structural and physical properties of materials under high-pressure conditions are of great interest in materials science and earth science. Although in situ neutron diffraction experiments under high pressure encounter many difficulties such as smallness of the samples and limitation of the diffraction window, these problems are anticipated to be overcome by high-flux neutrons and time-of-flight (TOF) measurements. TAKUMI has detector banks with the scattering angle of 90º and a large translation sample stage, which are suited to perform diffraction experiments with various high pressure-devices.

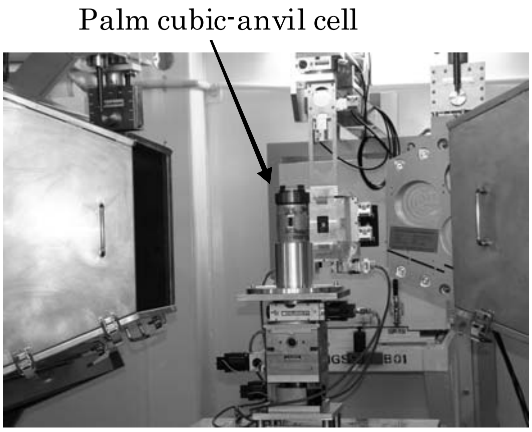

Fig. 4 Palm cubic-anvil cell

Three different types of high-pressure devices have been tested; a) a Paris-Edinburgh (P-E) Press [10], b) a palm cubic-anvil cell [11], and c) a new opposed-anvil cell with a nano-polycrystalline diamond (NPD cell) [12]. P-E press is a toroidal type high pressure apparatus and has been widely used for neutron experiments. Palm cubic-anvil cell is a multi anvil high-pressure device that has a small-scale and can be mounted into a cryogenic cooler. Figure 4 shows the palm cubic-anvil cell sitting on TAKUMI sample table. The nano-polycrystalline diamond in NPD cell has a Knoop-hardness value higher than single crystal diamond, and thus NPD cell is expected to generate high pressure (up to 60 GPa) with a large sample volume compared to ordinary diamond anvil cell [13]. Since these devices are very compact, the high-pressure techniques established at TAKUMI must be transferable to other instruments at MLF/J-PARC. We have also checked the performance of a small focusing mirror that was designed to gain the incident beam flux. Lead was selected as a standard sample for high pressure experiments at TAKUMI because of its large scattering length and low neutron absorption. In spite of small sample (<1 mm^3) and limitation of diffraction window, diffraction peaks from lead were observed successfully at high pressure conditions. The developments of high-pressure device components (TiZr alloy gasket, cBN anvil etc.) and careful shielding for reducing background and contaminated peaks made it possible to obtain the neutron diffraction patterns up to 5 GPa with a sufficient statistics at TAKUMI [14]. Furthermore, in situ experiments at simultaneous low temperature (60 K) and high pressure (1 GPa) have successfully been made with the palm cubic-anvil cell mounted in a cryostat.

2.4. General Use

TAKUMI is one of instruments at MLF having many users. Many proposals have been applied to TAKUMI since the user program at MLF/J-PARC was started. Proposals approved to be conducted during 2009 FY are shown in Table 1. As shown in the table, even there were only 16 proposals for general uses, 6 of them were proposed by users from industrial companies. However, since commissioning of TAKUMI is still in progress, beam time allocated for general uses can not achieved 50 %, as shown in Fig. 5. Beam time allocation for instrumentation use (commissioning) will be reduced gradually year by year and beam time for general uses will be increased to be majority.

Table 1 Approved proposal number at TAKUMI during 2009 FY

Proposal type	Number
Instrumentation use (IG)	1
Project use (PG) PG1 Stress/strain studies in superconducting composites PG2 High pressure neutron diffraction studies	2
General use from universities: 10 from industrial companies: 6	16

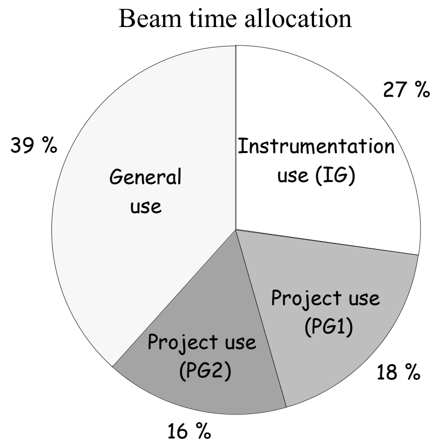

Fig. 5 Beam time allocation at TAKUMI during 2009 FY

3. Sample Environments in TAKUMI

To conduct experiments in various applications, developments of sample environmental (SE) devices are less than necessary. SE devices equipped at the present are a screw-typed loading machine with load capacity up to 50 kN, and a user-brought in dilatometer that can be used up to 1273 K. The loading machine is used to do in situ neutron diffraction measurements during tensile or compressive deformations with load or cross-head displacement controlled. Cyclic deformations with cross-head displacement speeds of less than 100mm/min can be also conducted. The dilatometer is used to do in situ neutron diffraction measurements during heating, hence neutron diffraction data and dilatation data can be obtained simultaneously. Other SE devices are now under development in the collaboration with users during 2009 FY. They are a Eurelian cradle (12kg capacity) for arbitrary sample rotation, a furnace that can be optionally added to the loading machine and a cryogenic load frame.

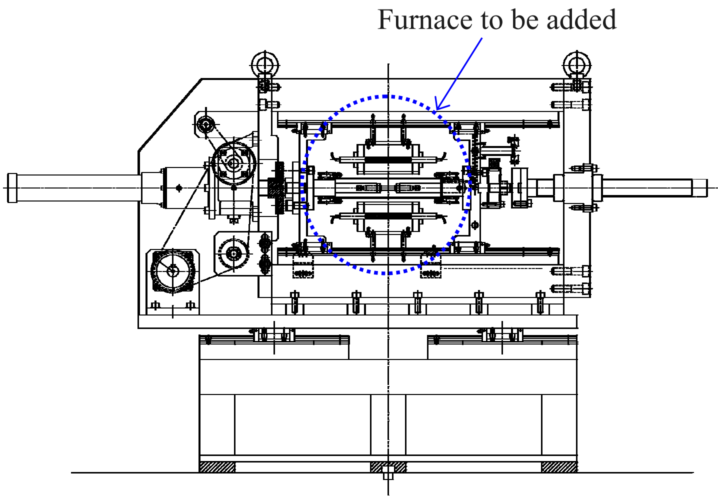

Fig. 6 Conceptual design of furnace (circled area) added to loading machine

A furnace to be added to loading machine, enabling in-situ measurements under loading at high temperatures, is under development. This will be delivered during March 2009, and preliminary studies using it will be performed after that. The conceptual design is shown in Fig. 6. A halogen-lamp typed furnace will be added to the loading frame in such a way that specimens will be heated from both top and bottom sides. The highest temperature is designed to be about 1273 K.

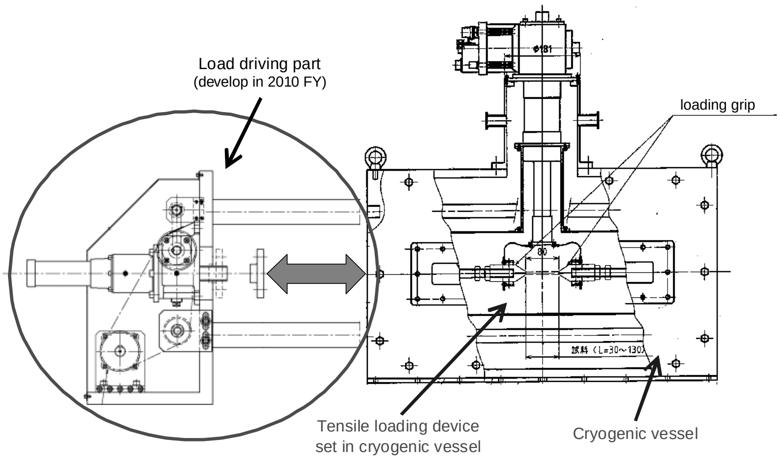

Fig. 7 Conceptual design of cryogenic loading machine

A cryogenic loadframe is developed under a collaboration with users joining in the research project of "Stress/Strain Studies in Superconducting Composites". In 2009 FY, a cryogenic vessel and a tensile loading device set in the vessel have been developed. The conceptual design is shown in Fig. 7, and they will be delivered on March 2010. The load-driving part will be developed during 2010 FY. A similar type of cryogenic loadframe has been developed at RESA, and could be used for tensile loading experiments at about 5 K. [15]

4. Summary

The Engineering Materials Diffractometer TAKUMI of MLF/J-PARC has finished the construction phase and started running user programs while the commissioning is still continuing. TAKUMI is changing to general use in majority gradually, and shifting to SE devices development and upgrading phase. Many scientific and/or industrial results with high impacts herewith are anticipated to be achieved.

5. References

1. S. Harjo, K. Aizawa, T. Ito, H. Arima, J. Abe, A. Moriai, K. Sakasai, T. Nakamura, T. Nakatani, T. Iwahashi and T. Kamiyama, to appear in Mater. Sci. Forum (2010).
2. T. Ito, T. Nakatani, S. Harjo, H. Arima, J. Abe, K. Aizawa and A. Moriai: to appear in Mater. Sci. Forum (2010)
3. T. Ito, S. Harjo, T. Nakatani, H. Arima, J. Abe, A. Moriai, K. Aizawa and M. Arai, submitted in this issue.
4. For instance: S. Awaji, H. Oguro, G. Nishijima, K. Takahashi, K. Watanabe, S. Machiya, H. Suzuki, S. Harjo, K. Aizawa, T. Ito, H. Arima, MecaSens V, Mito, Japan (2009), to appear in Mater. Sci. Forum (2010)
5. For instance: S. Machiya, K. Osamura, H. Suzuki, S. Harjo, T. Ito, Y. Tsuchiya, Abstract of CSJ Conference, **80** (2009) 8
6. S. Harjo, T. Ito, K. Aizawa, H. Arima, J. Abe, H. Kira, S. Takata, A. Moriai, T. Nakatani, K. Sakasai, T. Nakamura, T. Iwahashi, M. Arai and T. Kamiyama, submitted in this issue.
7. M. Hayashi et al., Proc. ICRS-5 (1997) 762
8. K. Osamura, S. Harjo, T. Ito, K. Aizawa, S. Awaji, G. Nishijima, H. Takahashi, H. Oguro, T. Hemmi, K. Matsui, Y. Tsuchiya, S. Machiya, H. Suzuki, M. Tomita and K. Suzuki, TEION KOGAKU **43** (2008) (in press)
9. N. Koizumi, et al., TEION KOGAKU **36** (2001) 478-485
10. J. M. Besson, R. J. Nelmes, G. Hamel, J. S. Loveday, G. Weill, and S. Hull: Physica B, **180&181** (1992),p. 907
11. Y. Uwatoko, K. Matubayashi, T. Matsumoto, N. Aso, M. Nishi, T. Fujiwara, M. Hedo, S. Tabata, K. Takagi, M. Tado, and H. Kagi: Rev. High Pressure Science & Technology **18** (2008) p. 230
12. T. Okuchi, S. Sasaki, T. Osakabe, Y. Ohno, S. Odake, and H. Kagi: J. Phys.: Conf. Ser. (in press)
13. T. Irifune, A. Kurio, S. Sakamoto, T. Inoue, H. Sumiya: Nature **421** (2003) p. 599
14. J. Abe, T. Hattori, K. Komatsu, H. Arima, M. Arakawa, A. Sano, H. Kagi, S. Harjo, T. Ito, A. Moriai, K. Aizawa, M. Arai and W. Utsumi: J. Phys.: Conf. Series (in press)
15. Y Tsuchiya,H Suzuki,T Umeno,S Machiya and K Osamura, Meas. Sci. Technol. **21** (2010) 025904-025907