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COMMISIONING OF THE ENGINEERING MATERIALS DIFFRACTOMETER TAKUMI OF J-PARC

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ABSTRACT

The Engineering Materials Diffractometer “TAKUMI” is designed and has been constructed at MLF of J-PARC to conduct various kinds of studies on materials science and engineering and to promote industrial applications, related with strain measurements. The commissioning of TAKUMI has been started from September 2008, and several user programs have been done. In the commissioning, the resolution $\Delta d/d$ of less than 0.2% was confirmed to achieve from diffraction measurements using 2 mm diameter of annealed piano wire, with combination of beam collimation (high resolution mode). The *d*-range measured by TAKUMI with single pulse frame, i.e. standard operation, was confirmed to be 0.05 nm to 0.27 nm, showing the optimum range for studies of materials covered by this machine. TAKUMI adopted an event mode data recording method. It was found that the recording method is very useful to manipulate data as we like, for instance, detector range, time of flight binning width and time resolved data, even the experiment has been finished.

1. Introduction

The Engineering Materials Diffractometer “TAKUMI” is decided to design and to construct at MLF/J-PARC to promote scientific and industrial studies in areas such as materials science and engineering and mechanical engineering. “TAKUMI” is a Japanese noun that means “engineering”, “skilled craftsman”, “master” or “professional”. This name is chosen to give to this diffractometer, and also to be the goal target of the diffractometer itself. TAKUMI is a time of flight (TOF) neutron diffractometer that 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.

The idea to build an engineering diffractometer at J-PARC has been discussed in a workshop in late 2000. The desired performances, basic design [1] and the Letter of Intent for the engineering diffractometer at MLF/J-PARC have been approved at 2004, and then the detailed design was fixed at the end of 2006 after an advisory meeting held on October 2006. This instrument was funded for construction in 2006, with a budget of J-PARC from Ministry of Education, Culture, Sports, Science and Technology, followed by the construction started from March 2007. This instrument was then named “The Engineering Materials Diffractometer” officially with “TAKUMI” as the nick-name. TAKUMI was officially completed on March 2009, while the commissioning has been started from September 2008 being parallel with the final stage of the construction. This paper reports TAKUMI design concept, the present status and the commissioning results.

2. Design Concept [2]

TAKUMI is designed to cover the applications described in the introduction. The performances required are to have a better strain resolution and to realize a lot shorter measurement time than similar existing intruments. Therefore, the primary performances are determined as follows: (i) the resolution at the 90 degree scattering detector is less than 0.2 % in $\Delta d/d$ (corresponding to $\Delta\varepsilon < 50$ με in steel), (ii) nominal measuring time is within 1 hour for volumetric strain measurement of 1 mm^3 in gauge volume, and (iii) to have enough space at the center of this instrument enabling to accommodate large scale components with the longest dimension of about 1 m.

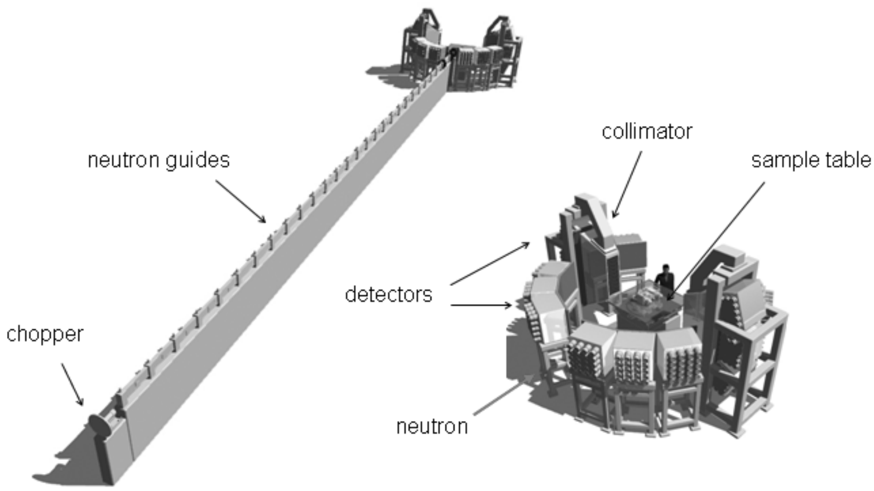

Fig.1 Three-dimensional drawing of TAKUMI design

This instrument is designed to place on beam line port 19 of the MLF and to view a decoupled-poisoned liquid H_2 moderator [3] which is newly developed specially for J-PARC project and can provide slow neutrons providing good symmetrical diffraction peak profiles throughout the acceptable wavelengths. The primary and the secondary flight paths are 40 m and 2 m, respectively. A three-dimensional drawing of the design is shown in Fig. 1. A single disk chopper is placed at 7.7 m apart from the moderator along the way of the primary flight path. A tapered steel-thimble collimator of about 4.9 m length is placed in the shutter and the biological shielding block to limit beam divergence and to reduce backgrounds. After the chopper, a 20 m long curved supermirror guide followed by a 10 m long straight supermirror guide are installed. Slit systems around the final straight guide part are installed to adjust the incident angular resolution, and the other one is placed just before the sample to define the incident gauge size, whose the location can be varied depending on the scale of the samples. Modified ENGIN-X type scintillator detectors with the horizontal spatial resolutions of 3 mm, which were planned to improve in horizontal channel range in collaboration between UK and Japan, are used for 90 degree scattering detector banks having a total solid angle of about 0.8 sr. Several sets of radial collimators with different definition gauge widths are prepared. A sample-translator table with *x*, *y*, *z* and θ axes being capable to handle large-scaled components up to 1 tone is installed at the center of this instrument. A load frame for applying uni-axial stress in tension, compression or low cyclic fatigue to carry out in situ strain measurements during deformation is also provided. An in situ measurement during deformation at a high/low temperature can be also performed by using a combination technique of applying stress and low/high temperature.

Several options including radiography and/or Bragg edge measurements are also considered to be implemented on this instrument for future upgrading. Data acquisition and analysis softwares including positioning for complex shape sample, profile analysis and visualization are designed to be user friendly.

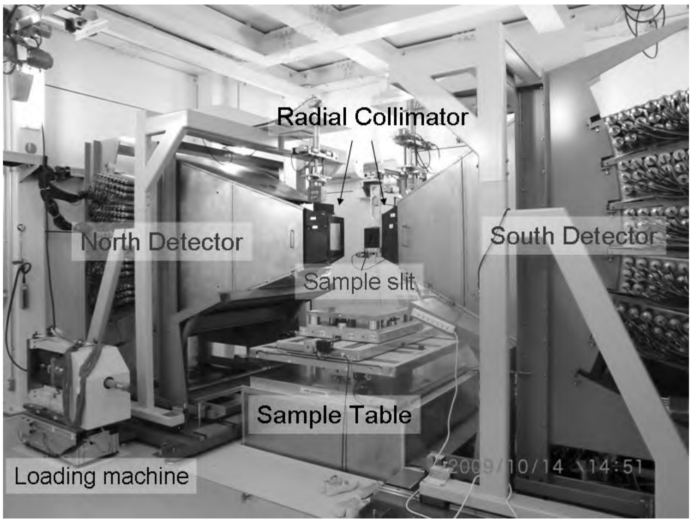

Fig. 2 Present status of TAKUMI

3. Present Status

Construction of TAKUMI has been finished on March 2009 with the detailed parts as described in section 2, except for detector ranges; approximately 72% coverage in 90 degree scattering banks than the designed due to budget limitation. ENGIN-X typed scintillator detectors [4] with larger horizontal coverage (360 channels per module with 3 mm wide and 200 mm high for each channel) [5] were succeeded to developed at J-PARC, and are now used in 90 degree scattering banks in combinations with new typed data acquisition electronics and an event mode data recording method. TAKUMI has only a pair of radial collimators for 2 mm gauge width at the present, but equipments of those for 5 mm, 3 mm and 1 mm gauge widths are planned from 2010 fiscal year onward. Fig. 2 shows the present status of TAKUMI experimental space.

Sample environments 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 100 mm/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 sample environments are now under development in the collaboration with users during 2009 fiscal year. They are a Eurelian cradle (12 kg capacity) for arbitrary sample rotation, a furnace that can be optionally added to the loading machine and a cryogenic cooler. The cryogenic cooler is planned to expand to be cryogenic load frame during 2010 fiscal year by installing a loading system.

TAKUMI developed also software application kit called EMAKi, which was designed to be user-friendly interface for novice users by graphical user interface (GUI). EMAKi comprises experimental support tools, device control interface, data acquisition system, data reduction, analysis and visualization tools. The details please refer Ref. [6]

4. Commissioning and Possibilities

4.1. Commissioning

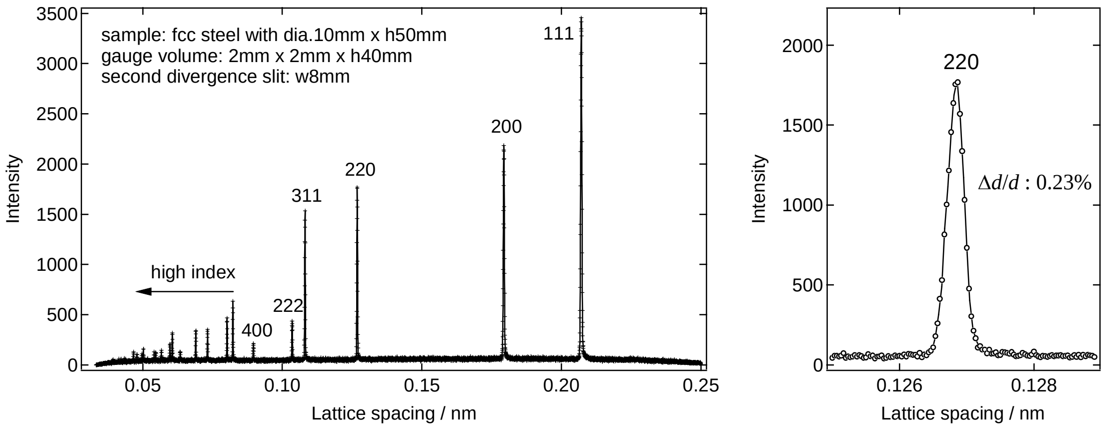

Fig. 3 Diffraction pattern of a 10 mm diameter austenitic steel measured with collimated beam and 2 mm radial collimator

TAKUMI started commissioning on September 2008 with only 2 detector modules, one at the north detector bank and another at the south. After checking the validity and the stability of the detectors and the data acquisition system, powder diffraction data of an austenitic steel alloy with 10 mm diameter without beam collimation (high intensity mode) was measured, and the resolution ($w = \Delta d/d$) of 0.4% was confirmed, as designed. When the gauge definition slit was set to be 2 mm wide and 40 mm high, and the 2 mm radial collimator was set in the scattering path, a powder diffraction pattern of the same sample was obtained as shown in Fig. 3. In this measurement the second divergence slit was set to be 8 mm wide, i.e., high resolution mode. The *w* value was found to be 0.23%, and the peak profile was almost symmetric. When we considered that the beam divergence between slit and sample must be existed, and the radial collimator viewing width should be wider than the designed parameter, the *w* value for a sample with 2 mm wide must be better. Lately, the radial collimator viewing width was confirmed to be about 2.5 mm.

A good matching between the peak height (*I*) and the peak resolution (*w*) will give good accuracy in data analysis. An experiment to find the good matching for the sample of 2 mm wide was performed and the obtained results are shown in Fig. 4. An annealed piano wire with 2 mm diameter was used as the sample, and the gauge definition slit and the radial collimators were not used in this experiment. The *w* value increases (the value becomes small) with narrowing the second divergence slit width, while the *I* value decreases. When we assume Figure of Merit (*FoM*) as a relation between the *I* value and the *w* value, that can be expressed as I/w^2 [7], *FoM* (normalized to that for 200 peak at the second divergence slit width of 20.2 mm) as a function of the second divergence slit width can be shown in Fig. 5. The *FoM* may be relaxed for the second divergence slit width of less than 7 mm with the w value of less than 0.2%. TAKUMI can provide a *d* range of 0.05 nm to 0.27 nm by adopting a delay time during measurement, as the standard operation. This *d* range can be expanded up to 0.47 nm, by measuring *TOF* range up to 80 ms and 12.5 Hz chopper rotation, but the intensity becomes half.

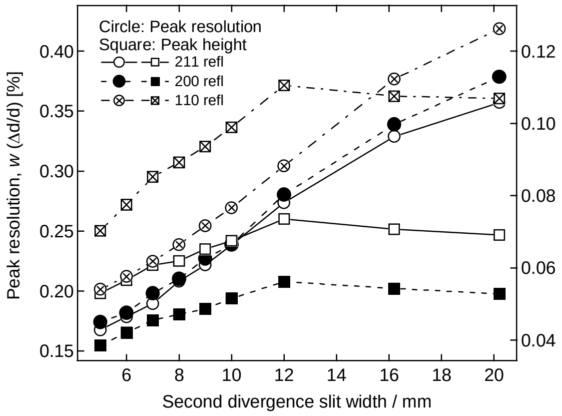

Fig. 4 Peak resolutions and peak heights obtained by changing the second divergence slit width. An annealed piano wire with 2mm diameter was used as the sample.

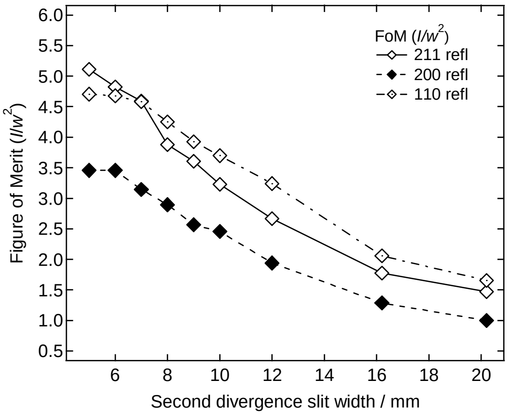

Fig. 5 Figure of Merit values evaluated from peak resolutions and peak heights shown in Figure 4.

4.2. Strain Mapping

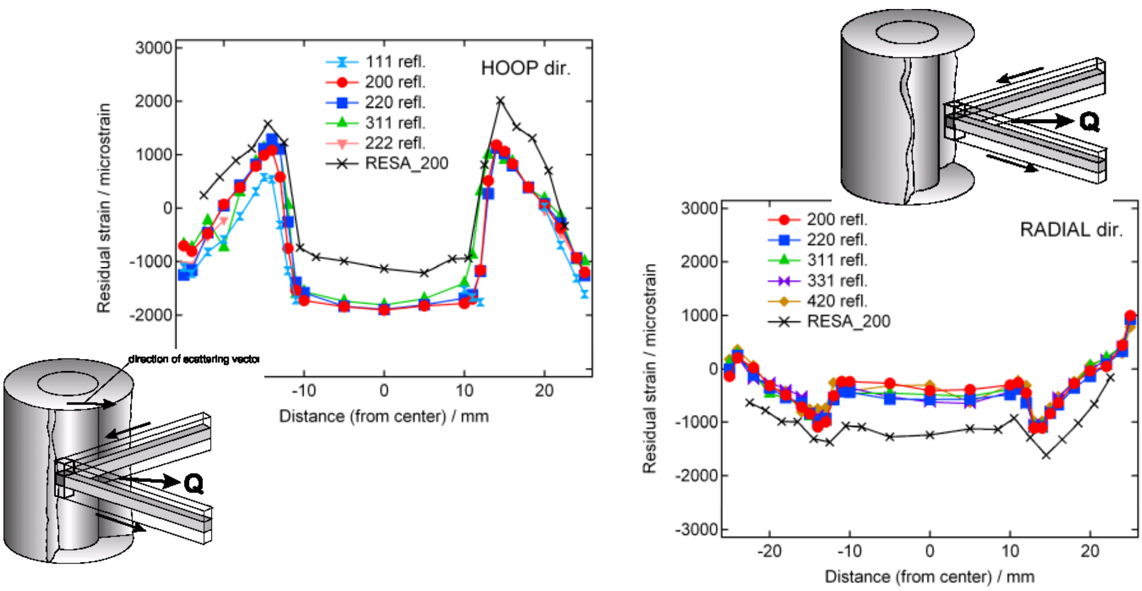

Fig. 6 Residual strain distribution in a shrink fit ring and plug aluminum alloy for hoop and radial direction.

To clarify performances for strain mapping, residual strain distributions for hoop and radial directions in a shrink fit ring and plug aluminum alloy were performed. The gauge volume for the measurements was 2×2×10 mm^3. The ring and plug aluminum was made and was previously measured by RESA [8] group of JRR-3, JAEA. Fig. 6 shows results measured at TAKUMI plotting together with those measured at RESA. As shown in the figure, tendencies of residual strains measured at both instruments show good agreement, and those measured at TAKUMI from different *hkl* reflections have very good agreement qualitatively and quantitatively.

4.2. 2-D Scattering Angle-TOF Diffraction

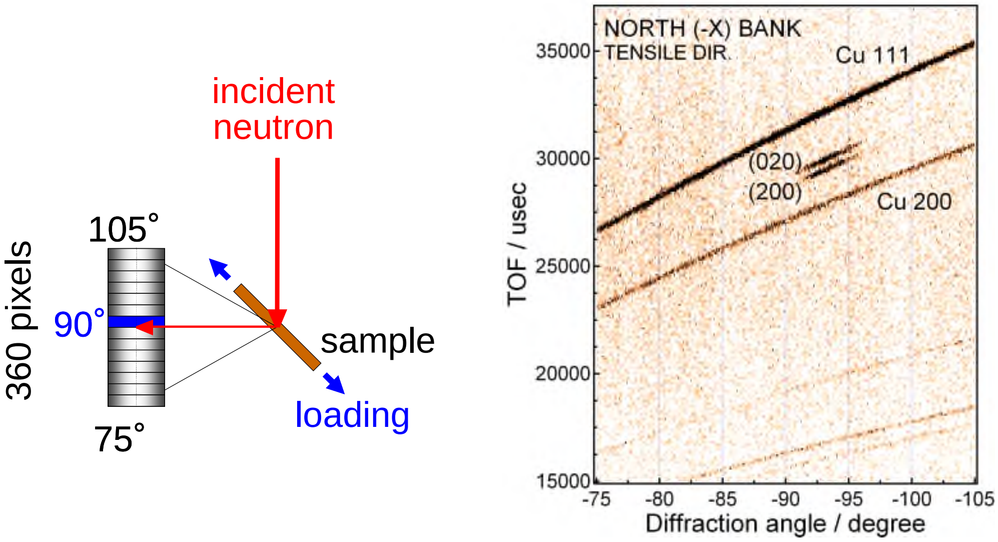

Fig. 7 Schematic geometry of $YBa_2Cu_3O_7$ tape setting for neutron diffraction measurement during tensile deformation (left), and 2-D diffraction pattern of $YBa_2Cu_3O_7$ tape at the north 90 degree scattering bank (right). Time focusing manipulation was not performed to the 2-D diffraction pattern data.

Fig. 7 shows a preliminary result from neutron diffraction measurements during tensile deformation of $YBa_2Cu_3O_7$ (YBCO) tapes [9], demonstrating how TAKUMI is appropriate for this kind of studies. The YBCO tape (4 mm width) used in the measurement had a total thickness of about 100 μm with only 1 μm thick YBCO layer. This tape used a Hastelloy substrate with about 40 μm thick, and was laminated by 20 μm thick Cu layers at both sides. Two YBCO tapes were superposed and were set at the loading machine in such a way that the loading axis was in the scattering plane and to be 45 degree to the incident beam direction (see Fig. 7 left). TAKUMI had only 1 detector module for each 90 degree scattering bank, and the beam power was 20 kW, during the experiments. Diffraction profiles were collected for 7.2 ks. When neutron data from all detector channels were binned together and time-focused, peaks from YBCO phase were difficult to identify, as ones expected. However, since TAKUMI adopted the event data recording method, the data could be re-manipulated to be data per channel and could be plotted as a 2-D diffraction pattern shown in Fig. 7 (right). The 2-D diffraction pattern was collected at the north 90 degree scattering bank, i.e., data for grains oriented along tensile direction. It was obviously seen that peaks from YBCO phase (020 and 200 peaks) were identified for only around 2θ range of 5 degree, showing a very strong texture. On the other hand, in 2-D diffraction pattern collected at the south bank, the YBCO 020 and 200 peaks were not observed, but other 00l peaks appeared at different 2θ .

4.3. Time-Slicing Measurement

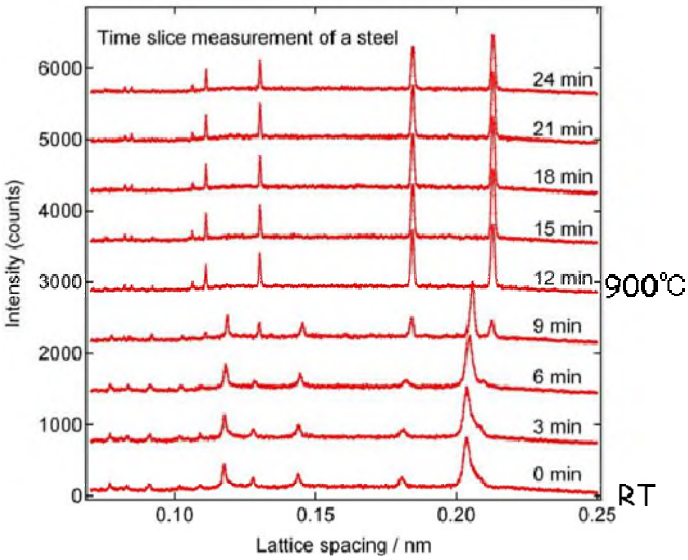

Fig. 8 Time slice diffraction patterns of a steel heating from RT up to 900 °C.

Fig. 8 shows a preliminary result from neutron diffraction measurements during heating of a bainitic steel, demonstrating how TAKUMI is also appropriate for this kind of studies. The steel used in the measurement had 5 mm diameter and 30 mm length, and was set at the dilatometer in such a way that the longitudinal axis was in the scattering plane and to be 45 degree to the incident beam direction. TAKUMI had only 3 detector modules for each 90 degree scattering bank, and the beam power was 20 kW, during the experiments. Diffraction patterns in Fig. 8 were those for only north detector bank, and were collected with 1 measurement and then were time-sliced every 3 minutes after the measurement and heating process were finished. Since TAKUMI adopted the event data recording method, the data could be time-sliced during measurement or after the measurement. When fast reaction or fast transformation is needed to observe, time-slicing with shorter time can be done. Principally diffraction pattern per 40 ms can be time-sliced because J-PARC operates at 25 Hz.

5. 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 was confirmed to be a neutron diffractometer having good performances to do many kinds of researches in materials science and engineering, during commissioning and running user programs. TAKUMI is open for users from everywhere, without any exceptions. Many scientific and/or industrial results with high impacts can be expected.

6. References

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