| MLF Experimental Report | 提出日 Date of Report<br>2012/11/08 |
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| 課題番号 Project No.<br>2012A0130<br>実験課題名 Title of experiment<br>Flux-pinning-induced stress and magnetostriction in bulk superconductors<br>実験責任者名 Name of principal investigator<br>Masaru Tomita<br>所属 Affiliation<br>Railway Technical Research Institute | 装置責任者 Name of responsible person<br>Kazuya Aizawa<br>装置名 Name of Instrument/ (BL No.)<br>Takumi (BL19)<br>実施日 Date of Experiment<br>2012/5/16,17,18 |

試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。(適宜、図表添付のこと)
Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.

**Samples**: In the top-seeded melt-textured (TSMT) technique, powders of $Y_2O_3$, $BaCO_3$ and CuO were weighed to obtain the composition $Y_{1.6}Ba_2Cu_3O_{7-x}$ and ground thoroughly followed by calcination at 900°C for 36 hours with three intermediate grindings, and then sintered at 940°C for 15 hours. The powder mixture was pressed into pellets of 30 mm and 50 mm in diameter and subject to a cold isostatic pressing under 200 Mpa. The differential thermal analysis (DTA) measurements were performed in air to determine the peritectic decomposition temperature, $T_p$. This temperature was then used to schedule the heat treatment profile of the melt growth process. Eventually, the melt textured samples were annealed at 450°C for 350 hours in flowing pure $O_2$ gas.

2. 実験方法及び結果 (実験がうまくいかなかった場合、その理由を記述してください。)
Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.

**1. Neutron diffraction measurements:** The "TAKUMI" a time of flight (TOF) neutron diffraction has been used to measure magnetic strains in high $T_c$ superconducting materials. For neutron diffraction measurements using Takumi, the sample was arranged as shown in Fig. 1. An incident beam is diffracted at a selected position of the melt textured Y-123 sample. The neutron diffraction with a time of flight diffracted beam is collected by a pair of detectors, which is located at 90 degree angles from the incident beam, in order to determine strains for both radial and hoop directions simultaneously. The picture of the experimental set-up and arrangement of cryostat containing the superconducting bulk magnet for neutron diffraction measurements are presented in Fig. 2. To determine the magnetostriction in the material, first the lattice spacing in various crystallographic directions of stress free bulk superconductor is determined. An incident beam is diffracted at a selected position of the melt textured Y-123 sample. The neutron diffraction with a time of flight diffracted beam is collected by a pair of detectors, which is located at 90 degree angles from the incident beam, in order to determine strains for both radial and hoop directions simultaneously. The picture of the experimental set-up and arrangement of

2. 実験方法及び結果（つづき） Experimental method and results (continued)

cryostat containing the superconducting bulk magnet for neutron diffraction measurements are presented in Fig. 2. To determine the magnetostriction in the material, first the lattice spacing in various crystallographic directions of stress free bulk superconductor is determined. The lattice spacing are derived from the diffraction shifts in the Bragg peaks of the trapped field superconductor. For any hkl diffraction peak, the lattice strain (ε) is given by

$$\varepsilon_{hkl} = (d_{hkl} - d^{o}_{hkl}) / d^{o}_{hkl}$$

where $d_{hkl}$ and $d^{o}_{hkl}$ are the average lattice spacing in the stressed and stress free superconductor, respectively. The hkl represents the Miller indices of the diffracting planes. Note that in the present case, the strain and strain-free material mean superconductor with and without the trapped magnetic field condition. With a pulsed source, changes in lattice spacing are related to TOF shifts in the Bragg peaks. The relationship of TOF to neutron wavelength is described by equation

$$\lambda = h / m_n \upsilon = ht / m_n L$$

where h is Planks constant, λ is the neutron wavelength, $m_n$ the neutron mass ($1.675 \times 10^{-27}$ kg), t is the time of flight for neutron to reach a detector after leaving its source, L is the total length of the flight path of the neutron (source to sample to detector). Bragg`s law, $2d_{hkl}\sin\Theta_{hkl} = \lambda$, can then be written as

$$d_{hkl} = ht / 2m_n L \sin\Theta_{hkl}$$

where $d_{hkl}$ is the lattice spacing, $2\Theta_{hkl}$ scattering angle, and $\varepsilon_{hkl} = \Delta d_{hkl} / d^{o}_{hkl}$.

**2. Magnetic Strain measurement at the neutron diffractoameter TAKUMI:** Stresses and strains in a bulk materials are usually directional and position dependent. This necessitates to measure strains at a number of locations in more than one direction. For this, we estimated the lattice spacing for the stress and stress free bulk samples at selected lattice planes in radial and hoop directions in the interval of 5mm. The neutron beam passes through the wall of vacuum vessel which is made of quartz glass. The first measurement was carried out on the sample A that had the trapped field of 4.5 T at approximately 45 K. Then, the cryo-cooler was turned off for de-magnetization. The sample temperature reached in 30 minutes to 110K above $T_c$ (transition temperature, 90 K). And at this temperature, the Y-123 superconductor generates electric resistance, and the super currents inside the bulk sample disappear. We turned on the cryo-cooler, for the next measurement condition that is for the sample that has no trapped field at about 45 K. The second measurement was started after the sample reached thermal equilibrium, which took 3.5 hours. Neutron diffraction peaks from the typical lattice plane which is measured at approximately 45 K before and after de-magnetization. The measured lattice planes were the (220), (110), and (200) (020) of Y-123 in sample A and B, respectively. This difference causes the different incident directions into each sample. For the sample A, the both selected (1 1 0) and (2 2 0) peaks in the radial directions showed small changes in lattice constants (see in Fig. 3). However, in the hoop direction, the lattice spacing was clearly changed (see in Fig. 3). Further, in sample B, we observed small changes in lattice spacing in hoop direction. The sample showed the trapped field of 2.2 T at approximately 45 K, which is half of the field as compared to the sample A. These results clearly indicate that trapped magnetic field values are the case sensitive for the generation of the magnetic forces inside the bulk superconducting material. For the sample A, the both selected (1 1 0) and (2 2 0) peaks in the radial directions showed small changes in lattice constants

2. 実験方法及び結果（つづき） Experimental method and results (continued)

(see in Fig. 3). However, in the hoop direction, the lattice spacing was clearly changed (see in Fig. 3). Further, in sample B, we observed small changes in lattice spacing in hoop direction. The sample showed the trapped field of 2.2 T at approximately 45 K, which is half of the field as compared to the sample A. These results clearly indicate that trapped magnetic field values are the case sensitive for the generation of the magnetic forces inside the bulk superconducting material.

Plane spacing was calculated from the fitted peak angle by Bragg's law. Finally, lattice strain was estimated as the difference of plane spacing with/without the trapped magnetic field in bulk superconductor. Fig. 4 shows the magnetic strain (%) as a function of trapped magnetic field at 45 K in the radial and hoop directions for sample A and B. From the figure, it is clear that when the trapped field is small then the magnetic strain is also less. Further, it can be seen that the magnetic strain of the bulk material rises with increasing of the trapped magnetic field. These results are important to enable validation of several industrial applications of superconducting materials before used to predict mechanical properties.

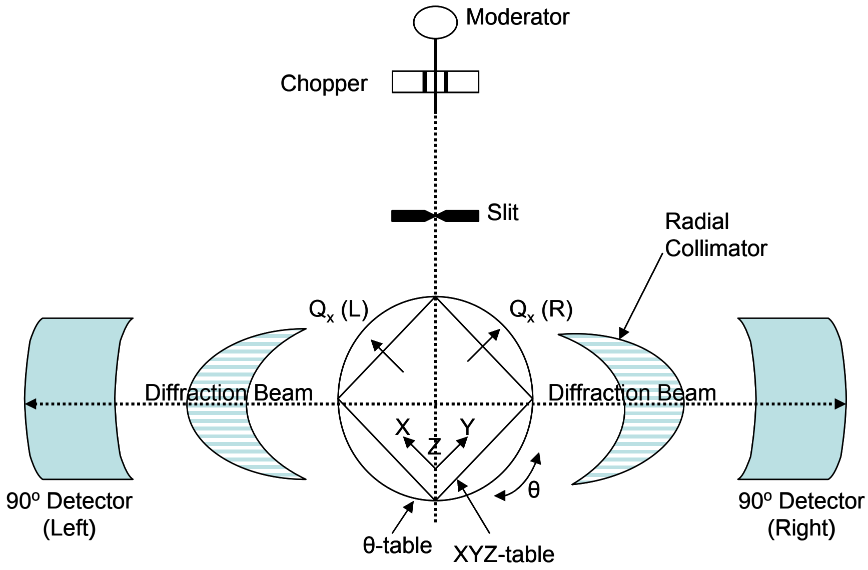


FIG. 1. Schematic of experimental set-up of the Engineering Material Diffractometer, TAKUMI.


FIG. 2. Photograph of experimental set-up for the Neutron diffraction time-of-flight in J-parc, TAKUMI.

## 2. 実験方法及び結果（つづき） Experimental method and results (continued)

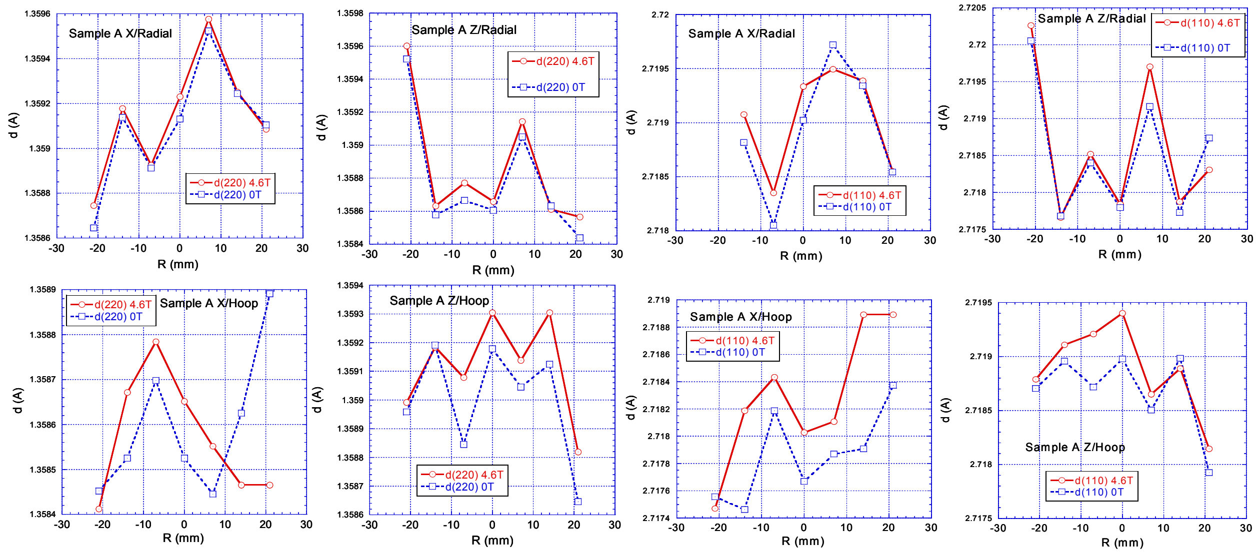


FIG. 3. Difference in the lattice spacing for the diffraction peak of the (2 2 0) of sample A with and without trapped field condition in the radial and hoop directions measured at 46 K. Note that the lattice spacing was clearly changed in the hoop direction (left-two figs); Difference in the lattice spacing for the diffraction peak of the (1 1 0) of sample A with and without trapped field condition in the radial and hoop directions measured at 46 K. Note that the lattice spacing was clearly changed in the hoop direction (right two figures).

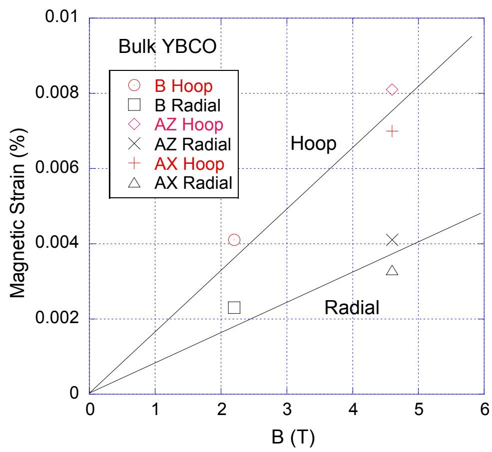


FIG. 4. The magnetic strain (%) as a function of trapped magnetic field at 45 K in the radial and hoop directions for melt processed Y-123 bulk material. Note that the magnetic strain of the bulk material rises with increasing of the trapped magnetic field.

**Proposed experiments at the TAKUMI**: When we measure the neutron diffraction measurements using the superconducting phase, there are already strains due to number of cracks formed during oxygenation specially, the interface between the $YBa_2Cu_3O_y$ “Y-123” matrix and $Y_2BaCuO_5$ “Y-211” secondary phase particles. Further, the strains also related with the grain shape and texture properties. Therefore, to gain much more information on the origin of the strains, samples must be prepared carefully without Y-211 secondary phase (or small quantity) and uniform performance. The next experiments, we will propose to use a specially designed Y-123 superconducting material for a time of flight (TOF) neutron diffraction at the TAKUMI.