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X-ray diffraction studies in pulsed high magnetic fields

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Abstract. X-ray diffraction studies have been performed up to 38 T in conjunction with synchrotron x-ray beams and a split-pair pulse magnet at the beamline BL19LXU of SPring-8. The magnet consists of coaxial two-coils which are made by winding CuAg wire up to 18 layers. The magnet has four windows with wide aperture angles and the height of 3 mm for passage of the x-ray beam. A pixel array detector was used to measure the diffraction patterns during an exposure of 5 millisecond around the peak field. The details of the instrumentations are presented.

1. Introduction

Recently synchrotron x-ray beams have been widely used for investigations of condensed matter physics. An x-ray diffraction gives us important information about symmetry and/or periodicity of the crystal and magnetic structures which can be changed at phase transitions induced by temperature, pressure and magnetic field. We have been developing x-ray diffraction techniques in conjunction with superconducting magnets as well as pulse magnets at the third generation synchrotron facility SPring-8, Japan. Two superconducting magnets producing magnetic fields up to 8 and 15 T have been respectively in steady operation. In order to exceed the limitation of the modern superconducting magnet technology, we must make another choice: a hybrid magnet or a pulse magnet. Considering a combination the x-ray diffraction technique with the high magnetic field, the hybrid magnet seems not to be realistic because of fair sizes of the magnet itself, the DC power supply and the cooling system, although it seems that some projects exist to construct DC field facilities except for Japan. On the other hand, instrumentations for pulsed high magnetic fields do not require such a huge space. Two Japanese groups including us [1-4] and one European group in ESRF [5] have been developing the pulse magnets and demonstrated significant performances of them. We have applied the pulsed magnetic field to a powder diffraction measurement using a pixel array detector and successfully observed a field induced lattice distortion of the antiferromagnet CoO [3,4]. A time-resolved data
acquisition is absolutely essential for performing x-ray diffraction measurements in pulsed high magnetic fields. Here we review several types of split-pair pulse magnet for x-ray diffraction studies. These magnets have been placed and operated in the experimental hutch EH4 of BL19LXU at SPring-8. First we mention the beamline BL19LXU and the apparatuses of EH4 except for the pulse magnets. Next we describe the detail of the split-pair pulse magnets and the pulsed power supply. Then the detailed description for taking x-ray diffractions with two types of detector systems is given. Finally we summarize and comment possible technical developments.

2. BL19LXU/SPring-8
The beamline equipped with an in-vacuum 27 m long undulator generates the World's brightest x-rays [6,7]. The x-ray beams are tuned by a Si(111) double-crystal monochrometer placed in the optics hutch. The four-circle diffractometer manufactured by Huber, Germany is installed at EH4 which is located downstream 135 m from the center of the undulator. At the entrance of EH4, the beam size is approximately 1 x 2 mm$^2$ and the photon flux is $5 \times 10^{13}$ photons/sec for the x-ray energy of 30 keV. A typical intensity of x-rays diffracted from a single crystal is $10^9$ photons/sec and $10^3$ photons/microsec. As a result, the time-resolved data acquisition in a few microsecond in pulsed high magnetic fields is feasible.

3. Pulsed magnetic field generation
The pulsed power supply with the storage energy of 500 kJ (10 kV) is installed at the BL19LXU. Two subunits of capacitor banks with 250 kJ can be operated independently. The pulsed current is released through thyristor switches synchronized with detector systems, mechanical shutters and a digital storage oscilloscope by a delay pulse generator.

At the beginning, we manufactured three tentative pulse magnets with pulse duration of about 6 msec [3]. Here we call them X1, X2 and X3. All the pulse magnets were designed and manufactured by collaboration between two high field facilities of ISSP, the university of Tokyo and KYOKUGEN, Osaka university. These pulse magnets are split-pair ones. Because this structure allows us to have wide aperture angles, it is effective in observing a lot of Bragg reflections. Two identical coils were made, because the magnet has a spacer between them as a passage for x-ray beams. Each coil consists of ten layers of coaxially wound CuAg wires. The wire with a cross section of 2 x 3 mm$^2$ has tensile strength of about 900 MPa. The coil was covered with glass fibers and reinforced by a vacuum impregnation with STYCAST$^{\text{TM}}$ 1266. After shaping the coil cylindrically, it was inserted into the Maraging steel pipe for a finished reinforcement. Before the insertion of the other coil, a fan-shaped spacer made of Maraging steel was inserted. Connecting the magnet with the capacitor bank of 250 kJ, magnetic fields up to 38 T with pulse duration of about 6 msec can be generated. It is noted that the lower part of the magnets is immersed in a liquid nitrogen bath so that the liquid does not disturb the x-ray beam. The pulse magnet X3 is now used in the margin of safety, because we examined the performance limitations of X2 and found it to be 48 T.

Next step is to manufacture a magnet with long pulse duration. Even though the synchrotron x-rays are extremely bright, it is obvious that the pulse duration should be long in the time-resolved data acquisition. On the other hand, the long pulse duration has an adverse influence on the cool-down time after discharging. We manufactured an 18 layers split-pair pulse magnet equipped with passages for liquid nitrogen. This is named X4. X4 also consists of two coils. Moreover each coil consists of four subunits(4 – 4 – 4 – 6 layers) reinforced independently by Maraging steel pipes. Between the second and the third subunits, a large number of holes were made in the pipe, through which liquid nitrogen can flow. Figure 1(a) shows a cut view of the split-pair pulse magnet and figure 1(b) is an extended figure of the spacer. X4 has a cold bore of 16 mm and four windows with a slit height of 3 mm. X4 is operated by the capacitor banks of 500 kJ. We succeeded in producing a magnetic field up to 38 T with pulse duration of 27 msec as shown in figure 2. The temperature of the magnet is monitored measuring a DC resistance of it. The maximum field of 38 T can be generated at intervals of about 20 minutes which is comparable to that of the magnet with the short pulse duration such as X3. Figure 3 shows the distribution of the magnetic field of X4 along the vertical axis of the magnet. The homogeneity is about 5% within 5 mm from the center of the magnet.
Figure 1. The cut view of the split-pair pulse magnet X4 with long pulse duration of 27 msec. Right figure shows the spacer for the passage of x-ray beams. The white area corresponds to the four windows.

Figure 2. Pulsed magnetic fields are plotted as a function of time. The solid line corresponds to the field pulse generated by X4. The dotted line represents the field pulse with short pulse duration.

Figure 3. The distribution of the magnetic field of X4 along the vertical axis of the magnet. The position of $z = 0$ corresponds to the middle of the two coils.

4. X-ray diffraction in pulsed high magnetic fields

X-ray diffraction measurements in pulsed high magnetic fields have been performed by using either a single photon counting pixel array detector or an avalanche photo diode (APD) detector. The pixel array detector PILATUS100K has been developed at Swiss Light Source. It consists of an array of 487 x 192 pixels, with a pixel size of 0.172 x 0.172 mm$^2$. Each pixel can count photons up to $10^6$ per one second. The detector is equipped with a gate circuit so that a synchronization with a pulsed magnetic field is easily done by an external trigger. The gate for an exposure is open in a certain term where the pulsed magnetic field reaches a peak value. The times are set to be 1 msec for the short pulse field and 5 msec for the long pulse field, respectively. The variations during the exposures are around 5 %. The long exposure time allows us to acquire diffraction patterns from powder samples with good statistics in just one measurement. The APD detector can also be used with a multi channel scalar (MCS)
module. Although the detector does not have an ability of a spacial resolution, it works faster than the pixel detector and collects whole profile in one field pulse. Therefore the APD is best for investigating field-induced phase transitions in detail. A cryostat made of glass can be installed into the magnet and a sample is immersed in liquid Helium to lower the temperature of it down to 1.3 K. The x-ray diffraction experiment on the powder sample of CoO using X3 was reported in Refs. [3,4]. Recently we have succeeded in taking diffraction data using the newly developed X4. The experimental result will be published elsewhere.

5. Summary
We have developed several types of split-pair pulse magnets for x-ray diffraction measurements. We succeeded in generating magnetic fields up to 38 T with pulse duration of about 27 msec leading to x-ray diffraction measurements in pulsed high magnetic fields with high sensitivity of photon counting.

We are planning to install a closed cycle refrigerator to cool the pulse magnet in order to control the coil temperature easily and decrease it down to around 30 K, where the resistance of the coil of CuAg is about 70 % that at 77 K. In addition, using the cryostat without liquid nitrogen, there are no liquid as well as by-product ice which disturb incoming and outgoing x-ray beams. Furthermore, in order to increase the maximum field, we will optimize parameters of the magnet: length of the coil, number of the layers and material of the wire etc. Some parts of the reinforcement made of Maraging steel can be replaced by high strength polymer fibers ZYLON, which has no influence on generating pulsed high magnetic fields. As a result of these improvements, pulsed magnetic fields beyond 50 T should be feasible.

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