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Improvement of beamline 4B9A and results of an X-ray diffraction experiment

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4B9A is a focusing and monochromatic photon beam at the BSRF, which was constructed in 1990. During the second phase of the BSRF program, the surface of the cylindrical mirror has been coated with Pt, covering the original Ni, and the monochromator has been upgraded. The maximum photon energy extends to 11 keV and the intensity has increased about tenfold with respect to the previous intensity at 6 keV. Synchrotron X-ray diffraction patterns for the Hg-1223 ($HgBa_2Ca_2Cu_3O_{8+\delta}$) superconducting bulk and thin film have been measured at 1.54014 Å. Results indicate that the bulk and film can be indexed as possessing tetragonal symmetry; lattice parameters a = 3.856 Å and c = 15.851 Å for the bulk Hg-1223 compound, and a = 3.8517 Å and c = 15.8511 Å for the film. Their structures are similar.

Keywords: beamlines; X-ray diffraction; superconductors.

1. Introduction

Beamline 4B9A is a focusing and monochromatic photon beam at the BSRF, which was constructed in 1990 (Shu *et al.*, 1986). A bent cylindrical mirror and monochromator are two important components of the beamline. The bent cylindrical mirror provides a vertical divergence of ±0.18 mrad and horizontal acceptance of 6 mrad, and was designed by Shu *et al.* (1986). The surface of the cylindrical mirror has been coated with platinum, covering the original nickel; Mr Peter Takacs at BNL, USA, helped us to perform a quality check of the mirror surface before and after the platinum-plating. In this way the maximum photon energy is extended to 11 keV, and the intensity is increased by tenfold compared with that of the 6 keV maximum energy used previously, when BEPC was operating at an energy of 2.2 GeV. In our case, the heat load illuminating the surface of the mirror is normally less than 20 W. It is delivered *via* beam optics to the non-cooled monochromator sitting in a high-vacuum chamber at 2×10^{-8} torr. To preserve a stable intensity at the exit of the light, it is necessary to overcome the mismatch between the emittance of the first crystal and acceptance of the second crystal. After improvement, the beamline and the diffraction station have been used for more experiments.

Since the discovery of superconductivity in the Hg–Ba–Ca–Cu–O systems (Cantoni *et al.*, 1993; Antipov *et al.*, 1993), many $HgBa_2Ca_{n-1}Cu_nO_{2n+2+\delta}$ (where n = 1, 2, . . . 5) compounds have been synthesized and studied. Here we present the results of the structure analysis of the $HgBa_2Ca_2Cu_3O_{8+\delta}$ (Hg-1223) superconducting bulk and thin film, of about 0.3 μm thickness, which were studied by synchrotron X-ray diffraction at the BSRF. The Hg-1223 superconducting bulk was synthesized under high pressure and the thin film was prepared by pulse laser deposition and the implantation technique. Analysis of data from the diffraction patterns shows that the superconducting phase and an impurity phase exist in the samples, with the main phase being the superconducting phase.

2. Experiment and results

Beamline 4B9A was constructed for X-ray diffraction and X-ray small-angle scattering experiments; two experimental stations share this photon beam. When the BEPC storage ring operates at dedicated mode 2.2 GeV for synchrotron radiation, the photon characteristic energy is 2.28 keV. In the first phase of the project, owing to the surface of the focusing mirror being coated with nickel, and by fixing a glancing incident angle of 0.5°, thus having an energy cut-off of ∼7 keV, the beamline actually provided a maximum available photon energy up to 6 keV. In this work the surface of the mirror was coated with Pt, ∼400 Å thick, to cover the nickel, and the beamline successfully presents a maximum photon energy up to 11 keV. The intensity of measured photons increases about tenfold at 6 keV with respect to the mirror coated with nickel.

The monochromator, consisting of an Si(111) double crystal, is an important optics element on beamline 4B9A. Thermal bump and global lattice expansion induced by the heat load are two dominant effects contributing to a thermally induced mismatch between the emittance of the first crystal and acceptance of the second crystal. The temperatures of the two crystals are different when the beam is working. A temperature difference between the two crystals may induce a misalignment. The misalignment will require further adjustment to guarantee double diffraction. To obtain a better X-ray beam from the monochromator, the angular alignment of the two crystals must be maintained to within a small fraction of the Bragg-reflection Darwin width, *e.g.* a few arcseconds. In addition, the asymmetry of the cutting crystal leading to mismatch between the two crystals has been resolved by an alignment of Cu $K\alpha$ as a source under a conventional X-ray machine.

The thermal bump produces a slope error on the illuminated area of the crystal surface, which is estimated by (Amenitsch *et al.*, 1995)

$$\omega \simeq 1.43\alpha_{\rm therm} QD^2/2kA,$$

where $\alpha_{\rm therm}$ is the thermal expansion coefficient, Q is the incoming power, D is the crystal thickness, k is the thermal conductivity and A is the area of the beam footprint on the crystal surface. In our experiment, the incoming thermal power load, Q, ranges up to 12 W, the size of the beam footprint A on the Si(111) crystal of the monochromator is about 40 mm^2, and a calculation using the above formula indicated that the slope error, ω, ranged from several hundred angstroms up to ∼1400 Å.

For the duration of the experiment, it is necessary to preserve the source brightness (photon flux per second per unit area per unit bandwidth per unit solid angle) in order to maintain a stable intensity within a deviation at a given energy. Our method involves mounting the piezoelectric (PZT) cera-

mics on the support of the first crystal of the monochromator. As mentioned previously, a thermal power load density of 0.3 W mm^{-2} on the first crystal is lower, so the alignment of the two crystals was carried out by using a PZT actuator with the feedback function, which is satisfactory and cheaper than the water-cooling system. The ceramics are normally polarized. When a voltage is applied to the PZT, the dimensions of the piezoelectric plate change – they expand or shrink. Controlling the value of the voltage (positive or negative) applied to the PZT, the head angle of the first crystal will be rotated with fine tuning; the second crystal is immovable. When the BEPC operating current decreases with time, the intensity also reduces, because the lifetime of the electron bunch at the BEPC is 6–8 h. In this case, by adjusting the voltage applied to the PZT ceramics, one can obtain stability of intensity. This method is successful and efficient when the slope error, produced by heat load and misalignment induced by a temperature difference between the two crystals, is small.

After improvement of the beam optics, the performance of the beamline should be as follows: photon energy range 3.5–11 keV; energy resolution $\Delta E/E \simeq 4 \times 10^{-4}$; intensity 5×10^9 photons s^{-1} at 6 keV; storage ring current 50 mA; $\Delta I/I \simeq$ 2–5% during a data-collection period of ~1 h. The diffraction pattern for the sample of Si powder was measured: the line width of the (111) reflection peak was 0.04°.

Several experiments have been performed at the beamline under the above-described conditions. One particular experiment was to determine the structure of superconductors by synchrotron X-ray diffraction. The superconducting bulk Hg-1223 was prepared under high pressure and annealed for 6 h, with oxygen flow. A pulse laser deposition technique was used to synthesize the precursor. This precursor has the nominal composition $Ba_2Ca_2Cu_3O_x$, which was prepared using the high-purity metal oxides BaO, CaO and CuO at 1073–1173 K for 48 h. The flow of oxygen was applied throughout the whole period. These calcined mixtures were pressed into a disc for use as a target for pulse laser deposition. By ablating $Ba_2Ca_2Cu_3O_x$ on the (100) $SrTiO_3$ substrate with the pulse laser deposition technique to form a thin film of ~1 μm thickness, and by implanting mercury ions into this film, the superconducting thin films are formed.

The structures of the Hg-1223 ($HgBa_2Ca_2Cu_3O_{8+\delta}$) superconducting bulk and thin film were determined using synchrotron radiation X-ray diffraction at a wavelength of 1.54014 Å. An X-ray diffractometer with an angular measured precision of 0.001° performs the angular scanning of the samples. To normalize the data collection, the incident intensity of photons was monitored using an ionization chamber, and a scintillation counter measured the diffraction X-ray intensity. Data were collected at room temperature by step scanning. The data-collection range was $20° \leq 2\theta \leq 70°$. A 2θ step size of 0.02° and a counting time of 1 s per point was used. X-ray diffraction patterns of Hg-1223 samples were obtained and are shown in Fig. 1 for the Hg-1223 bulk and in Fig. 2 for the superconducting film. The lattice parameters were calculated by fitting the X-ray diffraction peaks using a standard analysis program. The results indicate that the diffraction patterns of the superconducting bulk and film can be indexed in tetragonal symmetry. Lattice parameters a = 3.856 Å and c = 15.851 Å for the Hg-1223 superconducting bulk and a = 3.8517 Å and c = 15.8511 Å for the Hg-1223 superconducting film were obtained. The structures are found to be similar. The detected non-superconducting impurities in the film were identified as $BaCuO_2$, $CaHgO_2$ and CuO phases, which were calculated by utilizing a standard Rietveld program, similar to those impurities appearing in the bulk Hg-1223.

The superconducting transition temperature was 118 K with a transition width of ~9 K for the film, which was measured by magnetic susceptibility. Using a film size of 5.1 mm × 5.8 mm

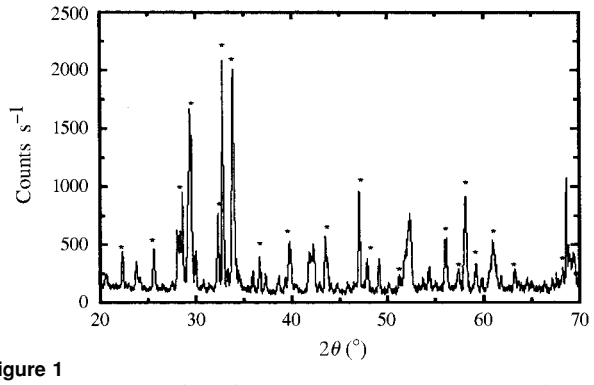

Figure 1
Synchrotron X-ray diffraction pattern of the superconducting bulk $HgBa_2Ca_2Cu_3O_{8+\delta}$ sample. The diffraction peaks of the superconducting phase are marked by small stars. The others are the diffraction peaks from impurities.

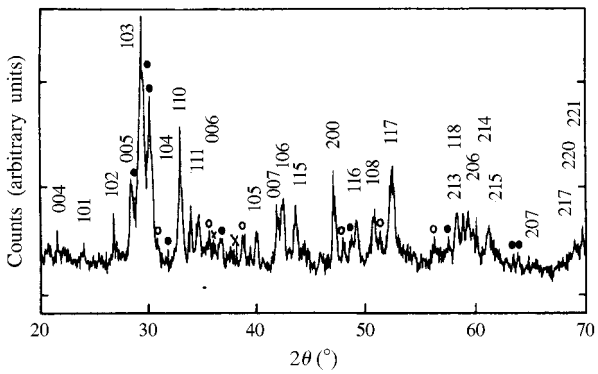

Figure 2
Synchrotron X-ray diffraction pattern of the Hg-1223 film sample at the optimum annealing condition. The impurity reflections are marked by dots (●) for $BaCuO_2$, circles (○) for $CaHgO_2$ and crosses (×) for CuO.

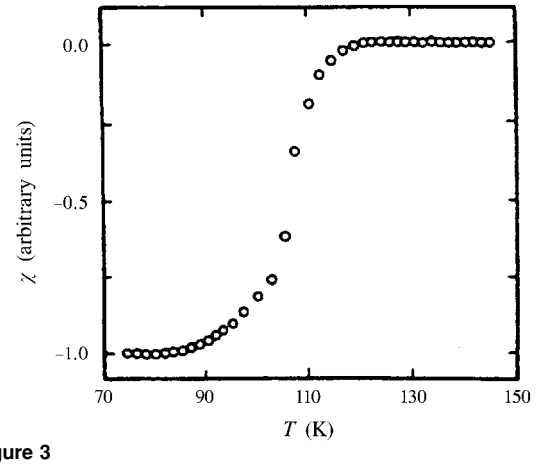

Figure 3
Susceptibility of the superconducting Hg-1223 film sample. The superconducting transition is 118 K and $\Delta T_c \simeq$ 9 K.

$\times$ 0.3 μm, a.c. susceptibility *versus* T for a superconducting thin film of Hg-1223 is shown in Fig. 3.

3. Conclusions

In our case, with the platinum-coated mirror, the range of photon energy was extended successfully. As for applying a positive or negative voltage to the PZT, it depends on the temperature decreasing or increasing on the surface of the crystal, which can improve the stability of the intensity at the beamline exit. The 0.3 μm samples of Hg-1223 superconducting thin film prepared by pulse laser deposition and the implanting technique were successfully analysed by synchrotron X-ray diffraction and indexed as possessing tetragonal symmetry. The lattice parameters are very similar for the thin film and bulk in our experiment. Measurements of several samples indicate that the optimum annealing conditions were achieved at 893 K for 60 h for the better superconducting film.

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