

# Element Substitution Effect on Superconductivity in BiS<sub>2</sub>-Based NdO<sub>1-x</sub>F<sub>x</sub>BiS<sub>2</sub>

Takafumi Hiroi, Joe Kajitani, Atsushi Omachi, Osuke Miura, and Yoshikazu Mizuguchi\*

Department of Electrical and Electronic Engineering, Tokyo Metropolitan University,  
1-1, Minami-osawa, Hachioji, 192-0397, Japan

Corresponding author: Y. Mizuguchi

E-mail: mizugu@tmu.ac.jp

Telephone: +81-42-677-2748

FAX: +81-42-677-2756

## Abstract

Recently, new layered superconductors having a BiS<sub>2</sub>-type conduction layer have been discovered. NdO<sub>1-x</sub>F<sub>x</sub>BiS<sub>2</sub> is a typical BiS<sub>2</sub>-based superconductor with a maximum  $T_c$  of 5.4 K. In this study, the effect of element substitution within the superconducting layer of BiS<sub>2</sub>-based NdO<sub>0.5</sub>F<sub>0.5</sub>BiS<sub>2</sub> was investigated. We systematically synthesized two kinds of polycrystalline samples of NdO<sub>0.5</sub>F<sub>0.5</sub>Bi(S<sub>1-x</sub>Se<sub>x</sub>)<sub>2</sub> and NdO<sub>0.5</sub>F<sub>0.5</sub>Bi<sub>1-y</sub>Sb<sub>y</sub>S<sub>2</sub> by a two-step solid-state reaction method. The phase purity and the changes in lattice constants were investigated by x-ray diffraction. The superconducting properties were investigated by magnetic susceptibility and electrical resistivity measurements. It was found that the partial substitution of S by Se resulted in the uniaxial lattice expansion along the  $a$  axis. The superconducting transition temperature were gradually degraded

with increasing Se concentration in  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}(\text{S}_{1-x}\text{Se}_x)_2$ . When Bi was partially substituted by Sb, the uniaxial lattice contraction along the *c* axis was observed in  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}_{1-y}\text{Sb}_y\text{S}_2$ . In the Sb-substituted system, a metal-insulator transition was observed with increasing Sb concentration.

Keywords: BiS<sub>2</sub>-based superconductor,  $\text{NdO}_{1-x}\text{F}_x\text{BiS}_2$ , element substitution, metal-insulator transition

## 1. Introduction

Since the discovery of the novel layered superconductor  $\text{Bi}_4\text{O}_4\text{S}_3$ , many studies for exploring new superconductors and clarifying the mechanisms of superconductivity in the  $\text{BiS}_2$  family have been carried out [1]. So far, three types of  $\text{BiS}_2$ -based superconductors,  $\text{Bi}_4\text{O}_4\text{S}_3$  [1],  $\text{REOBiS}_2$  ( $\text{RE} = \text{La, Ce, Pr, Nd, Yb}$ ) [2-9],  $\text{SrFBiS}_2$  [10,11], have been discovered. The highest record of the transition temperature ( $T_c$ ) is 11 K in  $\text{LaO}_{0.5}\text{F}_{0.5}\text{BiS}_2$  synthesized using a high-pressure technique. These  $\text{BiS}_2$ -based superconductors basically have a layered structure composed of common  $\text{BiS}_2$  superconducting layers and the blocking layers, which is quite similar to those of the cuprate and the Fe-based superconductors [13,14]. The superconducting properties of the  $\text{BiS}_2$  family can be tuned by changing the structure of blocking layer. To induce superconductivity in the  $\text{BiS}_2$ -based layered materials, electron doping into the  $\text{BiS}_2$  layers is required [15-20]. For example, in the  $\text{LaOBiS}_2$  system, superconductivity is induced by a partial substitution of  $\text{O}^{2-}$  by  $\text{F}^-$  or a partial substitution of  $\text{La}^{3+}$  by  $\text{M}^{4+}$  [2,21] at the blocking layers. Namely, the element substitution at the blocking layer is effective to induce/tune superconductivity in the  $\text{BiS}_2$ -based family. Furthermore, it was reported that the superconducting properties are sensitive to the change in the local crystal structures [22]. For the  $\text{REOBiS}_2$  systems ( $\text{RE} = \text{La, Ce and Pr}$ ), the optimal (the highest) superconducting properties are obtained by optimizing the crystal structures by using a high-pressure technique [2,4,6,23-27].

Although there are many reports on element substitution effects of blocking layers in  $\text{REO}_{1-x}\text{F}_x\text{BiS}_2$ , the effect of element substitution at the superconducting layer has been reported in only one report. In  $\text{Bi}_4\text{O}_4\text{S}_3$ , partial substitution of S by Se decreased its  $T_c$

[28]. In this study, the effect of element substitution at the superconducting layers on superconductivity of  $\text{NdO}_{0.5}\text{F}_{0.5}\text{BiS}_2$  was investigated. We synthesized Se-substituted  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}(\text{S}_{1-x}\text{Se}_x)_2$  and Sb-substituted  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}_{1-y}\text{Sb}_y\text{S}_2$ . These element substitutions basically generate the changes in the lattice constants without any change in the carrier concentration.

## 2. Experimental

In this study, the F concentration was fixed to be  $x = 0.5$  because the highest  $T_c$  was obtained for  $x = 0.5$  in  $\text{NdO}_{1-x}\text{F}_x\text{BiS}_2$ . The polycrystalline samples of  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}(\text{S}_{1-x}\text{Se}_x)_2$  and  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}_{1-y}\text{Sb}_y\text{S}_2$  ( $x, y = 0, 0.1$  and  $0.2$ ) were prepared by the two-step solid-state reaction method. The starting materials for  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}(\text{S}_{1-x}\text{Se}_x)_2$  are  $\text{Nd}_2\text{O}_3$  powders,  $\text{Nd}_2\text{S}_3$  powders,  $\text{NdF}_3$  powders, Bi grains,  $\text{BiSe}_2$  powders and S grains. The starting materials for  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}_{1-y}\text{Sb}_y\text{S}_2$  are  $\text{Nd}_2\text{O}_3$  powders,  $\text{Nd}_2\text{S}_3$  powders,  $\text{NdF}_3$  powders, Bi grains, Sb powders and S grains. The mixtures with the nominal compositions of  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}(\text{S}_{1-x}\text{Se}_x)_2$  and  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}_{1-y}\text{Sb}_y\text{S}_2$  were mixed-well, ground, pelletized, sealed in an evacuated quartz tube and heated at  $800^\circ\text{C}$  for 15 h. The obtained products were ground, pelletized, sealed in an evacuated quartz tube and annealed at  $800^\circ\text{C}$  for 15 h again. The obtained samples were characterized by powder X-ray diffraction (XRD) using the  $\theta - 2\theta$  method with a  $\text{CuK}\alpha$  radiation. The lattice constants were calculated using the peak positions. The temperature dependence of magnetization was measured by a superconducting quantum interface device (SQUID) magnetometer with an applied field of 5 Oe. The

temperature dependence of electrical resistivity was measured by the four-probe method.

### 3. Results

Figure 1 shows the powder XRD patterns of (a)  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}(\text{S}_{1-x}\text{Se}_x)_2$  and (b)  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}_{1-y}\text{Sb}_y\text{S}_2$  ( $x, y = 0, 0.1$  and  $0.2$ ). These XRD patterns were indexed using the  $P4/nmm$  space group. In the XRD patterns of  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}(\text{S}_{1-x}\text{Se}_x)_2$ , impurity peaks appeared in the doping range of  $y \geq 0.1$ . In contrast, the impurity peaks were not detected upon Sb substitution in the XRD patterns of  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}_{1-y}\text{Sb}_y\text{S}_2$ . Almost all of the peaks became slightly broader in the substituted samples. The lattice constants  $a$  and  $c$  were calculated using the positions of the (200) peaks and the (004) peaks, respectively.

The Se-concentration dependences of the lattice constants of the  $a$  axis and  $c$  axis for  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}(\text{S}_{1-x}\text{Se}_x)_2$  is shown in Figs. 2(a) and (b), respectively. With increasing Se concentration, the lattice constant of the  $a$  axis increased while the lattice constants of the  $c$  axis did not change obviously in  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}(\text{S}_{1-x}\text{Se}_x)_2$ . The Se substitution for the S site resulted in a uniaxial lattice expansion along the  $a$  axis.

The Sb-concentration dependences of the lattice constants of the  $a$  axis and  $c$  axis for  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}_{1-y}\text{Sb}_y\text{S}_2$  is shown in Figs. 2(a) and (b), respectively. With increasing Sb concentration, the lattice constants of the  $a$  axis did not change obviously while the lattice constant of the  $c$  axis decreased in  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}_{1-y}\text{Sb}_y\text{S}_2$ . The Sb substitution for the Bi site resulted in a uniaxial lattice contraction along the  $c$  axis.

The temperature dependences of magnetic susceptibility ( $\chi$ ) of  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}(\text{S}_{1-x}\text{Se}_x)_2$  ( $x = 0, 0.1$  and  $0.2$ ) are shown in Fig. 3. The inset shows the Se-concentration dependences of the  $T_{\text{c\_onset}}$  and the  $T_{\text{c\_10\%}}$ . The  $T_{\text{c\_10\%}}$  is defined as a temperature where the value of  $\chi$  becomes 10 % of  $\chi(2 \text{ K})$ . The  $T_{\text{c\_onset}}$  and  $T_{\text{c\_10\%}}$  decreased with increasing Se concentration. The shielding volume fraction of  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}(\text{S}_{1-x}\text{Se}_x)_2$  also decreased drastically. On the basis of these results, we concluded that the Se substitution for the  $\text{NdO}_{0.5}\text{F}_{0.5}\text{BiS}_2$  superconductor degraded superconducting properties and finally destroyed the superconducting states.

For the  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}_{1-y}\text{Sb}_y\text{S}_2$  system, superconductivity was strongly suppressed by the Sb substitution. Even for  $y = 0.1$ , superconducting transition was not observed in the magnetic susceptibility measurements. Therefore, we performed electrical resistivity measurements to investigate the Sb substitution effect on the physical properties. Figure 4 shows the temperature dependences of electrical resistivity for  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}_{1-y}\text{Sb}_y\text{S}_2$ . The resistivity drastically increased with increasing Sb concentration. When the Sb concentration was  $y = 0.1$ , the value of resistivity was  $1.90 \times 10^2 \text{ m}\Omega\text{cm}$  at 6 K. When the Sb concentration was  $y = 0.2$ , resistivity was  $1.35 \times 10^5 \text{ m}\Omega\text{cm}$  at 6 K. The value of resistivity for  $y = 0.2$  was about  $10^3$  times larger than that of  $y = 0.1$ . The remarkable change suggests that the Sb substitution for the Bi site results in the metal-insulator transition in the  $\text{NdO}_{1-x}\text{F}_x\text{BiS}_2$  system. Having considered the one-dimensional character of the electronic states in the  $\text{BiS}_2$ -based materials [15], we speculate that the observed metal-insulator transition would be related to the Anderson localization. To investigate further, detailed studies on transport properties and crystal structure using single crystals are needed.

#### 4. Conclusions

The effect of the element substitution within the superconducting layer on superconductivity in the BiS<sub>2</sub>-based superconductor NdO<sub>0.5</sub>F<sub>0.5</sub>BiS<sub>2</sub> was investigated. The polycrystalline samples of NdO<sub>0.5</sub>F<sub>0.5</sub>Bi(S<sub>1-x</sub>Se<sub>x</sub>)<sub>2</sub> and NdO<sub>0.5</sub>F<sub>0.5</sub>Bi<sub>1-y</sub>Sb<sub>y</sub>S<sub>2</sub> ( $x, y = 0, 0.1$  and  $0.2$ ) were synthesized by the two-step solid state reaction method. When S was substituted by Se, the lattice constant of the *a* axis increased, and the superconducting properties ( $T_c$  and shielding volume fraction) were degraded. When Bi was substituted by Sb, the lattice constant of the *c* axis decreased, and a metal-insulator transition was observed. The element substitution within the superconducting layer degrades superconductivity in the NdO<sub>1-x</sub>F<sub>x</sub>BiS<sub>2</sub> system.

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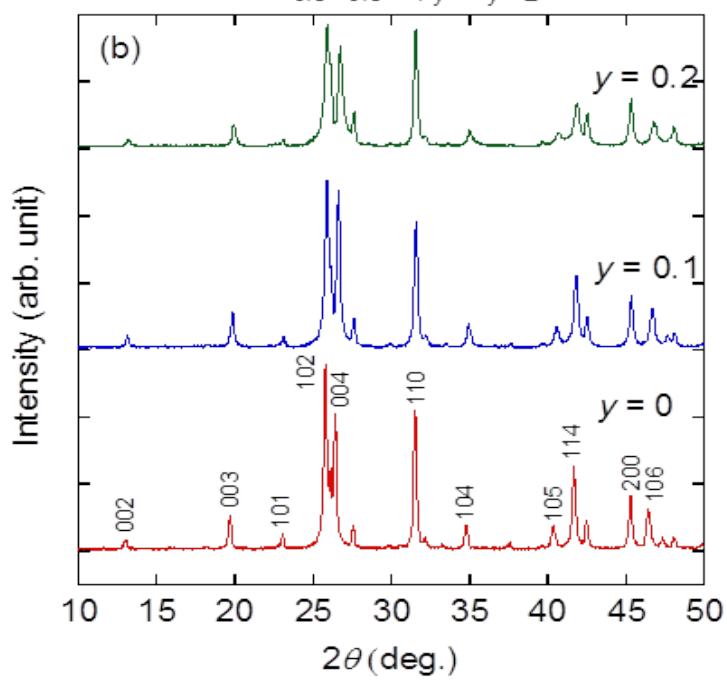
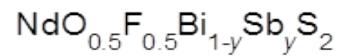
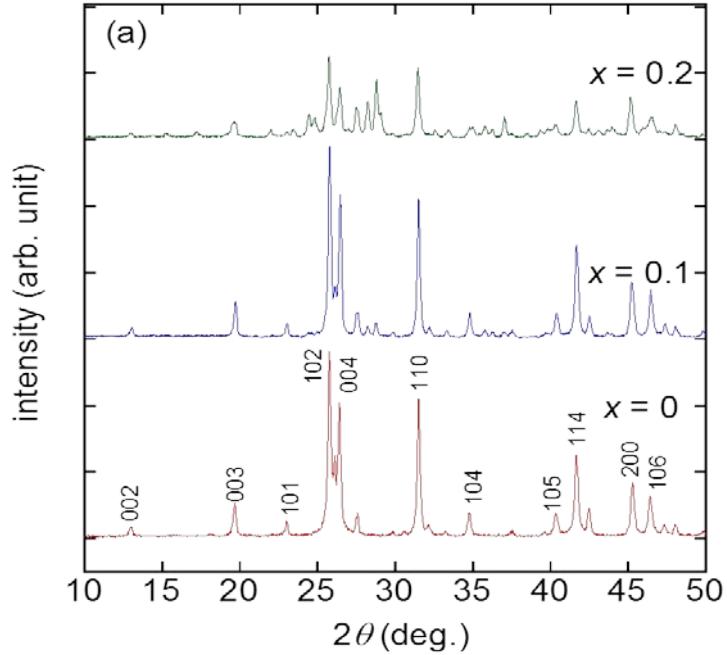


Fig. 1 XRD patterns of (a)  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}(\text{S}_{1-x}\text{Se}_x)_2$  ( $x = 0, 0.1, 0.2$ ) and (b)  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}_{1-y}\text{Sb}_y\text{S}_2$  ( $y = 0, 0.1, 0.2$ ).

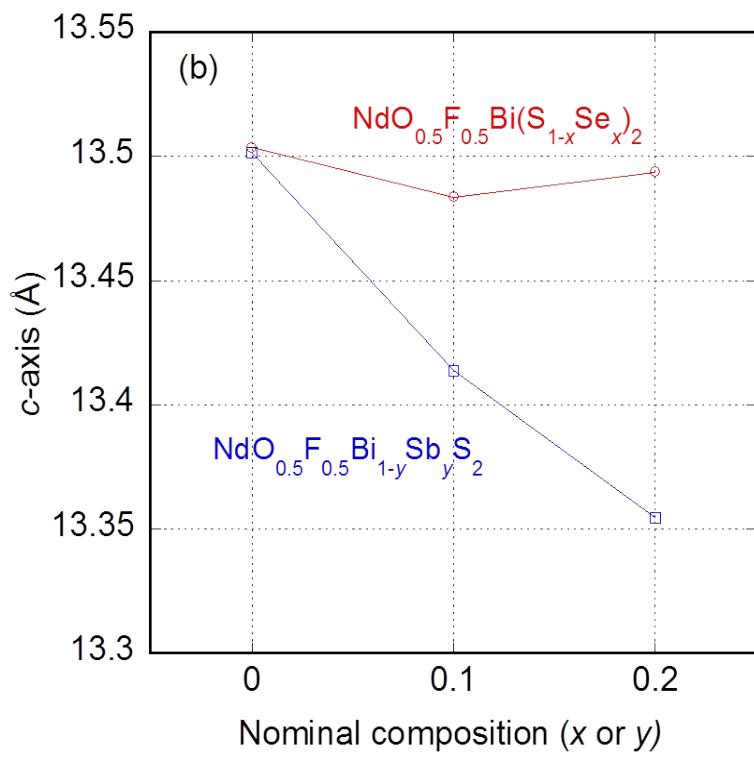
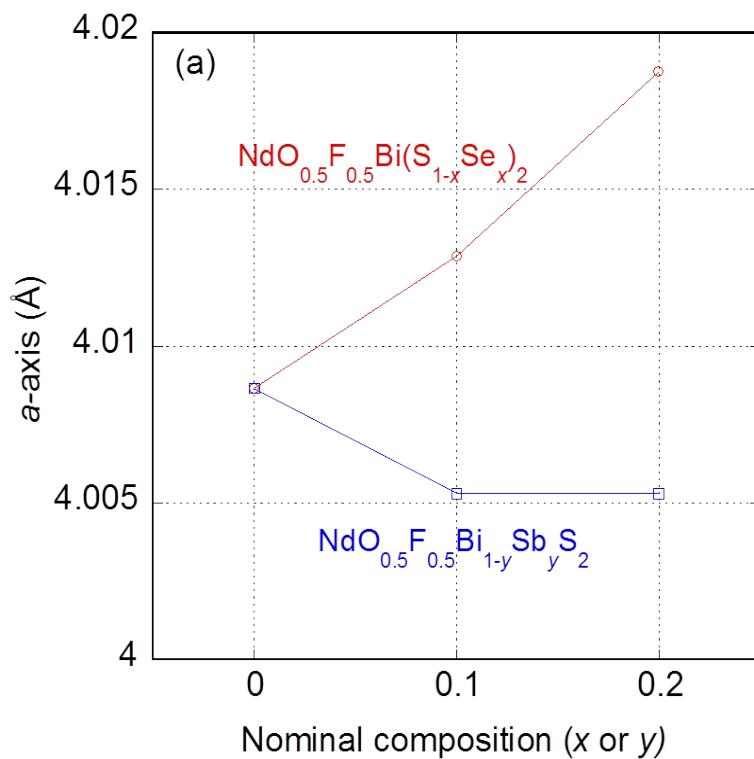


Fig. 2 Se-concentration  $x$  dependence (or Sb-concentration  $y$  dependence) of the lattice constants of (a)  $a$ -axis and (b)  $c$ -axis.

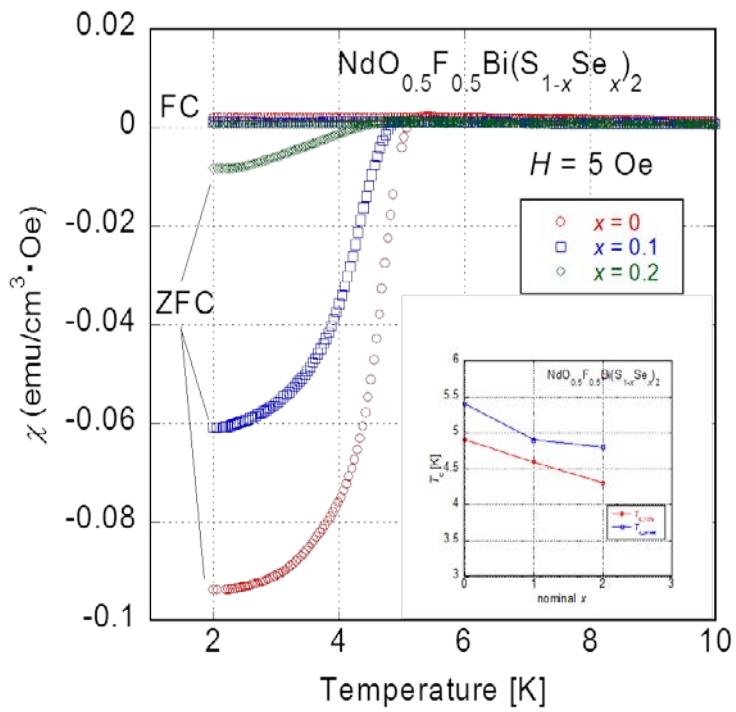


Fig. 3 Temperature dependences of the magnetic susceptibility ( $\chi$ ) of  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}(\text{S}_{1-x}\text{Se}_x)_2$  ( $x = 0, 0.1, 0.2$ ).

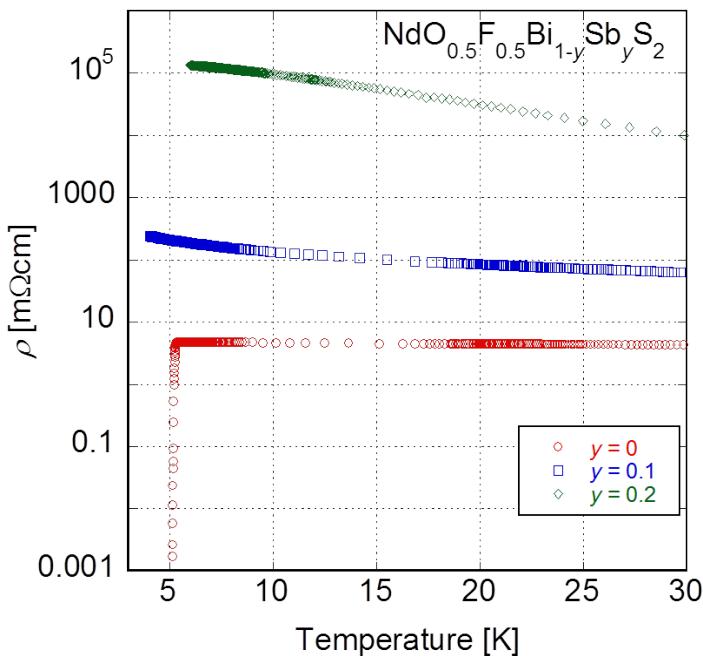


Fig. 4 Temperature dependences of electrical resistivity ( $\rho$ ) of  $\text{NdO}_{0.5}\text{F}_{0.5}\text{Bi}_{1-y}\text{Sb}_y\text{S}_2$  ( $y = 0, 0.1, 0.2$ ).