

Short Communication

Study of the structural, superconducting and electrochemical corrosion properties of F and Se co-doped LaOBiS₂ materials

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This study was conducted to successfully synthesize LaOBiS₂, F-doped LaOBiS₂, Se-doped LaOBiS₂, and F and Se-codoped LaOBiS₂ by the standard solid state reaction route, and study the structural, superconductivity, and electrochemical corrosion properties. The layered nature of the pure and doped LaOBiS₂ samples' crystals and their growth along the c-axis were revealed by structural investigations. Results revealed that Fe and Se doping caused expansion along the c-axis, respectively, confirming the effectiveness of the doping method. Studies on the relationship between resistance and temperature revealed a rapid superconducting transition for the F-doped LaOBiS₂ system with a T_c of 3.18 K, and F-ion substitution can increase the superconductivity. The results demonstrated that Se substitution could be the cause of the elevated carrier mobility by successfully suppressing local distortion. Results showed that F and Se doping increased the density of electron carriers. The temperature dependence of the ZFC and FC DC magnetic susceptibilities was studied, and the results revealed that the higher T_c of the F and Se-codoped LaOBiS₂ led to a diamagnetic transition into the superconducting state at T_c~3.8 K. According to electrochemical corrosion investigations, F and Se codoped LaOBiS₂ had a higher charge transfer resistance than other electrodes, showing a synergistic effect of F and Se on enhancing corrosion resistance.

Keywords: Superconductivity; LaOBiS₂; Se doping; Fe doping; Solid state reaction method; Electrochemical corrosion

1. INTRODUCTION

A superconductor is a perfect conductor of electric charge [1, 2]. It experiences no resistance and an electric current in a superconductor can persist indefinitely. It does not allow magnetic fields to penetrate [3, 4]. So, in addition to being incredibly effective, superconducting materials also have the added advantage of being able to float. When chilled below a specific temperature, certain materials have the ability to carry direct current electricity without losing energy (referred to as T_c) [5-7].

Superconducting wire can carry immense electrical currents with no heating, which allows it to generate large magnetic fields [8, 9]. An electromagnet with non-superconducting copper windings would melt with the same current [10, 11].

The world could change thanks to superconductors. A superconductor operating at ambient temperature would change technology [12, 13]. Since energy is not lost through resistance in a superconducting power grid, it would consume significantly less energy than current technologies. Applications of superconductivity include levitating trains, extremely precise magnetoencephalograms, and smaller and lighter engines, generators, and transformers [14]. Cellular phone towers now use high-temperature superconductors as electronic filters to separate radio signals from different phone calls. Conventional filters lose some of the signal due to electrical resistance, and the sharper the filter, the more of the signal it loses. Nuclear magnetic resonance (NMR) and electromagnetic resonance imaging (NMR) In order to create the large-volume, steady, and high-intensity magnetic fields required for MRI and NMR, superconductivity is most commonly used [15, 16]. Railguns, coilguns, cell phone base stations, quick digital circuits, and particle detectors are all powered by superconductors [17, 18].

Though there has been clear progress in research and innovation, superconductors are not a novel technology, and there is still much space for advancement. Aluminium, niobium, magnesium diboride, cuprates such as yttrium barium copper oxide and iron pnictides, as well as chemical substances with layered crystal structures, are prominent examples of superconductors [19, 20]. Superconductors face a serious problem when it comes to other practical applications: they operate at incredibly low temperatures [21, 22]. It is impossible to find superconductors at room temperature. Superconducting materials only really superconduct when kept below the transition temperature, which is a specified temperature. For known practical superconductors, the temperature is significantly lower than 77 K, the temperature of liquid nitrogen [23-25].

Recently, studies have shown that superconductivity was discovered in the BiS₂-based layered compounds such as Bi₄O₄S₃, Bi₃O₂S₃, Sr_{1-x}La_xFBiS₂, SrFBiS₂ and LnO_{1-x}F_xBiS₂ (Ln = La, Ce, Pr, Nd and Yb) [26, 27]. The crystal structure composed of an alternate stacking of the BiS₂ superconducting layers and blocking layers is quite similar to those of the Cu-oxide and Fe-based superconductors [28-30]. Thus, the BiS₂-based materials have been demonstrated to become superconducting upon electron doping into the Bi-6p orbitals within the BiS₂ layers [27, 31]. To the best of our knowledge, the electrochemical corrosion studies of superconductors based on BiS₂ are not successfully carried out. LaOBiS₂, F-doped LaOBiS₂, Se-doped LaOBiS₂, and F and Se-codoped LaOBiS₂ were effectively synthesized in this study, and its structural, superconducting, and electrochemical corrosion properties were investigated.

2. EXPERIMENT

2.1. Synthesis of superconductor

LaOBiS₂ was prepared using the standard solid state reaction route via vacuum encapsulation [32]. The powders of La (99%, Sigma-Aldrich), Bi (99%, Merck Millipore, Germany), S (97%, Merck

Millipore, Germany), LaF_3 (99%, Sigma-Aldrich), and La_2O_3 (99%, Sigma-Aldrich) and either F ($\geq 99.5\%$, Sigma-Aldrich) and Se (99.99%, Sigma-Aldrich) were weighed in a stoichiometric ratio of $\text{LaO}_{0.5}\text{F}_{0.5}\text{BiS}_2$ as perfect superposition of F-doped LaOBiS_2 which was confirmed in many studies [33-35]. The ground and homogenously mixed thoroughly in a very clean glove box which filled with a high purity argon atmosphere. Then, the mixed powder was pelletized in a quartz tube under 10^{-3} Pa which heated in a tube furnace at 850°C for 8 hours with a heating rate of $5^\circ\text{C}/\text{min}$.

2.2. Characterizations of instruments

The crystal structure of the starting material was examined by X-ray diffraction (XRD; MiniFlex-600, Rigaku Corporation, Tokyo, Japan). Scanning electron microscopy (SEM) experiments were performed on an environmental scanning electron microscope (FEI Model Quanta 450 FEG, Hillsboro, OR, USA). The temperature dependence of resistivity was recorded with a four-probe set-up in a Physical Property Measurement System (PPMS-9, Quantum Design Inc., USA). A Quantum Design PPMS-AG system (Quantum Design Inc., USA) was used for the measurement of temperature dependence of DC magnetization for the samples under 10 Oe in both zero fields cooled (ZFC) and field cooled (FC) modes. Electrochemical impedance spectroscopy (EIS) experiments were carried out using an electrochemistry workstation (CS1005, Zhengzhou CY Scientific Instrument Co., Ltd., China) equipped with a conventional three-electrode system containing a superconductor disc as the working electrode, platinum mesh as counter electrode, and a saturated calomel electrode as a reference electrode. EIS experiments were performed in 0.1 M Na_2SO_4 ($\geq 99.0\%$, Sigma-Aldrich) at frequency range of 10^{-2} – 10^5 Hz with ± 10 mV amplitude.

3. RESULTS AND DISCUSSION

3.1. Structural analyses

Figure 1 shows the XRD patterns of LaOBiS_2 , Se-doped LaOBiS_2 , F-doped LaOBiS_2 and F and Se-codoped LaOBiS_2 . As seen for all XRD patterns, there are the main diffraction peaks, which corresponded to the reported tetragonal crystal phase of CeOBiS_2 with the space group $P4/nmm$ [36]. For F-doped LaOBiS_2 and F and Se-codoped samples, small amounts of Bi_2O_3 and Bi_2Se_3 impurity phases are observed as indicated by an asterisk in the XRD patterns. By substituting the F atoms at the oxygen site, these impurity phases can be created because their synthesis temperature is relatively low and can break the chemical stoichiometry [37], similar to the F doping in the crystal structures of superconducting $\text{LaO}_{1-x}\text{F}_x\text{BiS}_2$ [38, 39]. The pure LaOBiS_2 indicates to refined lattice parameters of 'a' and 'c' which are lower than those of Se-doped LaOBiS_2 samples, illustrating to lattice expansion because the atomic radius of Se is bigger than that of S [40-42]. For studying the influence of F and Se doping, codoping, and structure modulation on LaOBiS_2 , the refined lattice parameters of LaOBiS_2 , F-doped LaOBiS_2 , Se-doped LaOBiS_2 , and F and Se-codoped LaOBiS_2 from the XRD data are depicted in Figure 2. It is observed that for F and Se-codoped LaOBiS_2 , the crystal structure shows a lattice shrinkage along the c-axis, while the a-axis remains almost unchanged. The Se substitution on S sites

may expand the lattice, and it is observed that there is separation between the (102) and (004) peaks which relates to a characteristic feature for F-doped LaOBiS_2 superconductors [43–45]. The (004) peak slightly shifts to higher angles after F doping at the O-site, indicating that the c axis decreases [43], while the position of the (110) peak (a -axis) remains almost unchanged attributed to the unchanged tetragonal crystal structure of LaOBiS_2 [46, 47].

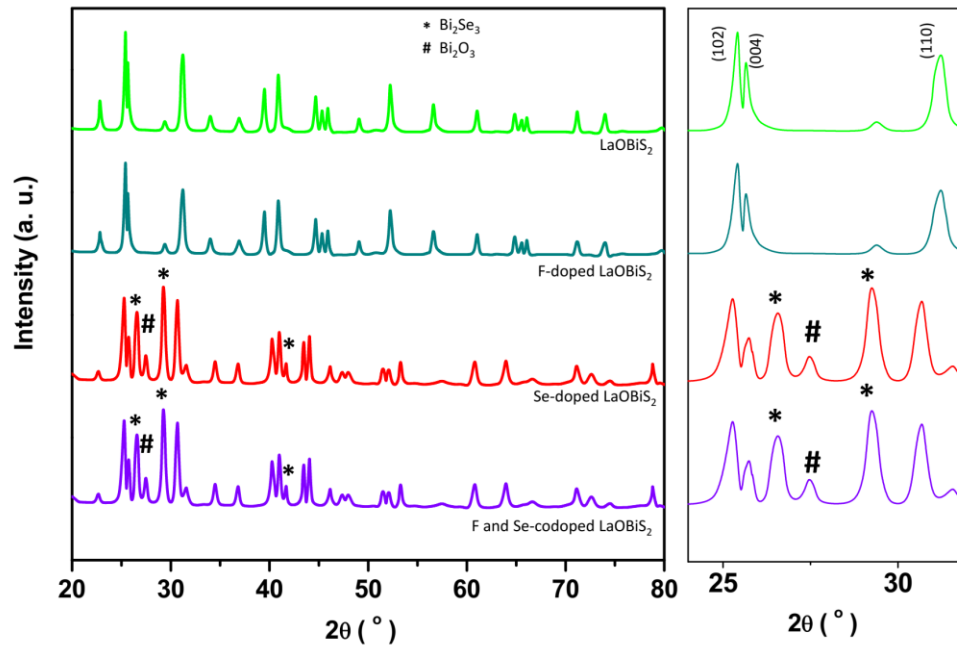


Figure 1. XRD patterns of LaOBiS_2 , Se-doped LaOBiS_2 , F-doped LaOBiS_2 and F and Se-codoped LaOBiS_2 .

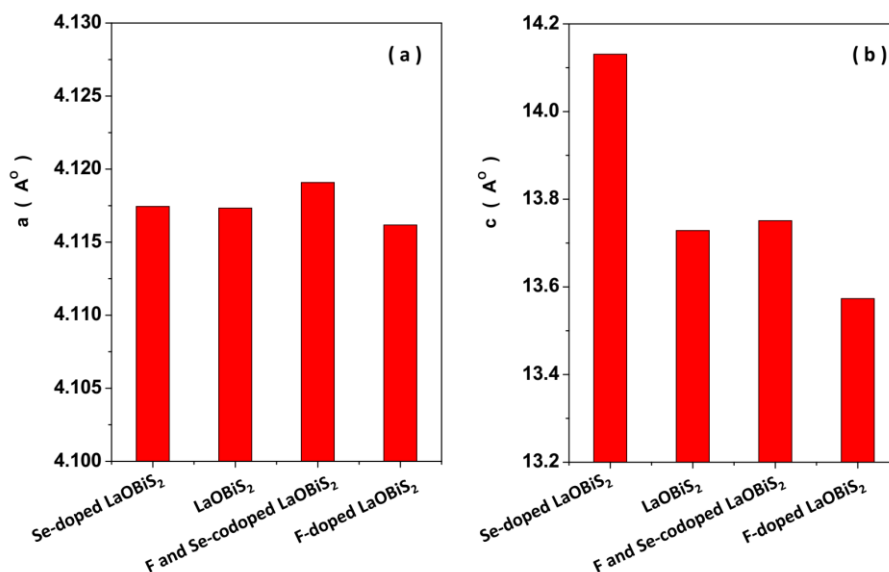


Figure 2. The refined lattice parameters of LaOBiS_2 , F-doped LaOBiS_2 , Se-doped LaOBiS_2 , and F and Se-codoped LaOBiS_2 from the XRD data (a) 'a' and (b) 'c'.

Figure 3 exhibits the SEM images of F-doped LaOBiS_2 , Se-doped LaOBiS_2 , and F and Se-codoped LaOBiS_2 . As seen, almost all of the samples are synthesized in crystal structure with slice-

shaped crystals in the main phase which mix with a few slice-shaped ones, illustrating the nature of the layered structure of LaOBiS_2 samples. The F and Se-codoped LaOBiS_2 sample shows a larger surface area and more porosity. However, the SEM image of Se-doped LaOBiS_2 displays a slice-shaped crystal that grew along the c-axis, indicating that the F doping leads to a remarkable shrinkage along the c-axis that is in agreement with the XRD results.

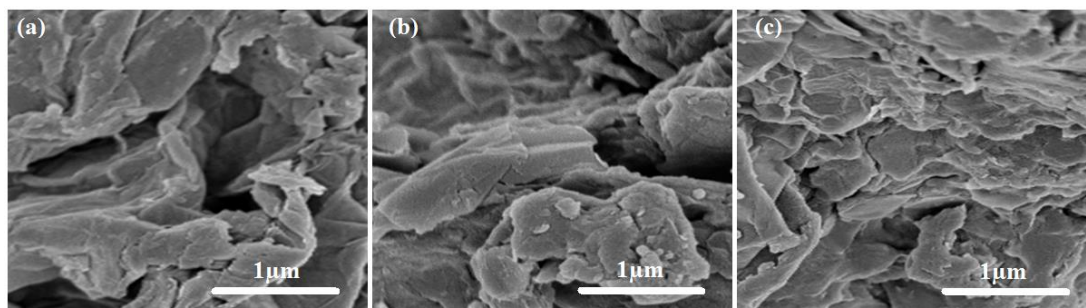


Figure 3. The SEM images of F-doped LaOBiS_2 , Se-doped LaOBiS_2 , and F and Se-codoped LaOBiS_2 .

3.2. Superconductivity analyses

Figure 4 depicts the in situ temperature dependence of the resistance of LaOBiS_2 , F-doped LaOBiS_2 , Se-doped LaOBiS_2 , and F and Se-codoped LaOBiS_2 measured at different temperatures from 2 K to 300 K. It is observed, the resistivity profiles of LaOBiS_2 , F-doped LaOBiS_2 samples exhibit semiconducting-like characteristics in normal state in the temperatures range from 2 K to 300 K, the resistivity increases with decreasing temperature, and the F-doped LaOBiS_2 indicates a sharp superconducting transition with T_c of 3.18 K, which is sizably higher than that of reported data for LaOBiS_2 (2.6 K) [48], which can be attributed to the main factors at F doping that included the increase of the charge transfer between adjacent blocks $[\text{LnO}] \rightarrow [\text{BiS}_2]$ ($\text{Ln}=\text{La}$) related to metallization of superconducting blocks $[\text{BiS}_2]$; and the reconstruction of the 2D-like Fermi surface associated with the nesting effect [49, 50]. It should be noted that the effect of chemical pressure on the superconductivity of doped LaOBiS_2 with F-ion substitution can enhance the superconductivity in the F-doped LaOBiS_2 system [51]. Moreover, studies have shown that an effective method for doping electrons into the BiS_2 conduction layer is the partial substitution of the O site at the blocking layer by F, and because of the difference in the valency between O^{2-} and F, electron doping into the BiS_2 layers is expected [52, 53]. As seen, the resistivity for doped and codoped samples is obviously smaller than that of undoped LaOBiS_2 , indicating that electron carrier density was enhanced by F and Se doping [52, 54]. Results show that the resistivity profiles of Se-doped LaOBiS_2 exhibit similar semiconducting behavior but the resistivity profiles of F and Se-codoped LaOBiS_2 demonstrate metallic behavior at normal state. The resistivity of F and Se-codoped LaOBiS_2 samples shows a linear temperature dependence as the temperature varied from 50 K to 300 K with superconducting phase transitions at low temperatures which is clearly different from the LaOBiS_2 , F-doped LaOBiS_2 samples, implying that Se substitution effectively suppresses local distortion and can be responsible for the increased carrier mobility together with a change in the Fermi level electronic structure [55, 56].

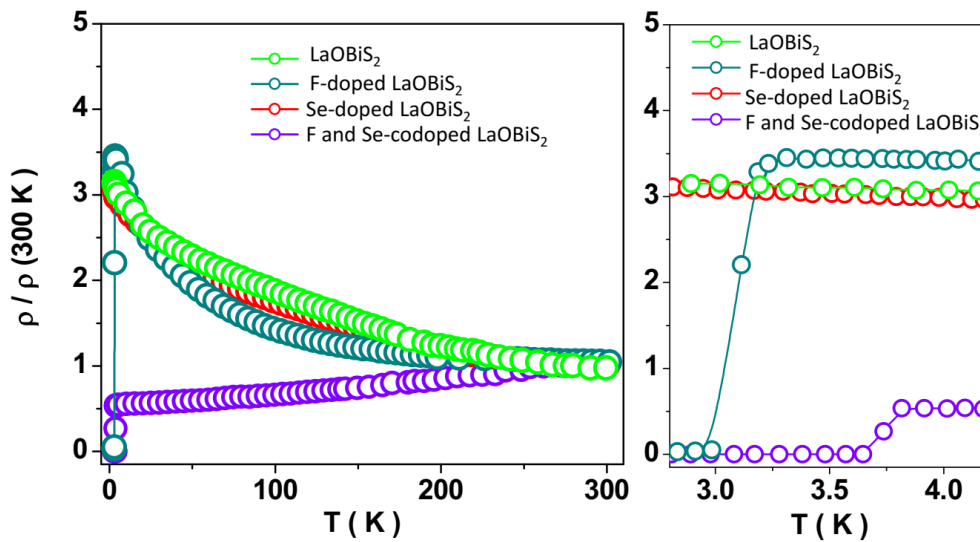


Figure 4. The temperature dependence of resistance of LaOBiS₂, F-doped LaOBiS₂, Se-doped LaOBiS₂, and F and Se-codoped LaOBiS₂ measured at different temperatures from 2 K to 300 K.

Figure 5a depicts the in situ temperature dependence of the zero-field cooled (ZFC) and field cooled (FC) DC magnetic susceptibility of F-doped LaOBiS₂, Se-doped LaOBiS₂, and F and Se-codoped LaOBiS₂ at temperatures ranging from 2 K to 6 K under a magnetic field of 10 Oe. It is indicated by a diamagnetic transition into the superconducting state in the low-temperature DC-susceptibility data (less than $T_c \sim 3.8 \text{ K}$), and the F and Se-codoped LaOBiS₂ sample shows an enhanced T_c . The T_c determined from resistivity of samples are presented in Figure 5b, indicating to the maximum T_c is determined for and F and Se-codoped LaOBiS₂ sample which in agreement with XRD results.

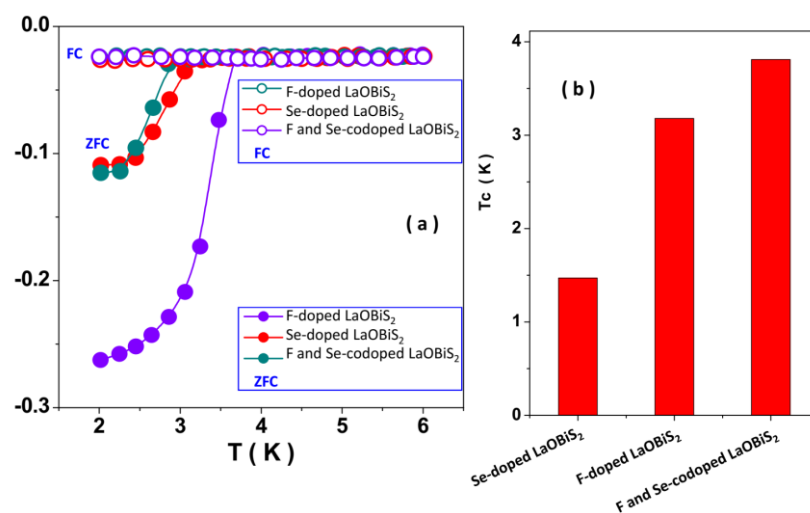


Figure 5. (a) The temperature-dependence of the zero-field cooled (ZFC) and field cooled (FC) DC magnetic susceptibility of F-doped LaOBiS₂, Se-doped LaOBiS₂, and F and Se-codoped LaOBiS₂ at temperatures range from 2 K to 6 K under a magnetic field of 10 Oe; (b) The T_c determined from resistivity of samples.

3.2. Electrochemical corrosion analyses

Figure 6 shows the Nyquist impedance responses of F-doped LaOBiS₂, Se-doped LaOBiS₂, and F and Se-codoped LaOBiS₂ electrodes in 0.1 M Na₂SO₄. A small incomplete capacitive semicircle is observed in the high frequency region, followed by a straight line with an angle close to 45° in the low frequency region that is attributed to the Warburg impedance [57]. The interfacial charge transfer resistance (R_{ct}) between electrode and electrolyte is responsible for the generation of the incomplete semicircle [58, 59]. The diffusion straight lines in the low frequency region indicated a faster ion diffusion rate on the F and Se-codoped LaOBiS₂ than other superconductor electrodes. The enhanced ion diffusion rate can be related to the larger effective surface area of the electrode which provides more surface area to be in contact with the electrolyte and forms shortcuts for ion diffusion between the electrode and the electrolyte [60]. It can be implied to highly capacitive behavior of the codoped electrode [61, 62]. The EIS data of F-doped LaOBiS₂, Se-doped LaOBiS₂, and F and Se-codoped LaOBiS₂ were commonly analyzed by fitting to an equivalent electrical circuit model which is presented in Figures 7a, 7b and 7c, respectively. These models are represented by a series and parallel resistor and capacitor, and Warburg impedance (W) [63], where R_s is solution resistance, CPE reflects the constant phase element, and R_{ct} is charge transfer resistance inversely proportional to corrosion rate. The fitting results are summarized in Table 1. As seen, F and Se-codoped LaOBiS₂ shows the higher charge transfer resistance than other electrodes, indicating to synergetic effect of the F and Se on enhancement of corrosion resistance. The drop in the non-ideal double layer capacitances (Q₁ and Q₂) can corroborate the enhancement of corrosion resistance in the F and Se-codoped LaOBiS₂ sample due to the generation of a film of corrosion products on the electrode surface [64]. It can be related to thickness of the protective part of the film is inversely proportional to its surface capacity [65]. Thus, the drop in Q₁ and Q₂ indicates an increase in the thickness of the protective film.

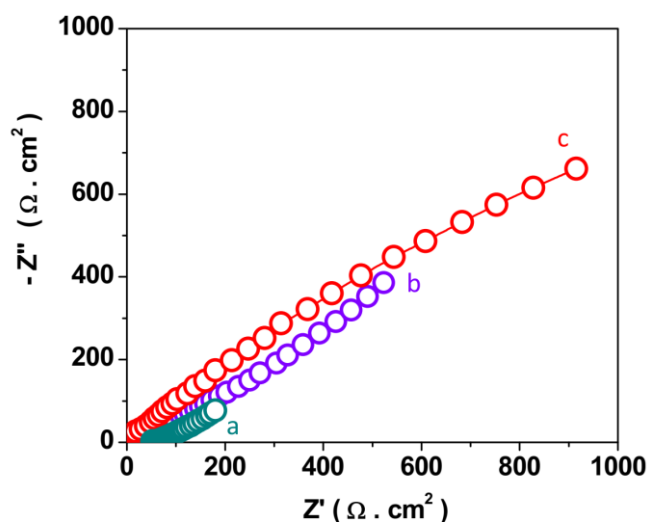


Figure 6. The Nyquist impedance responses of (a) F-doped LaOBiS₂, (b) Se-doped LaOBiS₂, and (c) F and Se-codoped LaOBiS₂ electrodes in 0.1 M Na₂SO₄.

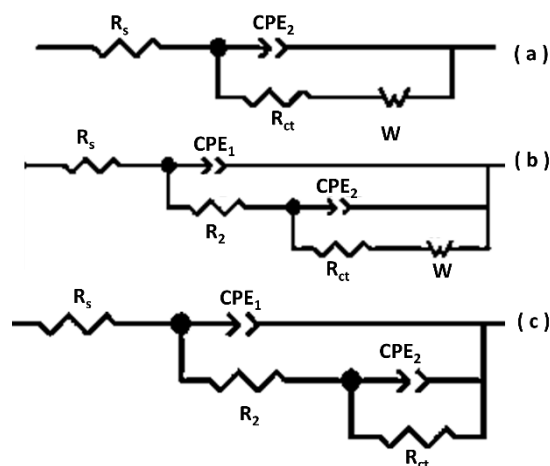


Figure 7. The equivalent electrical circuit model of (a) F-doped LaOBiS₂, (b) Se-doped LaOBiS₂, and (c) F and Se-codoped LaOBiS₂ samples used for fitting EIS data.

Table 1. The fitting results.

Sample	R_s ($\Omega \cdot \text{cm}^2$)	Q_1 ($\mu\text{F} \cdot \text{cm}^{-2}$)	R_2 ($\Omega \cdot \text{cm}^2$)	Q_2 ($\mu\text{F} \cdot \text{cm}^{-2}$)	R_{ct} ($\Omega \cdot \text{cm}^2$)
F-doped LaOBiS ₂	119	---	---	1792	155
Se-doped LaOBiS ₂	89	2345	54	72	2549
F and Se-codoped LaOBiS ₂	61	1741	1720	1805	4938

4. CONCLUSION

In conclusion, LaOBiS₂, F-doped LaOBiS₂, Se-doped LaOBiS₂, and F and Se-codoped LaOBiS₂ were successfully synthesized by a standard solid state reaction route. Structural analyses using SEM and XRD showed superconductors were synthesized in crystal structure and slice-shaped crystals, illustrating the nature of the layered structure of pure and doped LaOBiS₂ samples which grew along the c-axis, and that the Se doping and Fe doping led to expansion and growth along the c-axis, respectively that confirm the successful doping process. Temperature dependence of resistance studies indicated a sharp superconducting transition with T_c of 3.18 K F-doped LaOBiS₂ system, which can be attributed to the metallization of superconducting blocks [BiS₂] and F-ion substitution can enhance the superconductivity in the F-doped LaOBiS₂ system. Results demonstrated that Se substitution effectively suppressed local distortion and can be responsible for the increased carrier mobility together with a change in the Fermi level electronic structure. Results indicated that the resistivity for doped and codoped samples was obviously smaller than that of undoped LaOBiS₂, indicating that electron carrier density was enhanced by F and Se doping. Studies of the temperature dependence of the ZFC and FC DC magnetic susceptibilities showed that a diamagnetic transition into the superconducting state occurred at T_c~ 3.8 K for F and Se-codoped LaOBiS₂ which exhibited an

enhanced Tc. Electrochemical corrosion analyses showed that F and Se-codoped LaOBiS₂ displayed a higher charge transfer resistance than other electrodes, indicating a synergetic effect of F and Se on enhancement of corrosion resistance. The drop in the non-ideal double layer capacitances can corroborate the enhancement of corrosion resistance in the F and Se-codoped LaOBiS₂ sample due to the generation of a film of corrosion products on the electrode surface, and the drop in capacitances indicated that the thickness of the protective film had grown.

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