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Low-oxygen Annealing Process of FeSe Superconducting Materials

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Among various Fe-based superconducting materials, FeSe-based superconducting materials are considered to be ideal candidates for the exploring of superconducting mechanism due to their simplest crystal structure and tunable superconducting critical temperature. Besides, FeSe-based superconductors also can be adopted for practical applications based on their advantages, such as high upper critical field, high current capacity under high field, and low anisotropy. Therefore, it is necessary to optimize the fabrication process and superconducting performance of FeSe superconducting materials. Based on our previous study, with the introduction of high energy ball milling process and the change of initial Fe:Se ratio in the FeSe based superconductor, the content of tetragonal phase can be effectively improved. However, the existence of interstitial irons in the superconducting tetragonal phase β-FeSe can not be eliminated, which have obvious negative influence on the performance of the FeSe-based superconducting material. In this experiment, FeSe bulks with different Fe:Se ratios of 1.00, 1.05, 1.10, 1.15 and 1.20 were prepared with solid state sintering process. By comparing the superconducting properties of these sample both before and after annealing, the best Fe:Se ratio was determined to be 1.15. On this basis, many important parameters, including the annealing temperature, annealing time and oxygen partial pressure of the annealing atmosphere were systematically optimized. The phase composition and microstructure of the system were characterized after annealing, combined with the analysis of superconducting properties measurements. The results showed that during the low-oxygen annealing process, the interstitial irons inside the system was induced and diffused to the surface, which finally reduced the interstitial iron content of the tetragonal phase β-FeSe, thus increased the superconducting phase content and the critical transition temperature of the system. The optimum annealing process of 400 ^{o}C-5 %O_2-10 h for $Fe_{1.15}Se$ samples was obtained.

Keywords: Fe-based superconducting materials, FeSe, interstitial irons, annealing