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# The formation of nano-layered grains and their enhanced superconducting transition temperature in Mg-doped FeSe<sub>0.9</sub> bulks

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To search a proper dopant to further improve superconductivity in 11 type Fe-based superconductors makes sense to both their superconductivity mechanism and possible technological applications. In present work, Mg doped FeSe polycrystalline bulks were obtained by a two-step solid-state reaction method. Even though there are many MgSe and iron impurities existing in the Mg heavy doped FeSe bulks, they exhibit obviously increased  $T_c$  compared to undoped FeSe sample. It was found that Mg addition has little effect on the crystal lattice parameters of superconducting  $\beta$ -FeSe, whereas leads to the formation of nano-layered grain structure consisted of MgSe and  $\beta$ -FeSe with similar X-ray diffraction characteristics. Lots of nano-structural interfaces between FeSe and MgSe formed in this homogenous layered grain structure have significant effect on the superconducting properties and are responsible for the enhancement of  $T_c$ , as like the case of FeSe thin film on some specific substrates. Our work not only demonstrates a powerful way for raising  $T_c$  in bulk superconductors, but also provides a well-defined platform for systematic studies of the mechanism of unconventional superconductivity by considering interface effect.

The recent discovery of the Fe-based high temperature superconductors with superconducting transition temperatures as high as 55 K<sup>1,2</sup> have attracted world-wide attention despite the magnetism of Fe. Among them, 11 compounds with  $T_c$  of 8 K possesses the simplest crystal structure<sup>3</sup>, containing a single layer of tetrahedrally coordinated Fe atoms to chalcogen atoms (Se/Te). This simplicity of structure and presence of less toxic Se as compared to As make 11 compounds more attractive to study the superconductivity mechanism in iron based superconductors as well as their possible technological applications.

Even more remarkably, the onset  $T_c$  of binary  $\beta$ -FeSe<sub>1-x</sub> was shown to be as high as 27 K<sup>4</sup> with applied hydrostatic pressure, which suggests a possibility of increasing  $T_c$  via the chemical pressure route. Therefore, it is considered that chemical substitutions can be an alternative way to introduce pressure to change parameters and thereby affect superconductivity in the search for novel superconducting materials. Subsequently, several groups focused on different substitutions of ions with varied ionic radius upon either the Fe-site or the Se-site. Until now, the partial substitution of Se by Te and S has been found to significantly increase the  $T_c$ , e.g. with a maximum onset  $T_c$  of 15 K for FeSe<sub>0.5</sub>Te<sub>0.5</sub> or FeS<sub>0.2</sub>Te<sub>0.8</sub><sup>5-7</sup>.

Meanwhile, the effect of various metals dopants, such as Al, Ti, V, Cr, Mn, Co, Ni, Cu, Ga, In, Ba and Sm on the FeSe<sub>1-x</sub> superconducting polycrystalline samples obtained by a solid state reaction was investigated in previous study<sup>8,9</sup>. Among them, Co, Ni, Cu, Al, Ga or Sm doped samples consist of mainly the tetragonal phase, and these dopants occupied the Fe-sites and resulted in the changes in the lattice constants. On the other hand, the rest of the studied metal dopants did not incorporate into the host lattice of superconducting FeSe<sub>1-x</sub> and mainly formed inclusions with elements (Fe or Se) not incorporated into the matrix. However, in either case, the  $T_c$  in these doped FeSe<sub>1-x</sub> samples were generally suppressed, or even no longer emerged according to the resistivity as a function of temperature. Superconducting single crystal of pure FeTe<sub>1-x</sub>Se<sub>x</sub> ( $x = 0.3-0.55$ ) doped with Co, Ni, Cu, Mn, Zn, Mo, Cd, In, Pb, Hg, V, Ga, Mg, Al, Ti, Cr, Sr or Nd were also studied, and the similar results were found in these doped single crystals<sup>10</sup>.



Based on these backgrounds, doping of various metals in the FeSe polycrystalline bulks or single crystal samples seems to be a non-effective way to raise  $T_c$ . Consequently, lots of research teams around world lost their interest in exploring the doping of various metals and mainly focused on the research of substitution of Se-site by Te and S in recent years. Nevertheless, in present work, Mg doped FeSe polycrystalline bulks were prepared by two-step solid-state reaction method, which exhibits enhanced  $T_c$  compared to undoped FeSe sample. The corresponding enhancement mechanism is also discussed in detail, appearing to be quite different from that of the other metals doped FeSe polycrystalline bulks in previous studies.

## Experimental details

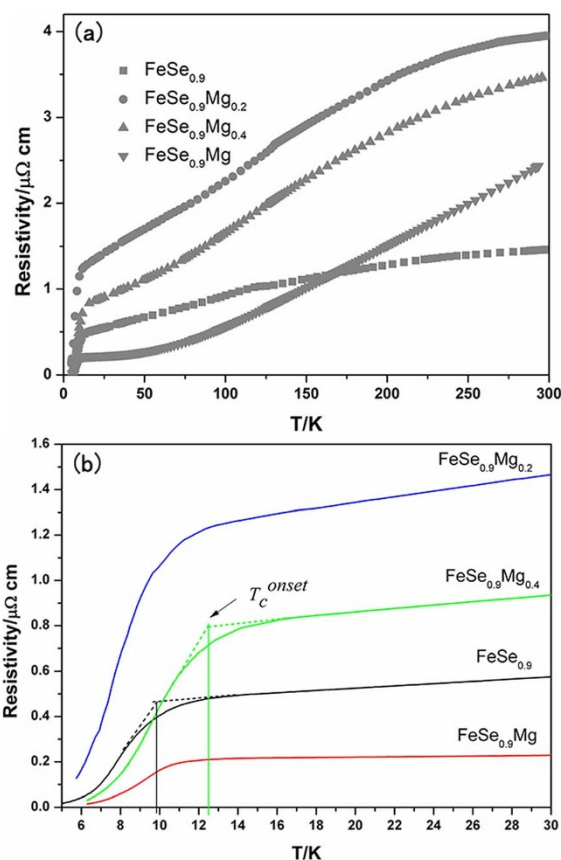
Polycrystalline bulk samples  $\text{FeSe}_{0.9}\text{Mg}_x$  ( $x = 0, 0.2, 0.4$  and  $1$ ) were prepared by two-step solid-state reaction method. The Fe powder (99.99% purity) and Se powder (99.5% purity) were firstly mixed in a molar ratio of  $\text{FeSe}_{0.9}$ . The mixed powders were cold-pressed into pellets of 5 mm in diameter and 2 mm in thickness under a uniaxial pressure of 5 MPa. All the pressed pellets were placed in an alumina crucible inside a quartz tube furnace and then sintered at  $650^\circ\text{C}$  for 24 h under protection of ultra-high purity Ar gas. After slowly cooling down to room temperature, these sintered  $\text{FeSe}_{0.9}$  bulks were reground into powders. Then these powders was mixed homogeneously with Mg powders (99.99% purity) in a molar ratio of  $\text{FeSe}_{0.9}\text{Mg}_x$  ( $x = 0, 0.2, 0.4$  and  $1$ ). The mixed powders were cold-pressed into pellets again. All the pressed pellets were placed in an alumina crucible inside a quartz tube furnace again and then sintered at  $750^\circ\text{C}$  for 0.5 h under protection of ultra-high purity Ar gas.

The phase composition of the sintered samples was determined by X-ray diffractometer (XRD) using Rigaku D/max2500 X-ray diffractometer with  $\text{Cu K}\alpha$  radiation. The morphologies were examined by field-emission scanning electron microscopes (SEM, S-4800, Hitachi). The sample resistance was measured using the standard four-probe method.

## Results and Discussion

Figure 1 shows the temperature dependence of electrical resistivity for the sintered  $\text{FeSe}_{0.9}\text{Mg}_x$  ( $x = 0, 0.2, 0.4$  and  $1$ ) samples. The positive slope of  $\rho(T)$  indicates that these samples exhibit metallic behavior. It is also found that Mg-doped samples possess higher resistivity than that of undoped samples at normal state, which can be attributed to the electron scattering effect owing to Mg doping. From this point of view, the resistivity of doped samples at normal state ought to increase as the amount of Mg addition increases. But their actual resistivity performance is out of our expectation. In fact, the resistivity of doped samples at normal state gradually decreased as the amount of Mg addition increased from  $\text{FeSe}_{0.9}\text{Mg}_{0.2}$  to  $\text{FeSe}_{0.9}\text{Mg}$ . Especially, the resistivity of  $\text{FeSe}_{0.9}\text{Mg}$  is even lower than undoped  $\text{FeSe}_{0.9}$  sample when the temperature is below 150 K. These unique results are totally different from the case of other metal doped FeSe samples reported in previous study<sup>8,9</sup>. Figure 1b presents the detailed temperature dependence of electrical resistivity for the sintered samples between 5 K and 30 K. It can be found that the onset of the transition temperature ( $T_c^{\text{onset}}$ ) of undoped  $\text{FeSe}_{0.9}$  is about 9.8 K, consistent with previous report<sup>3</sup>. As for  $\text{FeSe}_{0.9}\text{Mg}_{0.2}$  and  $\text{FeSe}_{0.9}\text{Mg}$  sintered samples, their  $T_c^{\text{onset}}$  are comparable to the undoped sample. More precisely, their  $T_c^{\text{onset}}$  are even a bit higher than that of undoped sample. On the other hand, the  $T_c^{\text{onset}}$  of  $\text{FeSe}_{0.9}\text{Mg}_{0.4}$  is significantly increased to 12.3 K, about 25% higher than that of undoped sample. This result is abnormal considering that most of metal dopants substituting on Fe site generally brought about the depression of  $T_c$ <sup>8–10</sup>.

The abnormal results of  $T_c$  obtained hereby imply that the introduction of proper amount of Mg addition by two-step sintering method seems open possible windows for the enhancement of  $T_c$  in FeSe superconductors via metal doping. In order to clarify the

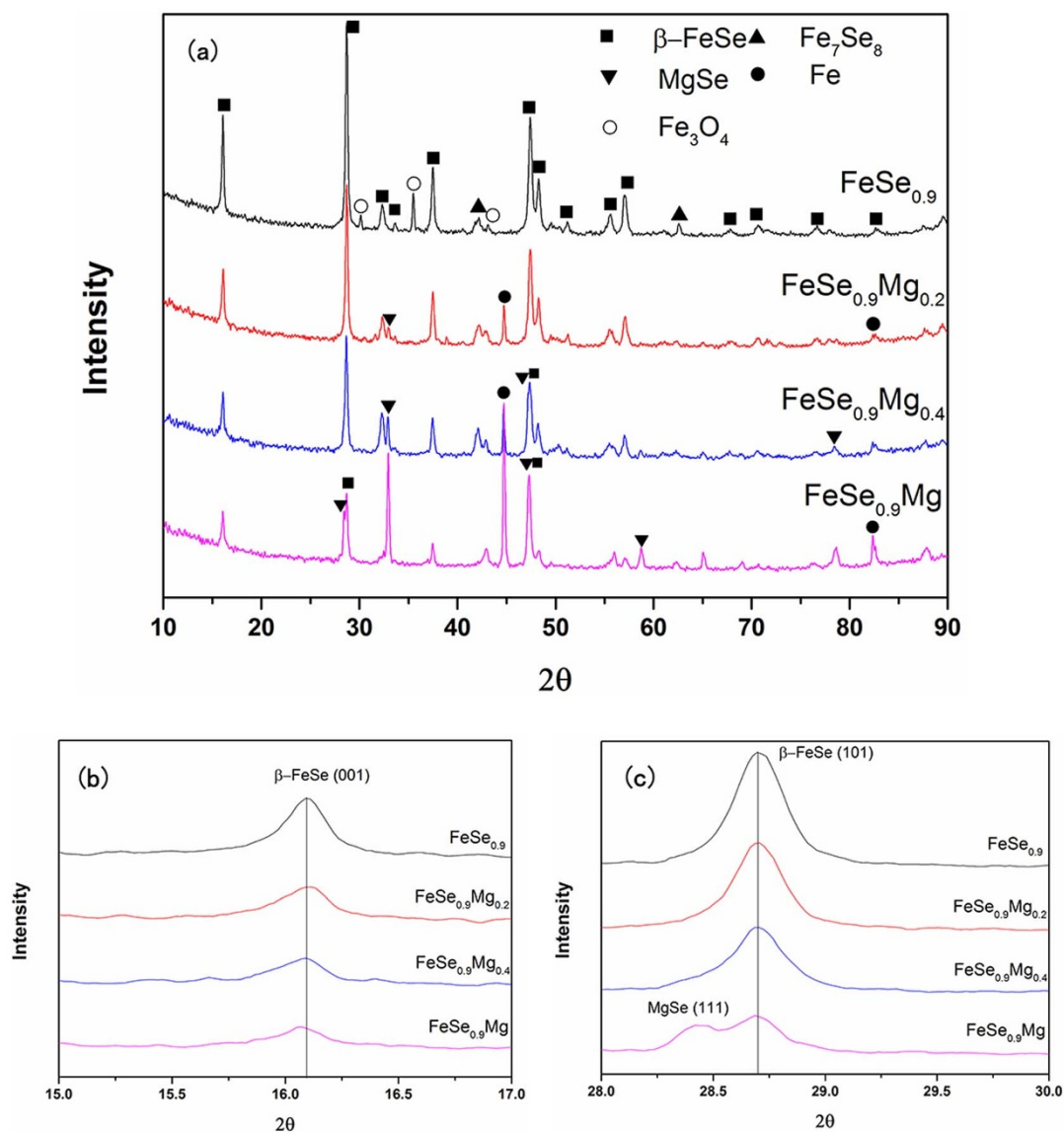


**Figure 1** | the temperature dependence of electrical resistivity for the  $\text{FeSe}_{0.9}\text{Mg}_x$  ( $x = 0, 0.2, 0.4$  and  $1$ ) samples prepared by two-step sintering, with (a) the whole measured temperature range from 5 K to 300 K and (b) the enlarged range between 5 K and 30 K.

enhancement mechanism in the  $T_c$  of the prepared sample, the phase composition and microstructure are investigated in detail.

Figure 2a shows the X-ray diffraction patterns of sintered  $\text{FeSe}_{0.9}\text{Mg}_x$  ( $x = 0, 0.2, 0.4$  and  $1$ ) samples. One can see that superconducting phase,  $\beta$ -FeSe, is the main phase in all the sintered samples. Some common impurities,  $\text{Fe}_7\text{Se}_8$  and  $\text{Fe}_3\text{O}_4$ , are also recognized in  $\text{FeSe}_{0.9}$  sample. As the amount of Mg dopant was added from  $\text{FeSe}_{0.9}\text{Mg}_{0.2}$  to  $\text{FeSe}_{0.9}\text{Mg}$ , the intensity of  $\beta$ -FeSe peaks decreased while the peaks corresponding to MgSe and Fe emerge and gradually become stronger. Observing Fig. 2a more carefully, one can also see that all the MgSe main peaks are very close to, and even overlap with  $\beta$ -FeSe peaks in the patterns (for example, MgSe (200) peak and  $\beta$ -FeSe (002) peak, MgSe (220) peak and FeSe (112) peak, MgSe (111) peak and FeSe (101) peak). Besides, it is noticed that the positions of (001) and (101) peaks corresponding to  $\beta$ -FeSe phase almost keep unchanged with the amount of Mg addition increasing, as shown in Fig. 2b and Fig. 2c, suggesting that the lattice parameters ( $a$  and  $c$ ) of  $\beta$ -FeSe almost remain unchanged. Based on the above XRD results, it can be concluded that Mg dopant mainly reacted with FeSe forming MgSe and Fe:  $\text{Mg} + \text{FeSe} = \text{MgSe} + \text{Fe}$ , and seldom entered into crystal lattice of  $\beta$ -FeSe and substitute of Fe-site.

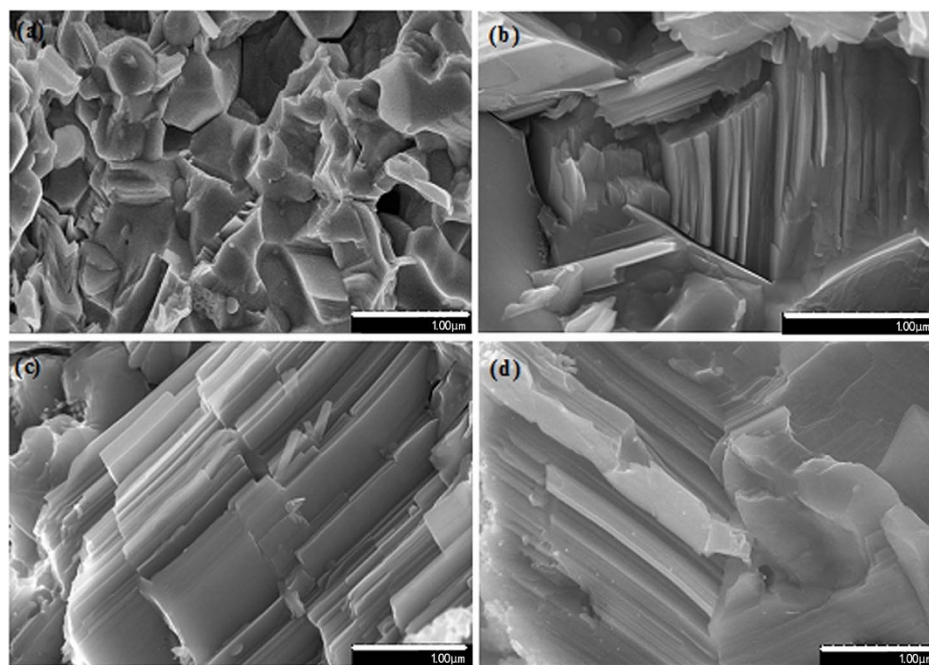
The SEM images of sintered  $\text{FeSe}_{0.9}\text{Mg}_x$  ( $x = 0, 0.2, 0.4$  and  $1$ ) samples are given in Fig. 3. It can be seen that the sintered undoped FeSe sample is dense and consisted of small randomly aligned grains (about 500 nm in size, see Fig. 3a), which is the typical characteristic of FeSe bulks prepared by traditional solid-state sintering<sup>11,12</sup>. On the other hand, regular layered grains are observed in the sintered Mg-doped samples. As the amount of Mg addition increases, the percent



**Figure 2** | The X-ray diffraction patterns of  $\text{FeSe}_{0.9}\text{Mg}_x$  ( $x = 0, 0.2, 0.4$  and  $1$ ) samples prepared by two-step sintering, with (a) The whole measured view, (b) Magnified view of  $\beta\text{-FeSe}$  (001) peak and (c) Magnified view of  $\beta\text{-FeSe}$  (101) peak.

of layered grains increased, and finally the layered grains dominate in the microstructure of sintered  $\text{FeSe}_{0.9}\text{Mg}_{0.4}$  and  $\text{FeSe}_{0.9}\text{Mg}$  samples. Moreover, the size of layered grains also enlarges as Mg addition increases. Interestingly, observing Fig. 3c and Fig. 3d more carefully, one can see that the thickness of each layer in these layered grains is very small (only about 100 nm) and also very homogeneous. This unique microstructure was seldom observed in other-metal doped FeSe samples. Combined with the XRD results, Mg addition mainly reacts with FeSe forming MgSe. The formation of these layered grains is supposed to be closely related with this reaction. Since the produced MgSe shares the similar x-ray diffraction characteristics with  $\beta\text{-FeSe}$ , it can incorporate into superconducting  $\beta\text{-FeSe}$  matrix, finally forming these multi-layered grain structure in the sintered Mg-doped sample. This speculation can be further proved by the elemental maps of Se, Fe and Mg for the microstructure of Mg doped sample, as shown in Fig. 4. It can be seen from Fig. 4 that Fe distributes uniformly in the whole SEM image while Mg and Se are relatively concentrated at the region of layered grains, as marked by white squares in Fig. 4. This result suggests that the layered grains are mainly consisted of MgSe and FeSe.

Combined with all the above results of superconducting properties, phase composition and microstructure, As Mg addition has little effect on the crystal lattice parameters of  $\beta\text{-FeSe}$ , but leads to the formation of unique layered grain structure consisted of MgSe and FeSe, it can be suggested that the enhancement of  $T_c$  in the  $\text{FeSe}_{0.9}\text{Mg}_{0.4}$  sample should be attributed to the multi-layered grain structure. Interestingly, Nabeshima et al have just reported that FeSe epitaxial thin film deposited on  $\text{CaF}_2$  (100) substrate exhibits significantly enhanced  $T_c$ <sup>13</sup>. In fact, the multi-layered grain structure in sintered  $\text{FeSe}_{0.9}\text{Mg}_{0.4}$  samples here is consisted of lots of homogeneous and thin MgSe and FeSe layers (as described above, the thickness of each layer is about 100 nm, see Fig. 3c), just like as the FeSe thin films on the MgSe substrates. MgSe is very similar with  $\text{CaF}_2$  in the crystal structure and crystal constants (Cubic,  $a = 5.463$  and  $5.462$  for MgSe and  $\text{CaF}_2$ , respectively). From this view, our result is coincidentally consistent with their research. They attribute the enhancement of  $T_c$  to the change of crystal lattice parameters  $a/c$  ratio. However, the lattice parameters of FeSe bulks almost keep unchanged by Mg doping in our work. There must be other mechanism underlying the enhancement of  $T_c$  here.



**Figure 3** | the typical SEM images of (a)  $\text{FeSe}_{0.9}$ , (b)  $\text{FeSe}_{0.9}\text{Mg}_{0.2}$ , (c)  $\text{FeSe}_{0.9}\text{Mg}_{0.4}$  and (d)  $\text{FeSe}_{0.9}\text{Mg}$  samples prepared by two-step sintering.

Besides Nabeshima et al's work,  $\text{FeSe}$  and  $\text{FeSe}_x\text{Te}_{1-x}$  thin films with much higher  $T_c$  than those of bulk superconductors have also been achieved by other groups recently, especially the thin films on the substrate with quite similar lattice constants with them<sup>14–16</sup>. In these films, it was found that the  $T_c$  of these films is significantly enhanced, whereas their lattice parameters changed very little, or even remained unchanged. The effect of the change in lattice parameters is too weak to account for the improvement of  $T_c$ . It was suggested that the interface effect plays an important role in increasing  $T_c$  of these thin films<sup>14,16</sup>. In fact, interface effect has always been an interesting topic in the superconducting field. A lot of previous

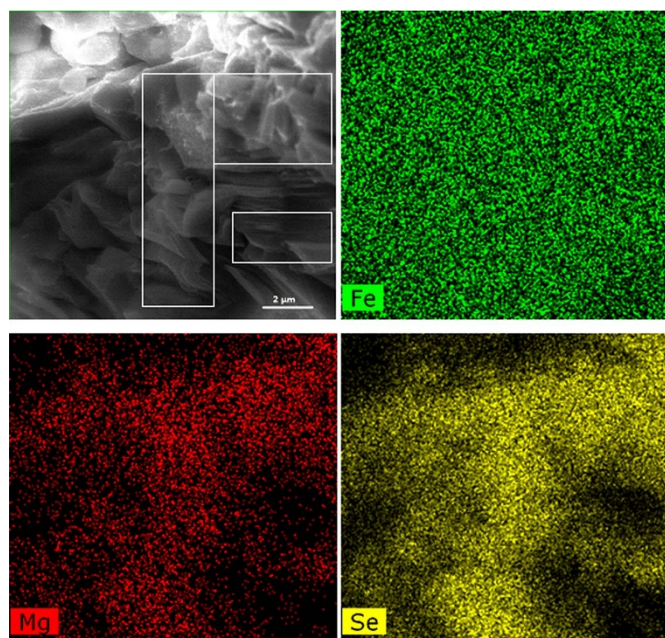
studies reported some unique superconducting phenomena at the various interfaces<sup>17–19</sup>.

In the case of  $\text{FeSe}_{0.9}\text{Mg}_{0.4}$  sample prepared in present work, there are a lot of nano-structural interface between  $\text{FeSe}$  and  $\text{MgSe}$  formed in the multi-layered grain structure of  $\text{Mg}$ -doped sample. As we all know,  $\text{MgSe}$  is wide band gap semiconductor with bandgap energy of about 4.0 eV. One can imagine that the interfaces between  $\text{MgSe}$  and  $\text{FeSe}$  forming in present work have an important effect on the superconducting properties of  $\text{FeSe}$ . These interfaces might enhance electron-phonon coupling, as reported in the interface of atomically uniform films of  $\text{Ag}$  on  $\text{Fe}$  substrate<sup>17</sup>. Another possibility is formation of two-dimensional electron gas at these interfaces, which may cause the higher  $T_c$ , as demonstrated in Ref. [18]. Further research need to be carried out to elucidate the mechanism underlying the enhancement of  $T_c$  observed here.

Based on the interface effect discussed above, it also explains well why the  $T_c$  performance in  $\text{FeSe}_{0.9}\text{Mg}_{0.4}$  sample is best among these three  $\text{Mg}$  doped samples. As for  $\text{FeSe}_{0.9}\text{Mg}_{0.2}$  sample,  $\text{Mg}$  addition is not enough and the interface between  $\text{MgSe}$  and  $\text{FeSe}$  in the layered grains is insufficient to affect the overall  $T_c$  of sintered sample. When it comes to the  $\text{FeSe}_{0.9}\text{Mg}$ , excess  $\text{MgSe}$  is formed and the superconducting  $\beta\text{-FeSe}$  is decreased too much, which also decrease the interfaces between  $\text{MgSe}$  and  $\text{FeSe}$ , and thus affects  $T_c$  performance.

## Conclusions

$\text{Mg}$  doped  $\text{FeSe}$  polycrystalline bulks were obtained by two-step solid-state reaction method, which exhibits increased  $T_c$  compared to undoped  $\text{FeSe}$  sample. It was found that  $\text{Mg}$  addition has little effect on the crystal lattice parameters of superconducting  $\beta\text{-FeSe}$ , whereas leads to the formation of nano-layered grain structure consisted of  $\text{MgSe}$  and  $\beta\text{-FeSe}$ . Lots of nano-structural interfaces between  $\text{FeSe}$  and  $\text{MgSe}$  formed in this homogenous layered grain structure have significant effect on the superconducting properties and are responsible for the enhancement of  $T_c$ . Although  $T_c$  in the  $\text{Mg}$  doped  $\text{FeSe}$  bulks in present work needs to be further enhanced via optimizing both the amount of dopants and the synthesis technique to form better layered structure, our work not only demonstrates a powerful way for raising  $T_c$  in bulk superconductors, but also provides a well-defined platform for systematic studies of the mechanism of unconventional superconductivity through considering



**Figure 4** | the elemental maps of  $\text{Fe}$ ,  $\text{Mg}$  and  $\text{Se}$  For the microstructure of  $\text{Mg}$  doped  $\text{FeSe}_{0.9}$  sample prepared by two-step sintering. The region of some layer grains was marked by white squares.



interface effect. Besides, FeSe thin film on MgSe substrate is also worth being studied in terms of interfacial superconductivity.

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## Author contributions

Z.M., Y.L. and H.L. discussed and designed the experimental scheme. F.L., N.C. and S.B. prepared the samples. Q.C. and D.P. did most of the characterizations. Z.M. analyzed the data and wrote the paper. M.H., J.H.K. and S.X.D. helped with the experiment and data analysis.

## Additional information

**Competing financial interests:** The authors declare no competing financial interests.

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