We report flux free growth of superconducting FeSe single crystals by an easy and versatile high temperature melt and slow cooling method for first time. The room temperature x-ray diffraction (XRD) on the surface of the piece of such obtained crystals showed single [101] plane of $\beta$-FeSe tetragonal phase. The bulk powder XRD, being obtained by crushing the part of crystal chunk showed majority (~87%) $\beta$-FeSe tetragonal (space group P4/nmm) and minority (~13%) $\delta$-FeSe hexagonal (space group P6$_3$/mmc) crystalline phases. Detailed high resolution transmission electron microscope images along with selected area electron diffraction showed the abundance of both majority $\beta$-FeSe and minority $\delta$-FeSe phases. Both transport ($\rho$-$T$) and magnetization exhibited superconductivity at below around 10 K. Interestingly, the magnetization signal of these crystals is dominated by the magnetism of minority $\delta$-FeSe magnetic phase, and hence the isothermal magnetization at 4 K was seen to be ferromagnetic like. Transport ($\rho$-$T$) measurements under magnetic field showed superconductivity onset at below 12 K, and $\rho = 0$ ($T_c$) at 9 K. Superconducting transition temperature ($T_c$) decreases with applied field to around 6 K at 7 T, with $dT_c/dH$ of ~0.4 K T$^{-1}$, giving rise to an $H_{c2}(0)$ value of around 50, 30 and 20 T for normal resistivity $\rho_n = 90\%$, 50% and 10% respectively, which are calculated from conventional one band Werthamer–Helfand–Hohenberg equation. FeSe single crystal activation energy is calculated from thermally activated flux flow model which is found to decreases with field from 1.2 meV for 0.2 T to 3.77 meV for 7 T.

Introduction

Discovery of superconductivity in O site F doped Fe-pnictides (REFeAsO: RE = rare earths) [1] had been of tremendous interest to both experimental and theoretical condensed matter physicists. In fact the superconducting transition temperature ($T_c$) of Fe-pnictides to the tune of above 50 K [2, 3] is second only to the famous high $T_c$ Cuprates [4, 5]. It looked like as if thunder struck again in year 2008 [1–3] after the discovery of high $T_c$ Cuprates in 1986 [4, 5]. Though a lot of research work is yet been carried out on both the above said superconducting compounds, the mechanism of superconductivity in them is yet very much elusive. This is unlike conventional superconductors including late entrant MgB$_2$ [6], which are to a large extent explainable by the electron–phonon mediated Bardeen–Cooper–Schrieffer (BCS) theory [7]. The Fe-pnictides case is even more interesting, because conventionally one feels that Fe based compounds would rather be more prone to be magnetic than superconducting. In this regards, another Fe based superconductor, namely the Fe chalcogenide i.e., FeSe entered the superconductivity kitchen in early 2009 [8]. Though the $T_c$ of FeSe and its variants viz. FeSe/Te is relatively lower to a maximum of around 20 K [8–10], the same increases tremendously under moderate applied pressures to the tune of above 30 K [11–13]. The $T_c$ of FeSe could in fact be increased to above 100 K for SrTiO$_3$ substrate grown ultra-thin films of the same, due to high tensile stress and thus the internal chemical pressure [14, 15]. The increase in $T_c$ for FeSe can also be achieved by favorable intercalation within adjacent FeSe layers to as high as above 50 K [16, 17].
It is clear that FeSe qualifies for its entry into the famous high $T_c$ club with its $T_c$ as high as up to 100 K [14–17]. Further, its simplistic crystal structure in comparison to Fe-pnictides and high $T_c$ Cuprates calls the same to be the ideal candidate for studying the superconductivity beyond BCS [7]. In this regards, ideally the theoreticians look for the physical property data including superconductivity characterization from the single crystalline samples. This is precisely because the crystalline materials are devoid of the grain boundary related complications. In fact, the physical property data being obtained from large crystals of materials with exotic properties viz. superconductivity, thermo electromotive or photoconductivity is a feast to the theoreticians. In this regards, though the FeSe seems to be the ideal case for studying mysterious superconductivity, the growth of reasonable size crystals of the same is yet elusive. Only tiny crystals (maximum mm size) of FeSe are yet grown, with added flux (NaCl/KCl) and that also in state of art crystal growth furnaces often involving complicated heat treatments [18–21]. Infact, it is previously known that FeSe cannot be gown directly from the high temperature melt [22]. On the other hand though the added flux (NaCl/KCl) decreases the melting temperature; the foreign contamination cannot be avoided completely.

Clearly, the single crystal growth of flux free large FeSe crystals had been a challenging problem for the experimental condensed matter physicists. In this regards, a recent article reporting successful flux free growth of large FeSe crystals had been an eye opener [23]. This work got the scientific community appreciation as well [24]. Though the crystals grown were of large size, the heating schedule thus envisaged was quite complicated and the furnace employed was state of art equipment based upon traveling-solvent floating-zone technique. We tried to grow flux free FeSe crystals of reasonable size from high temperature melt employing two step cooling method. Similar approach was applied recently by some of us for the growth of flux free large FeSe$_{1/2}$Te$_{1/2}$ superconducting crystals [25]. However, it is known previously that though large flux free crystals of FeSe$_{1/2}$Te$_{1/2}$ can be grown [26–29], the same is not true for FeSe [22]. After, several trials, we employed a relatively simple and easy heating schedule though with multiple steps and finally could grow flux free FeSe crystals from its melt at 1100 °C. The crystals thus grown are superconducting at below 10 K. In this article, the growth and characterization of such flux free large superconducting FeSe crystals is reported. High resolution transmission electron microscope (HRTEM) studies along with selected area electron diffraction (SAED) clearly approved the single crystalline nature with abundance of both majority ($\sim$87%) $\beta$-FeSe and minority ($\sim$13%) $\delta$-FeSe phases. Further studies to completely separate the majority ($\sim$87%) $\beta$-FeSe and minority ($\sim$13%) $\delta$-FeSe phases are yet underway. Worth mentioning is the fact, that the method thus reported is checked for repeatability couple of times. We believe our timely action related to growth of flux free FeSe crystals will catch the attention of scientific community and further refined crystals could emerge as a result.

**Experimental**

Basically, the constituent elements Fe and Se with 4N purity are grinded in the argon filled glove box, sealed in an evacuated quartz tube, heated (rate 1 °C min$^{-1}$) in a normal automated furnace to 1100 °C for 24 h with prior intermediate steps at 350 °C and 750 °C of 4 h each. This was followed by slow cooling (5 °C h$^{-1}$) to 460 °C and hold for 12 h, then to 250 °C with very slow cooling (2 °C h$^{-1}$), subsequently the furnace is allowed to cool naturally to room temperature over a span of around 10 h. The whole process took more than 10 days. The schematic flow chart of the employed heat treatment is shown in figure 1. X-ray diffraction (XRD) is done at room temperature using Rigaku x-ray diffractometer with CuKα radiation of 1.541 84 Å. The morphology of the obtained single crystal has been seen by scanning electron microscopy (SEM) images on a ZEISS-EVO MA-10 SEM, and energy dispersive x-ray spectroscopy (EDX) is employed for elemental analysis. Detailed micro-structural characterization of the FeSe crystal was carried out using a high resolution transmission electron microscope (HRTEM, model: Tecnai G2 F30 STWIN assisted with a field emission gun for the electron source at an electron accelerating voltage of 300 KV). Electrical and magnetic measurements were carried out respectively on quantum design magnetic property measurement system and cryogenics-PPMS down to 2 K in applied fields of up to 7 T.

**Results and discussion**

The as synthesized crystals are of few cm size (figure 2(a)). In fact when the quartz ampoule is broken, the whole material is one piece, shiny and looking to be in single crystalline form. Because the present FeSe crystals are grown from self flux method out of the stoichiometric FeSe, hence no pre-washing was required to remove the foreign flux. For various SEM measurements small pieces were taken from the as such obtained sample. Figures 2(b)–(f) show the room temperature SEM results for the studied FeSe single crystal. Figures 2(b) and (c) show the SEM micrographs obtained from various pieces of the FeSe chunk. Clearly the slab growth of FeSe crystals can be seen in figures 2(b) and (c) micrographs. The slab like layer by layer growth persists in the studied
FeSe crystal over a large area. This is similar to that as being seen recently for flux free grown FeSe\textsubscript{1/2}Te\textsubscript{1/2} single crystals\[25\]. The slab like growth for FeSe crystals is known earlier as well\[18–21, 23\]. The compositional analysis of selected area being carried by EDX is shown in figure 2(d). The crystal is found to be near stoichiometric i.e. close to nominal FeSe, with only a slight loss of Se. Further, figures 2(e) and (f) show near homogenous distribution of Fe and Se in the matrix. It is clear from figures 2(a)–(f) results that the presently self grown FeSe crystal is near stoichiometric with homogenous distribution of Fe, Se and the growth nature of the same is slab like.

Figure 3 exhibits detailed micro-structural characterization of FeSe single crystal being carried out using a high resolution transmission electron microscope (HRTEM, model: Tecnai G2 F30 STWIN assisted with the field emission gun for the electron source at an electron accelerating voltage of 300 kV). A uniform microstructure was observed throughout in the sample under the electron beam (figure 3(a)). At further higher magnifications a compact microstructure without the porosity was delineated throughout in the specimen (inset (B) in figure 3(a)). At low magnification, a FeSe crystal of size about 16 μm in length has been displayed as inset (C) in figure 3(a). A gray-level contrast observed in the microstructure distinguishes the presence of a minor phase (δ-phase of hexagonal crystal structure of FeSe) in the matrix constituted of a t-phase of tetragonal crystal.
structure as a major phase (t- and δ-phases marked in figure 3(a)). A SAEDP of δ-phase along [2110] zone axis of a hexagonal crystal structure of FeSe (lattice parameters: \(a = 0.37\) nm, \(c = 0.58\) nm, space group: P63/mmc, reference: JCPDS card no. 86-2246) has been displayed as inset (A) in figure 3(a). Correspondingly, a set of important planes of δ-phase of hexagonal hkl indices: 0002, 0112, 0111, 0110 are marked as points 1, 2, 3, 4 on the electron diffraction spots in reciprocal space in inset (A) of figure 1(a). Similar to δ-phase, a SAEDP of t-phase along [001] zone axis of a tetragonal crystal structure of FeSe (lattice parameters: \(a = 0.38\) nm, \(c = 0.55\) nm, space group: P4/nmm, reference: JCPDS card no. 85-0735) has been displayed as inset (D) in figure 3(b). Correspondingly, a set of important planes of t-phase of tetragonal hkl indices: 020, 110, 200 are marked as points 5, 6, 7 on the electron diffraction spots in reciprocal space in inset (D) of figure 3(a). Further to resolve the presence of both major tetragonal t- and minor hexagonal δ-phases in the specimen, atomic scale imaging was performed. Figure 3(b) shows a set of 200 atomic planes of tetragonal crystal structure of FeSe with the inter-planar spacing of 0.19 nm, stacked at lattice scale in throughout the region. Inset (E) in figure 3(b) exhibits the inter-planar spacing of 0.29 and 0.22 nm of corresponding hexagonal atomic planes of 0002 and 0112 at lattice scale.

The room temperature XRD patterns of the FeSe crystal being taken after powdering the same and also such on surface of the same are shown in figure 4 and its inset respectively. The surface of FeSe crystal is aligned at [101] plane, see inset figure 4. The same orientation is seen in [23] as well. The powder XRD of the studied FeSe crystal being shown in main panel figure 4, though mainly corresponds to the majority tetragonal (β-FeSe) phase, yet the minority hexagonal (δ-FeSe) being marked with * can also be seen. To further elucidate and quantify the presence of both phases, we carried out detailed Rietveld analysis on the powder XRD pattern of the
studied FeSe crystal and the results are shown figure 5. The majority tetragonal phase ($\beta$-FeSe) is $\sim 87\%$ and the minority hexagonal ($\delta$-FeSe) one is $\sim 13\%$. The refined lattice parameters are $a = b = 3.7707(3)\AA$ and $c = 5.512(3)\AA$ for majority phase and $a = 3.623(3)\AA$ and $c = 5.877(3)\AA$ for the minority one. The rietveld refined co-ordinate positions for majority tetragonal ($\beta$-FeSe) phase are Fe (2a) at $3/4$, $1/4$, $0$ and Se (2c) at $1/4$, $1/4$, $0.2594(2)$. The same for minority hexagonal ($\delta$-FeSe) are Fe (2a) at $0$, $0$, $0$ and Se (2c) at $1/3$, $2/3$, $1/4$. The schematic unit cells for both the $\beta$-FeSe and $\delta$-FeSe phase are shown in inset of figure 4. Further, the rietveld refined data for both the phases are shown in table 1. Interestingly, the abundance of both the $\beta$-FeSe and $\delta$-FeSe phases is seen in our HRTEM results as well, see details in figure 6. The majority tetragonal phase (FeSe) is superconducting at below $9\,\text{K}$. To further elucidate upon this point, we also carried out the isothermal magnetization measurements, because the former is superconducting (diamagnetic) and later is known to be magnetic.

The zero-field-cooled (ZFC) and field-cooled (FC) DC magnetic susceptibility of FeSe crystal at $10\,\text{Oe}$ applied field in temperature range to $2$–$15\,\text{K}$ is shown in figure 6. Though, clear branching of ZFC and FC is seen below $10\,\text{K}$, the moment is negative. This is puzzling, because ideally if the studied crystal is superconducting, the same must have conformed with $\text{ve}$ moment as sign of the diamagnetism. Interestingly, the observed result (figure 6) is possible if $\text{ve}$ magnetic background could be riding on the diamagnetic signal from superconductivity. This superimposition is clear as the ZFC moment becomes $\text{ve}$ below around $5\,\text{K}$ due to increasing superconducting volume fraction below $T_c$. In present case, where both majority ($\sim 87\%$) superconducting ($\beta$-FeSe) and minority ($\sim 13\%$) magnetic ($\delta$-FeSe) are present (XRD and HRTEM results), the magnetization outcome in figure 6 is not surprising. In fact, in [23] as well, the DC moment outcome on similar flux free grown FeSe crystals would have been the same, but unfortunately in that article only the AC susceptibility measurements ($299\,\text{Hz}$, $1\,\text{Oe}$) are shown. The AC susceptibility measurements ($333\,\text{Hz}$, $10\,\text{Oe}$) on present FeSe crystal are shown in inset of figure 6. These measurements clearly demonstrate that our FeSe crystal is clearly superconducting at below $9\,\text{K}$. To further elucidate upon this point, we also carried out the isothermal...
magnetization (MH) measurements well below the superconducting transition temperature i.e., at 4 K and the results are shown in figure 7. Clearly a ferromagnetic (FM) loop is seen with saturation moment above 1 T with negligible coercively of few Oe only. The expanded part of MH at near origin is shown in inset of figure 7, indicating clearly dominating diamagnetic part for low fields (below 70 Oe) and for higher fields the moment becomes +ve due to overriding positive moment from minority magnetic ($\delta$-FeSe) phase. The lower critical field ($H_{c1}$) of the studied crystal seems to be around 25 Oe, which is being marked in inset of figure 7.

To ascertain, if the ZFC and FC branching at 10 K arises out of the superconducting transition, we carried out the resistivity versus temperature ($\rho$–$T$) measurements on FeSe crystal and the results are shown in figure 8. The superconductivity onset is seen at 12 K and $\rho = 0$ is obtained at around 9 K. The studied FeSe crystal is superconducting at below 9 K and the normal state conductivity is of metallic nature. The magneto transport measurements i.e., $\rho(T)H$ in superconducting transition region (4 K–15 K) are shown in figure 9. The $T_c(\rho = 0)$ is decreased monotonically from 9 K to around 5.8 K under applied field of 7 T. The $dT_c/dH$ is $\sim$0.4 K T$^{-1}$. The zero temperature upper critical field $H_{c2}(0)$ is calculated applying the conventional one-band Werthamer–Helfand–Hohenberg (WHH) equation, i.e., $H_{c2}(0) = -0.693T_c(dH_{c2}/dT)_{T=T_c}$. The calculated $H_{c2}(0)$ with different criterion of $\rho = 10\%$, 50\% and 90\% of the normal state resistivity is around 20, 30 and 50 T respectively. The WHH plots are shown in figure 10. Importantly, the $H_{c2}(0)$ values are outside the Pauli paramagnetic limit of 1.84$T_c$. This indicates that the studied crystals are heavily pinned possibly due to the presence of minority ($\sim$13\%) magnetic ($\delta$-FeSe) phase along with majority ($\sim$87\%) superconducting ($\beta$-FeSe) phase.

To further elucidate upon the $\rho(T)H$ behavior of FeSe crystals the thermally activated flux flow thermally activated flux flow (TAFF) plots ($\ln\rho$ versus $1/T$) at various fields for studied FeSe crystal are shown in figure 11.
Figure 8. Temperature dependent electrical resistivity from temperature range 300–5 K of FeSe single crystal.

Figure 9. Electrical resistivity with temperature under various magnetic fields up to 7 T in temperature range of 15–4 K for FeSe single crystal.

Figure 10. Upper critical field ($H_{c2}$) calculated from $\rho(T,H)$ data with 90%, 50% and 10% $\rho_n$ criteria of FeSe single crystal.
According to TAFF theory\cite{30, 31}, the Arrhenius relation is given by the equation\cite{32}
\[
\ln \rho(T, H) = \ln \rho_0(H) - \frac{U_0(H)}{k_B T}.
\]
where \(\ln \rho_0(H)\) is the temperature independent constant, \(k_B\) is the Boltzmann's constant and \(U_0(H)\) is TAFF activation energy. The \(\ln \rho\) versus \(1/T\) plot with field in TAFF region remains linear. This linear region with fields is fitted very well and is shown in figure 11. The extrapolated fitted lines intercept at the same temperature i.e., at bulk \(T_c\) of the crystal, which is around 11.62 K. With an increase in the magnetic field, the resistivity broadening takes place due to thermally assisted flux motion\cite{32}. As far as Fe based superconductors are concerned, the Ba-122 compound shows least broadening due to lower thermal fluctuations, while iron pnictides i.e. ReO\(_{1-x}\)F\(_x\)FeAs (Re-1111) show wider resistivity broadening similar to that as for Cuprate YBCO compounds with an increase in field\cite{32–34}. Excitingly FeSe\(_{1-x}\)Te\(_x\) based superconductors show intermediate resistivity broadening with increases in magnetic field\cite{35}. The present case of self flux grown FeSe crystals seems to be closer to Ba-122\cite{32}.

The activation energy is calculated for the different magnetic fields in the range of 0.2–7 T. The variation of activation energy with field is very wide i.e., from 12.1 meV for 0.2 T to 3.77 meV for 7 T, this shows how the magnetic field affect the creep of thermally activated vortices. Interestingly, this activation energy is far below than the activation energy of FeSe\(_{0.5}\)Te\(_{0.5}\) single crystals\cite{25}, suggesting single vortex pinning for FeSe single crystalline compound. With increasing magnetic field, the thermally activation energy follows power law i.e. \(U_0 = K \times H^{-\alpha}\) where \(U_0\) is field dependent. Value of \(\alpha\) vary with field i.e., lower for lower fields and higher for higher fields, see figure 12. \(U_0\) is calculated for different fields with \(\alpha = 0.21\) for lower field i.e., up to 2 T and \(\alpha = 0.62\) for higher field i.e. from 3 to 7 T. In lower fields, the weak power low decreases of \(U_0(H)\) denotes single vortex pinning\cite{31, 36}. The TAFF behavior of FeSe seems closer to Ba-122 Fe based superconductor than the Fe-pnictide and HTSc Cuprates ones.

**Figure 11.** \(\ln \rho\) versus \(1/T\) for different magnetic fields of FeSe single crystal, corresponding solid line are fitting of Arrhenius relation.

**Figure 12.** Field dependent of activation energy \(U_0(H)\) with solid lines fitting as power law of \(U_0(H) \sim H^{-\alpha}\).
Conclusions

We reported flux free growth of FeSe single crystal without any complicated heating schedule in a simple automated furnace for first time. The XRD result of FeSe single crystal shows that the crystal growth is in [101] plane. Powder XRD result showed both majority (~87%) β-FeSe tetragonal and minority (~13%) δ-FeSe hexagonal crystalline phases. HRTEM results though suggests towards the single crystalline nature but with presence of two phases. The superconductivity at around 10 K is confirmed by the both MT and μ-T measurements. $H_c(0)$ value, which is calculated from conventional one-band WHH equation comes around 50 T for 90% of $\rho_0$ criterion. Activation energy is estimated up to 7 T magnetic fields with the help of TAFF model, which showed that single vortex pinning dominants in low field region. Separating the two phases i.e., (~87%) β-FeSe tetragonal and (~13%) δ-FeSe hexagonal is the priority task at hand. In an earlier study [37], the two phases were separated from flux (KCl–AlCl₃) grown melt of FeSe. The flux free growth of FeSe from direct melt has yet been elusive, only scant reports with specialized techniques like chemical vapor transport on AlCl₃/KCl eutectic melt do exist [38]. In this regards, the present study is an eye opener.

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