Solid state synthesis and characterization of bulk FeTe$_{0.5}$Se$_{0.5}$ superconductors

K. Onar$^1$ and M. E. Yakinci$^{1,2}$

$^1$İnönü Üniversitesi, Fen Edebiyat Fakültesi, Fizik Bölümü, 44280-Malatya, TURKEY
$^2$İnönü Üniversitesi, Mühendislik Fakültesi, Biyomedikal Mühendisliği Bölümü, 44280-Malatya, TURKEY

E-mail: kubra.onar@inonu.edu.tr

Abstract. FeTe$_{0.5}$Se$_{0.5}$ polycrystalline superconductor samples were synthesized by solid-state reaction method at different heating temperatures. The morphological and structural characterization of FeTe$_{0.5}$Se$_{0.5}$ samples were carried out by X-rays Diffraction, Scanning Electron Microscope and Energy Dispersive X-ray Spectroscopy. The electrical, magnetic and thermal transport properties were investigated up to 8 T by using physical property measurement system. The results reveal that the sensitivity of electrical and magnetic properties strongly depends on the heat treatment cycles. The upper critical field, $H_{c2}(0)$, was determined with the magnetic field parallel to the sample surface. It gives a maximum value of 36.3 T. The lower critical field, $H_{c1}(T)$, was obtained as 210, 140 and 70 Oe at 5, 8 and 12 K, respectively. The coherence length, $\xi$, at the zero field, was calculated to be 1.94 nm and suggested a transparent intergrain boundaries peculiarity. The $\mu_0H_{c2}(0)/k_BT_c$ rate shows higher value (3.36 T/K) than the Pauli limit (1.84 T/K) which suggests unconventional nature of superconductivity for the polycrystalline FeTe$_{0.5}$Se$_{0.5}$ superconducting samples.

1. Introduction

Superconductivity and magnetism are two different concepts in physics and they are not considered together, because superconductivity is disappeared by the magnetism, until late 1990. When Ru based materials were first discovered in 1997, the scientists were surprised due to existence of both magnetism and superconductivity together in the sample [1]. Then in 2008, discovery of LaFeAsO$_1$, F$_x$ with superconducting transition temperature $T_c$ up to 26 K attracts a big attention due to existence of magnetic Fe element and superconductivity in the material [2]. After that different phases of iron based superconductors were discovered, Ba$_{1-x}$K$_x$Fe$_2$As, the “122-phase” with $T_c$ of ~38 K [3], Li$_{1-x}$FeAs, so-called “111-phase” with $T_c$=18 K [4] and FeSe, so-called “11-phase” with $T_c$=8 K [5]. The superconducting transition temperature has been increased up to ~55 K by replacing or substituting/doping of La with other rare earth elements such as, Ce, Sm, Nd, Pr, Gd, Eu and Tm. The obtained phase is then called “1111-phase” [6, 7]. The FeSe (11-phase) has the most simple
crystal structure, $T_c$’s up to 37 K under high pressure, and most importantly it has not contain toxic materials such as, As and it is easy to obtain in single crystalline form [8-13].

In general, iron-based superconductors have layered crystal structure [14]. Fe-As layers, which are negatively charged, and Ln-O layers (Ln: lanthanides), positively charged, are simply arranged along the c-axis. The Ln-O layers are insulating nature and the Fe-As layers are responsible from the conducting mechanism [14,15]. The FeSe phase, called as “11-phase” with $T_c=8$ K, which is a derivative of FeAs family, is composed of tetrahedral stacks of Fe$_2$Se$_2$ layers which have edge sharing peculiarity and behave as conducting layers similar to B$_2$ layers in MgB$_2$ metallic superconducting alloys [7,16]. Recently, alkaline metals such as, K, Rb, Cs, Li and Na have been added successfully into FeSe materials. The physical properties of obtained materials show that all additives behave as a charge carriers and (Fe$_2$Se$_2$)$_{\delta}$ layers are responsible at the conductivity [17-20]. Mizuguchi et. al. obtained FeTe material which has tetragonal crystal structure that is similar to FeSe superconductors [21] and it has a magnetic or a structural phase transition at around 70 K [22, 23]. However, FeTe does not show superconductivity. The other investigation indicates that Se substitution suppressed and causing superconductivity [24-27]. The FeTe materials can be prepared either in polycrystalline or in single crystal form by using different methods [28-30]. In the application side of FeSe and FeTeSe materials, various forms such as, thin/thick films [31, 32] and wires/tapes [33-36] can be prepared and they have capabilities to meet today’s technological needs.

In this work, polycrystalline FeTe$_{0.5}$Se$_{0.5}$ samples have been prepared by using conventional solid state reaction method. To understand better the physical and magnetic properties, we have focused on the estimations of the upper critical field ($H_{c2}$), lower critical field ($H_{c1}$), activation energy ($E_a$) and the coherence length, $\xi$, as well as the structural and electrical transport properties.

2. Experimental Details

Polycrystalline FeTe$_{0.5}$Se$_{0.5}$ material was prepared using conventional solid-state reaction method. High purity powders of the Iron (99.999% Alfa Aesar), Tellurium (99.999% Alfa Aesar) and Selenium (99.999% Alfa Aesar) were used as raw materials. Appropriate amount of Fe/Te/Se:1/0.5/0.5 was ground by agate mortar to obtain a homogenized mixture and then transferred into quartz tubes and sealed.

Then the quartz tubes were placed inside a tube furnace and heated up with a rate of 2°C/min at 600, 650 and 700°C (so called samples FTS-1, FTS-2 and FTS-3 respectively) for 24 h. After slow cooling to room temperature the first heating cycle was repeated again with an intermediate grinding in a glove box under Ar atmosphere. In the third and final heating stage, sample was re-grounded and pressed isostatically in the glove box at 65 MPa and annealed at fixed 400°C for 24 h with 2°C/min heating and cooling ramps in the evacuated quartz tubes, Table1. After the heating cycles, structural properties of the FeTe$_{0.5}$Se$_{0.5}$ samples obtained were examined by X-Rays Powder diffractometer (XRD). A Rigaku RadB DMaxII diffractometer and Jade 6+ refinement program was used to analyze phase formation. The microstructural formations of the samples prepared were investigated using Scanning Electron Microscope (SEM) (Leo EVO 40VPX)

Table 1. Heat treatment cycles of polycrystalline FeTe$_{0.5}$Se$_{0.5}$ samples.

| Sample | First heat treatment | Second heat treatment | Third heat treatment |
|--------|----------------------|-----------------------|---------------------|
|        | Temp. (°C) | Time (h) | Temp. (°C) | Time (h) | Temp. (°C) | Time (h) |
| FTS-1  | 600      | 24       | 600      | 24       | 400      | 24       |
| FTS-2  | 650      | 24       | 650      | 24       | 400      | 24       |
| FTS-3  | 700      | 24       | 700      | 24       | 400      | 24       |
and Energy Dispersive X-rays spectroscopy (EDX) (Bruker 125 eV). The electrical, thermal and magnetic properties were carried out using Quantum Design Physical Properties Measurement Systems, (PPMS-9T) with applied fields up to 8 Tesla. To determine the upper critical field $H_{c2}(0)$, the $R-T$ curves, measured under applied fields up to 8 T and Werthamer-Helfand-Hohenberg (WHH) formula, were used. The superconducting coherence length was estimated using Ginzburg Landau (GL) equation. The $I-V$ characteristics of the samples were determined using 1 $\mu$V criteria. The activation energy of the samples was calculated using the Arrhenius equation.

3. Results and discussions

3.1. X-ray analysis (XRD)

Figure-1 shows the XRD patterns of the polycrystalline FeTe$_{0.5}$Se$_{0.5}$ samples prepared by solid state reaction method. Heat treatment cycles of the samples prepared are summarized in Table 1. A slight difference between the x-ray patterns of samples heat treated at 600 to 700 $^\circ$C was obtained. As seen in Figure-1, all of the polycrystalline FeTe$_{0.5}$Se$_{0.5}$ samples prepared contain small amount of impurity phases. These phases are mainly antiferromagnetic hexagonal FeTe$_{0.5}$Se$_{0.5}$ phase (marked with H) and unreacted Fe element (marked as asterisks). We did not detect common impurity phases such as, FeSe$_2$, Fe$_7$Se$_8$ and $\delta$-FeSe and also oxide peaks as obtained in previous works by other groups [27, 37, 38]. The main peaks are indexed as tetragonal FeTe$_{0.5}$Se$_{0.5}$ superconducting phase with a space group of P4/nmm. Lattice parameters and unit cell volume of the samples prepared were calculated to be $a=3.7885(5)$ Å, $c=5.9453(5)$ Å and 85.03 Å$^3$ respectively. After the optimization of heating cycles the intensity of the impurity phases was decreased but, single phase samples were not obtained, Figure 1. This indicates that the structural formation of the polycrystalline FeTe$_{0.5}$Se$_{0.5}$ samples was not largely depending on the heat treatment cycles. Similar results were obtained previously by different groups and can be associated with the large melting temperature difference between the Fe, Se and Te materials [36, 39].

3.2. SEM and EDX analysis

Figure 2a and 2b show the surface micrographs of the sample FTS-2 in different magnifications as an example. Strongly connected but randomly oriented granular formation with an average grain size of 2 $\mu$m, which is the typical growth characteristics of polycrystalline FeTeSe material prepared with the solid state reaction method, were obtained.

Figure 1. XRD graphs of the polycrystalline FeTe$_{0.5}$Se$_{0.5}$ samples heated at different temperatures.
Microstructural formation, show that there is no effect of thermal treatment on the microstructural evolution as obtained on the XRD results. We also noticed that the density of the samples was not high comparing to other metallic structures or single crystalline form of same material. According to EDX results calculated ratio of Fe:Te:Se was found as 49.6:25.2:25.2 for sample FTS-2 and FTS-3. These data are very close to the 1:0.5:0.5 starting ratio and indicating that the good reaction between the Fe, Te and Se in the quartz tubes. The sample FTS-1 showed slightly Fe rich and Te deficient phase formation and the deviation from the actual composition is about 3%, however, which has no effect on the microstructural formation of the samples. The EDX dot mapping for sample FTS-3 are given in Fig. 2c as an example and homogenious FeTe$_{0.5}$Se$_{0.5}$ phase distribution was obtained.

3.3 Transport Properties

Figure 3a shows temperature dependence of resistivity for the polycrystalline FeTe$_{0.5}$Se$_{0.5}$ samples at zero field. The region around the $T_c^{(onset)}$ is magnified and shown in the inset of Figure 3a. In generate the results obtained are summarized in Table 2. According to Figure 3a, all samples have a metallic behavior down to their $T_c^{(onset)}$ temperature. Samples FTS-2 and FTS-3 are showed a sharp resistivity drop after $T_c^{(onset)}$ showing that the purity and the quality of the samples. A double transition was observed for the sample FTS-1, indicating a multiphase nature as obtained in the XRD and EDX results in the previous sections. The first transition, obtained at around 14 K, is believed to be due to the tetragonal FeTeSe superconducting phase, and the second transition, obtained at around 10.3 K, indicates the existence of the impurities, mainly hexagonal FeTeSe phase and unreacted Fe in micro-level. This indicates insufficient ionic diffusion process due to low temperature heating of this sample comparing to the FTS-2 and FTS-3. Although anomalies such as, structural or magnetic phase transformation between 50 K and $T_c^{(onset)}$ for all samples was observed. In order to determine superconducting transition temperature of the samples precisely $d\rho(T)/dT$ values were calculated.

![Figure 2. SEM micrographs of the polycrystalline FeTe$_{0.5}$Se$_{0.5}$ samples a) high magnification image b) Low magnification image c) EDS mapping image.](image-url)
Figure 3. a) Temperature dependence of resistivity for the polycrystalline FeTe\(_{0.5}\)Se\(_{0.5}\) samples, b) derivative of resistivity \(d\rho/dT\) as a function of temperature.

Figure 3b shows the \(d\rho(T)/dT\) versus \(T(K)\) graphs of samples and the results are given in Table-2. Figure 3b indicates that, sample FTS-2 has much sharper peak than those of FTS-3 and FTS-1, suggesting that the heat treatment condition of the sample is the optimum. This peculiarity is also confirmed in the graphs of \(\rho(T)-T(K)\), in Figure 3a, as high \(T_{c}^{(\text{onset})}\) and \(T_{c}^{(\text{zero})}\) values with a sharp transition. However, sample FTS-1 was showed a double peak, means that the double transition as obtained in the graphs of \(\rho(T)-T(K)\), in Figure 3a, suggest that the multiphase nature as mentioned before. Figure 4a shows the resistivity transition at magnetic field up to 8 T with applied field, \(H\), parallel to the sample surface for sample FTS-2 as an example. The other samples were showed similar trends. In general, the resistive transition was shifted to a lower temperatures and superconducting transition is slightly broadened by increasing the magnetic field as expected. Because, an increase on the magnetic field, attenuate the electron pairs by enforcing to bring the opposite spins in the same direction. This causes a reduction on the electron pairs population, reduces the \(T_{c}\) and delays the transition to the superconducting state. To determine the upper critical field \(H_{c2}(0)\), we used 10% \(T_{c}^{(\text{offset})}\), 50% \(T_{c}^{(\text{midpoint})}\) and 90% \(T_{c}^{(\text{onset})}\) criterion and Werthamer-Helfand-Hohenberg (WHH) formula. The WHH formula is given as [39];

\[
H_{c2}(0) = -0.693 T_{c}\left(\frac{dH_c(T)}{dT}\right)_{T=T_{c}}
\]  

(1)

By using Eq-1, the zero temperature value of upper critical field, \(H_{c2}(0)\), is calculated to be 87.5, 69.9 and 36.3 T for 10% \(T_{c}^{(\text{offset})}\), 50% \(T_{c}^{(\text{midpoint})}\) and 90% \(T_{c}^{(\text{onset})}\) respectively, Figure 4b. These values are slightly higher than the literature value [40]. The theoretical value of \(H_{c2}(T)\) was also calculated using an equation which is developed by Wen et.al. [41];

Table 2. Superconducting properties of polycrystalline FeTe\(_{0.5}\)Se\(_{0.5}\) samples prepared.

| Sample No | \(T_{c}^{(\text{onset})}\) (K) | \(T_{c}^{(\text{zero})}\) (K) | \(\Delta T\) (K) | \(H_{c2}(0)\) (T) | \(\xi(0)\) (nm) | \(E_{a}(H)\) (0T) | \(J_{c}^{\text{trans}}\) (A/cm\(^2\)) at 4.2 K (0T) |
|-----------|----------------|----------------|--------------|----------------|--------------|----------------|-------------------------------|
| FTS-1     | 14.3/10.3      | 5.2           | 9.1          | 66.6 - 50.6 - 23.6 | 2.22 - 2.55 - 3.73 | 2.610\(^{13}\) | 0.28                          |
| FTS-2     | 15.6           | 9.5           | 6.1          | 87.5 - 69.9 - 36.3 | 1.94 - 2.17 - 3.01 | 1.310\(^{12}\) | 6.06                          |
| FTS-3     | 14.9           | 8.3           | 6.6          | 84.1 - 57.7 - 32.8 | 1.98 - 2.38 - 3.17 | 9.410\(^{13}\) | 2.09                          |
\[ H_{c2}(T) = H_{c2}(0) \left( \frac{1 - \left( \frac{T}{T_c(0)} \right)^2}{1 + \left( \frac{T}{T_c(0)} \right)^2} \right) \]  

(2)

For sample FTS-2, the graph of calculated values are given in insert (lower left) of Figure 4b, as an example. At 0 K, calculated values were found to be 84.04 T, 57.60 T and 32.59 T for 10\% \( T_c \) (offset), 50\% \( T_c \) (midpoint) and for 90\% \( T_c \) (onset) respectively. These figures are found close to the results calculated by using the WHH equation. The superconducting coherence length, \( \xi(0) \), along the \( c \)-axis, was estimated using Ginzburg Landau (GL) equation. The GL equation is given by:

\[ H_{c2}(0) = \frac{\phi_0}{2\pi^2 \xi(0)} \]  

(3)

\[ \xi(0) = \left( \frac{\phi_0}{2\pi H_{c2}(0)} \right)^{1/2} \]  

(4)

here, \( \Phi_0 = 2.07 \times 10^{-15} \) Wb is the flux quantum [42, 43]. The calculated values of \( \xi(0) \) for 10\% \( T_c \) criterion are obtained to be 1.94, 1.98 and 2.22 nm for sample FTS-2, FTS-3 and FTS-1 respectively, Table-2. These results were found close to the previously calculated values by other research groups and also comparable to the single crystal samples [43]. We also calculated the \( \mu_0 H_{c2}(0)/k_B T_c \) value, which gives valuable information about the nature of the materials by using \( H_{c2}(0) \) obtained from the WHH theory. The \( \mu_0 H_{c2}(0)/k_B T_c \) results obtained showed comparably higher value (3.36 T/K) than the Pauli limit (1.84 T/K). This suggested unconventional nature of superconductivity in the sample as obtained in the FeTeSe single crystal family and also FeSe polycrystalline materials [30,36,44].

Figure 4. a) Field dependence of the resistivity of sample FTS-3 b) the upper critical field, \( H_{c2} \), as a function of critical temperature. The insert shows calculated theoretical values.

3.4. The Activation Energy Calculations

The activation energy, \( E_a \), is an important parameter for the description of the flux dynamics in the mixed state and revealing the height of the energy barriers which controls the vortex motion in the vortex liquid. In the thermally activated flux flow (TAFF) region, the thermally activated resistivity is defined as [45],

\[ \rho = \rho_0 \exp \left[ -\frac{E_a(J,H,T)}{k_B T} \right] \]  

(5)
where $\rho_0$ is the pre-exponent factor, $k_B$ the Boltzmann constant and $E_a$ the activation energy for the flux motion that generally depends on the temperature, $T$, the current density, $J$, and the applied magnetic fields, $H$. At constant current, the functional dependence of $E_a(H, T)$, is believed to give information about the dissipation mechanism and can be calculated from the slope of $\ln(\rho/\rho_0)$ versus $1/T$ graphs [45]. Figure 5 shows the $E_a$ versus $H(T)$ graphs of the samples FTS-1, FTS-2 and FTS-3. In general, the variation of $E_a$ is explained as an increase of flux trapping due to increased magnetic field. It is also well known that the decrease of the activation energy promotes the thermally activated dissipation due to the vortex motion in a single-crystal sample [45]. In contrast, for polycrystalline superconductors, dissipation begins in intergrain boundaries instead of sample surface or inside the sample. Thus, low value of $E_a$ can be explained with the transparent intergrain boundaries.

![Figure 5](image_url)

**Figure 5.** Calculated activation energy of the samples as a function of magnetic field.

### 3.5. Magnetic Properties

Figure 6a shows the temperature dependence of the magnetization ($M-T$) measurements under 10 Oe for samples prepared with different heating cycles. Above $T_c$, sample FTS-1 showed Pauli paramagnetic type behavior up to ~125 K and then remain unchanged down to ~10 K, then dropped to diamagnetic phase just after $T_c$. This indicates the role of impurities ie: unreacted Fe and hexagonal non-superconducting phase of FeTeSe material on the magnetic properties. An anomaly, which is obtained at around 125 K, is believed to be related with the structural transformation. However, sample FTS-2 and FTS-3 showed negative ferromagnetic sign down to $T_c$ and then sharp drop was obtained just after $T_c$, Figure 6a.

![Figure 6](image_url)

**Figure 6.** a) Temperature dependence of Magnetization and b) M-H curves at different temperatures for sample FTS-2. The first quadrants of the M-H curves are given in the left insert.
The controversy between the sample FTS-1 and both FTS-2 and FTS-3 suggests the role of different spin-spin and/or spin-orbital interaction of different phases, even under small magnetic fields (10 Oe) during the measurements. Magnetic hysteresis curves of sample FTS-2 as an example at 5, 8 and 12 K are given in figure 6b. It can be seen that polycrystalline FTS-2 sample has mainly a ferromagnetic background due to ferromagnetic nature of Fe, which yields more dominant ferromagnetic signal than the diamagnetic signal. In general, this indicates the absence of bulk superconductivity in the samples, as obtained by the other research groups [35-41]. The lower critical field \( H_{c1} \) is an important superconducting parameter where the magnetic field starts to penetrate in to the sample and accordingly vortices consist of on the sample surface as expected. The first quadrant of the M-H curves at 5, 8 and 12 K were measured to determine the lower critical field. The results of sample FTS-2 are given in Figure 6b (the left insert) as an example. The lower critical field values of sample FTS-2 were calculated as 210, 140 and 70 Oe for 5, 8 and 12 K, respectively. These values were found to be lower than the \( H_{Tc} \) materials but are comparable to low \( T_c \) conventional superconductors and FeSe based materials.

3.6. I-V Measurements
The \( I-V \) characteristics of the samples were measured by the four probes technique with the current flow along the sample surface. The measurements were carried out at 4.2 K with magnetic fields ranging from 50 Oe to 1T. \( I-V \) curves of the sample FTS-2 at 4.2 K, under different magnetic fields. The results are given in Figure 7 and summarized in Table 2. The \( I-V \) characteristic of the samples appear as flux creep behavior as obtained for the \( H_{Tc} \) BSCCO material [45]. The highest \( J_{c,\text{trans}} \) value was calculated to be 60.6 A/cm² at zero field and 4.2 K and 19.7 A/cm² at 1 T and 4.2 K. When the external field increases, the critical current density, \( J_{c,\text{trans}} \), decreases in the flux creep regime. However, the subsidence is found to be very slow comparing with the \( H_{Tc} \) and also other conventional superconductors and suggested strong grain connectivity properties in this material. In addition we believed that The small amount of impurity phases, which provides pinning centers on the surface, play a crucial role on the \( J_{c,\text{trans}} \) limit particularly for sample FTS-2 and FTS-3.

3.7. Thermal properties
The Seebeck coefficient, \( S \), of the polycrystalline FeTe\(_{0.5}\)Se\(_{0.5}\) samples are given as a function of temperature in Figure 8a. The Seebeck coefficient, \( S \), of samples FTS-2 and FTS-3 was started from negative value (approximately \(-2\mu\text{V/K}\)) at high temperatures and then slightly increases between 250 and 200 K. After the certain temperature it drops to \(-25\text{K}\) and then increases again. At low temperature region, the sample FTS-1 shows similar trend like FTS-2 and FTS-3 samples. But a positive \( S \) value is obtained between 180 and 290 K and then it turns to negative value at 300 K. This anomaly can be explained using drag mechanism which is related with the exchange of momentum between charge carriers and magnetic fluctuations of particularly ferromagnetic phase that is dominant all over the sample. The temperature dependence of thermal conductivity, \( \kappa \), are shown in Figure 8b. The \( \kappa \) value of the samples show a variation temperature at room temperature. For the sample FTS-2, the optimally treated sample, indicates comparably high \( \kappa \) value at room temperature. It decreases slowly from the room temperature to 75 K and then rapid reduction is observed to the minimum value. The other sample show similar peculiarity. In general, the obtained results show a classical property of metals and alloys and suggesting two important points. Firstly; at high temperature region, thermal conductivity dominates by phonons because electron contribution evaluates by the Wiedermann-Franz law being negligible as suggested by other research groups [46]. Secondly, at low temperature region the main scattering mechanism of phonons was charge carriers and structural defects such as, a large Fe vacancies and relatively large \( c \)-axis disorders.
4. Conclusion

The Polycrystalline FeTe$_{0.5}$Se$_{0.5}$ superconductors have been prepared by conventional solid-state reaction method in the evacuated quartz tube. Structural, physical, magnetic and transport properties were investigated. The results show that FeTeSe polycrystalline samples are very sensitive to the magnetic field and this intricacy can be related with insufficiency of bulk superconductivity in the samples where different magnetic behaviors can be occurred. For optimally treated sample (FTS-2) the upper critical field, $H_{c2}(0)$, was obtained to be 87.5 T, at the zero field coherence length, $\xi$, value was calculated to be 1.94 nm and the lower critical field, $H_{c1}(T)$, is obtained to be 210, 140 and 70 Oe at 5, 8 and 12 K, respectively.

The $\mu_0 H_{c2}(0)/k_B T_c$ value, which gives more information about the nature of the superconductor materials showed higher value (3.36 T/K) than the Pauli limit (1.84 T/K). In overall, the results indicate the unconventional nature of superconductivity in the polycrystalline FeTe$_{0.5}$Se$_{0.5}$ superconductor samples. The $J_c$ value of samples is still low but it is convenient for bulk applications such as, large magnets or flywheels.

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