



UNIVERSITY
OF WOLLONGONG
AUSTRALIA

University of Wollongong
Research Online

Australian Institute for Innovative Materials - Papers

Australian Institute for Innovative Materials

2010

Synthesis and physical properties of $\text{FeSe}_{1/2}\text{Te}_{1/2}$ superconductor

V P. Awana

National Physical Laboratory, New Delhi

Anand Pal

National Physical Laboratory, New Delhi

Arpita Vajpayee

National Physical Laboratory, New Delhi

Monika Mudgel

National Physical Laboratory, New Delhi

H Kishan

National Physical Laboratory, New Delhi

See next page for additional authors

Publication Details

Awana, VPS, Pal, A, Vajpayee, A, Mudgel, M, Kishan, H, Husain, M, Zeng, R, Yu, S, Guo, YF, Shi, YG, Yamaura, K & Takayama-Muromachi, E (2010), Synthesis and physical properties of $\text{FeSe}_{1/2}\text{Te}_{1/2}$ superconductor, *Journal of Applied Physics*, 107(9), pp. 09E128-1-09E128-3.

Research Online is the open access institutional repository for the University of Wollongong. For further information contact the UOW Library:
research-pubs@uow.edu.au

Synthesis and physical properties of FeSe_{1/2}Te_{1/2} superconductor

Abstract

One of the most important properties of very recently reported FeSe based superconductors is the robustness of their superconductivity under applied magnetic field. The synthesis and control of superconductivity in FeSe based compounds is rather a difficult task. Synthesis and physical property characterization for optimized superconductivity of FeSe_{1/2}Te_{1/2} at 13 K is reported here. The compound crystallized in a tetragonal structure with lattice parameters $a=3.8015(2)$ and $c=6.0280(4)$ Å. Magnetization measurements indicated bulk superconductivity with lower critical field (H_{c1}) of around 180 Oe. By applying Ginzburg–Landau theory, the $H_{c2}(0)$ value is estimated to be ~ 1840 kOe for the 90% of resistive transition. A heat capacity measurement revealed bulk superconductivity by a hump at T_c near 13 K and an expected decrease in the same was observed under an applied magnetic field.

Keywords

Synthesis, physical, properties, FeSe₁, Te₁, superconductor

Disciplines

Engineering | Physical Sciences and Mathematics

Publication Details

Awana, VPS, Pal, A, Vajpayee, A, Mudgel, M, Kishan, H, Husain, M, Zeng, R, Yu, S, Guo, YF, Shi, YG, Yamaura, K & Takayama-Muromachi, E (2010), Synthesis and physical properties of FeSe_{1/2}Te_{1/2} superconductor, *Journal of Applied Physics*, 107(9), pp. 09E128-1-09E128-3.

Authors

V P. Awana, Anand Pal, Arpita Vajpayee, Monika Mudgel, H Kishan, Mushahid Husain, Rong Zeng, S Yu, Y F. Guo, Y.G Shi, K Yamaura, and E Takayama-Muromachi

Synthesis and physical properties of FeSe_{1/2}Te_{1/2} superconductor

V. P. S. Awana,^{1,a)} Anand Pal,¹ Arpita Vajpayee,¹ Monika Mudgel,¹ H. Kishan,¹ Mushahid Husain,² R. Zeng,³ S Yu,^{4,5} Y. F. Guo,^{4,6} Y. G. Shi,^{4,6} K. Yamaura,^{4,5} and E. Takayama-Muromachi^{4,5,6}

¹National Physical Laboratory (CSIR), Dr. K.S. Krishnan Marg, New Delhi 110012, India

²Department of Physics, Jamia Millia Islamia, New Delhi 110025, India

³ISEM, University of Wollongong, New South Wales 2522, Australia

⁴Transformative Research-Project on Iron Pnictides (TRIP), JST, Tsukuba, Ibaraki 305-0044, Japan

⁵Superconducting Materials Center, National Institute for Materials Science, 1-1 Namiki, Tsukuba, Ibaraki 305-0044, Japan

⁶International Center for Materials Nanoarchitectonics (MANA), National Institute for Materials Science, Tsukuba, Ibaraki 305-0044, Japan

(Presented 21 January 2010; received 30 October 2009; accepted 11 December 2009; published online 22 April 2010)

One of the most important properties of very recently reported FeSe based superconductors is the robustness of their superconductivity under applied magnetic field. The synthesis and control of superconductivity in FeSe based compounds is rather a difficult task. Synthesis and physical property characterization for optimized superconductivity of FeSe_{1/2}Te_{1/2} at 13 K is reported here. The compound crystallized in a tetragonal structure with lattice parameters $a=3.8015(2)$ and $c=6.0280(4)$ Å. Magnetization measurements indicated bulk superconductivity with lower critical field (H_{c1}) of around 180 Oe. By applying Ginzburg–Landau theory, the $H_{c2}(0)$ value is estimated to be ~ 1840 kOe for the 90% of resistive transition. A heat capacity measurement revealed bulk superconductivity by a hump at T_c near 13 K and an expected decrease in the same was observed under an applied magnetic field. © 2010 American Institute of Physics. [doi:10.1063/1.3366602]

I. INTRODUCTION

The recent discovery of iron-based chalcogenide^{1–3} and the iron-based pnictide^{4–6} superconductors has generated a great deal of interest in the scientific community. The parent compounds of the FeAs based superconductors do not exhibit superconductivity but possess stripe type commensurate antiferromagnetic spin order accompanied by a structural transition at around 150 K.^{7,8} The superconductivity was obtained by either fluorine doping at the oxygen site or by the deficiency of oxygen ions in the system.^{4–9} The highest $T_c = 55$ K has been reported in SmFeAS(O_{1-x}F_x).¹⁰ Very recently, another iron based superconductor FeSe was reported with a T_c of 8 K.¹ FeSe is the simplest-structured among the iron-based superconductors. The T_c of FeSe is increased up to 15 K by partial substitution of Te or S for Se and it is also increased up to ~ 27 K by applying hydrostatic pressure of 1.48 GPa.^{3,11–15} The effect of pressure on superconductivity, i.e., dT_c/dP is around 9.1 K/GPa, which is four times more than for any other known superconductor until date. Very high dT_c/dP of FeSe warrants large scope for increasing its T_c by chemical pressure through on site substitutions. Garbarino *et al.*¹⁶ found that under compression, FeSe transforms from the original tetragonal structure to an orthorhombic high pressure phase above 12 GPa. The high-pressure orthorhombic phase has a higher T_c reaching 34 K at 22 GPa.

Here, we report synthesis and physical properties of FeSe_{1/2}Te_{1/2}. The structural magnetotransport and heat capacity measurement are reported for title compound.

II. EXPERIMENTAL

A Polycrystalline sample of title compound was synthesized by the solid-state reaction route. The stoichiometric ratio of high purity (>3 N) Fe, Se, and Te were ground, palletized, and encapsulated in an evacuated quartz tube. The encapsulated tube was then heated at 750 °C for over 24 h and slowly cooled to room temperature. The sintered pellets were again ground, pelletized in a rectangular shape, sealed in an evacuated quartz tube, and resintered at 750 °C for 12 h. Unlike the highly reactive RE (rare earths) and alkaline metals (Sr, Ba, and Ca), the Fe, Se, and Te were weighed and mixed in open with inhaling (mask) and skin touch (gloves) precautions. The x-ray diffraction patterns of the samples were obtained with the help of a Rigaku diffractometer. The resistivity measurements were recorded for temperatures down to 4.2 K via a four-probe method. The temperature dependence of dc magnetization of the FeSe_{1/2}Te_{1/2} sample was analyzed using a superconducting quantum interference device magnetometer (Quantum Design) both in zero-field and field-cooled configurations. Heat capacity measurements were carried out on a Quantum Design Physical Property Measurement System (PPMS).

III. RESULTS AND DISCUSSION

The x-ray diffraction pattern of FeSe_{1/2}Te_{1/2} compound and its Rietveld refinement are shown in Fig. 1. All the peaks

^{a)} Author to whom correspondence should be addressed. FAX: 0091-11-45609310. Tel.: 0091-11-45608329. Electronic mail: awana@mail.nplindia.ernet.in. URL: www.freewebs.com/vpsawana.

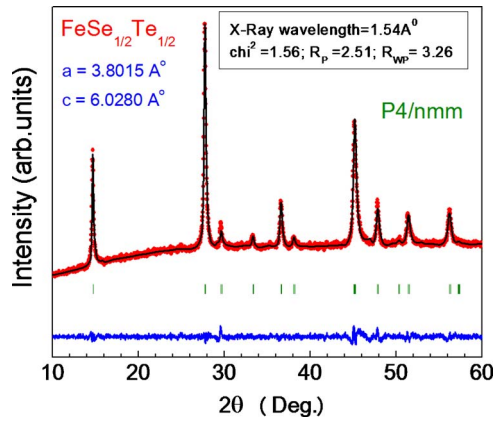


FIG. 1. (Color online) The observed (dots), calculated (solid line), and differences diffraction (bottom solid line) profiles at 300 K for $\text{FeSe}_{1/2}\text{Te}_{1/2}$.

are well indexed using a space group of $P4/nmm$, and no impurity phases are found. Fe is located at coordinate position $(\frac{3}{4}, \frac{1}{4}, 0)$ and Se/Te at $(\frac{1}{4}, \frac{1}{4}, 0.2778)$ in $\text{FeSe}_{1/2}\text{Te}_{1/2}$ compound. The compound crystallizes in a tetragonal structure and no secondary phase is observed. Lattice parameters are found to be $a=3.8015(2)$ and $c=6.0280(4)$ Å. The lattice parameters are in good agreement with previously reported literature.^{11,16}

The magnetic susceptibility at 10 Oe is shown in Fig. 2 for both zero-field cooling (ZFC) and field cooling (FC) measuring conditions. Sample shows clear superconducting diamagnetic response below this onset temperature T_c^{dia} , i.e., below 13 K. This value is in confirmation with earlier reports.¹⁷⁻¹⁹ This figure confirms the bulk superconductivity in the present sample. The hysteresis loops (M-H) measured at temperatures 2, 5, and 8 K are shown in lower inset of Fig. 2. The upper inset of Fig. 2 shows only the first quadrant of the M(H) loop at 2, 5, and 8 K. The M(H) plot inverts from

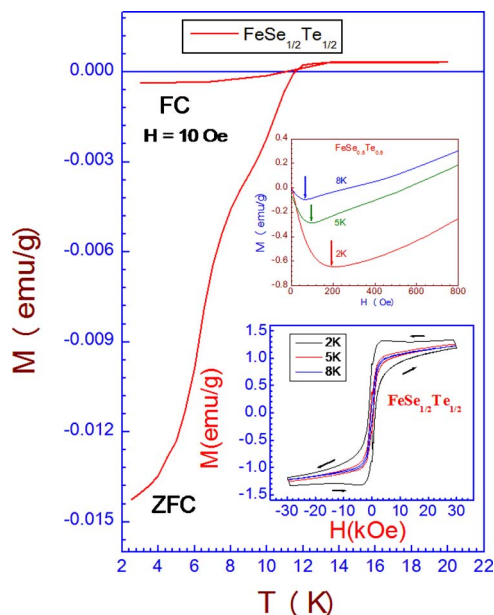


FIG. 2. (Color online) Temperature dependence of magnetization under 10 Oe field measured in both FC and ZFC conditions. Lower inset shows the complete M(H) loop at 2, 5, and 8 K and upper inset shows the first quadrant of the M(H) loop.

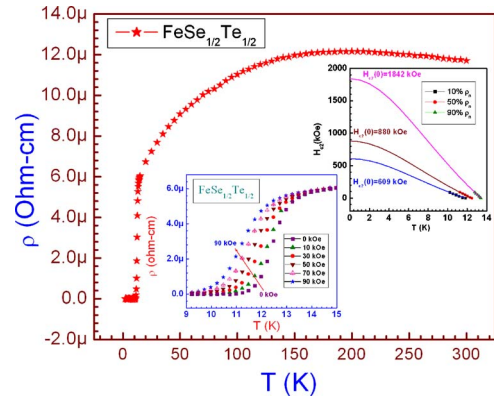


FIG. 3. (Color online) Temperature dependence of the resistivity of $\text{FeSe}_{1/2}\text{Te}_{1/2}$ measured in 0 kOe field; lower inset shows the temperature dependence of resistivity measured in fields up to 90 kOe for the same compound and upper inset shows the H_{c2} vs T plots derived from measurements of resistivity against temperature and magnetic field [$\rho(T)/H$ plots].

around 180 Oe at 2 K. For higher T of 5 and 8 K, obviously the inversion field and hence H_{c1} decreases. This shows that the lower critical field (H_{c1}) of the studied $\text{FeSe}_{1/2}\text{Te}_{1/2}$ sample is around 180 Oe at 2K. The lower inset depicts wide open M(H) plots in all four quadrants at 2, 5, and 8 K. The opening of the M(H) loops decrease with increase in temperature. Further the positive moment of the Fe magnetic ion is riding over the diamagnetic signal and hence results in some positive background.

Figure 3 shows the temperature dependence of the resistivity $\rho(T)$ of the $\text{FeSe}_{1/2}\text{Te}_{1/2}$ sample. This figure depicts that the studied sample has a somewhat semiconducting behavior above 150 K; below this temperature it shows the metallic behavior in the normal state. Mizuguchi *et al.*¹¹ also got the semiconducting type behavior for Te substituted FeSe compounds. With a decrease in temperature below 14 K, the resistivity of the sample starts vanishing abruptly due to occurrence of superconductivity. The onset and the zero resistivity temperatures of the sample are determined to be 13.5 and 11.5 K, respectively.

In order to determine the upper critical field of the sample $\rho(T)$ curves are measured under different magnetic fields up to 90 kOe. The superconducting transition zone of $\rho(T)/H$ measurements is shown in lower inset of Fig. 3. The superconducting transition temperature decreases with an increase in applied magnetic field with a rate of decrement, i.e., $dT_c/dH \sim 0.03$ K/kOe. Such a low value of dT_c/dH makes this compound superior against the Nb based BCS type, HTSc, borides, or even the recently invented FeAs superconductors.

The upper critical field is determined using different criterion of $H_{c2}=H$ at which $\rho=90\%\rho_N$ or $50\%\rho_N$ or $10\%\rho_N$, where ρ_N is the normal resistivity or resistivity at about 15 K. The H_{c2} variation with temperature is shown in upper inset of Fig. 3. To determine $H_{c2}(0)$ value, we applied Ginzburg-Landau (GL) theory. The GL equation is

$$H_{c2}(T) = H_{c2}(0) * (1 - t^2)/(1 + t^2),$$

where $t=T/T_c$ is the reduced temperature.²⁰ The fitting of experimental data is done according to the above equation,

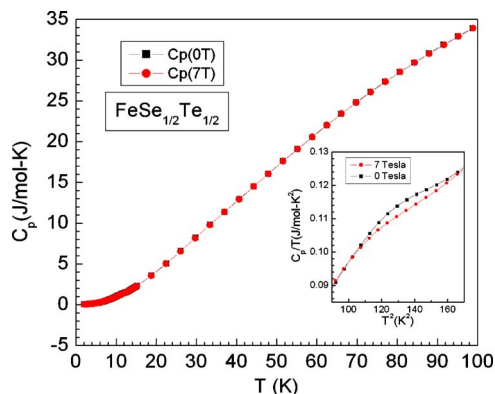


FIG. 4. (Color online) Heat capacity variation with temperature (C_p - T) in zero field and 70 kOe field; inset shows the enlarged view of the anomaly present in C_p/T vs T^2 .

which not only determines the H_{c2} value at 0 K [$H_{c2}(0)$] but also determines the temperature dependence of critical field for the whole temperature range. $H_{c2}(10\%)$, $H_{c2}(50\%)$, and $H_{c2}(90\%)$ are estimated to be 608, 880, and 1842 kOe, respectively, at 0 K. These values of upper critical field are comparable with the reported in literature.^{15,21,22} Yadav *et al.*²² estimated the values of $H_{c2}(10\%)$, $H_{c2}(50\%)$, and $H_{c2}(90\%)$ as 690, 880, and 1840 kOe, respectively, for $\text{FeTe}_{0.60}\text{Se}_{0.40}$ sample.

In order to study the nature of magnetic anomalies we further performed specific heat measurement under a magnetic field of 0 and 70 kOe, as shown in Fig. 4. The specific heat in zero field displays a small hump at $T_c \approx 13$ K. This is in agreement with literature.¹⁷ After applying the magnetic field of 70 kOe the small hump is observed at the same temperature but with slightly decreased magnitude. This is obvious for the case of a superconducting transition. Generally, specific heat of a superconductor decreases with applied field. The enlarged view of this anomaly present in C_p/T versus T^2 is shown in inset of Fig. 4. There is no report available on heat capacity measurement under applied magnetic field in this temperature range; in Ref. 17, Sales *et al.*¹⁹ reported the $C_p(T)$ plot only at zero field. Very recently,¹⁹ incommensurate magnetic excitations are reported for $\text{FeSe}_{1/2}\text{Te}_{1/2}$ just below its T_c , which could also contribute to $C_p(T)$ H anomaly.

IV. CONCLUSIONS

In summary, we have studied the structural, electronic transport and magnetic properties of $\text{FeSe}_{1/2}\text{Te}_{1/2}$ superconductor. The result of resistivity and magnetization measurements clearly shows that title compound becomes a superconductor below ~ 14 K. $\rho(T)H$ measurements show that the superconductivity transition temperature (T_c) barely decreases with applied field. This is unlike the Nb based BCS

type, HTSC, borides or even the recently invented FeAs superconductors. The high $H_{c2}(0)$ value renders this superconductor as powerful competitor, which will be potentially useful in very high field applications.

ACKNOWLEDGMENTS

The work is supported by Indo-Japan (DST-JSPS) bilateral exchange research program. V.P.S.A. further thanks NIMS for providing him with the MANA visiting scientist position for three months. Authors from NPL would like to thank Professor Vikram Kumar (DNPL) for his constant encouragement. Anand Pal, Arpita Vajpayee, and Monika Mudgel are thankful to CSIR for providing the financial support during their research.

- ¹F. C. Hsu, L. Y. Luo, K. W. Yeh, T. K. Chen, T. W. Huang, and P. M. Wu, *Proc. Natl. Acad. Sci. U.S.A.* **105**, 14262 (2008).
- ²T. M. McQueen, Q. Huang, V. Ksenofontov, C. Felser, Q. Xu, and R. J. Cava, *Phys. Rev. B* **79**, 014522 (2009).
- ³Y. Mizuguchi, F. Tomioka, S. Tsuda, and T. Yamaguchi, *Appl. Phys. Lett.* **93**, 152505 (2008).
- ⁴Y. Kamihara, T. Watanabe, M. Hirano, and H. Hosono, *J. Am. Chem. Soc.* **130**, 3296 (2008).
- ⁵H. Takahashi, K. Igawa, K. Arii, Y. Kamihara, M. Hirano, and H. Hosono, *Nature (London)* **453**, 376 (2008).
- ⁶G. F. Chen, Z. Li, D. Wu, G. Li, W. Z. Hu, J. Dong, P. Zheng, J. L. Luo, and N. L. Wang, *Phys. Rev. Lett.* **100**, 247002 (2008).
- ⁷C. R. de la Cruz, Q. Huang, J. W. Lynn, J. Li, W. Ratcliff, J. L. Zarestky, H. A. Mook, G. F. Chen, J. L. Luo, N. L. Wang, and P. Dai, *Nature (London)* **453**, 899 (2008).
- ⁸M. R. Norman, *Physics* **1**, 21 (2008).
- ⁹H. Kito, H. Eisaki, and A. Iyo, *J. Phys. Soc. Jpn.* **77**, 063707 (2008).
- ¹⁰Z. A. Ren, W. Lu, J. Yang, W. Yi, X. L. Shen, Z. C. Li, G. C. Che, X. L. Dong, L. L. Sun, F. Zhou, and Z. X. Zhao, *Chin. Phys. Lett.* **25**, 2215 (2008).
- ¹¹Y. Mizuguchi, F. Tomioka, S. Tsuda, T. Yamaguchi, and Y. Takano, *J. Phys. Soc. Jpn.* **78**, 074712 (2009).
- ¹²K.-W. Yeh, T.-W. Huang, Y.-L. Huang, T.-K. Chen, F.-C. Hsu, P. M. Wu, Y.-C. Lee, Y.-Y. Chu, C.-L. Chen, J.-Y. Luo, D.-C. Yan, and M.-K. Wu, *Europhys. Lett.* **84**, 37002 (2008); e-print arXiv:0808.0474v2.
- ¹³M. H. Fang, H. M. Pham, B. Qian, T. J. Liu, E. K. Vehstedt, Y. Liu, L. Spinu, and Z. Q. Mao, *Phys. Rev. B* **78**, 224503 (2008).
- ¹⁴T. Imai, K. Ahilan, F. L. Ning, T. M. McQueen, and R. J. Cava, *Phys. Rev. Lett.* **102**, 177005 (2009).
- ¹⁵T. Kida, T. Matsunaga, M. Hagiwara, Y. Mizuguchi, Y. Takano, and K. Kindo, *J. Phys. Soc. Jpn.* **78**, 113701 (2009).
- ¹⁶G. Garbarino, A. Sow, P. Lejay, A. Sulpice, P. Toulemonde, M. Mezouar, and M. Nunez-Regueiro, *EPL* **86**, 27001 (2009).
- ¹⁷B. C. Sales, A. S. Sefat, M. A. McGuire, R. Y. Jin, and D. Mandrus, *Phys. Rev. B* **79**, 094521 (2009).
- ¹⁸T. Geetha Kumary, D. K. Baisnab, J. Janaki, A. Mani, A. T. Satya, R. M. Sarguna, P. K. Ajikumar, A. K. Tyagi, and A. Bharathi, *Supercond. Sci. Technol.* **22**, 095018 (2009).
- ¹⁹H. A. Mook, M. D. Lumsden, A. D. Christianson, S. E. Nagler, B. C. Sales, R. Jin, M. A. McGuire, A. Sefat, D. Mandrus, T. Egami, and C. dela Cruz, *Phys. Rev. Lett.* (in press); e-print arXiv:0911.5463v1.
- ²⁰X. Wang, S. R. Ghorbani, G. Peleckis, and S. Dou, *Adv. Mater. (Weinheim, Ger.)* **21**, 236 (2009).
- ²¹K. W. Yeh, H. C. Hsu, T. W. Huang, P. M. Wu, Y. L. Luo, T. K. Chen, J. Y. Luo, and M. K. Wu, *J. Phys. Soc. Jpn.* **77**, 19 (2008).
- ²²C. S. Yadav and P. L. Paulose, e-print arXiv:0907.0147.