

Effects of Toluene (C₇H₈) Addition on Microstructure and Superconducting Properties of Bulk C₆H₁₀O₆Fe + SiC-doped MgB₂

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Abstract

Superconductors can reduce the energy loss and consumption, help to build high efficiency computers, power plant and store electric energy and can solve the energy crisis partially at room temperature. Synthesis and characterization of bulk MgB₂ superconductor co-doped with C₆H₁₀O₆Fe+SiC and MgB₂ samples co-doped with C₆H₁₀O₆Fe+SiC +(C₇H₈) is reported in this paper. Effects of C₆H₁₀O₆Fe+SiC and (C₇H₈) addition during sample preparation on microstructure and superconducting properties are investigated. Decrease of the *a*-axis lattice parameter and degradation of the crystalline of the samples suggested successful substitution of C for B. C₆H₁₀O₆Fe+SiC with C₇H₈ treated samples showed best pinning performance, while C₆H₁₀O₆Fe+SiC treated samples still showed an increased in the values of critical current density J_c as compared to un-doped samples. At 5K and 8T, the highest critical current (J_c) reached 1.85×10^4 A/cm² and 0.95×10^4 A/cm² for 10 wt% C₆H₁₀O₆Fe+SiC treated samples with and without C₇H₈, respectively. At 20K, the highest critical current (J_c) reached 2.85×10^2 A/cm² at self field for 10 wt% C₆H₁₀O₆Fe+SiC treated samples with and without C₇H₈. It is concluded that C₆H₁₀O₆Fe+SiC is an excellent co-doping for MgB₂ lattice, while C₆H₁₀O₆Fe+SiC+C₇H₈ is effective in improving the homogeneity of C₆H₁₀O₆Fe+SiC-doped MgB₂ phases.

Key Words

Superconductors, MgB₂, Synthesis, characterization, Pinning.

Introduction

MgB₂ is advantageous to other conventional metallic superconductors for applied superconductivity due to the significantly high transition temperature of 39 K [1], low cost and high abundance of Mg and B, and the large coherence length. Doping with various elements or compounds as well as optimization of synthesis conditions has been found to be effective for enhancing the critical current properties for MgB₂ bulks and wires. C is one of the most effective dopants for such purposes showing significant enhancement in the electromagnetic properties of MgB₂ [2-13]. Improvement of both upper critical field (H_{c2}) and critical current density (J_c) is essential to fully utilize MgB₂'s T_c of 39K. Attempts to accomplish this have led to the introduction of numerous techniques including ball milling [14, 15], chemical doping [3, 4, 6-13], irradiation [16,34,35], and various thermo-mechanical processing techniques [17-20]. Among these, chemical doping is a simple and readily scalable technique. Due to relatively large coherence length and small anisotropy of MgB₂, the fluxoids to be pinned are string-like and could be pinned by inclusions and precipitates in the grains, which makes chemical doping highly anticipated [8]. Among large number of compounds used for chemical C doping into MgB₂, SiC is the most popular, being effective for enhancement of the irreversibility field (H_{irr}) and J_c under magnetic fields [3,4,12,21]. However, the best high field J_c values achieved were compromised by the reduction in self-field and low-field J_c . In most of the cases samples are prepared by solid state mixing of starting materials. It is important to emphasize the fact that achievement of homogeneous mixtures using solid state mixing is a great deal of challenge, especially when nano-size precursors are used. Agglomeration of nano-additives in the precursors is a common phenomenon. Xu et al. already reported that the C₇H₈ is the best as a media for improving the superconducting properties and homogeneity of the powder compared to other hydrocarbon solution [14,36]. In addition, poor reactivity of various forms of C makes substitution of C for B in MgB₂ at temperatures that are close to the formation temperature of MgB₂ very ineffective. Thus, in order to decrease sintering temperature and improve effectiveness of C substitution, active C species are required. Hydrocarbons such as benzene have been used for C substitution [13]. However, high volatility of these compounds limits the C substitution level significantly. We have shown that carbohydrates such as

$C_6H_{10}O_6Fe + SIC$ [22] are effective for C doping and the C substitution level can be achieved higher than that reported in [13]. There are several advantages of carbohydrate doping we already discussed in our previous paper [22]. In this article, we report on the fabrication of bulk C-doped MgB_2 samples with $C_6H_{10}O_6Fe + SIC$ used as a C source material. Effects of C_7H_8 addition to the mixture of precursors and preheating effect is analyzed as well. The lattice parameters, critical temperature (T_c), J_c , H_{irr} , H_{c2} , and microstructures of both methods are compared to that of un-doped MgB_2 used as a reference.

Experimental Procedures

For C_7H_8 containing samples, the selected amount of $C_6H_{10}O_6Fe + SIC$ (99%), from 0 to 30 wt%, was dissolved in C_7H_8 (99.5%). The solution was mixed with an appropriate amount of B (99%) powder. This slurry was dried in a vacuum furnace to achieve maximum coating of B powder particles by $C_6H_{10}O_6Fe + SIC$. This uniform composite was then mixed with a stoichiometric amount of Mg (99%) powder. The resulting powders were ground, pressed, and then sintered at 900°C for 30 min under high purity argon gas. The heating rate was 5°C/min. Phase purity, crystal structures and morphology of the samples were characterized by x-ray diffraction (XRD) and scanning electron microscopy (SEM). For structural parameters the x-ray diffraction patterns were refined using Reitica Rietveld analysis software suite and self-developed diffraction analysis software. The T_c was defined as the onset temperature at which diamagnetic properties were observed. H_{c2} (T) and H_{irr} (T) were defined as the fields where the temperature dependent resistance at constant magnetic field R (H_{c2} , T) = 0.9 Rns and R (H_{irr} , T) = 0.1 Rns with Rns being the normal state resistance at 40 K. The magnetization (M) was measured at 5 and 20 K using a Physical Properties Measurement System (PPMS, Quantum Design) in a time-varying magnetic field with sweep rate 50 Oe/s and amplitude of 8.5 T. In order to avoid the sample size effect on the J_c , all the samples were shaped to have same sizes. The magnetic J_c was derived from the width of the magnetization loop using Bean's model. J_c versus magnetic field was measured up to 8.5 T.

Results and Discussions

Figure 1 shows the x-ray diffraction for MgB_2 bulks doped with $C_6H_{10}O_6Fe + SIC$ dissolved in C_7H_8 , respectively. The reference sample, i.e. un-doped as well as C-doped with C_7H_8 samples was of hexagonal MgB_2 phase having minute amounts of MgO impurity. As we can see the position of (110) peak for all samples has been systematically shifted towards higher 2θ values with increasing C doping level. On the other hand, no change was observed in the position of the (002) reflection. These behaviors can be well explained by C substitution for boron (B) in the MgB_2 crystal lattice [24-27] as the a -axis length decreases with increasing C doping level. Furthermore, the peaks of the C-doped MgB_2 samples were broader than those of the un-doped sample, indicating a lattice disorder caused by C doping. The dependences of lattice parameter a on the C doping level for all samples are summarized in Table 1. The relationship between the length of lattice parameter a and actual C substitution suggests that only some part of raw C coming from carbohydrate is doped into the MgB_2 lattice. From fig. 3, it can also be clearly seen that C_7H_8 treated samples contain less MgO impurity than those in without C_7H_8 samples, proving that C_7H_8 is effective for prevention of the absorption of oxygen from atmosphere during sample preparation procedure. Hence, this fact might have been crucial for better performance of C_7H_8 treated samples.

Figure 2 shows the comparison of T_c and FWHM measured for the un-doped and C_7H_8 treated and untreated C-doped MgB_2 samples. It is clear that T_c systematically decreases with increasing C-doping level for both groups of samples as compared to that of un-doped sample. The effect of band filling of the σ band by electron doping can originate from the difference in the valences of B and C, and thus explain a systematic decrease of T_c values [28, 33-36]. One should note that the decrease in T_c correlates well with decrease in a -axis lattice parameter. Thus, the decrease of the a -axis length and T_c observed in C-doped samples can be simply explained on the basis of the actual C substitution effect [29]. It is also noted that the T_c suppression rate in dry mixing sample was higher than sample treated with C_7H_8 i.e. the rate of change of T_c in C_7H_8 treated sample was almost 1K but in dry mixing sample that was more than 3K. This result strongly suggested that the homogeneity of C_7H_8 treated samples was improved which is very useful in high field applications. This effect can also be explained by the fact that C source material, i.e. $C_6H_{10}O_6Fe + SIC$, may easily dissolve in C_7H_8 . Thus, being in the form of solution it can coat the surface of B powders, filling up the voids and gaps in the powders as well. This is probably because large fraction of $C_6H_{10}O_6Fe + SIC$ does not take part in substitution reaction and directly go away with C_7H_8 under this specific experiment condition. After the decomposition of $C_6H_{10}O_6Fe + SIC$ the produced fresh C reacts with a higher surface area of B resulting in more effective C substitution. The FWHM values for both samples with and without C_7H_8 [23] consistently increased with increasing C-doping level indicating poorer crystallinity as compared to the

reference sample. C substitution in MgB_2 is considered to introduce lattice distortion and reduction of mean free path and coherence length. This result indicates that introduction of lattice defects and/or decrease of the crystal size of MgB_2 occurred upon $\text{C}_6\text{H}_{10}\text{O}_6\text{Fe} + \text{SICS}$ doping.

In Figure 3 we have shown the comparison of MgO amount in dry and wet mixing with C_7H_8 . It should note that the MgO amount was significantly decreased in C_7H_8 treated sample because in dry mixing when $\text{C}_6\text{H}_{10}\text{O}_6\text{Fe} + \text{SIC}$ decomposes, it might produce large amount of H_2O and CO which reacts with Mg to form MgO but when $\text{C}_6\text{H}_{10}\text{O}_6\text{Fe} + \text{SIC}$ reacts with C_7H_8 it might produce less amount of H_2O and CO which might cause to form less MgO and FeO . As a consequence, any water molecules that are produced during decomposition of $\text{C}_6\text{H}_{10}\text{O}_6\text{Fe} + \text{SIC}$ cannot be absorbed by the powders and thus the amount of formed MgO are lower as compared to the samples prepared via dry processing route. Actual C substitution versus amount of C source for both dry mixing sample and C_7H_8 treated sample is shown in fig 4. C substitution was calculated from a -axis lattice parameter. The a -axis lattice parameter, decreased from 3.0836 (3) Å for a well developed un-doped sample sintered at 900°C for 30 min to 3.0781(33), 3.0779(31), 3.0777(29) Å for 10 wt%, 20 wt% and 30 wt% $\text{C}_6\text{H}_{10}\text{O}_6\text{Fe} + \text{SIC}$ samples with C_7H_8 sintered at 900°C for 30 min, respectively. Actual C substitution increased slightly in C_7H_8 treated samples but in dry sample it increased very rapidly due to large change in a -axis lattice parameter. This is probably because the large fraction of $\text{C}_6\text{H}_{10}\text{O}_6\text{Fe} + \text{SIC}$ does not take part in substitution reaction and directly goes away with C_7H_8 under this specific experiment condition. Fig. 5 shows the dependence of critical current density (J_c) on applied magnetic field (H) for C doped MgB_2 samples treated with C_7H_8 at 5 K and 20 K. The 10 wt% (nominal) sample showed the best J_c , reaching $1.14 \times 10^4 \text{ A cm}^{-2}$ at 5 K and 8 T which was higher than dry mixing samples [23]. Kim et al. [22] reported that the particular carbohydrate chosen had an influence on the homogeneity of the samples. In addition, C_7H_8 solution is also found to be effective for preparing homogeneous powder in experiments [14]. During the decomposition of the carbohydrate, some C can be coated on the surface of B powder, which also could affect the T_c . In our previous study we reported $\text{C}_6\text{H}_{10}\text{O}_6\text{Fe} + \text{SIC}$ doped MgB_2 mixed with C_7H_8 [22] showed much higher J_c in both high and self field than $\text{C}_6\text{H}_{10}\text{O}_6\text{Fe} + \text{SIC}$ doped with and without C_7H_8 . Different factors may affect on J_c such as chemical composition and structure of doping, preheating temperature, decomposition temperatures of both doping etc. On the other hand, a high level of doping of $\text{C}_6\text{H}_{10}\text{O}_6\text{Fe} + \text{SIC}$ with C_7H_8 samples degraded the J_c characteristics, mainly due to the decrease of T_c caused by large amounts of C substitution. It should be noted that J_c under low fields for the $\text{C}_6\text{H}_{10}\text{O}_6\text{Fe} + \text{SIC}$ doped samples with and without C_7H_8 was also closed to our best un-doped sample. The improved J_c under low fields is considered to be derived from the high bulk density and smaller amount of impurity phases [9].

The microstructures of the un-doped and $\text{C}_6\text{H}_{10}\text{O}_6\text{Fe} + \text{SIC}$ doped samples with C_7H_8 are shown in Fig. 8. The behavior of these SEM images is likely to be related to the grain size. For instance, FWHM of the $\text{C}_6\text{H}_{10}\text{O}_6\text{Fe} + \text{SIC}$ doped samples (both treated and untreated C_7H_8) increased with amount of doping level, indicating that grain size decreases. Many groups have reported that the increase of grain size in MgB_2 is an important factor in the suppression of J_c , since grain boundary pinning is an important mechanism in controlling J_c [4, 30-34]. Note that a decrease in the grain size could also result in peak broadening. However, the grain size seemed to remain the same for all the samples, doped and un-doped. The significant broadening of the peaks in the doped MgB_2 samples can be explained by the lattice distortion originating from the C substitution for B and also the degradation of the crystallinity of the MgB_2 . The normalized temperature (T) dependence of H_{irr} and H_{c2} for all samples is shown in Figures 6 and 7. As we can see the H_{irr} and H_{c2} properties of the doped MgB_2 samples are significantly enhanced for both samples treated and untreated [23] with C_7H_8 . This enhancement in H_{irr} and H_{c2} is a result of C substitution into B sites of the MgB_2 lattice. The higher values for the doped MgB_2 samples indicate that the impurity scattering is stronger due to the C substitution into B sites. The residual resistivity ratio (RRR) values decreased with increasing doping level (Table 1). The decreased RRR for doped sample indicates the increased impurity scattering as a result of C substitution into B site.

Conclusion

In the present study, we have systematically studied the effects of $\text{C}_6\text{H}_{10}\text{O}_6\text{Fe} + \text{SIC}$ doping on microstructures, crystal structure and critical current properties of MgB_2 bulks. Decreases of the a -axis length and T_c due to the C substitution were observed in the $\text{C}_6\text{H}_{10}\text{O}_6\text{Fe} + \text{SIC}$ doped with and without C_7H_8 samples. Substantially enhanced J_c properties under high magnetic fields were observed in the $\text{C}_6\text{H}_{10}\text{O}_6\text{Fe} + \text{SIC}$ doped samples. Medium temperature sintering, low temperature preheating, and C substitution effects are considered to contribute to the high J_c properties of the $\text{C}_6\text{H}_{10}\text{O}_6\text{Fe} + \text{SIC}$ doped samples. On the other hand, the values of the FWHM of the MgB_2 for the $\text{C}_6\text{H}_{10}\text{O}_6\text{Fe} + \text{SIC}$ doped all samples increased with increase of its doping levels, suggesting the introduction of

lattice defects and/or decrease of the crystal size of MgB_2 occurring upon $\text{C}_6\text{H}_{10}\text{O}_6\text{Fe}+\text{SIC}$ doping. The reduced coherence length and mean free path caused by C substitution are considered to strengthen the grain boundary flux pinning in the $\text{C}_6\text{H}_{10}\text{O}_6\text{Fe} + \text{SIC}$ doped samples. Therefore, it is concluded that $\text{C}_6\text{H}_{10}\text{O}_6\text{Fe} + \text{SIC}$ which is under the carbohydrate group is an excellent candidate C source doping for improving the in-field J_c of MgB_2 materials.

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Biography

MR. Syed Quddus is a PhD student. He is an author of this paper. He was born in Pakistan. He have completed Master degree in Physics (Electronics Materials). M.Phil Degree in Electronics (Materials, Lasers, etc), Master degree in Information Technology and a Postgrade Diploma in IT/Education. His interests are Electronics Material Engineering.

Table Caption:

Table 1: Lattice parameters, critical temperature (T_c), residual resistivity ratio (RRR), actual C content and critical current density (J_c) at self-field (20K) and 8T (5K) for the un-doped and $C_6H_{10}O_6Fe + SiC$ doped without (A) and with (B) $C_7H_8 MgB_2$ samples.

Figure Captions:

Fig. 1: The X-ray diffraction patterns for the un-doped and 10 wt%, 20 wt% and 30wt% $C_6H_{10}O_6Fe + SiC$ doped without (A) and with (B) $C_7H_8 MgB_2$ samples.

Fig. 2: The Full-Width at Half Maximum (FWHM) as a function of $C_6H_{10}O_6Fe + SiC$ content for the un-doped and 10 wt%, 20 wt% and 30 wt% $C_4H_6O_6$ doped without (A) and with (B) $C_7H_8 MgB_2$ samples.

Fig. 3: The ac susceptibility vs critical temperature (T_c) patterns for the un-doped and 10 wt%, 20 wt% and 30 wt% $C_6H_{10}O_6Fe + SiC$ doped without (A) and with (B) $C_7H_8 MgB_2$ samples.

Fig. 4: The magnetic critical current density (J_c) for the un-doped and 10 wt% (A), 20 wt% (B) and 30 wt% (C) $C_6H_{10}O_6Fe + SiC$ doped without and with $C_7H_8 MgB_2$ samples.

Fig. 5: Temperature dependence of upper critical field (H_{c2}) and irreversibility field (H_{irr}) for the un-doped and 10 wt% (A), 20 wt% (B) and 30 wt% (C) $C_6H_{10}O_6Fe + SiC$ doped without and with $C_7H_8 MgB_2$ samples.

Fig. 6: SEM images for the un-doped (A), 10 wt% $C_6H_{10}O_6Fe + SiC$ doped without C_7H_8 (B), 30 wt% $C_6H_{10}O_6Fe + SiC$ doped without C_7H_8 (C), 10 wt% $C_6H_{10}O_6Fe + SiC$ doped with C_7H_8 (D) and 30 wt% $C_6H_{10}O_6Fe + SiC$ doped with C_7H_8 (E) MgB_2 samples.

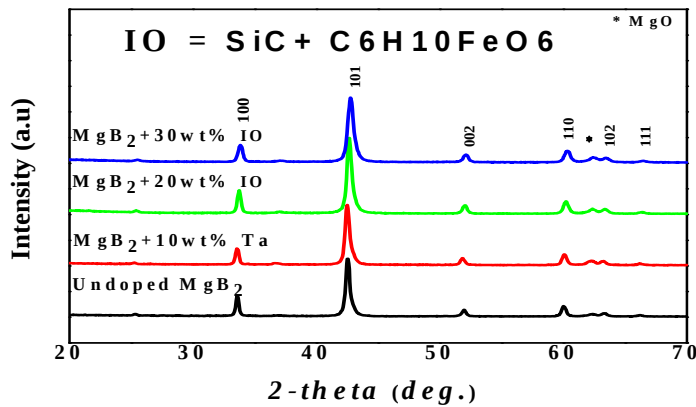


Fig. 1

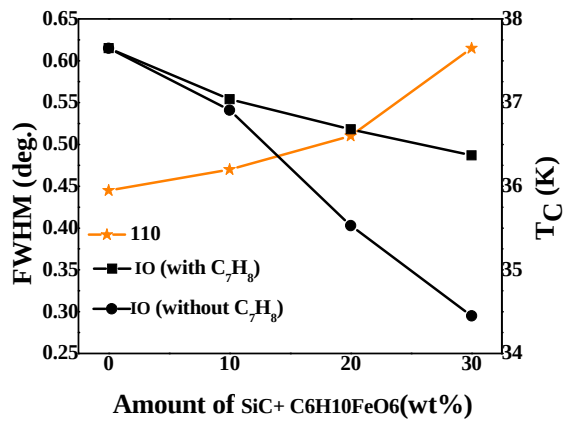


Fig. 2

Fig. 3

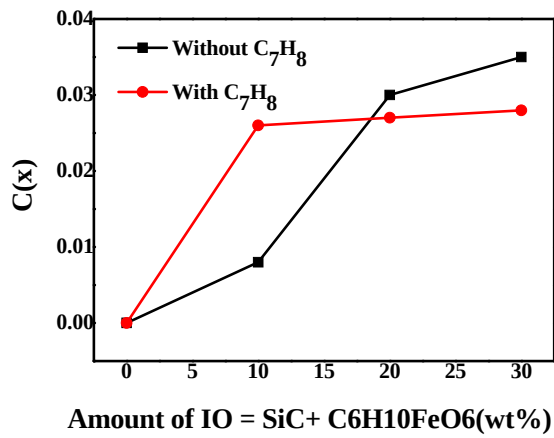


Fig. 4

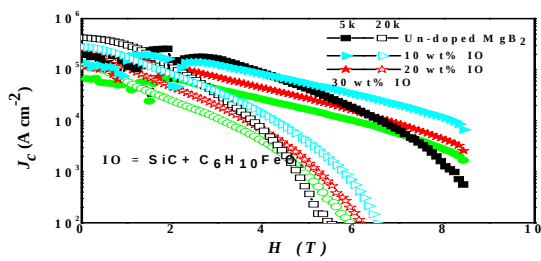


Fig. 5

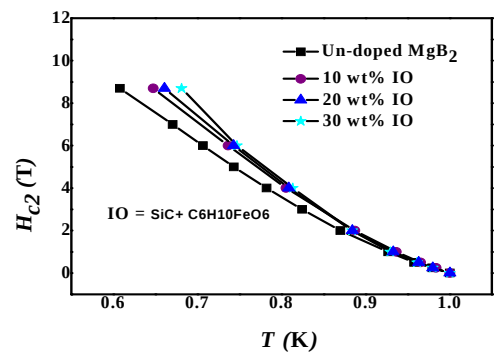


Fig. 6

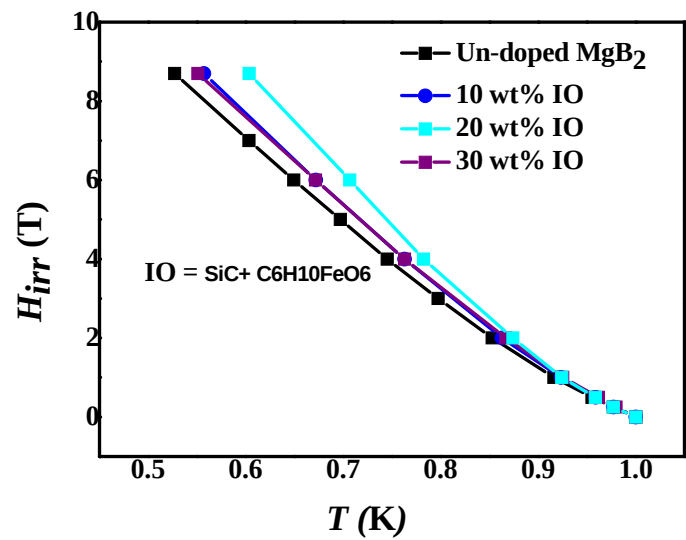


Fig. 7

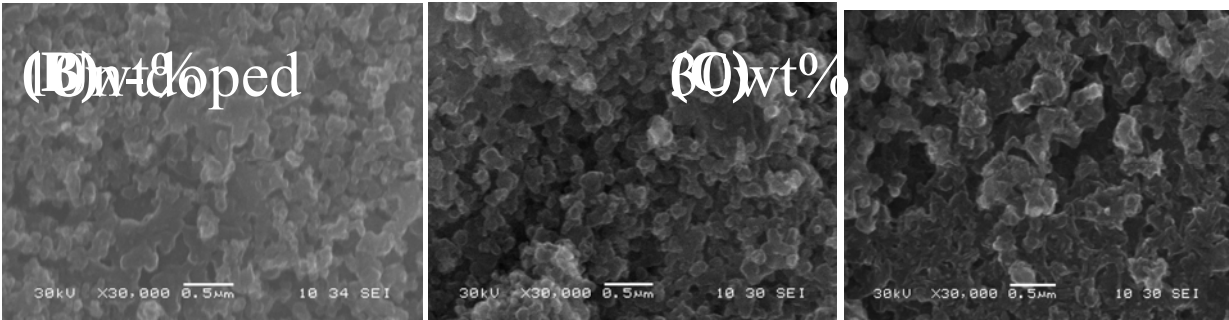


Fig. 8

Table: 1

C ₄ H ₆ O ₆ (wt%)	Lattice parameter		Actual C (x)*	T_c (K)	RRR	J_c (Acm ⁻²)	
	a (Å)	c (Å)				Self-field (20K)	8 T (5K)
0	3.0836(03)	3.5223(4)		37.75	2.15	4.5×10^5	0.15×10^4
10	3.0781(33)	3.5255(40)	0.0265	37.03	1.79	2.90×10^5	1.17×10^4
20	3.0779(31)	3.524(46)	0.0273	36.69	1.98	2.07×10^5	0.48×10^4
30	3.0777(29)	3.5243(48)	0.0282	36.38	1.96	1.36×10^5	0.29×10^4