Effects of neutron irradiation on carbon doped MgB$_2$ wire segments

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Abstract

We have studied the evolution of superconducting and normal state properties of neutron irradiated Mg(B$_{0.962}$C$_{0.038}$)$_2$ wire segments as a function of post-exposure annealing time and temperature. The initial fluence fully suppressed superconductivity and resulted in an anisotropic expansion of the unit cell. Superconductivity was restored by post-exposure annealing. The upper critical field, $H_{c2}(T = 0)$, approximately scales with $T_c$, starting with an undamaged $T_c$ near 37 K and $H_{c2}(T = 0)$ near 32 T. Up to an annealing temperature of 400$^\circ$C there recovery of $T_c$ tends to coincide with a decrease in the normal state resistivity and a systematic recovery of the lattice parameters. Above 400$^\circ$C a decrease in ordering along the c-direction coincides with an increase in resistivity, but no apparent change in the evolution of $T_c$ and $H_{c2}$. To a first order approximation, it appears that carbon doping and neutron damage affect the superconducting properties of MgB$_2$ independently.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Neutron irradiation and carbon doping of MgB$_2$ are two methods by which defects can systematically be introduced. Carbon enters the structure, replacing boron [1, 2], and electron dopes the system, which results in a suppression of $T_c$ [3]. Carbon presumably acts as a point defect, enhancing scattering primarily within the $\pi$ band [3], which leads to an enhancement of $H_{c2}$ at low doping levels [4] in accordance with theoretical calculations [5]. Up to a doping level of 10%, two-gap superconductivity is preserved [6] and evidence suggests that it may persist down to $T_c = 0$ [7].

Due to the high neutron capture cross section of $^{10}$B, neutron irradiation studies have varied as researchers have employed different techniques to ensure uniform damage throughout samples. The superconducting properties differ depending upon the irradiation conditions. Enhancements in $H_{c2}$ have been observed for neutron irradiation on isotopically enriched Mg$_{11}$B$_2$ for fluences of order $10^{17}$ cm$^{-2}$ [8], and for irradiation of MgB$_2$ containing natural boron with a fast neutron fluence of $10^{19}$ cm$^{-2}$ [9]. Defects introduced by neutron irradiation, regardless of irradiation conditions, are effective at pinning vortices, and samples which exhibit a $T_c$ above approximately 30 K show enhanced $J_c(H)$ values in the vicinity of 20 K [8, 10, 11].

We have previously reported on neutron irradiation of MgB$_2$ filaments [12]. Although the filaments contained natural boron and MgB$_2$ containing natural boron has a slow neutron half depth of 130 $\mu$m, they were 140 $\mu$m in diameter and, given that an isotropic neutron flux was used, the damage is believed to be essentially uniform throughout the samples. Irradiating with a fluence level of $4.75 \times 10^{15}$ cm$^{-2}$ resulted in a suppression of $T_c$ to below 5 K. Superconductivity was restored...
by performing post-exposure anneals. Upper critical field values were found to approximately scale with \( T_c \). It is believed that the suppression of \( T_c \) is a result of both an increase in interband scattering and a decrease in the electron density of states at the Fermi surface. The temperature dependence of the upper critical field exhibits Werthamer, Helfand, and Hohenberg (WHH-) [13] like behaviour, which suggests the bands become fully mixed, only when \( T_c \) is near or below 10 K. The field dependence of the critical current density was found to depend largely upon the superconducting transition temperature. Generally speaking, \( J_c (H, T) \) increases for samples with higher \( T_c \) values. For example, the field at which filaments could carry in excess of 10⁸ A cm⁻² was extended from near 1 T in an undamaged wire to above 1.5 T in the case of a sample whose annealing profile resulted in a \( T_c \) near 37 K.

Given that carbon doping appears to weakly decrease \( T_c \) and increase \( H_{c2} \) by preferentially increasing scattering within the \( \pi \) band, whereas neutron damage appears to decrease \( T_c \) and \( H_{c2} \) by increasing the inter-band scattering, the focus of this paper is to examine the effects of combining these two scattering mechanisms by inducing and removing the effects of neutron damage in carbon doped MgB₂ samples. This will allow us to add and subtract inter-band scattering in samples with already enhanced intra-\( \pi \)-band scattering.

2. Experimental methods

Carbon doped Mg(B\(_{0.96}C_{0.038}\))₂ was prepared in a two step reaction process as described in detail elsewhere [14]. Carbon doped boron filaments, produced by Specialty Materials, Inc., were exposed to excess Mg vapour while the temperature was ramped from 650 to 1200 °C over 96 h. Three filaments, each approximately 5 mm in length, were sealed in quartz ampoules under a He atmosphere and irradiated with an isotropic fluence of 7.13 × 10¹⁹ cm⁻² reactor neutrons at the Missouri University Research Reactor (MURR), as described in [12]. The Mg(B\(_{0.96}C_{0.038}\))₂ filaments had a diameter of 110 µm, indicating the isotropic irradiation should result in essentially uniform damage. The boron filaments used in this study contain a tungsten boride core. Fast neutrons colliding with \(^{182}\)W atoms, which have a natural abundance of 26.3%, can be absorbed into the nucleus, causing the emission of a proton and transforming the tungsten into \(^{182}\)Ta. \(^{182}\)Ta decays back to \(^{182}\)W, with a half life of 181 days. As a result the filaments were mildly radioactive and required appropriate safety measures in handling.

Normal state and superconducting properties were determined for a series of post-exposure annealing profiles. One set consisted of 24 h anneals at temperatures up to 600 °C. In the second set the annealing temperature was held constant at 500 °C while the annealing time was varied from 1 to 1000 h. In all cases the anneals were performed by placing samples, still sealed with their quartz ampoules, into a Lindberg model 55035 Mini-Mite tube furnace that was preheated to the desired annealing temperature. After the samples were annealed for the desired length of time, the ampoules were removed from the furnace and air quenched to room temperature.

Powder x-ray diffraction (XRD) measurements were made at room temperature using Cu Ka radiation in a Rigaku Miniflex Diffractometer. Measurements were performed on six filaments from two ampoules. Peak positions were determined by fitting each peak with a pseudo-Voigt function using Jade analysis software. A silicon standard was used to calibrate each pattern. The experimentally determined Si peak positions were found to be offset from their known values by a constant amount. Within each spectrum, the peaks varied about some constant offset and this variation was used to estimate experimental uncertainty in the lattice parameters. Lattice parameters were determined from the position of the (002) and (110) peaks. DC magnetization measurements and magnetization hysteresis loops were done in a Quantum Design MPMS-5 SQUID magnetometer. For magnetization measurements, individual wires, which have mass of approximately 0.15 mg, were oriented along the direction of the applied field. Transport measurements were made using a standard four probe AC technique, with platinum wires attached to the samples with Epotek H20E silver epoxy. Typical samples had voltage contacts that were 2–3 mm apart. Resistivity versus temperature in applied fields up to 14 T was measured in a Quantum Design PPMS-14 system and resistivity versus field was measured up to 32.5 T using a lock-in amplifier technique at the National High Magnetic Field Laboratory in Tallahassee, FL.

3. Structural properties

Figure 1 presents the (002) and (110) x-ray peaks for the series of neutron irradiated Mg(B\(_{0.96}C_{0.038}\))₂ samples annealed for 24 h at various temperatures.
observed in pure MgB$_2$ samples even when they are exposed to a higher fluence level.

As the annealing temperature is increased up to a temperature of 400°C we see a systematic shift of both the (002) and (110) peaks to higher 2θ, indicating a contraction of both the $a$- and $c$-lattice parameters. At 400°C the $a$-lattice parameter appears to reach a minimum, measuring 3.0769(11) Å, which is 0.0020(13) Å or 0.065% larger than the undamaged sample. The $c$-lattice parameter decreases monotonically as a function of temperature up to 400°C, where it has a value of 3.5466(12) Å, which is 0.0314(14) Å or 0.89% larger than the undamaged value. For anneals at 450°C and above, a qualitative change in the evolution of the x-ray peaks occurs. The (002) peaks begin to broaden substantially (figure 2). For the 450 and 500°C anneals, the stable peak refinements for the (002) peak were unattainable, preventing us from attaining estimates of the $c$-lattice parameter. The corresponding (110) peaks shift to lower 2θ, indicating that the $a$-lattice parameter may be expanding slightly. After annealing at 600°C for 24 h, the (002) peak appears to bifurcate, and indexing as two different peaks yields one $\Delta c$ value which is comparable to that attained for an annealing at 400°C and another which is considerably lower (figure 3). The (110) peak position, and hence calculated $\Delta a$, is comparable to that of the 400°C anneal. The full set of calculated changes in lattice parameters relative to the undamaged samples for the series of 24 h anneals is plotted in figure 3. Included in figure 3 is the evolution of the relative change in the lattice parameters as a function of annealing temperature for a pure MgB$_2$ sample exposed to a fluence of $4.75 \times 10^{18}$ cm$^{-2}$ from [12].

The (002) and (110) x-ray peaks for a series of samples annealed at 500°C for times up to 1000 h are shown in figure 4(a). The (002) peaks continue to broaden as the annealing time at 500°C is increased. Stable peak refinements for the (002) peaks were unattainable due to their distorted shape. The (110) peaks monotonically shift to higher 2θ as a function of annealing time up to 96 h, at which point the $a$-lattice parameter is within experimental error of the undamaged value (figure 4(b)). While the $a$-lattice parameter is larger for the case of the 24 h anneal at 500°C relative to the 24 h anneal at 400°C, increasing the annealing time to 96 h at 500°C results in a continued contraction of $a$. Extending the annealing time an additional order of magnitude results in a negligible change in $a$. It appears that continued annealing at 500°C causes the structure to become more disordered in the $c$-direction while the $a$-lattice parameter is returned to near the undamaged value.
4. Thermodynamic and transport measurements

Magnetization and transport measurements were performed to determine the evolution of $T_c$, $H_{c2}$, and normal state resistivity as a function of annealing time and temperature. $T_c$ was determined using an onset criterion in resistivity measurements and a 1% screening criterion in magnetization. Figure 5(a) presents normalized magnetization curves for the entire set of one day annealed samples. As the annealing temperature is increased, the transition temperature monotonically approaches the undamaged value of 36.8 K. It is worth noting that all of the $M(T)$ curves show sharp transitions.

Resistivity versus temperature data are plotted in figure 5(b). Normal state resistivity values decrease monotonically as a function of annealing up until $T_{\text{anneal}} = 450^\circ$C, at which point $\rho_0$ increases approximately by a factor of four. The exact cause of this jump in $\rho_0$ is unknown, but it coincides with the broadening of the (002) x-ray peak. Subsequent increases in the annealing temperature result in a decrease in $\rho_0$ relative to the $T_{\text{anneal}} = 450^\circ$C sample. It has to be mentioned that residual resistivities of samples annealed at 200, 450, 500, and 600 $^\circ$C are monotonic as a function of the annealing temperature, raising the question as to whether it is the resistivities of the 300 and 400 $^\circ$C annealed samples that are in fact anomalous. Multiple measurements of samples annealed at 600 $^\circ$C for 24 h are included in figure 5(b) and illustrate the spread in the data. It should be noted that all three of these filaments are from the same quartz ampoule. All three samples show a decrease in resistivity relative to that of the 500 $^\circ$C annealed sample, but are still above the minimum attained by annealing at 400 $^\circ$C.

A plot of the normalized, $\rho/\rho_0$ (300 K), resistivity shows that there are two interesting features in the evolution of the normal state resistivity as a function of annealing temperature (figure 5(c)). For the samples annealed at 300 and 400 $^\circ$C, the temperature dependence, $\rho(T)$, has an odd hump in the 100–150 K range. Such a hump has not been observed in pure MgB2 wires [15] but has been reported for neutron damaged pure MgB2 annealed up to a temperature of 400 $^\circ$C for 24 h [12]. The trends seen in the calculated normal state resistivity values are also seen in normalized resistivity (figure 5(c)), indicating the apparent increase in resistivity occurring at 450 $^\circ$C is a real effect and not the result of some type of geometrical effect, such as cracking. It should be noted that whereas the transport measurements show changes in the evolution of both $\rho_0$ and $\rho(T)$ as a function of annealing temperature for samples annealed from 300 to 450 $^\circ$C the magnetization data showed smooth, sharp transitions with monotonic increases in $T_c$ throughout this range of annealing temperatures.

Magnetization and transport curves for the series of samples annealed at 500 $^\circ$C for various times are plotted in figure 6. Multiple measurements of samples annealed for 1 and 24 h were performed. The 1 h anneal transport data consists of sets of two wires from two different ampoules. One set was then used for magnetization measurements and is included along with a filament from a third ampoule. The 24 h data consist of two wires from a single ampoule and an additional filament from a second ampoule. The normalized magnetization transitions for the 1 h anneals all fall below those of the 24 h anneals (figure 6(a)). However, for the transport data, the range of $T_c$ values is broader and several of the transitions occur at temperatures greater than is seen for the 24 h anneals (figure 6(b)). It should be noted that for the samples annealed at 500 $^\circ$C for 1 h for which both magnetization and transport measurements were performed, in both cases the transport data showed a $T_c$ that was approximately 0.5–1 K higher than was observed in the magnetization measurements. Such a spread was also observed in the case of neutron irradiation on pure MgB2 [12], suggesting the spread in the data is a reflection of sample to sample variation.

Figure 5. Normalized magnetization (a) and zero field resistivity curves (b) for samples annealed at various temperatures for 24 h. (c) Normalized resistivity curves for neutron irradiated Mg(B$_{0.96}$C$_{0.038}$)$_2$ samples annealed for 24 h at various temperatures.

Figure 6. Magnetization and transport curves for the series of samples annealed at 500 $^\circ$C for various times.
The multiple transport measurements on samples annealed at 500 °C for 1 h also exhibit considerable spread in the normal state resistivity values, and no systematic evolution of $\rho_0$ as a function of annealing time is observed. Residual resistivity ratios of these samples indicate that the observed spread in resistivity values represents true sample to sample variation in $\rho_0$ and is not the result of pathologic geometric problems such as cracks.

In the case of samples annealed at 500 °C for various times, generally speaking, extending the annealing time tends to decrease $\Delta T_c$, although there is considerable spread in the data (figure 7). For neutron irradiated pure MgB$_2$ wires, $\Delta T_c$, which was taken as a measure of the defect concentration, was found to decrease exponentially with time [12], indicating defects are being annealed out by a single activated process with random diffusion [16]. For such samples, the defect concentration, $n$, obeys the relation

$$n = n_0e^{\lambda t}$$  

where $n_0$ is the initial defect concentration, $\lambda$ is a rate constant, and $t$ is the time. $\lambda$ is a function of the activation energy, $E_a$, and diffusion coefficients. For samples which do show an exponential behaviour in the decrease of $\Delta T_c$ (and by presumption the defect density) as a function of annealing time, the activation energy can be determined by the so-called cross-cut procedure [16]. This involves comparing the annealing time for which different temperature anneals reached the same

$$\ln \frac{t_1}{t_2} = \frac{E}{k} \left( \frac{1}{T_1} - \frac{1}{T_2} \right).$$  

Unfortunately, we did not have a sufficient number of samples to perform studies of the temperature dependence of $\Delta T_c$ as a function of annealing time at any temperatures other than 500 °C. However, if we assume that the primary difference between neutron irradiation of pure and carbon doped MgB$_2$ is in the activation energies, then we can gain some insight by comparing $\lambda$ values attained from the two sets of samples. For the case of the carbon doped samples, since the spread in $T_c$ for a given annealing time is so large, we can obtain only a rough estimate, based on a fitting of the midpoint of the $T_c$ values. Such a calculation yields a rate constant of $\lambda = 1.61$ s$^{-1}$. For irradiation on pure MgB$_2$, anneals at 200, 300, and 400 °C yielded rate constants of 2.77, 2.21, and 3.51 s$^{-1}$ respectively [12]. Although there is considerable spread in the rate constants for the pure MgB$_2$ case, and considerable uncertainty in the rate constant associated with the carbon doped MgB$_2$ samples (estimates of $\lambda$ from raw data range from 1.00 to 2.03 s$^{-1}$) analysis of equation (2) shows that since carbon doped samples have a lower rate constant than pure MgB$_2$ samples the activation energy is higher for carbon doped samples. It should be noted that higher reaction temperatures were required to synthesize carbon doped MgB$_2$ than pure MgB$_2$ [14], suggesting that there are different energy scales associated with carbon doping.

Upper critical field values were determined using the onset criteria in both resistivity versus temperature in applied fields up to 14 T and resistivity versus field in field sweeps up to 32.5 T (figure 8). The complete $H_{c2}(T)$ curves for the entire set of annealing profiles are given in figure 9. $H_{c2}(T = 0)$ approximately scales with $T_c$. Such behaviour was also observed for neutron irradiation and post-exposure anneals on pure MgB$_2$ filaments [12].

Critical current densities were estimated from magnetization hysteresis loops using the Bean critical state model [17].
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Figure 8. Resistivity versus temperature (a) and resistivity versus field (b) for a sample annealed at 500 °C for 1000 h.

Figure 9. Upper critical field curves for samples annealed for 24 hours at various temperatures and samples annealed for various times at 500 °C. Multiple data sets exist for the 450 °C/24 h and 500 °C/96 h anneals. \(H_{c2}(T = 0)\) approximately scales with \(T_c\).

Figure 10. Critical current densities at 5 K inferred from magnetization hysteresis loops. (a) Samples annealed at various temperatures for 24 h. (b) Samples annealed at 500 °C for various times.

for cylindrical geometry. Figure 10(a) shows the field dependence of \(J_c\) at \(T = 5\) K for the entire set of 24 h anneals at various annealing temperatures. For the set of one day anneals, the in-field performance improves with annealing temperature, up to 500 °C. Presumably the increased transition temperature and hence decreased reduced temperature \((T/T_c)\) of the measurement is the primary cause of the enhancement. However, there exists a much greater increase in in-field \(J_c\) values between the sample annealed at 400 °C and the sample annealed at 450 °C than between the sample annealed at 300 °C and the sample annealed at 400 °C. Whereas increasing the annealing temperature from 300 to 400 °C results in a near 7 K or 36% increase in \(T_c\) but only a factor of two improvement in \(J_c\) at all fields, increasing the annealing temperature from 400 to 450 °C results in only an additional 5 K or 17% increase in \(T_c\), but nearly an order of magnitude increase in in-field \(J_c\) values. This suggests the increase in disorder between the hexagonal planes, which coincided with an increase in resistivity, may also play a role in enhancing the flux pinning. The sample annealed at 600 °C shows slightly reduced \(J_c(H)\) values relative to the 500 °C anneal. Thus, at 600 °C, either the increased order along \(c\) is diminishing \(J_c\), or we are beginning to anneal out some of the defects which are effective at pinning vortices, or some combination thereof.

Extending the annealing time from 1 to 96 h at 500 °C results in relatively minor increases in in-field \(J_c\) values (figure 10(b)). The data for the 500 °C and 96 h anneal are particularly noisy and may not truly be enhanced relative to the 500 °C/103 h anneal. All of these 500 °C anneals show \(J_c\) remaining fairly constant over the field range of 1–4 T.

At 5 K, the best in-field performance, which results from the 96 and 1000 h anneals at 500 °C, yields a \(J_c\) value which
is maintained at $10^8$ A cm$^{-2}$ in an applied field of 4 T. In comparison, the best in-field performance at 5 K for a sample of neutron irradiated pure MgB$_2$ dropped below $10^8$ A cm$^{-2}$ in an applied field of approximately 1.5 T [12] and an undamaged carbon doped filament dropped below this level near 1 T [14].

5. Discussion

Up to an annealing temperature of 400 °C, the behaviour of irradiated carbon doped samples is quite similar to that of irradiation of pure MgB$_2$ filaments. As-damaged samples have expanded unit cells and suppressed superconducting transition temperatures. Post-exposure annealing tends to return the lattice parameters and $T_c$ towards the undamaged values. In both cases, the upper critical field values, $H_{c2}(T = 0)$, tend to scale with $T_c$. The intriguing aspect of neutron damage in the carbon doped samples is the temperature induced decrease in structural order in the c-direction which occurs near $T_{anneal} = 450$ °C. No such effect was seen in the case of neutron irradiation in pure MgB$_2$ filaments. The feature does not appear to be a phase segregation as no double transition is seen in magnetization. Furthermore, samples annealed at 500 °C showed sharp, single transitions in both magnetization and resistivity, while having broad (002) peaks. The sample annealed for 24 h at 600 °C showed evidence of having two distinct phases with different c-lattice parameters but the same a-lattice parameter. Here again, both the magnetization and transport data showed only single transitions. Thus the transition temperature appears to be insensitive to the c-lattice parameter. The a-lattice parameter is presumably not the sole determining factor for $T_c$, as samples annealed for 24 h at 450 and 500 °C showed increases in $\Delta a$ relative to the sample annealed for 24 h at 400 °C, while also having increased $T_c$ values. It should be noted that although measurements on single filaments showed sharp, single transitions, there was significant spread in the $T_c$ values between different samples.

The broadening of the (002) peak coincides with a near fourfold increase in the normal state resistivity and an approximate order of magnitude increase in in-field $J_c$ values. Increases in normal state resistivity for annealing temperatures above 400 °C have been observed in neutron irradiation of pure MgB$_2$ [18]. In this case, the authors report a continued increase in $\rho_0$ as a function of annealing temperature above 400 °C and attribute it to a loss of intergrain connectivity. In contrast, our data show a decrease in $\rho_0$ for 24 h anneals at 500 and 600 °C, which supports the notion that a different mechanism is responsible for the anomalous behaviour in the normal state resistivity of neutron irradiated carbon doped samples.

Despite the decrease in structural order along the c-axis and large increase in resistivity at $T_{anneal} = 450$ °C, no qualitative changes to the evolution of $T_c$ and $H_{c2}$ as a function of annealing temperature were observed. The transition temperature tends to approach that of the undamaged sample and $H_{c2}$ continues to roughly scale with $T_c$. Even the 500 °C, 1000 h anneal shows a suppressed $H_{c2}(T = 0)$ value relative to the undamaged sample. This is in contrast to the results obtained with irradiation of pure MgB$_2$ filaments [12]. For fluence levels of $4.75 \times 10^{18}$ and $9.50 \times 10^{18}$ cm$^{-2}$, two values which bracket the $7.13 \times 10^{18}$ cm$^{-2}$ fluence level used in this study, $H_{c2}(T = 0)$ was enhanced by approximately 2 and 3 T respectively.

Both pure and carbon doped samples show increases in normal state resistivity after annealing (figure 11). Pure MgB$_2$ wires irradiated with a fluence of $4.75 \times 10^{18}$ cm$^{-2}$ and subsequently annealed for 24 h at various temperatures showed an increase in $\rho_0$ at an annealing temperature of 200 °C. In contrast, for Mg(B$_{0.96}$C$_{0.04}$)$_2$ filaments exposed to a fluence of $7.13 \times 10^{18}$ cm$^{-2}$ and subsequently annealed for 24 h at various temperatures, the apparent anomalous increase occurs for an annealing temperature of 450 °C. For both sets of samples, following the abrupt increase, the normal state resistivity decreases monotonically as a function of annealing temperature. It should be noted however, that, in the case of the carbon doped samples, it is not clear whether or not there exists some correlation between the change in the temperature dependence of the normal state resistivity for the 300 and 400 °C anneals and the reduced $\rho_0$ values for these anneals. That is, rather than the jump in $\rho_0$ at 450 °C representing an anomalous increase, the $\rho_0$ values associated with the samples annealed at 300 and 400 °C may in fact be anomalously low.

These data, coupled with the analysis of the rate constants, suggest that in the case of neutron irradiated carbon doped MgB$_2$ samples, there is a different, and higher, energy scale associated with the annealing of defects. This is perhaps not altogether surprising, as it was shown that higher reaction temperatures were necessary to form the carbon doped phase. Whereas pure boron filaments can be fully converted to MgB$_2$ in as little as 2 h at 950 °C [19], isothermal reactions required 48 h at 1200 °C to convert carbon doped boron fibres to Mg(B$_{1-x}$C$_x$)$_2$, even for doping levels as low as $x = 0.004$ [14]. Although diffusion of Mg vapour into a carbon doped boron matrix and repair of neutron induced damage are indeed two, rather different, phenomena, this correlation is at least worth noting.

The temperature dependence of the upper critical field shows positive curvature near $T_c$ for samples with a $T_c$ near 26 K and above (figure 9). For the samples annealed for 24 h at 200 and 300 °C $H_{c2}$ approaches $T_c$ linearly. If the two bands are fully mixed, the temperature dependence of the upper critical field should follow WHH [13] behaviour, where $H_{c2}(T = 0)$
is given by

\[ H_{c2}(T = 0) = 0.69 T_c \frac{dH_{c2}}{dT} \]  \hspace{1cm} (3)

Calculating \( H_{c2}(T = 0) \) using equation (3) for the samples annealed for 24 h at 200, 300, and 400°C yields values of 2.4, 5.2, and 6.5 T. Experimentally, the lowest temperature \( H_{c2} \) values we could reliably attain from transport measurements for each of these samples were 2.1, 6.5, and 10 T, occurring at temperatures of 4.2, 5.2, and 4.6 K respectively. Only for the sample annealed at 200°C does the WHH fit yield an \( H_{c2}(T = 0) \) value which is consistent with the experimentally observed data. Thus it is likely that only for samples with a \( T_c \) below 10 K are the bands fully mixed. Such a result was also obtained in the case of the neutron irradiation on pure MgB\(_2\) [12]. This implies that the inter-band scattering rates resulting from defects associated with the neutron irradiation are comparable in both pure and carbon doped samples, suggesting that the scattering associated with the two different sources of defects acts independently. This is not altogether surprising as the suppression in \( T_c \) for carbon doped samples is believed to result from changes in the Fermi surface, rather than increases in inter-band scattering, and scattering associated with carbon doping is believed to be primarily within the \( \pi \) band [3]. Thus if inter-band scattering is introduced primarily through defects resulting from neutron damage, if pure and carbon doped samples have similar initial \( T_c \) values, then they should also exhibit fully mixed bands at similar \( T_c \)s, which was experimentally observed. A similar result was found in the case of neutron irradiation on pure MgB\(_2\) filaments, implying that the scattering associated with carbon doping and neutron irradiation act independently.

6. Conclusions

We have studied superconducting and normal state properties of neutron irradiated carbon doped MgB\(_2\) filaments as a function of post-exposure annealing time and temperature. In spite of anomalous behaviour in the evolution of the c-lattice parameter and normal state resistivity, \( T_c \) tended to return towards the undamaged value with increased annealing time and temperature. Upper critical field values were found to approximately scale with \( T_c \) and exhibited WHH-like behaviour, suggesting a complete mixing of the two bands, for samples with \( T_c \) near 10 K. Neutron irradiation of pure MgB\(_2\) also led to a complete mixing of the bands near 10 K, suggesting that the scattering associated with carbon doping and neutron irradiation act independently.

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