Effect of fabrication route on density and connectivity of MgB$_2$ filaments

J Viljamaa, P Kováč, I Hušek, T Melišek, V Štrbík and E Dobročka

Institute of Electrical Engineering, Slovak Academy of Sciences, Dúbravská cesta 9, 841 04 Bratislava, Slovakia

E-mail: jonna.viljamaa@gmail.com

Abstract. Series of samples was manufactured to test the effect of fabrication route on the density of the polycrystalline core, the connectivity between MgB$_2$ grains and also the critical current density of undoped MgB$_2$ conductors. All the samples had titanium sheaths and were manufactured using the Powder-in-Tube method. The Ti tubes were filled with either in situ, ex situ, or 60%–40% mixture of in situ and ex situ powders. After powder packing, the samples went through different deformation steps such as rotary swaging, two-axial rolling, pressing, cold isostatic pressing, or some combination of these, to form monofilamentary tapes. The core density was qualitatively estimated by measuring the Vickers microhardness from several locations of the ceramic filament. The changes in connectivity, or effective cross-section, were evaluated by performing resistivity measurements on the MgB$_2$ cores after removal of the metallic sheath. The $J_c$ characteristics were measured at 4.2 K and at variable external magnetic flux densities.

1. Introduction

Research within the superconducting properties of magnesium diboride (MgB$_2$) has been intensive since its discovery in 2001 [1]. Although the interest has also moved onto the application perspectives because of the very promising superconducting properties of MgB$_2$, there is still much work to be done concerning the study and enhancement of the basic material properties, such as the connectivity of the grains and the density of the filament.

Numerous attempts to improve the critical current density ($J_c$) under an applied magnetic field ($B$) have been made in both in situ and ex situ wires prepared by the standard Powder-in-Tube (PIT) method [2]–[7]. In the in situ method, a mixture of Mg and B powders is used, whereas in the ex situ method the MgB$_2$ powder has already been prepared externally. The main difference between these two methods is that the final heat treatment (HT) is not necessary for the ex situ method. In the ex situ route, the purpose of the HT is to increase the area of connections between the powder grains by sintering them together. For this reason, the HT temperature is usually high, 950 °C [3, 5], while for the in situ route much lower temperatures of 640–750 °C can be used to form the MgB$_2$ phase [6, 7].

The volume occupied by the MgB$_2$ phase is smaller than that of the unreacted Mg + B mixture and therefore the in situ wires are accompanied by porous cores, typically reaching only 50% [9] of the theoretical density of 2.62 g/cm$^3$ [8]. On the other hand, for the ex situ samples, the ‘closest packing of spheres’ model indicates that the highest density that can be obtained is 74% [9]. However, the final density of the filaments may be affected, for example, by applying pressure. Therefore, after powder
packing, our samples were subjected to different deformation steps such as rotary swaging, two-axial rolling, pressing or cold isostatic pressing to test the effect of these augmentations on the core density.

On the other hand, the surfaces of MgB$_2$ grains are coated with insulating layers composed most importantly of oxides such as MgO and amorphous BO$_x$. Both the pores and these impurity layers reduce the current path and decrease the effective conductivity of the filament as a whole when compared to the conductivity of an individual grain. The combination of the two aforementioned phenomena, the current percolation caused by the voids and the impurity phases between the MgB$_2$ grains are the probable reason for the observed poor $J_c$ values in all the current MgB$_2$ wires and tapes [9, 10].

To improve the $J_c$, it is necessary to increase the connectivity of the MgB$_2$ filaments. This needs to be combined with the highest possible density of the core which for the PIT method can theoretically be achieved by using the ex situ fabrication route. On the other hand, the substitution of B by the most common addition for MgB$_2$, carbon, to achieve improved $J_c(B)$ performance, especially for high values of $B$, is more efficient by using the in situ route [11, 12]. To realize the optimal combined benefits of these, it has been suggested to use a mixture of in and ex situ powders with a composition of 60%–40%, respectively [13]. Thus, the purpose of this study is to determine how the core density and conductivity using the method suggested by Rowell [14] are affected by different deformation methods or the use of mixed in and ex situ powders.

2. Experiment

Titanium tubes of 5.8/4.1 mm in outer/inner diameter were filled with three types of powder: in situ, ex situ and 60%–40% mixture of these two, respectively, in wt%. The different precursor powder compositions are denoted as I, E and M, respectively. The Ti sheath is used because of its claimed inert properties and chemical stability in terms of reaction and diffusion at the interface [15]. As received MgB$_2$ (98% purity) powder was purchased from the company Alfa Aesar and used for the ex situ tapes. This powder has very wide grain size distribution ranging from 0.1 up to 100 µm and a minority phase of MgO up to 5% [16]. For the fabrication of the in situ powder, Mg (99% purity, ~20 µm particle size) and B (99%, ~1 µm) powders in stoichiometric ratio 1:2 were milled together for 40 minutes in a planetary ball mill in Ar gas. To prepare the mixed powder, 60% of the in situ and 40% of the ex situ powders were mixed and further ball milled for 20 minutes.

The three filled Ti tubes were deformed by rotary swaging (RS) and two-axial rolling (TAR) into monofilamentary tapes with a rectangular cross-section of 0.815 x 2.53 mm$^2$ (sample 0). A portion of these tapes were further deformed either by cold isostatic pressing (CIP, denoted as C) at 1.5 GPa (sample 1), or by uniaxial pressing (P) with a hydraulic press with the pressing applied perpendicular to the surface of the tape at 1.3 GPa (2) which is the maximum applicable pressure for specimens the size of our samples, and at 0.65 GPa (3). All of the samples from 0 to 3 were heat treated under typical conditions, the I and M samples at 650 °C, and the E samples at 950 °C, all for 30 minutes under an Ar atmosphere. Pieces of the samples that only went through the RS + TAR deformation were also heat treated at two different temperatures, specifically 750 °C (sample 4, only I and M) and 850 °C (5). See table 1 for gathered information about the differences in the fabrication processes of all the samples.

Figure 1 shows an example of a cross-section of an as-deformed MgB$_2$ RS + TAR tape prepared using the mixed precursor powder. The core structures and core areas were studied with the polarized light optical microscope Olympus BX51M.

Transport critical current ($I_c$) values were derived in the external magnetic field which range from 2.5 to 8.5 T with steps of 0.5 T at 4.2 K by using the 1 µV/cm criteria as the critical electric field criterion. The measurements were performed with current flow both parallel and perpendicular to the applied field. The $n$-values were then estimated from the slopes of these $I$-$V$ curves for every value of applied $B$. To acquire the $J_c$, the $I_c$ values were divided by the filament cross-section area. In addition, the $J_c$ anisotropy, $\Gamma \equiv J_{c,par}/J_{c,perp}$, was also determined from $I_c$ measurements in parallel and perpendicular fields. Refer to table 1 for these quantities measured at 7 T and 4.2 K together with the acquired microhardness (HV0.05) data.
Table 1. The fabrication routes with HV and $J_c(4.2\text{ K}, 7\text{ T})$ characteristics for MgB$_2$/Ti tapes.

| Sample | Deformation | $p$ [MPa] | HT [°C][min] | HV$_{ave}$ Before HT | HV$_{ave}$ After HT | $J_{c,par}$ [A/cm$^2$] | $\Gamma$ [-] | $n$-value [-] |
|--------|-------------|-----------|--------------|----------------------|---------------------|---------------------|----------|-------------|
| I-0    | RS+TAR     | –         | 650/30       | 98                   | 5533                | –                   | 14.6     |             |
| M-0    | RS+TAR     | –         | 650/30       | 168                  | 5496                | 1.59                | 18.6     |             |
| I-1    | RS+TAR+C   | 1500      | 650/30       | 140                  | 6030                | 1.56                | 24.3     |             |
| M-1    | RS+TAR+C   | 1500      | 650/30       | 167                  | 5817                | 1.67                | 23.4     |             |
| E-1    | RS+TAR+C   | 1500      | 950/30       | –                    | 84                  | –                   | 4.1      |             |
| I-2    | RS+TAR+P   | 1300      | 650/30       | –                    | 4870                | 1.44                | 13.7     |             |
| M-2    | RS+TAR+P   | 1300      | 650/30       | –                    | 6208                | 1.57                | 25.1     |             |
| E-2    | RS+TAR+P   | 1300      | 950/30       | –                    | 490                 | 3.92                | 9.2      |             |
| I-3    | RS+TAR+P   | 650       | 650/30       | –                    | 248                 | 5238                | 1.52     | 18.8        |
| M-3    | RS+TAR+P   | 650       | 650/30       | –                    | 210                 | 5434                | 1.60     | 19.0        |
| I-4    | RS+TAR     | –         | 750/30       | –                    | 250                 | 653                 | 1.14     | 13.2        |
| M-4    | RS+TAR     | –         | 750/30       | –                    | 398                 | 1457                | 1.28     | 8.9         |
| I-5    | RS+TAR     | –         | 850/30       | –                    | 714                 | –                   | 10.5     |             |
| M-5    | RS+TAR     | –         | 850/30       | –                    | 1322                | 1.31                | 9.1      |             |
| E-5    | RS+TAR     | –         | 850/30       | –                    | 75                  | 1.81                | 5.1      |             |

Figure 1. The cross-section of a sample tape with mixed in and ex situ powders before the heat treatment.

Figure 2. Close-up of the interface of tape I-2 after the heat treatment.

Resistive four-probe measurement with a DC current varying from 10 to 80 mA (depending on the sample) was used for measuring the $R(T)$ behavior of extracted MgB$_2$ cores. The cores were prepared by mechanically removing the Ti sheath. The short pieces of core were connected to the current and potential contacts of sample holder by colloid silver. The resistances were then measured in He vapor in temperatures ranging from 10 to 300 K. The areas of the extracted cores and the distances of the potential contacts were measured with the help of the optical microscope to transform the resistance data to resistivity values. The sample holder for the resistance measurements has several positions for the core sample varying by their distances between contacts, so the closest voltage contact distance to that of the measured was chosen as the value used in the transformation. This can cause an error of maximally 25% in the converted resistivity values. In addition, the resistivity data might have been affected by a possible error in core area prediction due to the difficulties of uniformly removing the Ti sheath.

All the resistivity curves were normalized to the resistivity of the corresponding sample at 40 K. From this data, the critical temperature ($T_c$) was determined as the temperature, where the resistivity had dropped to 50% from the value at 40 K, and the width of the transition ($\Delta T_c$) as the difference of temperatures at which the values 90% and 10% of the normalized resistivity values were achieved. The residual resistivity ratio, RRR, is determined as the normalized resistivity value at 300 K.

In addition, Vickers microhardness measurements (HV0.05–50g) were performed for the cross-sections of the filaments in order to study the development of the density for different deformation.
methods. The phases of the filaments were analyzed using the X-ray diffraction (XRD) method for ground extracted MgB$_2$ cores although detection of MgO is difficult due to its small size and partly amorphous state which cannot be identified with XRD.

3. Results and discussion

Before the heat treatment, the microhardness of the cores was studied for the normal 0 samples and for the CIPped 1 samples. As can be seen from table 1, the HV has increased considerably after CIPping for the I tape but for the M tape, there is no difference in the averaged value over the whole core. However, even for the M-1, the HV near the interface has increased from 153 to 235 while for the inner part the HV of the M-1 has decreased.

After the HT, the HV was measured for samples 0 (only M), 1, 3 and 4. It is apparent, as expected, that the HV increases during the heat treatment. The highest HV is achieved for the CIPped M-1 tape whereas surprisingly the I-1 sample has the lowest HV. The lower pressure of 0.65 GPa did not affect the HV of the M sample. The higher HT temperature, on the other hand, has a larger effect. The values obtained for the I tapes do not vary greatly, only ranging from 172 to 250. The reason for this kind of behavior can be that the lower HT temperatures are high enough for sintering the I samples while for the M samples the sintering process is not carried out far enough so that the ex situ grains present in the core could connect with each other or with the newly created MgB$_2$ phase resulting in an inhomogeneous core structure [13]. No clear correlation between the HV values and the $J_c(B)$ dependence could be shown, probably due to the large differences in precursor powders and fabrication methods between samples.

A close-up of the interface of the tape pressed at 1.3 GPa is shown in figure 2. Clearly, a darker region of reacted part of the filament can be seen next to the sheath. It is proposed, that elevated pressures and possibly also elevated HT temperatures [15] can lead to an enhanced diffusion between Ti in the sheath and B in the core. This phenomenon has been difficult to perceive in previously made samples because of the usage of more typical preparation conditions and HT temperatures. The thickness of the reaction layer seems to vary greatly, from less than 20 to more than 50 µm, possibly due to unevenly distributed pressure. It is, however, very important to notice that Ti might not be good enough sheath material after all—especially when studying or using more intensive fabrication conditions. It is expected that the diffusion is more profound in I than in M or E wires since the ex situ powder would have to decompose first to release the B taking part in the reaction.

In figure 3, the two best measured $J_c(B)$ performances are shown for both I and M samples. In addition, the inset shows how these compare with the M (lower curve) and E (lowest curve) samples which were heat treated at the temperature of 850 °C. It is apparent that the samples with M precursor powder have steeper $J_c(B)$ curves. This behavior is common to all our samples, which indicates that the I tapes exhibit stronger pinning which starts to have an effect at higher values of $B$. One possible reason for this is in the differences in grain size that is smaller for I thank for M samples, as shown by [13]. In addition, increased amount of MgO at the grain boundaries decrease the current between grains but at the same time can improve the pinning thus leading to less steep $J_c(B)$ curve [17]. It is also apparent from table 1 that there is a strong correlation between the values of $J_c(B)$ and the $n$-values—the higher $J_c(B)$ performance being achieved, as expected, for a sample having the higher $n$-value.

The samples exhibiting these elevated characteristics shown in figure 3 were pressed at high pressures, except the I-0 sample which went through the typical fabrication route. It should also be noted that in tapes M-1 and M-2, cracks in the sheath were observed at the corners near the interface that could have decreased the observed performances for these samples.

The reason for the obviously lower $J_c$ for tape I-2 (see table 1) is not yet analyzed. The observed reaction layer is certainly not expected to have such a dramatic influence. The uniaxial applied pressure of 1.3 GPa is most probably introducing cracks into the most stressed parts of the core thus reducing the effective core area for current path. The dissimilar behavior of I and M samples could be explained by different response to the applied deforming pressure due to differences in hardness and
structure of the core. It has been shown previously that the effect of pressure can fluctuate [15], [18], the pressure thus leading to different powder core densities.

The anisotropies for a few selected tapes are shown in figure 4. What is interesting to notice is that throughout all the samples, the following behavior prevails: i) all M samples have higher $\Gamma$ than the equivalent I samples, ii) higher HT temperature leads to lowered $\Gamma$ for both I and M samples and iii) typically for E samples, the $\Gamma$ rises exponentially with $B$. A possible explanation for this kind of behavior is that the usage of I as the precursor powder and the higher HT temperature enhances the homogeneity of the core. It has been shown before that tapes with enhanced pinning have lower anisotropies [19] which is consistent with our less steep I samples having lower $\Gamma$ values.

The $\Gamma$ also increases predominantly with the applied pressure: samples 1 and 3 exhibit higher $\Gamma$ values than the unpressed samples 0 due to improved texture [20] but samples 2 have slightly lower anisotropies (not shown in the figure). This is anticipated since higher pressure increases the texturing of the filament thus leading to higher anisotropy. For all I and M samples, the $\Gamma$ values are below 1.7 at 7 T which are usual values for MgB$_2$ tapes [19], [21]–[22]. This was expected because the extent of the applied deformations was not very intensive due to the fairly small starting diameter of the Ti tube and its softness after annealing.

The so called Rowell analysis [14] was applied to all samples for which reliable data could be measured to receive information about the connectivity inside the MgB$_2$ filaments. With Rowell analysis, the fraction of effective current carrying cross-sectional area, $A_F$, or connectivity, is estimated by comparing the phonon term resistivity ($\Delta \rho$) [9] of a perfectly connected ideal specimen with that of the observed value of the sample. The $\Delta \rho$ is taken to be the change in the resistivity values at room temperature and just above the $T_c$, or at 40 K in the case of MgB$_2$. The $\Delta \rho$ value to which the measured sample should be compared has been under some discussion [23]–[28]. The value of 4.3 $\mu\Omega$cm used by Rowell has been estimated as the in-plane resistivity for a single-crystal which neglects the anisotropy and the polycrystalline nature of a real MgB$_2$ filament. The value of ideal $\Delta \rho$ used in this article is 7.3 $\mu\Omega$cm [23] which better takes into account the previously mentioned difficulties. The measured resistivity values, with calculated values for $\Delta \rho$, $A_F$ and RRR, are all gathered in table 2.

The very low values of $A_F$, varying from connected areas of less than 1 to 6%, indicate weak grain connectivity likely due to the presence of non-superconductive areas such as macropores and insulating phases like MgO. The general inspection of $A_F$ reveals that the intermediate pressing seems to enhance the connectivity between grains whereas for higher pressure the values of $A_F$ become lower which might indicate that the applied pressures were too high and introduced some cracks into the MgB$_2$ core. This prediction is supported by the higher effective area of I-2 than M-2 where the latter has harder ex situ grains within the core during the application of pressure. In addition, the $A_F$ values
for M samples are always lower than those of the equivalent I samples. This can partly be due to the HT temperature that is not high enough to sinter the \textit{ex situ} grains.

### Table 2. The measured resistivity properties for MgB$_2$/Ti tapes.

| Sample | $T_c$ [K] | $\Delta T_c$ [K] | $\rho_{40K}$ [µΩcm] | $\rho_{300K}$ [µΩcm] | $\Delta \rho$ [µΩcm] | $A_F$ [-] | RRR [-] |
|--------|-----------|------------------|------------------|------------------|------------------|--------|--------|
| I-0    | 38.39     | 1.9              | 227.16           | 477.53           | 200.37           | 0.036  | 1.72   |
| M-0    | 38.14     | 1.8              | 1166.66          | 1984.12          | 817.46           | 0.009  | 1.70   |
| I-1    | 36.84     | 2.6              | 444.11           | 712.96           | 268.85           | 0.027  | 1.61   |
| M-1    | 36.46     | 3.0              | 538.42           | 887.52$^a$       | 349.09           | 0.021  | 1.65   |
| I-2    | 37.48     | 1.9              | 280.87           | 434.01           | 153.14           | 0.048  | 1.55   |
| M-2    | 34.39     | 3.0              | 702.00           | 1141.73          | 439.76           | 0.017  | 1.63   |
| I-3    | 37.68     | 2.3              | 146.50           | 267.31           | 120.82           | 0.060  | 1.82   |
| M-3    | 36.07     | 2.7              | 222.52           | 380.36           | 157.84           | 0.046  | 1.71   |
| I-4    | 38.23     | 1.7              | –                | –                | –                | –      | –      |
| M-4    | 37.70     | 1.8              | 456.62           | 859.48           | 402.86           | 0.018  | 1.88   |
| M-5    | 38.38     | 2.0              | 642.68           | 1054.46          | 411.78           | 0.018  | 1.64   |
| E-5    | 38.54     | 2.0              | 5575.47          | 6734.15          | 1158.68          | 0.006  | 1.21   |

$^a$ Expected from $\rho_{40K}$ and the general form of the resistivity curves.

As can be seen from table 2, there is a clear correlation between $T_c$ and $\Delta T_c$: the higher the $T_c$ the narrower the $\Delta T_c$ for both I and M tapes. The M samples always have lower $T_c$ and wider $\Delta T_c$ than the equivalent I samples. This can again be explained by the lower connectivity between the grains in the M tapes caused by the relatively low HT. The higher $T_c$ and narrower $\Delta T_c$ for samples 4 and 5 heat treated at higher temperatures support this prediction. However, the $J_c(B)$ dependences for both of these samples are degraded.

The lowest $T_c$ is obtained for the tape M-2 although it has the best $J_c(B)$ characteristics of all the samples. Otherwise, the range of $T_c$ values is rather narrow, the difference being only 2.3 K. The I-0 tape has the highest $T_c$ of the I samples and the $T_c$ of M-0 is also relatively high. The application of pressure therefore decreases the $T_c$ whereas higher HT temperature increases it. Since usually the $T_c$ correlates well with the phase purity and homogeneity of the core for both \textit{ex situ} and \textit{in situ} wires [17], [28]–[30], the observations would indicate that pressing causes small cracks inside the filament which decrease the $T_c$. In addition, the lower $T_c$ values for samples heat treated at higher temperatures could be due to higher MgO content at grain boundaries.

The basic structural parameters and the content of phases for selected samples acquired using the XRD analysis are presented in table 3. None of the analyzed cores contained detectable amounts of MgB$_4$ but the amount of MgO varies from 5 to 10.6%. However, the volume of MgO does not correlate with the values of $\Delta \rho$. Particularly noticeable is the high $\Delta \rho$ value achieved for M-0 which has a relatively low MgO content. It is also interesting to notice that even though M-5 has both high MgO content and elevated $\Delta \rho$, the $\Delta \rho$ of M-0 is much higher.

### Table 3. The lattice constants for MgB$_2$ with the core composition.

| Sample | $a$ [nm] | $b$ [nm] | % MgB$_2$ | % MgO | % MgB$_4$ |
|--------|----------|----------|-----------|-------|-----------|
| I-0    | 0.30864  | 0.35230  | 92.8      | 7.2   | –         |
| M-0    | 0.30862  | 0.35239  | 93.8      | 6.2   | –         |
| I-1    | 0.30880  | 0.35239  | 93.0      | 7.0   | –         |
| I-5    | 0.30838  | 0.35269  | 91.3      | 8.7   | –         |
| M-5    | 0.30847  | 0.35263  | 89.4      | 10.6  | –         |
| E-5    | 0.30856  | 0.35240  | 95.0      | 5.0   | –         |
The samples in which a higher HT temperature was applied (I-5 and M-5) have the lowest in-plane lattice parameter, \( a \), but highest out-of-plane lattice parameter, \( b \). The sample E-5 can be viewed belonging to the same group even though the employed HT temperature was lower than normally applied for \textit{ex situ} conductors. The elevated HT temperature has also increased the concentration of MgO. The degraded transport current density properties of samples with higher HT temperature might therefore be explained by the increased amount of insulating MgO, like proposed earlier. Conversely, the highest \( a \) with a relatively low \( b \) was observed for the CIPped tape when compared to other samples. It is therefore probable that pressing has a widening effect on \( a \).

The effect of different pressing methods and applied pressures on \( J_c(B) \) and resistivity behavior for the M samples is shown in figures 5 and 6, respectively. The same, but for the I samples, is shown in figures 7 and 8. The pressing generally seems to have an enhancing effect on the transport properties even though the differences are not especially prominent. The highest \( J_c \) values were achieved by CIPping for the I tapes and by the higher uniaxial pressing for M tapes. Peculiarly, the \( A_F \) values appear to have an inverse correlation with \( J_c(B) \) behavior. The samples I-2, I-3 and M-3 with the highest \( A_F \) values have similar or lower transport current density performance than their unpressed equivalents. While the resistivity and \( A_F \) values are very sensitive to the used powder quality and deformation method, the \( J_c(B) \) of these tapes is changed only slightly. It is therefore apparent that the \( J_c(B) \) values are not dominantly influenced by the connectivity evaluated by the Rowell analysis.

What is interesting to notice is the effect of pressure on the resistivity near the offset of transition to superconducting phase. For M tapes, a second step is clearly visible for all CIPped and pressed samples whereas for the I samples, only the CIPped tape shows such a response. This kind of behavior...
could be explained by a network of microfractures which extend throughout the core, possibly caused by too high pressure or the harder \textit{ex situ} grains in M tapes. In any case, the step in I-I cannot be due to the MgO content since its concentration does not greatly differ from that of the other samples. No correlation between the \(J_c(B)\) dependence with RRR, \(T_c\) or \(\Delta T_c\) could be established.

The effect of heat treatment temperature on \(J_c(B)\) and resistivity behavior of M tapes is given in figures 9 and 10, respectively. Unfortunately, no reliable data about the effect of HT temperature on the resistivity of I tapes could be measured. Instead, the effect on transport properties is shown in figure 11. The higher than the normal HT temperature of 650 °C degrades the \(J_c(B)\) performance considerably which is in agreement with previously published behavior for addition free samples [12]. This effect is more profound in the I samples. Opposite responses could be expected, however, for samples with either carbon or SiC additions [13]. The more intensive HT conditions have reduced the \(A_F\) value compared to the averaged value which is in correlation with the observed \(J_c(B)\).

Figure 9. The effect of HT temperature on the \(J_c(B)\) behavior of M samples at 4.2 K.

Figure 10. The effect of HT temperature on the resistive transition of M samples.

Figure 11. The effect of HT temperature on the \(J_c(B)\) behavior of I samples at 4.2 K.

Figure 12. The resistivity curves for E-5 and M-5 tapes fabricated in similar ways, and the I tape with the lowest resistivity.

Figure 12 shows the resistivity curves for the M-5 and E-5 samples that went through the same fabrication route in addition to the sample that has the lowest resistivity, I-3. The differences are surprisingly high; please note the logarithmic scale. The \(\Delta \rho\) and therefore the \(A_F\) values correlate well with the transport properties (refer to figure 3 for the general \(J_c(B)\) behavior) but the \(T_c\) and \(\Delta T_c\) values are very similar to each other for all the three samples.
4. Conclusion
The effect of fabrication routes on density, connectivity and the critical current density was studied for monofilamentary MgB$_2$/Ti tapes. The PIT method was used to make the tapes which are filled with either in situ, ex situ, or a 60%–40% mixture of both in situ and ex situ powders.

Results show that differences in $J_c(B)$ values are relatively small for the majority of the tapes, excluding the samples heat treated at higher temperatures; the best samples reached 10 kA/cm$^2$ between 6 and 6.2 T which is fairly high for undoped MgB$_2$ tapes. It was also observed that the anisotropy increased with addition of ex situ powder, application of pressure or the lower heat treatment temperature. This is because these aforementioned circumstances improve the texture of MgB$_2$ and degrade the homogeneity of the core. Generally, the values of the effective cross-section area, $A_F$, acquired through the Rowell analysis were very low for all of the samples, indicating very weak connectivity between grains.

The data acquired through the transport measurements is more reliable than the calculated resistivity values due to the possible errors in measuring the core cross-section area and the voltage contact distances. Unfortunately, the values of $\rho$ are strongly influenced by the dimensions of the sample. For unknown reasons, in our experiment, the $\rho(T)$ data does not correlate with the $J_c(B)$ behavior as expected. Possible explanations for this incongruity could be either the formation of the microfractures which are a result of the intensive pressing and possibly the errors in evaluated sample dimensions.

It should be noted nevertheless that when measuring a $J_c$ in an applied field ($B \neq 0$), the $J_c$ is additionally influenced by flux pinning and upper critical field, $B_{c2}$. If these properties vary with the changed processing parameters, the measured $J_c(B > 0)$ also varies. To eliminate the effect of pinning and $B_{c2}$, it would thus be more beneficial to compare the $\Delta \rho$ values with $J_c(B = 0)$ values. Unfortunately, however, the high $J_c$ values at zero field cannot presently be measured due to the lack of stabilizing material in the samples.

Acknowledgement
The authors wish to thank Wacław Pachla for his kind assistance with the CIP treatment of the samples, and Ľubomír Kopera and Miloslav Kulich for their instructions and help with practical work. This work was supported by the European Commission under contract number MRTN-CT-2006-035619.

References
[1] Nagamatsu J, Nakagawa N, Muranaka T, Zenitany I and Akimitsu J 2001 Nature 410 63–4
[2] Zhao Y, Feng Y, Cheng C H, Zhou L, Wu Y, Machi T, Fudamoto Y, Koshizura N and Murakami M 2001 Appl. Phys. Lett. 79 1154–6
[3] Suo H L, Beneduce C, Dhallé M, Musolino N, Genoud J E and Flükiger R 2001 Appl. Phys. Lett. 79 3116–8
[4] Cimberle M R, Novak M, Manfrinetti P and Palenzona A 2002 Supercond. Sci Technol. 15 43–7
[5] Kováč P, Hušek I and Melišek T 2002 Supercond. Sci. and Technol. 15 1340–4
[6] Dou S X, Horvath J, Soltanian S, Wang X L, Qin M J, Zhou S H, Liu H K and Munroe P G 2003 IEEE Trans. on Appl. Supercond. 13 3199–202
[7] Fu B Q, Feng Y, Yang G, Liu C F, Zhou L, Cao L Z, Ruan K Q and Li X G 2003 Physica C 392–396 1035–8
[8] Liu C F, Yan G, Du S J, Xi W, Feng Y, Zhang P X, Wu X Z and Zhou L 2003 Physica C 386 603–6
[9] Yamamoto A, Shimoyama J, Kishio K and Matsushita T 2007 Supercond. Sci. Technol. 20 658–66
[10] Eisterer M 2007 Supercond. Sci. Technol. 20 R47–73
[11] Ma Y, Zhang X, Nishijima G, Watanabe K, Awaji S and Bai X 2007 *Appl. Phys. Lett.* **88** 072502–3
[12] Yeoh W, Horvat J, Kim J H, Xu X and Dou S X 2007 *Appl. Phys. Lett.* **90** 122502–1–3
[13] Kováč P, Reissner M, Melišek T, Hušek I and Mohammad S 2009 *J. Appl. Phys.* **106** 013910–7
[14] Rowell J M 2003 *Supercond. Sci. and Technol.* **16** R17–27
[15] Kováč P, Hušek I, Melišek T and Holubek T 2007 *Supercond. Sci. Technol.* **20** 771–6
[16] Kováč P, Hušek I, Pachla W, Melišek T, Diduszko R, Fröhlich K, Morawski A, Presz A and Machajdik D 2002 *Supercond. Sci. Tehcnol.* **15** 1127–32
[17] Kováč P, Hušek I, Melišek T, Grivel J C, Pachla W, Štrbík V, Diduszko R, Homeyer J and Andersen N H 2004 *Supercond. Sci Technol.* **17** L41–6
[18] Hancock M H and Bay N 2007 *Supercond. Sci. Technol.* **20** 886–90
[19] Kováč P, Melišek T and Hušek I 2005 *Supercond. Sci Technol.* **18** L45–8
[20] Kováč P, Hušek I, Fedor J, Melišek T, Cambel V, Morawski A and Kario A 2009 *Physica C* **469** 713–6
[21] Kumakura H, Matsumoto A, Fujii H, Kitagushi H and Togano K 2002 *Physica C* **382** 93–7
[22] Lezza P, Gladyshevskii R, Senatore C, Cusanelli G, Suo H L and Flükiger R 2005 *IEEE Trans. Appl. Supercond.* **15** 3196–9
[23] Wilke R H T, Bud’ko S L, Canfield P C, Finnemore D K, Suplinskas R J and Hannahs S T 2005 *Physica C* **424** 1–16
[24] Eltsev Y, Lee S, Nakao K Chikumoto N, Tajima S, Koshizuka N and Murakami M 2002 *Phys. Rev. B.* **65** 140501–1–4
[25] Jiang J, Senkowicz B J, Larbalestier D C and Hellstrom E E 2006 *Supercond. Sci Technol.* **19** L33–6
[26] Canfield P C, Finnemore D K, Bud’ko S L, Ostenson J E, Lapertot G, Cunningham C E and Petrovic C 2001 *Phys. Rev. Lett.* **86** 2423–6
[27] Gozzelino L, Minetti B, Ummarino G A, Gerbaldo R, Ghigo G, Laviano F, Lopardo G, Giunchi G, Perini E and Mezzetti E 2009 *Supercond. Sci Technol.* **22** 065007
[28] Tarantini C et al. 2006 *Phys. Rev. B.* **73** 134518–1–11
[29] Soltanian S et al. 2001 *Physica C* **361** 84–90
[30] Jiang C H, Hatakeyama H and Kumakura H 2005 *Physica C* **423** 45–50