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Effect of Rapidly Annealing Process on MgB_2 Superconducting Wires

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Abstract

The present study has reported the effect of rapidly annealing and cooling process on the transport and morphological properties of Fe/MgB_2 wires. Transport properties like critical transition temperature, transition width and engineering critical current density of the obtained wires at different annealing and durations were determined for superconducting wires. The results show that the annealing temperature is more dominant to accelerate the reaction rate of Mg and B in the wires in comparison with annealing duration. Among the studied wires, a highest J_c (T = 36K) value >150 A/cm^2 was achieved for the wires at 900ºC and 1000ºC for small durations (15 minutes). In the study, it was investigated whether fast annealing and cooling is a possible candidate to fabricate fast the requested superconducting MgB_2 long length wires for coils by React&Wind method in continuous system or not.

Keywords: MgB_2 wire, critical current density, heat treatment.

1. INTRODUCTION

Critical current density (J_c), critical transition temperature (T_c) and upper critical field (H_{c2}) parameters of superconducting materials are those of the most prominent properties in industrial applications. Since discovery of MgB_2 superconductors [1], has still interesting in terms of higher critical transition temperature than Nb based inter-metallic superconductors and workable above ~20 K(liquid hydrogen) as cryogen-free [2]. Many researchers have widely studied on the different application areas such as the materials engineering and heavy-industrial technology of superconducting materials namely, YBCO, BSSCO and especially MgB_2 superconducting materials [3-5]. Besides, nowadays the scientific studies on the wire or tape formations for the intermetallic binary superconductor MgB_2 as Magnetic resonance imaging [6,7], high power wind turbines [8,9], hydrogen level sensor [10] and space satellite [11].

The fabricated wires commonly by means of powder-in-tube process (PIT) [12-15] have been improved to be applied into industrial by utilizing different preparations such as reactive liquid infiltration(RLI) method [16,17], internal magnesium diffusion (IMD) process [18], cold high pressure densification (CHPD) method [19], and advanced or modified internal magnesium diffusion (AIMD) procedure [20] in according to strengths and weaknesses. The manufacture process is categorized in two main processes as the in-situ and ex-situ MgB_2 method in terms of the initial filling powders. In-situ MgB_2 in the fabrication of superconductor wires has preferred due to the benefit as possible heat treatment at low temperature, comfortable doping, control of particle size, and high critical current density

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under applied external magnetic field [21]. Additionally, there are various parameters that may control and affect the formation rate of the MgB_2 layer as annealing temperature and time [22], cooling and heating rate [23, 24], morphology of the B powder [25] thickness of the B layer [26] and also density of the B layer [27]. The factors affecting J_c performance are weak inter-grain connectivity, porosity, and low MgB_2 core density [28, 29]. Up to now, various manufacture methods [30,31], as several sheath materials [32], efficient dopants [33], and heat treatment conditions [34] have been tested and achieved. Fe sheathed MgB_2 wire or tape has been interested in fabrication due to useful in cold drawing process and relatively high critical current density in comparison with the used other sheaths, etc. [15]. On the other hand, low cost production of a single MgB_2 phase is hard due to the formation of different phases after reaction such as MgB_6, MgB_{12}, and MgO [35]. Heat-treatment, size and purity of constituents, and operational steps affect directly a pure MgB_2 formation. Oxidation and grain connectivity are grave questions in the preparation of MgB_2 samples. Since the critical current density being directly relation with grain connectivity is the most important parameter in applications. The increment of transport critical current density may be possible; however it still requests to diminish several extrinsic factors in the fabrication of wires and other devices [36, 37]. The major challenge is how to overcome the major limitations of inter-grain connectivity in MgB_2 formation to enhance critical current density (J_c). Hence, it is necessary to study the production variables relating to the superconductivity of MgB_2 in detailed. Further studies are needed concerning short annealing steps and short annealing times to obtain the optimum grain structure and to discover and to develop the performance of the MgB_2 sample [38]. The basic studies to deal with difficulties and to transfer gained experience to industrial applications have still been continued.

In the study, the electrical and morphological properties of various superconducting Fe sheathed MgB_2 wires produced with rapidly annealing and cooling via solid state reaction method have been examined by DC electrical resistivity depending on temperature, critical current values at constant temperature closed to critical transition temperature(T_c) and scanning electron microscopy (SEM). The obtained results showed that the annealing temperature is more dominant according to annealing duration to increase reaction rate between Mg and B. High annealing temperature for small duration is needed to complete formation of MgB_2. The wires annealed at 900ºC and 1000ºC for small durations (15 minutes) have the highest J_c value >150 A/cm^2 at 36K temperature closed to critical transition temperature (T_c).

2. EXPERIMENTAL PROCEDURES

A high purity atomized spherical Mg powder (99.0% - 100-200 mesh) and elemental amorphous boron(95-97% pure) bought by Pavezyum Company were weighed in 1:2 ratio as stoichiometric and mixed with a ball milling machine for 3 hours for preparation of in-situ mixture. Then, iron tube OD/12.0mm and ID/9.00mm were cut 200mm in length and cleaned in alcohol with ultrasonic system for 30 minutes. After that, the Fe tube being the aluminium foil was pressed as stopper in both sides of iron tube was filled with densification about 1.30 g/cm^3 by weighing 6 g of the obtained mixture by means of powder in tube method. Outer diameter of the prepared iron tube was drawn from 12.0mm to 1.00mm with the diameter 15 percent reduction and intermediate annealing processes. The monocore MgB_2/Fe superconducting wires after drawing process were obtained by conventional solid state reaction. The solid state reaction of the drawing wires was performed by annealing at different temperatures(in the range of 650ºC and 1000ºC) and durations (for 15 and 30 minutes) in a Protherm tube furnace PZF 12/75/700 with suddenly heating and cooling under 5 bar high purity argon pressure. The microstructural properties (grain connectivity, phase formation, densification, and surface morphology) were investigated by a scanning electron microscopy (SEM, JEOL 6390-LV). The DC resistivity measurements (*R-T*) and current carrying capacity measurement(*I-V*) of the samples were carried out by the standard dc four probe technique between

10K and 50K and at close to T_c^{offset} values and constant temperature, respectively in CRYO Industries system (closed-cycle cryostat). The nano-voltmeter and the current source were programmable Keithley 2182A and Keithley 220, respectively.

3. RESULTS and DISCUSSIONS

Figure 1 shows the change of the resistivity values depending on temperature of the MgB_2 wires 1.00mm in diameter annealed between 650ºC - 1000ºC for 15 minutes in the range of 10 to 50 K. It can be said that resistivity value systematically increases with rising of annealing temperature for 15 min. On the other hand, the normal state resistivity of the MgB_2 wires was linearly increased at below 750ºC for short time due to the metallic behaviour causing unreacted Mg. Additionally, the critical transition temperature (T_c) of the studied wires at different annealing temperatures for 15 min was the same (about 37.5K) excluding the wire annealed at 650ºC for 15 min, because the annealing temperature is not enough for the completely reaction and it has extremely poor connectivity [39].

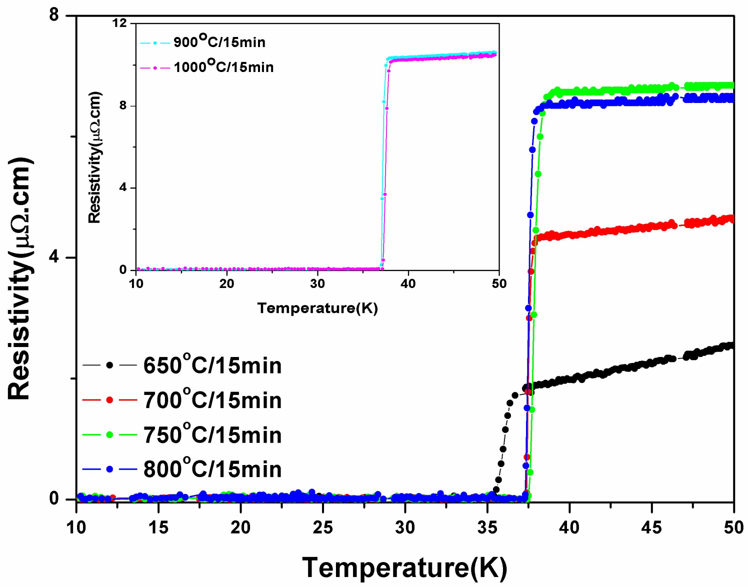


Figure 1. Resistivity vs temperature (ρ-T) curves of superconducting MgB_2/Fe wires obtained at 650 ºC/15 min, 700 ºC/15 min, 750 ºC/15 min and 800 ºC/15 min. The inset shows resistivity vs temperature (ρ-T) curves for the wires at 900 ºC/15 min, and 1000 ºC/15 min.

The resistivity curves depending on temperature of the MgB_2 wires 1.00mm in diameter annealed between 650ºC and 800ºC for 30 minutes in the range of 10 to 50 K were seen in Figure 2. It can be seen clearly that resistivity value rises with ascending of annealing temperature for 30 min. Furthermore, the normal state resistivity of the MgB_2 wires was also linearly increased due to the unreacted Mg at below 750 ºC for 30 min. So, it can be said that the change of the annealing temperature is more dominant to accelerate the reaction rate of Mg and B in the wires according to annealing duration. In addition, the critical transition temperature (T_c) of the studied wires at different annealing temperatures for 30 min was the same (about 37.5K). The normal state resistivity value (ρ_{40}) was mostly increased with annealing temperature and duration due to the formation of smaller grains.

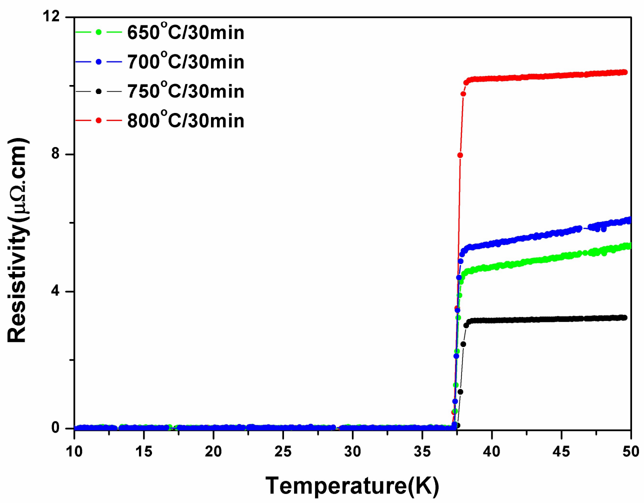


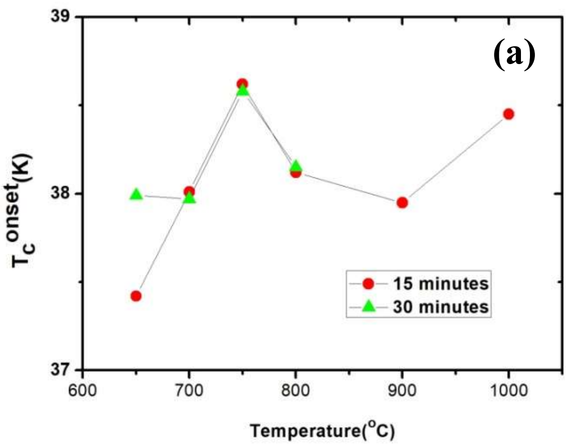
Figure 2. Resistivity vs temperature (ρ-T) curves of superconducting MgB_2/Fe wires obtained at 650 ºC/30 min, 700 ºC/30 min, 750 ºC/30 min and 800 ºC/30 min.

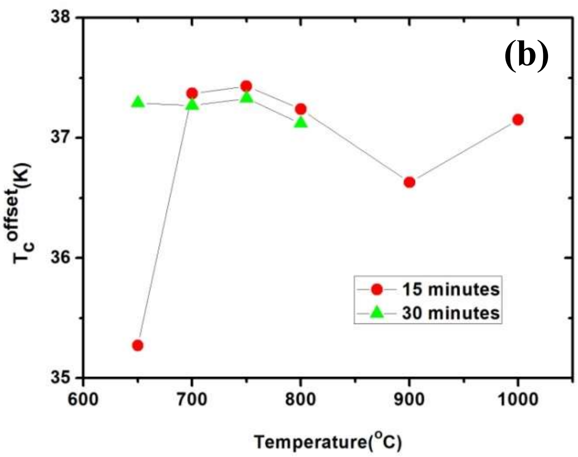
As indicated in Table 1, T_c^{onset}, T_c^{offset}, ΔT, T_c, ρ_{40} and slope of the normal state resistivity values of all studied MgB_2 wires can be seen in detailed. T_c values were obtained from the derivative of ρ-T curves. T_c^{offset} and T_c^{onset} values of the wires alter 35.27K to 37.43K and 37.42K to 38.62K, respectively. While T_c^{onset} value does not changes with increment of the annealing duration [38], the change of T_c^{offset} value depending on annealing duration can be significantly seen only at 650ºC for 15 min and others are the same, because low annealing temperature(650ºC) is near the melting point of Mg [39]. ΔT value being relation with grain connectivity change 0.64 K for the wire at 750ºC for 15 min. to 2.15 K for the wire at 650ºC for 15min. Also, the obtained results show that the maximum value of the normal state resistivity (ρ_{40}) is 10.38 μΩ.cm and ρ_{40} value is approximately the same for greater annealing temperatures than 950ºC for 15 min and 800 ºC for 30 min. Furthermore, slope of the normal state resistivity altering linearly with temperature is max. 0.0618 μΩ.cm/K and 0.0707 μΩ.cm/K for the wires 650ºC for 30 min. and 700ºC for 30 min, respectively. So, the longer time below 700ºC is needed for comparison with the obtained wires at higher annealing temperature. The results indicate that annealing temperature (750 and 800ºC) and duration (15 and 30 min.) are suitable for the production of the wires without unreacted Mg. Among the all studied samples, highest resistivity values are the same (10.25 μΩ.cm) with negligible deviation (±0.1 μΩ.cm) for the wires at 800ºC for 30 min, 900ºC for 15 min and 1000ºC for 15 min. In the results, it can be said that poor connectivity in the wires obtained at the higher annealing temperature occurs with increment of normal state resistivity as based on formation of the dense filament [40]. Also, increasing of resistivity or reducing of effective cross-section area of the samples is relation with MgO formation in the grain boundaries at high annealing temperature. Moreover, the results indicate that ascending of the annealing temperature and duration increases the normal state resistivity being directly relation with the amount of the grain boundaries and reversely proportional with grain-size.

Table 1. The obtained electrical values from the ρ-T curve of the studied MgB_2/Fe wires.

Samples	T_c^{onset} (K)	T_c^{offset} (K)	ΔT (K)	T_c (K)	ρ_{40} (μΩ.cm)	Slope of normal state resistivity (μΩ.cm/K)
650 °C/15 min	37.42	35.27	2.15	36.01	1.98	0.0564
700 °C/15 min	38.01	37.37	0.64	37.51	4.39	0.0363
750 °C/15 min	38.62	37.43	1.19	37.84	6.56	0.0144
800 °C/15 min	38.12	37.24	0.88	37.49	6.73	0.0137
900 °C/15 min	37.95	36.63	1.32	37.14	10.38	0.0233
1000 °C/15 min	38.45	37.15	1.30	37.37	10.25	0.0228
650 °C/30 min	37.99	37.29	0.70	37.37	4.68	0.0618
700 °C/30 min	37.97	37.27	0.70	37.47	5.37	0.0707
750 °C/30 min	38.58	37.33	1.25	37.72	3.13	0.0084
800 °C/30 min	38.15	37.12	1.03	37.49	10.19	0.0195

The effect of annealing temperature and short duration on superconducting properties of in situ MgB_2/Fe monocore wires is investigated. Figure 3a, b presents the T_c^{onset} and T_c^{offset} critical temperature values of the wire samples as a function of annealing temperature and duration. In the results, while the T_c^{offset} values(37.27K with negligible deviation) of the wires obtained for 30 minutes do not depend on the annealing temperature up to 800°C, value of ones for 15 minutes changes significantly at temperature being greater than 800°C(36.63K and 37.15K) and smaller than 700°C(35.27K). Also, the T_c^{offset} values of the wires obtained at temperature between 700°C and 800°C are not different in annealing durations for 15 and 30 minutes [40]. Moreover, when the Figure 3a,b and c are examined in detailed, the annealing duration in the range of 700°C and 800°C annealing temperature is not effective in terms of the T_c^{onset}, T_c^{offset}, ΔT values. In Figure 3c, the change of ΔT values indicates that the broadening of transition width (2.15K) for the wire at 650°C for 15 min. may be due to the uncompleted MgB_2 formation or inhomogeneity for short time heat treatment. The transition width (ΔT) relation with critical current density (J_c) and grain connectivity of the other wires changes with small variation in the range of 0.64K to 1.32K.





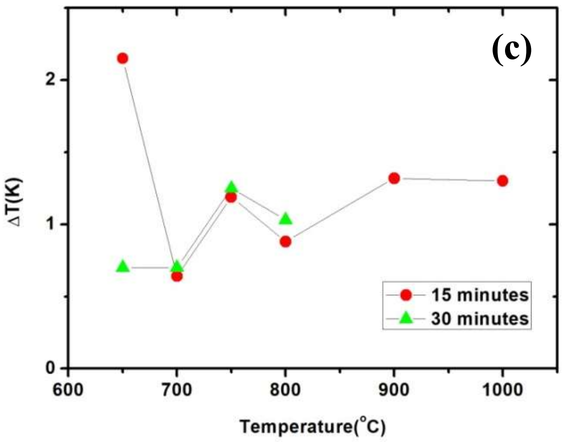


Figure 3. a) T_c^{onset}, b) T_c^{offset} *and* c) *ΔT* behaviour of the obtained MgB_2 wires with different annealing temperature and duration.

In the Figure 4, it can be seen that the critical current density (J_c) values of the wires obtained at 750ºC and 800ºC for 30 min and 900ºC and 1000ºC for 15 min. were measured at constant temperature (at 36 and 37 K) closed to critical transition temperature (T_c) by applying maximum 1 Amper(A). When the J_c value was measured at a certain temperature range being below 1 K of the T_c value of the studied wire, the result could be analysed easier by applying 1 A. Because, the J_c value of the wire decreases as it gets closer to T_c value. The performed previous our work with heating and cooling rate 5ºC/min presents critical current density values in the range of 25A/cm^2 and 125A/cm^2 at different temperatures (800 ºC - 1000ºC) for 1 hour[28]. The results show that the highest J_c values(>150 A/cm^2) at 36 K belong to the wires at the high annealing temperature (900 and 1000ºC) for 15 min. although T_c value of the wires at 750ºC and 800ºC for 30 min. were higher than those of annealed for 15 min. This may be attributed to the increment of J_c value with decrement of T_c value. The J_c values of the wires at 750ºC for 30 min, 800ºC for 30 min., 900ºC for 15 min. and 1000ºC for 15 min. were measured 18.88 A/cm^2, 6.88 A/cm^2, 32.63 A/cm^2 and 25.34 A/cm^2 at 37 K and 69.52 A/cm^2, 29.11 A/cm^2, >150 A/cm^2 and >150 A/cm^2 at 36 K, respectively. The J_c values decline with increment of annealing temperature at the same duration as Figure 4b, it may be due to the starting formation of Fe_2B phase [41] and MgB_4 phase [35] as impurity. Hence, it can be said that the J_c value decreases with long annealing duration at high annealing temperature above 900ºC due to more impurity phase formation[39].

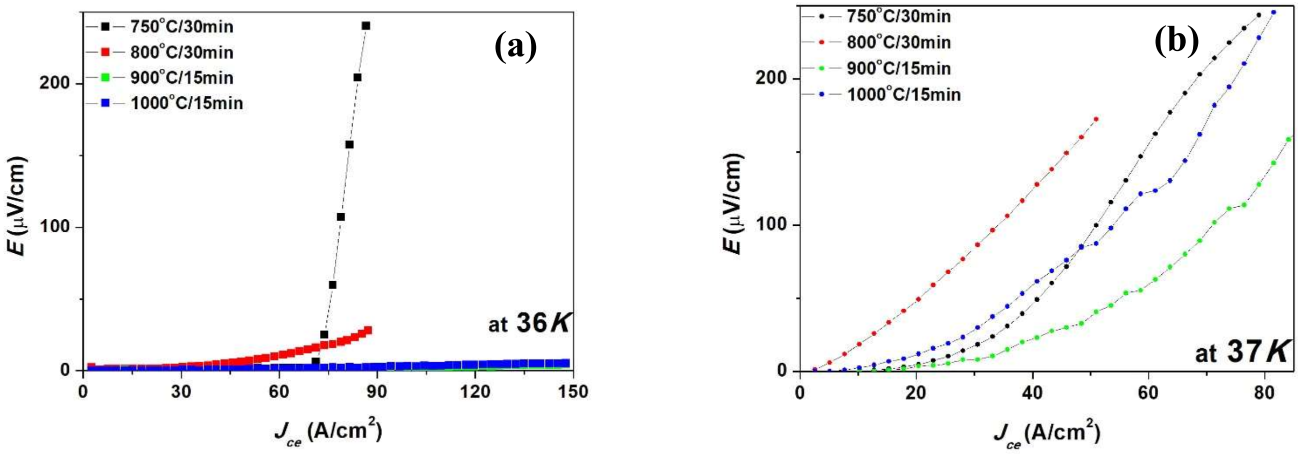


Figure 4. Electric field vs Critical current density (E-J_c) graph obtained at constant a-) 36K and b-) 37K for the wires at 750ºC and 800ºC for 30 min and 900ºC and 1000ºC for 15 min.

The Figure 5 (a)-(d) shows the cross-sectional SEM images taken from the polished surface of the superconducting wires 1.00mm in diameter at the different annealing temperatures an durations. The amount of the formed holes in the core of the wires mean that reaction between Mg and B is possible even at 650ºC being melting point of Mg for short duration (15 min.) by rapidly heating and cooling. Reaction rate depends on the annealing temperature and duration, however annealing temperature is more effective in accordance with the annealing duration.

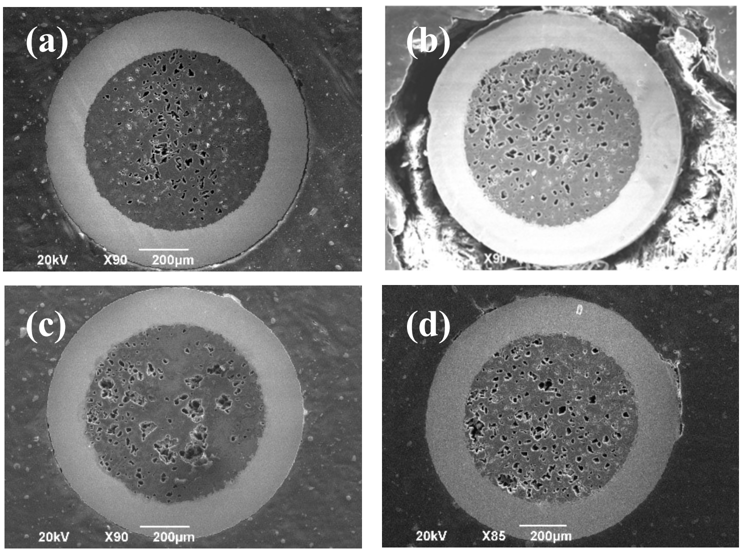


Figure 5. The cross-sectional SEM images of the wires (a) at 650ºC-15min, (b) 700ºC-15min, (c) 650ºC-30min, and (d) 700ºC-30min.

In Figure 6, it can be seen the SEM images with x1000 in magnification of the wires at different annealing temperatures for 15 min. The diameter of the holes in the core of the wire is relation with reaction process between Mg and B powders. The brightness in the core indicates the uncompleted reaction or the presence of Mg after annealing in the Figure 6 (a), (b) and this is compatible with the behaviour normal state resistivity depending on temperature in the electrical measurements. Also, while the size of the holes formed after reaction changes 10 to 15 µm for the wires at 650ºC for 15 min, it alters 15 to 20 µm for the wires at 700ºC for 15 min. As the annealing temperature increases 750ºC to 900ºC for 15 min, the size of the pores raises up to 30 µm. The SEM images shows the completed reaction between Mg and B due to no change significantly of the size of pores above 750ºC and no detectible of the brightness at the near to holes.

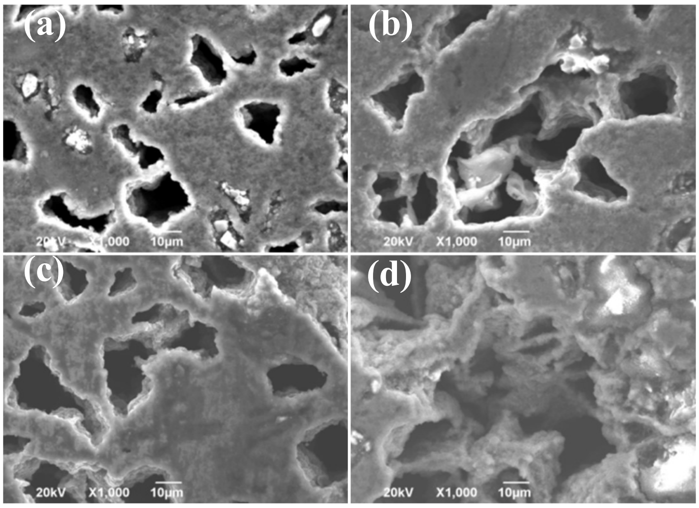
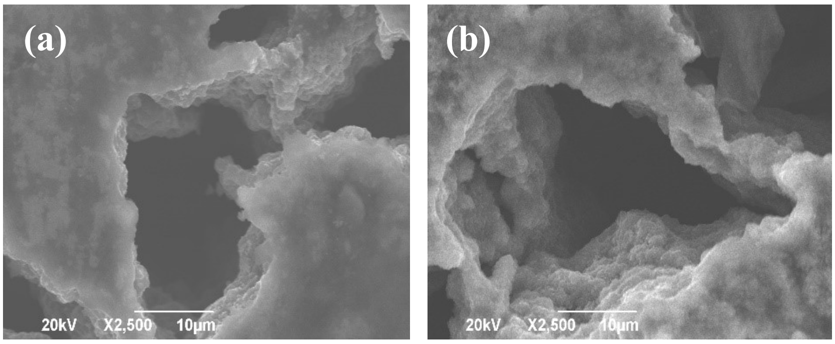


Figure 6. The SEM images of (a) 650 ºC -15min, (b) 700 ºC -15min, (c) 750 ºC -15min, and (d) 900 ºC -15min.

Figure 7 presents the SEM images with x2500 in magnification of the wire having high performance. Figure 7 (a) and (b) shows the size of pores does not alter with the short annealing duration at 750ºC and it can be said that short annealing duration is recessive according to the annealing temperature. The surface morphology of the wires at the various annealing temperature and duration has almost the same up to 800ºC as Figure 7 (a), (b) and (c). Moreover, the smaller grain size occurs at high annealing temperature above 800ºC and this causes more grain boundaries and resistivity. The observations in the SEM images are overlap with the other results. The pore size remains almost constant while the number of voids increases with increasing the annealing temperature. Finally, the structure in Figure 7(d) with well-connected small grains and less porosity supports to be perfect current ways causing high J_c.



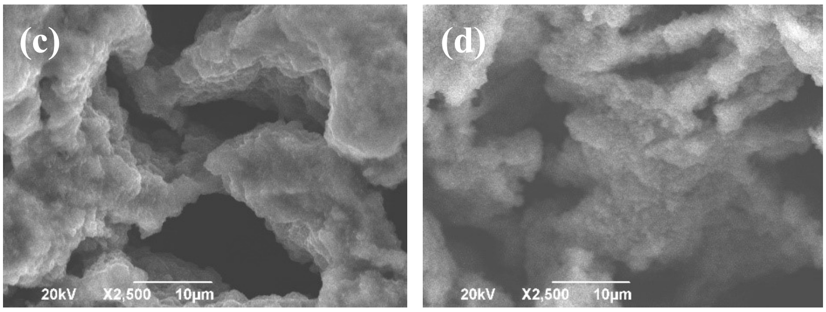


Figure 7. The SEM images of (a) 750 ºC -15min, (b) 750 ºC -30min, (c) 800 ºC -30min, and (d) 900 ºC -15min.

4. CONCLUSIONS

The effect of the rapidly annealing process on the superconducting wires was investigated by utilizing electrical and morphological results. The critical transition temperature (T_c) of all wires for 15 min. was about 37.5K excluding the wire annealed at 650ºC causing extremely poor connectivity and no completely reaction. The annealing temperature is more dominant to accelerate the reaction rate of Mg and B in the wires in comparison with annealing duration. The wires at the high annealing temperature (900 and 1000ºC) for 15 min have the highest J_{ce} values (>150 A/cm^2) at 36 K although they were not the highest T_c value among the studied wires. The annealing temperature increases 750ºC to 900ºC for 15 min, the size of the pores rises up to max. 30 µm and the surface morphology of the wires at the various annealing temperature and duration have almost the same up to 800ºC. It can be concluded that the rapidly annealing process has no negative effect on the electrical and morphological properties of the superconducting wires and this process may be possible for coil fabrication by using long wire to anneal rapidly in continuous systems with the React&Wind method.

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