Microstructure Dependence of Mechanical Property of Commercial MgB₂ Composite Wires

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Synopsis: It is known that four kinds of MgB₂ composite wires have been commercialized. The present study aims to compare the difference of microstructure dependence of mechanical property of those wires. The volume fraction of MgB₂ filaments in the wire was designed in the range between 0.1 and 0.19. The voids existing in MgB₂ filaments distributed in the scale smaller than a few ten micron meter. The tensile test was carried out at room temperature. Young's modulus, 0.2 % proof stress and strain were determined. The calculated Young's modulus was larger than the observed one for all four different wires. This discrepancy was attributed to the poor sintering of MgB₂ filaments as the existence of voids. This situation was proved by the numerically calculated results. A way to realize the higher ductility MgB₂ wire was proposed. The proof stress and strain shall be enhanced by replacing to other constituent metallic element with high thermal expansion coefficient and high Young's modulus. The further requirement is to densify MgB₂ polycrystalline filaments for reducing the void fraction.

Keywords: magnesium diboride, Young's modulus, 0.2 % proof stress, voids

1. Introduction

Magnesium diboride (MgB₂) wire is used in a variety of commercial application under liquid helium-free condition: magnetic resonance imaging¹⁻⁴⁾, fault current limiters⁵⁾, transformers⁶⁾, motors⁷⁾, generators⁸⁾, adiabatic demagnetization refrigerators⁹⁾, superconducting magnetic energy storages¹⁰⁾, cables¹¹⁾ and high-energy physics applications^{12,13)}. So far MgB₂ has good potentiality for various applications mentioned above due to its much higher critical transition temperature (39 K), the light weight of constituent elements and their abundances in the earth's crust, and the relatively easier manufacturing processes of conductors to use in cables and magnets¹⁴⁻¹⁶⁾.

To realize its industrially available wire in practice, however, the following issues shall be still overcome: long-length wire fabrication and mass production with desirable current carrying

capacity, homogeneity along the conductor, and stability performance¹⁷⁻¹⁹⁾. In practice, MgB₂ is brittle intermetallic compound and is not possible to deform plastically by itself. Therefore, practical SC wires shall be fabricated as a composite with several metallic components in order to manufacture a long wire. Similar situations have been experienced when fabricating the commercial Al₅²⁰⁾, BSCCO²¹⁾ and REBCO²²⁾ wires. Further the conjugation with metallic elements brings other advantages of electromagnetic stabilization and so on²³⁾.

Till now, four kinds of composite MgB₂ wire are commercialized²⁴⁻²⁷⁾. In the present study, the difference of microstructures in those commercialized composite wires have been clarified. In particular, the distribution of voids existing in MgB₂ filaments was precisely studied, which influence both mechanical and electromagnetic properties. The mechanical property is an important parameter to certify stability against the hoop stress generating in magnetic coil under high magnetic field. Through the tensile test, the mechanical properties of Young's modulus and 0.2 % proof load were determined. Their results were compared among four different MgB₂ wires for making clear the microstructure dependence. Further the Young's modulus was numerically evaluated by using the linear summation rule and the results were compared with the observed ones. Comparing both observed and calculated Young's moduli, we could get information on the degree of sintering of MgB₂ filaments.

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2. Experimental Procedure

2.1 Commercial MgB₂ wires

The composite MgB₂ wires are commercialized by four manufacturers by different ex-situ method (Columbus) and in-situ one (Sam-Dong, Hyper Tech and Hitachi). Their general cross sectional view is shown in **Fig. 1**. The filament (1) indicates a sintered MgB₂ component, which is surrounded by the inner sheath for preventing any chemical reaction with the matrix. In some cases, the filament (2) consisting of metallic component is installed in order to enhance the mechanical property as a whole. The outer sheath is important to keep uniform superconducting and mechanical properties of such long wire. The practical construction for four kinds of commercialized MgB₂ wires is listed in **Table 1** and **Fig. 2**.

2.2 Microstructure analysis

The wire sample was mounted in the thermosetting resin and polished finally to get mirror surface by using the milling machine IM3800 PLUS (Hitachi High-Tech). By using the SEM microscope, the secondary electron and characteristic X-ray images were observed. For measuring void fraction in the sintered MgB₂ component, the back scattered electron image was carefully analyzed.

2.3 Mechanical test

The tensile test at room temperature was carried out by using the testing machine Shimadzu AG-50kNIS. The stress vs strain curve was continuously recorded up to about 1 % elongation. The detail of the present procedure was followed to the standard process²⁸⁾. For measuring the strain precisely, the paired Nyilas type strain gauge was attached on the center part of the wire

sample. The 0.2 % proof point ($A_{0.2}$, $R_{0.2}$) as well as the Young's moduli (E_0 , E_u) were evaluated.

3. Experimental Results

3.1 Microstructure of MgB₂ composite wires

Fig. 2 shows the backscattered electron image of respective MgB₂ composite wires. The area fraction of each component was measured. Their result is listed in **Table 1**.

As the present MgB₂ composite wire consists of each long continuous component, its volume fraction is equal to the observed area fraction. The volume fraction of MgB₂ component has been designed in the range between 0.1 and 0.19. The highest volume fraction is found in Hitachi wire and the lowest one is in Hyper Tech one. The result on the volume fraction of each component is summarized in **Table 1**. Only Hyper Tech includes the filament 2 (Cu). Different kinds of matrix material are used for different wires, which influence mechanical properties as mentioned in the following section. The same kind of outer sheath is utilized for all the present commercial MgB₂ wires.

Fig. 3 shows the back scattered electron image inside the MgB₂ filament area. The filament has been produced through the sintering of MgB₂ powder or a mixture of Mg and B ones. The black dots are voids surrounded by MgB₂ grains. The size of voids distributes in the scale smaller than a few ten micron meters. The size distribution and volume fraction look to differ for different wires, respectively. At least, however, two dimensional void density is in the range between $4\text{--}25 \times 10^4 \text{ mm}^{-2}$ and the one dimensional size (α) distributes between $0.1\text{--}10 \mu\text{m}$. However the contour of void image was not so clear because Mg and B are light elements and the void size is relatively small for getting high

Fig. 1 General view of cross section of MgB₂ composite wires.

Fig. 2 Cross-sectional view of four MgB₂ wires.

Table 1 Components and their volume fraction of the composite wires.

Manufacturer	Filament (1)	Filament (2)	Inner Sheath	Matrix	Outer Sheath
Columbus ²⁴⁾	MgB ₂ (0.160)	-	-	Ni (0.314)	Ni-Cu-Fe (0.525)
Sam Dong ²⁹⁾	MgB ₂ (0.132)	-	Nb (0.165)	Cu (0.320)	Ni-Cu-Fe (0.382)
Hyper Tech ³⁰⁾	MgB ₂ (0.099)	Cu (0.149)	-	Nb (0.369)	Ni-Cu-Fe (0.382)
Hitachi ³¹⁾	MgB ₂ (0.189)	-	Fe (0.296)	Cu (0.224)	Ni-Cu-Fe (0.291)

Fig. 3 Cross sectional surface of the MgB₂ filament area.

resolution. As mentioned later, we discussed how the void distribution affects the elastic property with the help of the graphical expression. It is suggested that their different distribution of voids is pointed out to relate with each different sintering schedule. The existence of voids influences the mechanical behavior, which differs from that of the fully sintered polycrystalline MgB₂ bulk as mentioned later.

3.2 Result of room temperature tensile test

The result of tensile test at room temperature is reported here. A typical stress vs strain curve for the Columbus wire is shown in **Fig. 4(a)**, where the left graph is the enlarged picture up to 0.2 % elongation and the right is the whole R vs A curve. The straight dotted line is drawn along the initial part. The dotted line deviates soon from the observed curve over about 0.03 % elongation, over which the elasto-plastic deformation starts to take place. The behavior of full plastic deformation is observed in the strain region over the 0.2 % proof point. Some parameters representing the characteristics of the stress vs strain curve were determined as Young's moduli, E_0 and E_u and the 0.2 % proof stress and strain. Their result is indicated in **Table 2**.

Fig. 4 Stress vs strain curve at room temperature for four kinds of commercial MgB₂ wires.

Fig. 4(b) gives the stress vs strain curve for the Sam Dong wire. When comparing with the Columbus data, the elasto-plastic deformation region takes place up to 0.8 %. The result for the Hyper Tech wire is indicated in **Fig. 4(c)**. The elasto-plastic behavior is observed in the strain region over the 0.2 % proof

point. Even in the plastic deformation region, the stress increases gradually with increasing strain.

The result for the Hitachi wire is indicated in **Fig. 4(d)**. The elasto-plastic behavior appears in the strain region over the 0.2 % proof point. In the plastic deformation region, the stress increases gradually with increasing strain.

For obtaining accurate results, the measurement was repeated five times by using different samples from the same wire. The average value and the standard deviation were calculated as shown in Table 2. This requirement is based on the

Table 2 Summary of mechanical test at room temperature.

Sample #	E_0 (GPa)	E_u (GPa)	$R_{p0.2}$ (MPa)	$A_{0.2}$ (%)
Columbus-1	166	166	216	0.333
Columbus-2	172	168	217	0.340
Columbus-3	162	166	216	0.331
Columbus-4	169	167	216	0.320
Columbus-5	164	166	218	0.345
average	166	167	217	0.334
standard deviation	3.5	0.8	0.8	0.008

Sample #	E_0 (GPa)	E_u (GPa)	$R_{0.2}$ (MPa)	$A_{0.2}$ (%)
SamDongHT1	109	105	174	0.359
SamDongHT2	100	106	169	0.370
SamDongHT3	107	106	173	0.372
SamDongHT4	99	100	170	0.374
SamDongHT5	91	102	168	0.366
average	101.2	104	171	0.368
standard deviation	6.6	2.4	2.5	0.005

Sample #	E_0 (GPa)	E_u (GPa)	$R_{0.2}$ (MPa)	$A_{0.2}$ (%)
HyperTech-5	116	126	213	0.388
HyperTech-6	105	125	214	0.391
HyperTech-7	125	127	207	0.368
HyperTech-8	129	125	209	0.368
HyperTech-9	118	125	207	0.367
average	119	126	210	0.376
standard deviation	8.2	0.8	3.0	0.010

Sample #	E_0 (GPa)	E_u (GPa)	$R_{p0.2}$ (MPa)	$A_{0.2}$ (%)
Hitachi-2	132	142	208	0.353
Hitachi-3	138	144	204	0.347
Hitachi-4	143	145	205	0.346
Hitachi-5	145	147	207	0.341
Hitachi-6	139	143	211	0.357
average	139	144	207	0.349
standard deviation	4.5	1.7	2.4	0.005

standardization procedure mentioned in IEC-TC90²⁸⁾. When looking at the standard deviation for both E_0 and E_u , the E_u 's value is lower than the E_0 's one. So hereafter the E_u is taking into consideration for discussion. The result is summarized in **Table 3**. The calculated Young's modulus is attached here. It is interesting that the Young's modulus differs largely depending on the wire prepared by each manufacturer. For all wires, the observed E_{obs} is smaller than E_{cal} , which is numerically obtained.

As shown in Table 3, 0.2 % proof stress resulted in a large change depending on the sort of tapes, while the 0.2 % proof strain keeps a similar value. This large change of 0.2 % proof stress correlates with the amount of Cu content. Comparing two tapes of Sam Dong and Hyper Tech, a large 0.2 % proof stress results in the less amount of Cu. Similar tendency meets for Hitachi wire. Those facts might link to the easier plastic deformation of Cu in the small strain region.

3.3 Analysis of Young's modulus

The present MgB₂ composite wires consist of continuous long components. Due to such characteristic structure, it is reasonable to evaluate the Young modulus by the Voigt rule as indicated,

$$E_{cal} = \sum f_i E_i \quad (1)$$

where f_i and E_i are volume fraction and Young's modulus of each component. Their necessary data are listed in Tables 1 and 4, respectively.

The calculated result is listed in Table 3. It looks that all the calculated Young's moduli are larger than the observed ones. Those discrepancy is suggested to occur relating to the term of volume fraction of MgB₂ filament. The MgB₂ filament part has

Table 3 Summary of tensile test for four kinds of commercial wires.

Manufacturer	E_u (E_{obs}) (GPa)	$R_{0.2}$ (MPa)	$A_{0.2}$ (%)	E_{cal} (GPa)	E_{obs}/E_{cal}	na
Columbus	167	217	0.334	198	0.84	0.16
Sam Dong	104	171	0.368	152	0.68	0.32
Hyper Tech	126	210	0.376	147	0.86	0.14
Hitachi	144	207	0.349	181	0.79	0.21

Table 4 Reference data of Young's modulus.

Component	E (GPa)	Reference
MgB ₂	224	[32]
Cu	110	[33]
Nb	103	[33]
Ni	207	[33]
Fe	205	[34]
Monel400	185	[35]

Fig. 5 Distribution of voids in MgB₂ area.

been not fully sintered reaching to 100 % densification, but includes some voids as shown in Fig. 3.

4 Discussion

4.1 Relation between the observed E and the distribution of voids

As listed in Table 3, the observed Young's modulus of MgB₂ filament is smaller than the calculated one. The reason is suggested to be related to the existence of voids in MgB₂ filament as shown in Fig. 3. Here the influence of void distribution is analyzed as follows. At first, a single inclusion with the Young's modulus E_v distributes in the matrix with E_m where its size is expressed as $\alpha \times \beta$ as shown in Fig 5(a). We suppose that the stress R is applied along the horizontal direction. Then the total Young's modulus is given by Eq. (2),

$$E_p = (1-\alpha)E_m + \frac{\alpha E_m E_v}{(1-\beta)E_m + \beta E_i} \Rightarrow (1-\alpha)E_m \quad (2)$$

When the inclusion is replaced to the void with $E_v = 0$, the second term of the right hand side is obtained.

In the next step, a system including fine inclusions is analyzed. An example of $n=4$ is indicated in Fig. 5(b), where the size of each inclusion is settled as the $1/n^2$ area of the original single inclusion given in Fig. 5(a). This means that the integrated total area of fine inclusions is equal to the one inclusion given in Fig. 5(a)

$$E_p = (1-n\alpha)E_m + \frac{n\alpha E_m E_v}{\left(1-\frac{\beta}{n}\right)E_m + \frac{\beta}{n}E_v} \Rightarrow (1-n\alpha)E_m \quad (3)$$

Replacing the inclusions to voids ($E_v=0$), the total Young's modulus is given by the second term of the right hand side equation.

When comparing both equations of (2) and (3), the total Young's modulus is made clear to become smaller when the inclusions (voids) distribute finer. The E_p given by Eqs. (2) and (3) is the expected Young's modulus from the observation. Thus we may put as follows,

$$\frac{E_{obs}}{E_{cal}} = \frac{E_p}{E_m} = 1 - n\alpha \quad (4)$$

The rough estimation from Fig. 3 gives that one dimensional void density (n) is $0.2-5 \times 10^2 \text{ mm}^{-1}$ and the size (α) is $0.1-10 \times 10^{-3} \text{ mm}$. Their distribution is supposed to obey a log normal distribution function. Then the typical combination of n and α gives the $n\alpha$ in the average value of 0.10 and the 50 % probability range of 0.02-0.5. As shown in Fig. 5(b), the Young's modulus becomes smaller, when the size of voids decreases and the density increases. The $n\alpha$ estimated here is quite close to the observed data listed in Table 3. Thus it is concluded that the fine distribution of voids affects the decrease of Young's modulus. It is suggested that the coarsening of voids is effective during sintering process for improving the Young's modulus, because the area fraction of matrix including no void increase. On the other hand, the larger voids produce incoherent strain around them and influence the mechanical property in the elastic and plastic regions. In the elastic region, its influence has been discussed just in the present context. The increase of such incoherent strain relates to an earlier fracture behavior. As a whole, we suggest an optimum condition about the distribution of voids, in which the sintered MgB₂ elements in the composite wire keep normal Young's modulus and avoid an early fracture.

4.2 Way to realize the higher ductility MgB₂ wire

One of targets to improve the commercial MgB₂ wire is that the 0.2 % proof stress and strain shall be increased up to 600 MPa and 0.6 %, respectively, which have been already achieved for the REBCO wire^{36,37}.

The proposed processes for these requirements are suggested as follows,

(1) Thermal strain in MgB₂ filaments shall be increased for increasing reversible stress and strain. This suggestion might be referred to the report³⁸.

(2) Densification and alignment of MgB₂ filaments shall be improved for increasing critical current density and fracture strain.

In order to increase thermal strain³⁸, one of solutions is to replace the outer sheath material to stainless steel (SUS), which has higher Young's modulus and higher thermal expansion coefficient. The thermal residual strain induced in the MgB₂ filament is given, for instance, in the present case of Hitachi wire,

$$A_{MgB_2}^T = \frac{\{(\alpha_2 - \alpha_1)E_2V_{f2} + (\alpha_3 - \alpha_1)E_3V_{f3} + (\alpha_4 - \alpha_1)E_4V_{f4}\}(T - T_o)}{E_c} \quad (5)$$

where α_i and V_i are thermal expansion coefficient, and volume fraction where 1, 2, 3 and 4 are MgB₂, inner sheath, matrix and outer sheath, respectively as listed in Table 1. The result for the present Hitachi wire is

$$A_{MgB_2}^T = 0.114 \% \quad (6)$$

When replacing Monel 400 to SUS with thermal expansion coefficient of $17.3 \times 10^{-6} \text{ K}^{-1}$ and Young's modulus of 200 GPa, then the thermal residual strain is reduced to

$$A_{MgB_2}^T = 0.061 \% \quad (7)$$

According to the further calculation³⁹, the 0.2 % proof strain, $A_{0.2}$ increases up to the target value of 0.6 % . Recently Tanaka et al⁴⁰ reported the improved MgB₂ composite wire by replacing Monel 400 to stainless steel, where the 0.2 % proof strain reached 0.46 %. Further we need to look for other high performance material for reaching the final target.

5. Summary

At the first step, the difference of microstructure was investigated among four kinds of commercialized composite wires. The volume fraction of MgB₂ component was found in the range between 0.1 and 0.19. The distribution of voids existing in MgB₂ filaments was studied. The voids distributed in the scale smaller than a few ten micron meters.

The tensile test was carried out at room temperature to investigate the mechanical property of those composite wires. Young's modulus, 0.2 % proof stress and strain were determined. The Young's modulus was numerically evaluated based on the linear summation rule. The calculated modulus was larger than the observed one for all four different wires. This discrepancy was attributed to the poor sintering of MgB₂ filaments as the existence of voids. This situation was proved by the numerically calculated results. The fine distribution of voids affects the decrease of Young's modulus. So it is suggested that the coarsening of voids is effective during sintering process for improving the Young's modulus.

The way to realize the higher ductility MgB₂ wire was considered. It was suggested that the proof stress and strain shall be enhanced by replacing to another constituent metallic element with high thermal expansion coefficient and high Young's modulus. The second requirement is to densify MgB₂ polycrystalline filaments for reducing the void fraction and achieving better grain connectivity.

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