Microstructure and texture evolution of pure niobium in cold-drawn Mg+B/Nb/Cu wires

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Abstract
In this study, Mg+B wires were prepared by powder in tube method using Nb and Cu tubes as barrier and sheath, respectively, followed by cold drawing. Microstructural, textural, and mechanical properties of the Nb barrier at different drawing strains ($\varepsilon_d$) were investigated. The results showed that the Nb barrier demonstrated a saturation hardness of 159.4 HV. The microstructure of the Nb barrier became elongated along the drawing direction increasing $\varepsilon_d$. Sub-grains existing inside the deformed grains rotated from low-angle grain boundaries to high-angle grain boundaries and developed into new grains. The main textural components of the Nb barrier were {111} $\gamma$-fiber and {hkl} (110) $\alpha$-fiber. Recrystallized grains exhibited a low maximum orientation distribution function intensity, weak {100} (110) $\alpha$-fibers, and strong {111} (110) $\gamma$-fibers as compared to those of the deformed grains. The relationship between the microstructure evolution and mechanical properties of the Nb barrier and the changes in the cross-sectional area fractions of the materials constituting the Mg+B composite wire are discussed. The current study provides details about the misorientation profile inside deformed grains and continuous dynamic recrystallization mechanism of the cold-drawn Nb barrier.

1. Introduction

Niobium has a body centered cubic (BCC) structure and unique properties such as being non-magnetic, chemical compatibility, and high ductility at cryogenic temperatures. Materials such as Nb$_3$Sn and NbTi that contain Nb as the main element exhibit superconductive properties below 20 K. MgB$_2$/Nb/Cu wire, (with $T_c$ of 39 K) in which Nb is used in bulk, exhibits the highest critical temperature among materials incorporating Nb [1–3]. In order to use MgB$_2$/Nb/Cu wire for a superconducting magnet for magnetic resonance imaging (MRI) machines, an appropriate technique for fabrication of long-length wire is required.

Powder-in-tube (PIT) is one of the methods for manufacturing MgB$_2$/Nb/Cu wire that is several kilometers in length [4]. In this method, a mixture of mixed Mg+B powders (Mg+B core) is filled into Nb/Cu tubes, with Nb and Cu tubes used as barrier and sheath, respectively, and the composite is converted into a wire by a multi-pass of cold-drawing process. This extensive cumulative strain on the barrier in wire making causes the formation of variety of microstructures, which alter mechanical properties. The resulting high strength of the multi-pass deformed barrier improves superconducting properties of MgB$_2$ wire by increasing the powder filling density of the core [5]. Studies have reported that MgB$_2$/Nb composite wires manufactured using cold-drawing are continuous; however, non-uniform interfaces are observed between the Mg+B core and the Nb barrier in wires deformed under high strains [6–8]. Q. Wang et al [7] carried out bending tests on a MgB$_2$/Nb/Cu wire and observed a rapid degradation in critical current density ($I_c$) due to cracking in the barrier around non-uniform MgB$_2$/Nb interfaces during the bending. Therefore, barrier stability at large deformations is essential for improving the formability and superconducting properties of wires. However, few studies have investigated Nb barrier properties such as microstructure and hardness.

In this study, we investigated microstructural evolution of the Nb barrier in cold-drawn Mg+B/Nb/Cu wires and hardness variation with increasing cumulative strain; we also discuss the relationship between Nb barrier microstructure and the stability of the MgB$_2$/Nb interface.
Table 1. Chemical composition (ppm) of Nb.

| Nb   | Ti  | C   | O   | W   | Fe  | Mo | N   | H   | Ta | Si | Ni |
|------|-----|-----|-----|-----|-----|----|-----|-----|----|----|----|
| Bal. | <1  | 29  | 92  | 39  | 4   | <10| 1   | 5   | 356| <1 | 1  |

Table 2. Summary of the diameter, reduction of area, and drawing strain of Mg+B/Nb/Cu in each pass of drawn wires.

| Pass | D (mm) | RA (%) | ε          |
|------|--------|--------|------------|
| 0    | 25     | 0      | 0          |
| 7    | 15.57  | 61.2   | 0.95       |
| 15   | 8.92   | 87.3   | 2.06       |
| 23   | 5.46   | 95.2   | 3.04       |
| 30   | 3.37   | 98.2   | 4          |
| 37   | 2.07   | 99.3   | 4.98       |
| 44   | 1.27   | 99.7   | 5.96       |

2. Experimental

The chemical composition of Nb used as a diffusion barrier in this study is presented in table 1. The outer/inner diameters of the Nb and Cu tubes were 21/16 and 25/22 mm, respectively. Mg (~75 μm) and B (~200 nm) powders were mixed in Mg:B ratio of 1:2. The mixed Mg+B powder was filled in a Nb/Cu tube at an initial powder filling density of 30% with respect the theoretical density (Mg+2B; 2.01 g cm⁻³) followed by sealing. The Mg+B/Nb/Cu billet with an initial diameter of 25 mm was drawn into a Mg+B/Nb/Cu wire with a final diameter of 1.27 mm and 43 passes at room temperature. Reduction in area (RA) can be calculated as follows:

\[ \text{RA} = \frac{A_0 - A_i}{A_0} \times 100 \]  

where \( A_0 \) and \( A_i \) are the cross-sectional areas of the initial and drawn wires after \( i \) drawing passes, respectively. Cumulative strain (namely, \( \varepsilon_{\text{cum}} \)) is defined as follows:

\[ \varepsilon_{\text{cum}} = \ln \left( \frac{A_0}{A_i} \right) = 2 \ln \left( \frac{D_0}{D_i} \right) \]  

where \( D_0 \) and \( D_i \) are the diameters of the initial and drawn wires after \( i \) drawing passes, respectively. The detailed drawing schedule, diameter, and RA of the drawn wire are provided in table 2.

Vickers micro-hardness (HV) of the cold-drawn Nb barrier was measured using a Vickers hardness tester (HM-221, Mitutoyo Co., Ltd, Kanagawa, Japan) under a load of 500 g for 10 s.

The microstructures of the drawn Nb barrier were analyzed by electron back-scattering diffraction (EBSD). EBSD using a field-emission scanning electron microscope (FE-SEM) equipped with a TESL SEM Laboratores (TSL) EBSD system. To prepare the samples for EBSD, the surfaces of the samples were mechanically ground using SiC paper, followed by cross-sectional ion beam milling (Ion Milling System, E-3500, Hitachi, Japan). The measurement plane for EBSD was observed in the longitudinal section parallel to the drawing axis. The EBSD scanning step size was 0.2 μm. EBSD data were processed using TSL-orientation imaging microscopy analysis software. EBSD results from the drawing direction (DD)-the radial direction (RD) planes of the cold-drawn Nb barrier, which is the longitudinal section, are shown in figure 1. The grain orientation spread (GOS) method was used to determine the fraction of the dynamic recrystallized (DRXed) grains. The grains having a GOS value of \( \leq 2.5^\circ \) were considered as DRXed grains.

3. Results

3.1. Area fraction

Figure 2 shows the changes in the area fractions of the Mg+B core, Nb barrier, and Cu sheath calculated from the cross-sectional image of the Mg+B/Nb/Cu wires according to \( \varepsilon_{\text{cum}} \). Insert shows the cross-sectional images of the Mg+B/Nb/Cu wires at \( \varepsilon_{\text{cum}} = 2.06, 4, \text{and} 5.96 \). In \( \varepsilon_{\text{cum}} \) range of 0–2.06, the area fraction of the Nb barrier increased from 33 to 42%, whereas that of the Mg+B core decreased from 41 to 33.1%. It was considered that in the Mg+B core with a low powder filling density of 30%, voids were reduced by the compressive stress applied during cold drawing, thereby increasing the powder filling density of the Mg+B core. In \( \varepsilon_{\text{cum}} \) range of 2.06–4, the area fractions of all materials were almost constant. In \( \varepsilon_{\text{cum}} \) range of 4–5.96, the area fraction of the Nb barrier decreased from 40 to 29%, whereas that of the Mg+B core increased from 33.8 to 36.3%. Additionally, the interface between Mg+B core and Nb barrier became non-uniform as increasing drawing strain.
3.2. Microstructure evolution

To understand the change in the area fraction changes of the cold-drawn Nb barriers following different εd, we initially focused on the variation in microstructure of Nb. Figures 3(a)–(d) shows the inverse pole figure (IPF) maps of the as-received and drawn Nb barriers at different drawing strains. The inset shows a standard stereographic triangle. The color of the grains is correlated with the standard inverse pole figure stereographic triangle. The as-received Nb barrier exhibited an equiaxed microstructure, the average grain size and aspect ratio were 96.2 μm and 0.53, respectively (figure 3(a)). In addition, there was no color gradient inside these grains, reflecting the completely recrystallized as-received Nb.

The microstructure of the Nb barrier at εd = 2.06 consists of grains with a lamellar structure aligned parallel to the DD (figure 3(b)), which is typically observed in cold-deformed BCC metals [9, 10]. The grains with lamellar structure were gradually refined and thinly elongated along the DD, the average grain size from equivalent diameters and aspect ratio decreased from 14.2 to 6.9 μm and 0.16 to 0.13 with increasing in the εd from 4 to 5.96, respectively (figures 3(c) and (d)). Furthermore, these grains had a high color gradient, which indicated that the dislocation density accumulated inside the grains caused lattice distortion, thereby increasing the misorientation angle.

Figure 4(a) shows the fractions of high angle boundaries (HABs-misorientation angle larger than 15°), intermediate angle boundaries (IABs-misorientation angle between 5 and 15°) and low-angle boundaries (LABs- misorientation angle less than 5°) of the Nb barriers at different drawing strain. The fraction of HABs rapidly decreased, whereas that of LABs and IABs increased at a drawing strain of 2.06. The fraction of LABs gradually decreased, whereas that of IABs and HABs increased with a further increase in the drawing strain. Figure 4(b) shows geometrically necessary dislocation (GND) density and fraction of DRXed grains of the Nb barriers at different drawing strains. The GND density of the Nb barrier gradually increased with increasing drawing strain, indicating increasing lattice curvature and stored energy in the Nb barrier. The fraction of
Figure 3. Inverse pole figure (IPF) maps of the (a) as-received Nb barrier, and Nb barriers with a drawing strain of (b) 2.06, (c) 4, and (d) 5.96.

Figure 4. (a) Fraction of high-, intermediate- and low-angle boundaries and (b) Average GND density and fraction of recrystallized grains of Nb barriers at different drawing strains.
DRXed grains greatly decreased due to the development of a deformed structure at a drawing strain of 2.06, but its fraction gradually increased as the drawing strain increased. It was considered that the sub-grains (=LABs) existing inside the grains with lamellar structures rotated from the IABs to the HABs and developed into new grains due to increased dislocation density.

3.3. Texture evolution
The texture evolution of \{110\} in Nb barriers at different \(\varepsilon_d\) was examined, and the results are shown in figures 5(a)–(d). The \{110\} //DD texture intensities of the Nb barriers strongly developed with an increase in \(\varepsilon_d\). The results of the \{110\} pole figure are similar to those of the asymmetrically deformed BCC metals \[10, 11\], and \(\alpha\)-fiber (DD // \{110\}) is evidently developed.

Cold deformed BCC metals generally form \(\alpha\)- and \(\gamma\)-fibers, and Nb with a BCC structure exhibits important fiber textures in Euler space (\(\varphi_1 = 0–90^\circ\), \(\Phi = 0–90^\circ\), \(\varphi_2 = 45^\circ\)) \[9, 12\]. Orientation distribution function (ODF) was expressed using Miller index \(\{hkl\}\langleuvw\rangle\) using the formula described by Kou et al \[13\]. Figures 6(a)–(d) shows the ODF contours (\(\varphi_2 = 45^\circ\)) of the as-received Nb barrier and Nb barriers at the drawing strains of (b) 2.06, (c) 4, and (d) 5.96. Ideal orientation positions for the main texture components are shown in (e).

**Figure 5.** \{110\} Pole figures of the (a) as-received Nb barrier, and Nb barriers with a drawing strain of (b) 2.06, (c) 4, and (d) 5.96.

**Figure 6.** ODF contours (\(\varphi_2 = 45^\circ\)) of the (a) as-received Nb barrier and Nb barriers at the drawing strains of (b) 2.06, (c) 4, and (d) 5.96. Ideal orientation positions for the main texture components are shown in (e).
progressive sub-grain rotation that exceeds a misorientation angle of 10°. Features consistent with this mechanism were experimentally observed via the EBSD misorientation profile owing to their rapid recovery after large strains. 

\[ \theta = \text{value converted to radians from the accumulated misorientation angle}, \quad b = \text{Burgers vector} \]

\[ \text{s} = \text{distance} \]

In addition, the texture of the Nb barrier was rotated along the \( \varphi_1 \)-axis (\( \Phi = 55^\circ \)) in the range from 0 to 60°, which represented a \{111\} \( \{110\} \) \( \gamma \)-fiber parallel to the normal direction. \( \alpha \)- and \( \gamma \)-fibers developed with an increase in \( \varepsilon_d \) from 2.06 to 5.96, and the maximum ODF intensity orientation shifted from \{112\} \( \{110\} \) to \{100\} \( \{110\} \).

For a better understanding of the texture evolution, the main BCC fiber textures were calculated and plotted in figure 7. The texture of the Nb barrier exhibited a strong \{111\} \( \{110\} \) \( \gamma \)-fiber and a weak \{100\} \( \{110\} \) \( \alpha \)-fiber with an increase in \( \varepsilon_d \) from 4 to 5.96. P. Qu et al. [9] applied accumulative roll bonding (ARB) to Ti/Al/Nb composite and found a weak \( \alpha \)-fiber and strong \( \gamma \)-fiber in ARB-processed Nb sheet. For BCC metals, it is well known that the effect of recrystallization is to sharpen the \( \gamma \)-fiber at the expense of the \( \alpha \)-fiber [14].

4. Discussion

4.1. Microstructure and texture evolution by CDRX

Nb is a representative metal with a high SFE (200 mJ m\(^{-2}\)) than Al (166 mJ m\(^{-2}\)), Mg (125 mJ m\(^{-2}\)), and ferritic iron (180 mJ m\(^{-2}\)) [15, 16]. Cold-deformed metals with high SF Es easily undergo Continuous DRX (CDRX) owing to their rapid recovery after large strains [17, 18]. The CDRX nucleation mechanism is activated by progressive sub-grain rotation that exceeds a misorientation angle of 10°–15° within the grains. Microstructural features consistent with this mechanism were experimentally observed via the EBSD misorientation profile [19].

To understand the behavior of CDRX occurring inside the grains with lamellar structures, point-to-point (local) and point-to-origin (cumulative) misorientations were calculated in the regions labelled ‘A’ and ‘B’ in figures 3(c) and (d), magnified at a higher magnification in figures 8(a) and (b), respectively. Figures 9(a)–(d) shows the misorientation profiles along arrows in figures 8(a) and (b). Here, A1–B1 and A2–B2 represent directions parallel and perpendicular to the elongated direction (DD) of the deformed grains, respectively. Figures 9(a)–(d) shows the misorientation profiles along arrows in figures 8(a) and (b). The local misorientation (black line) for all arrows was less than 10°, implying that there were no other grains inside the existing grain. The cumulative misorientation (red line) of the Nb barrier at \( \varepsilon_d = 4 \) exceeded 12° only along the A2 arrow, indicating that the gradual sub-grain rotation was well developed perpendicular to the DD. The cumulative misorientation of the Nb barrier at \( \varepsilon_d = 5.96 \) for all arrows surpassed 12°, implying that the gradual sub-grain rotation was established in directions parallel and perpendicular to the DD of the deformed grain. As the microstructure evolution is closely related to the change in dislocation density, the GND density was calculated using equation (3):

\[ \text{GND} = \frac{\theta}{b\delta} \]

where \( \theta \) is the value converted to radians from the accumulated misorientation angle, \( b \) is the Burgers vector (For Nb: \( b = 0.29 \text{ nm} \)) [20], and \( \delta \) is the distance. The GND density is higher when the accumulated misorientation angle is larger and \( \delta \) is shorter. Cumulative misorientation along the A1 and A2 arrows were 4.4° at 20 \( \mu \text{m} \) and 48.4° at 7.8 \( \mu \text{m} \), respectively, and the calculated GND for the A2 arrow (37 \( \times \) \( 10^{13} \text{ m}^{-2} \)) was approximately 37 times that along A1 arrow (1 \( \times \) \( 10^{13} \text{ m}^{-2} \)). The microstructure of the Nb barrier at \( \varepsilon_d = 4 \) developed into
elongated coarse grains with an increase in εd because the deformed grains with relatively low dislocation density along the A1 arrow do not undergo CDRX. The cumulative misorientations for the B1 and B2 arrows were 12.3° at 8.8 μm and 12.9° at 2.1 μm, respectively. The calculated GND density for the B1 arrow \( (8 \times 10^{13} \text{ m}^{-2}) \) was approximately eight times that along the A1 arrow, and the GND density for the B2 arrow was equal to that along the A2 arrow. These results showed that CDRX occurred along the B1 and B2 arrows at εd = 5.96. S Y Park et al \[21\] applied high-ratio differential speed rolling (HRDSR) to a pure Nb sheet at thickness reduction ratio of 80% and found grain refinement by CDRX occurred, which indicates that high shear deformation was induced by HRDSR. However, while the drawing process imposes a lower shear deformation on the sample than HRDSR, drawn Nb in this experiment has a large amount of dislocation density due to the higher cumulative deformation than HRDSR. It is considered that the high dislocation density acted as the driving force of CDRX.

To compare the textural behaviors of the grains recrystallized by CDRX (=CDRXed grain) and the deformed grains in figure 3(d), the EBSD data for the CDRXed and deformed grains were separately extracted and
analyzed. Figures 10(a and c) and (b and d) show the EBSD maps and corresponding ODF sections ($\varphi_2 = 45^\circ$) of Nb at $\varepsilon_d = 5.96$: (a) and (b) the deformed grains only and (c) and (d) the recrystallized grains only, which have a lower texture intensity.

Figure 10. EBSD maps and corresponding ODF sections ($\varphi_2 = 45^\circ$) of Nb at $\varepsilon_d = 5.96$: (a) and (b) the deformed grains only and (c) and (d) the recrystallized grains only, which have a lower texture intensity.

Figure 11. Vickers hardness of Nb barriers and Mg+B core at different drawing strain.

Figure 11. Vickers hardness of Nb barriers and Mg+B core at different drawing strain.

analyzed. Figures 10(a and c) and (b and d) show the EBSD maps and corresponding ODF sections ($\varphi_2 = 45^\circ$) for the deformed and CDRXed grains of the Nb barrier at $\varepsilon_d = 5.96$, respectively. The main textures of all the grains are \{hkl\} $\langle 110 \rangle$ $\alpha$-fibers and \{111\} $\langle 110 \rangle$ $\gamma$-fibers. The texture of the CDRXed grains exhibited weak \{100\} $\langle 110 \rangle$ $\alpha$-fibers and strong \{111\} $\langle 110 \rangle$ $\gamma$-fibers as compared to that of the deformed grains, which is similar to the texture of recrystallized BCC metals [14]. In addition, the CDRXed grains exhibited a relatively low maximum ODF intensity compared to that of the deformed grains. The reason for the decrease in the maximum ODF intensity of the Nb barrier at $\varepsilon_d = 5.96$ in figure 6(d) is that the fraction of DRXed grains with low ODF intensity increased as shown in figure 4(b).

4.2. Vickers hardness

Figure 11 shows the Vickers hardness values of the cold-drawn Nb barriers and Mg+B core at different drawing strains. The hardness of the cold-drawn Nb rapidly increased at $\varepsilon_d = 3.04$, and then maintained with a further increase in drawing strain. The hardness of the Nb barrier did not increase with an increase in $\varepsilon_d$ from 4 and 5.96 likely due to a dynamic balance between continuous dislocation generation during plastic deformation and dislocation annihilation by DRX. The hardness of the Mg+B core increased steadily with increasing drawing strain, which indicates that the Mg+B core was densified. Z. Han et al [22] found that the critical powder filling density of cold drawn powder/metal composite wire was 80%. It is considered that the hardness of the Mg+B
core was steadily increased by the increased powder filling density with an increase in drawing strain, but that of the Nb barrier not increases after $\varepsilon_d = 4$. Thus, cold-drawn Mg+B/Nb/Cu wires at $\varepsilon_d = 5.96$ had a non-uniform interface between the Mg+B core and Nb barrier as shown in figure 2.

5. Conclusions

In this study, the relationship between the microstructural evolution of the Nb barrier and the variation in the cross-sectional area fractions of the materials constituting the Mg+B/Nb/Cu wire was investigated at different $\varepsilon_d$. The main conclusions are summarized below.

1. The cross-sectional area fraction of the Nb barrier decreases after $\varepsilon_d = 4$ and a non-uniform interface between the Mg+B core and the Nb barrier was observed in the cold-drawn Mg+B/Nb/Cu wire at $\varepsilon_d = 5.96$.

2. Grains of the cold-drawn Nb barrier gradually elongated along the DD with increasing drawing strain. Recrystallized grains were observed inside the deformed grains. The sub-grains existing inside the deformed grains rotated from the LABs to the HABs and transformed into new grains. The volume fraction of the HABs and CDRXed grains gradually increased after $\varepsilon_d = 4$.

3. The main texture components of the cold-drawn Nb barrier are $\{111\}\{110\}$ $\gamma$-fibers and $\{hkl\}\{110\}$ $\alpha$-fibers. After an $\varepsilon_d$ of 4, the texture of the Nb barrier exhibited strong $\{111\}\{110\}$ $\gamma$-fibers and weak $\{100\}\{110\}$ $\alpha$-fibers. CDRXed grains demonstrated a low maximum ODF intensity, weak $\{100\}\{110\}$ $\alpha$-fibers, and strong $\{111\}\{110\}$ $\gamma$-fibers as compared to those of the deformed grains.

4. The hardness of the Nb barrier remained constant after $\varepsilon_d = 4$. This steady-state of hardness was due to a dynamic balance between continuous dislocation generation during plastic deformation and dislocation annihilation by DRX.

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Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.

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