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Fabrication of Nb_3Al superconducting wires by utilizing the mechanically alloyed $Nb(Al)_{ss}$ supersaturated solid-solution with low-temperature annealing

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ABSTRACT

High-performance Nb_3Al superconducting wire is a promising candidate to the application of high-field magnets. However, due to the production problem of km-grade wires that are free from low magnetic field instability, the Nb_3Al wires made by rapid heating, quenching and transformation (RHQT) are still not available to the large-scale engineering application. In this paper, we reported the properties of the *in situ* powder-in-tube (PIT) Nb_3Al superconducting wires, which were made by using the mechanically alloyed $Nb(Al)_{ss}$ supersaturated solid solution, as well as the low temperature heat-treatment at 800 °C for 10 h. The results show that Nb_3Al superconductors in this method possess very fine grains and well superconducting properties, though a little of Nb_2Al and Nb impurities still keep being existence at present work. At the Nb_3Al with a nominal 26 at.% Al content, the onset T_c reaches 15.8 K. Furthermore, a series of Nb_3Al wires and tapes with various sizes have been fabricated; for the 1.0 mm-diameter wire, the J_c at 4.2 K, 10 T and 14 T have achieved 12,700 and 6900 A/cm^2, respectively. This work suggests it is possible to develop high-performance Cu-matrix Nb_3Al superconducting wires by directly using the $Nb(Al)_{ss}$ supersaturated solid-solution without the complex RHQT heat-treatment process.

1. Introduction

Since higher upper critical field B_{c2} and much better strain tolerance properties, Nb_3Al superconductor is considered as a promising alternative for Nb_3Sn at high-field applications, such as next-generation ITER DEMO reactor, GHz NMR, and high-energy particles accelerators [1]. However, because of the strict Nb_3Al phase formation conditions, it is still difficult to produce the high-performance Nb_3Al superconducting wires at industrial environment. Extensive efforts have been made to develop the practical Nb_3Al superconducting wires [2–5].

At early studies, Webb et al. firstly reported that a large single-crystal grain of bcc $Nb(Al)_{ss}$ supersaturated solid-solution produced by quenching from under a solidus was able to be rolled into a tape at room temperature and then be transformed to nearly stoichiometric A15 Nb_3Al phase after an additional heat treatment. The resultant Nb_3Al tape showed excellent critical current density J_c, about 1000 A/mm^2 at 4.2 K and 20 T [7]. However, the fabrication of ductile $Nb(Al)_{ss}$ rod was quite difficult, because a relatively slow cooling rate for bulk rods directly formed A15 and Nb_2Al sigma phase at the grains boundary of $Nb(Al)_{ss}$ during quenching process. After that, Lijima et al. invented a rapid heating, quenching and transformation method (RHQT) to prepare the high-performance Nb_3Al superconducting wires by utilizing the Nb/Al composite precursor wires [8]. In this method, the Nb_3Al superconductors are with fine grains and nearly stoichiometric; and consequently they have an excellent T_c, J_c and H_{c2}. By optimizing the RHQT fabrication process, km-grade Nb_3Al wires with extremely high transport J_c have been successfully fabricated. However, for the RHQT way, there are still some unavoidable problems, while it was used to produce the practical Nb_3Al superconducting wires.

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Firstly, the wrap of copper at Nb_3Al superconducting wire is high-cost and time-consuming. Since the maximum temperature of RHQT is up to 2000 °C that is far above the melting point of copper, the copper coat, which is necessary to the preparation of Nb/Al composite precursor wires and also the use of Nb_3Al superconducting wires, should be removed before RHQT process and enwrapped after RHQT process. And especially, the cover of copper to Nb surface is not easy. Secondly, it is well-known that at the RHQT process, the Nb–25 at.% Al composite precursor wire has to be fired from 40 °C to 2000 °C during 0.1 s, and immediately quenched to the room temperature. The above process highly depends on the uniformity of the Nb/Al precursor wires and RHQ heat-treatment equipment. Therefore, in order to fabricate the practical high-performance Nb_3Al superconducting wires, another method is necessary to be developed.

As we know, the key that the RHQT Nb_3Al wires possess excellent properties is the uniform high-quality $Nb(Al)_{ss}$ supersaturated solid-solution. Certainly, besides the liquid-quenching way, mechanical alloying (MA) is another effective approach to prepare the $Nb(Al)_{ss}$ supersaturated solid-solution by utilizing the high-energy ball milling. For example, Peng et al. systematically studied the mechanical alloying of Nb–Al powders, and Nb_3Al superconductors have been successfully produced by an annealing of 825 °C for 2 h [9]. Tachikawa et al. have prepared the $Nb_3(Al, Ge)$ tapes by using the MA powders of raw Nb_2Al and Nb mixtures, with a J_c of over 2.7×10^4 A/cm^2 at 23 T and 4.2 K [10]. In addition, by using the MA Nb–Al powders as well as RHQT heat-treatment, Sumption et al. has successfully fabricated the Nb_3Al superconductor with a T_c of 17–19 K [11]. The above results imply that it is possible to develop Nb_3Al wires by using MA $Nb(Al)_{ss}$ supersaturated solid-solution, combing with powder in tube (PIT) method, which is widely used to fabricate the Bi2223, Bi2212, MgB_2 and Fe-based superconducting wires [12–15]. However, the research about the MA $Nb(Al)_{ss}$ supersaturated solid-solution and PIT Nb_3Al superconducting wires are still not intensive by far. And especially, by using the MA $Nb(Al)_{ss}$ supersaturated solid-solution and PIT method, we could easily produce the Cu-matrix Nb_3Al wires, which largely benefits the practical application of Nb_3Al superconducting wires and simplifying the Nb_3Al wires fabrication process.

Based on the above-mentioned motivations, we detailedly studied the MA method to make $Nb(Al)_{ss}$ supersaturated solid-solution and superconducting properties of the MA Nb_3Al superconductors; and also a series of Nb_3Al superconducting wires and tapes with various sizes had been made by conventional PIT method in this work. The results suggest that the MA Nb_3Al superconductors have very high purities and good T_c and J_c properties; and it is very promising to develop high-performance Cu-matrix Nb_3Al superconducting wires for the use of high-field superconducting magnet by this route.

2. Experimental detail

Niobium (−325 mesh, 99.8% purity, Alfa Aesar) and aluminum (−325 mesh, 99.5% purity, Alfa Aesar) powders with nominal compositions, $Nb_{1-x}Al_x$ ($x = 0.23$, 0.25, 0.26, and 0.27) were mixed by mechanical alloying way, which was carried out in a SPEX 8000 M shaker mill. For each run, the mixture powders of approximately 2.0 g and harden steel balls of 20 g (ball-to-powder weight ratio 10:1) were loaded into a hardened steel vial in an argon-filled glove box and milled for different time (0, 2.5, 5.0 and 10 h). During the milling process, no process control agents were added. After milling, the powders were pressed into cylinder with 10 mm in diameter under 10 MPa pressure; and then the cylinders were sintered at 800 °C or 950 °C for different time in flowing Ar atmosphere. Vacuum annealing of $Nb(Al)_{ss}$ supersaturated solid-solution will result in the loss of Al and thus significant degradation of the Nb_3Al superconducting phase.

The preparation conditions of all the bulk samples and their onset and middle T_c are shown at the Table 1. The AMT-1 sample is without mechanical milling, but with hand milling for 3 h. After annealing, phase compositions and microstructure of Nb_3Al superconductors were analyzed by X-ray diffraction (XRD) and scanning electron microscopy (SEM). The magnetic properties of samples were measured over temperature range of 7–30 K using a physical properties measurement system (9T-PPMS, Quantum Design). The magnetic J_c was calculated from the width, ΔM, of the magnetization loops (M–H) using the Bean model, $J_c = 20\Delta M/[a'(1 - a'/3b')]$, where a' and b' are the dimensions of the sample perpendicular to the direction of the applied magnetic field with $a' < b'$.

Single filament Nb_3Al wires and tapes have been made by conventional powder in tube (PIT) method with an Nb barrier and copper sheath. The powders used at all these wires and tapes are the Nb $(Al)_{ss}$ supersaturated solid solution with 25 at.% Al content and a milling time of 2.5 h. The Nb_3Al superconducting wires and tapes were annealed at 800–850 °C for 2–10 h under a flowing Ar. Transport I_c measurements of wires were carried out at 4.2 K in external magnetic fields up to 15 T by using a standard four-probe technique. A magnetic field was applied both perpendicular to the wires and current flow. The I_c was defined with a criterion of 1 μV cm^{-1} and transport critical current density, J_c, was obtained by dividing I_c by cross-sectional area of the Nb_3Al core.

3. Results and discussion

Fig. 1(a and b) shows the SEM pictures of raw Nb and Al powders. The Nb powders present diverse and irregular shapes and the grains of Al powders are spherical. Both of them have an average grain size of about 10 μm. Fig. 1(c and f) shows the pictures of MA Nb–25 at.% Al powders with different milling times (MT), 1.0 h, 2.5 h, 5.0 h and 10.0 h, respectively. From these pictures, it can be seen that with the increase of milling time, average grain size of the Nb–Al powders obviously decreases. For the MT1.0 h sample shown in Fig. 1(c), most of the grains have a planar shape, which suggests for the MA process, within a short time, alloying of Nb and Al powders mainly depends on the press of ball-to-ball or ball-to-vial. At the MT2.5 h and MT5.0 h, both of them display the quasi-spherical grains, but the latter has much smaller grain size. For the MT10.0 h sample in Fig. 1(f), most of the powders present spheral and very fine grains. Interestingly, some planer grains like those in Fig. 1(c) still exist at the MT10.0 h sample,

Table 1
The Al at.% content, milling time of powders, annealing conditions and onset and middle superconducting transition temperature, T_c for all the bulk Nb_3Al samples.

Samples	Al content (at.%)	Milling time of powders (h)	Annealing conditions (°C/h)	$T_{c,onset}$	$T_{c,mid}$
AMT-1	25	0	800/10	9.8	9.0
AMT-2	25	2.5	800/10	15.3	14.2
AMT-3	25	5.0	800/10	15.4	14.4
AMT-4	25	10.0	800/10	/	/
PMT-1	27	2.5	950/24	14.2	13.0
PMT-2	25	2.5	950/24	14.4	11.8
PMT-3	23	2.5	950/24	13.8	11.2
CMT-1	23	2.5	800/10	15.4	13.0
CMT-2	24	2.5	800/10	/	/
CMT-3	26	2.5	800/10	15.8	14.8
CMT-4	27	2.5	800/10	/	/
RMT-1	24	2.5	800/40	15.6	14.4
RMT-2	27	2.5	800/40	15.5	14.2

$T_{c,onset}$ means the onset superconducting transition temperature.
$T_{c,mid}$ means the mid superconducting transition temperature.

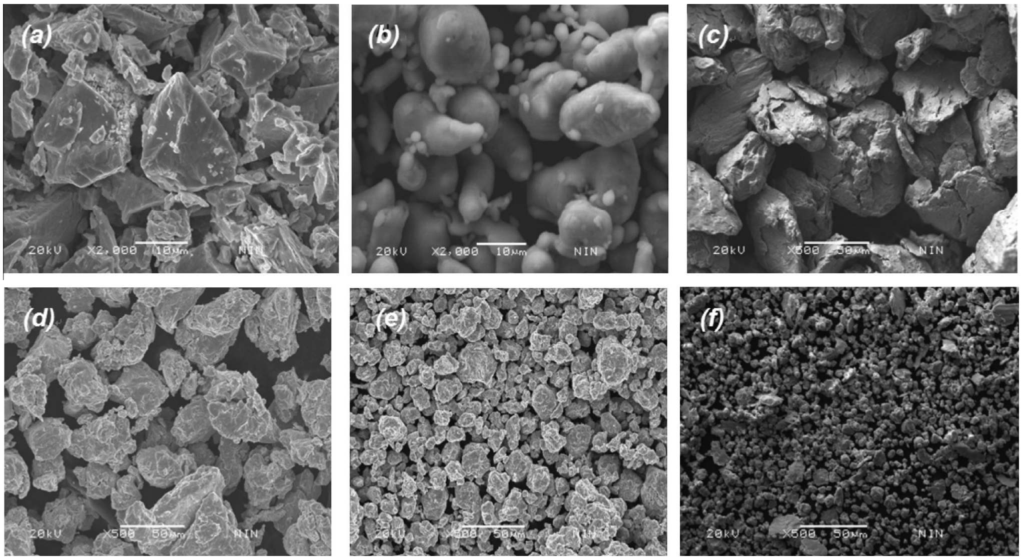

Fig. 1. SEM pictures of different Nb/Al powders; (a) raw Nb powders; (b) raw Al powders, and (c–f) Nb–25 at.% Al powders with milling time of 1.0 h, 2.5 h, 5.0 h and 10.0 h, respectively.

which suggests that planer Nb–Al grains are unavoidable in the MA powders, since Nb and Al powders are soft and ductile.

Fig. 2(a) shows the XRD patterns of MA Nb–25 at.% Al mixture powders with the milling time of 0–10 h. From this picture, it can be seen that after a milling of 2.5 h, the Al peaks disappear, implying that all the Al powders have already been alloyed into Nb matrix. With the increase of milling time, Nb peaks obviously broaden and shift to the higher degree. While the milling time reaches 10 h, the Nb/Al mixture will form the nanocrystalline powders [6], which make against to produce Nb_3Al superconductor. Fig. 2(b) shows the XRD results of these MA powders after a annealing of 800 °C for 10 h. Both in the AMT-2 and AMT-3, Nb_3Al is the main phase, though a little of Nb_2Al and Nb impurities seem to be existing; and intensity of Nb_3Al peaks at AMT-3 is much higher than that at AMT-2. In contrast, at the AMT-1 and AMT-4, only few Nb_3Al phase forms and the main phase is Nb or Nb_2Al. It suggests that mechanical alloying is an effective method to accelerate the formation of Nb_3Al phase, but long-time milling will result in the production degradation of Nb_3Al phase. Clearly, before annealing, for the AMT-2 and AMT-3, the Nb–Al powders are supersaturated solid-solution state, but comparably at the AMT-4 it is quasi-amorphous state. Since the formation of Nb_3Al phase is the Nb–Al diffusion and ordering process, it is reasonable that the AMT-4 is more difficult to form Nb_3Al than the AMT-2 and AMT-3.

Fig. 3 shows the effects of Nb/Al stoichiometry and annealing conditions on phase formation of Nb_3Al superconductor. All the samples at Fig. 3 are with a milling time of 2.5 h. As shown in Fig. 3(a), all the samples have main Nb_3Al phase and a little of Nb_2Al and Nb impurities. With the increase of nominal Al at.% content, the volume of Nb_2Al phase obviously tends to increase. The Nb_2Al impurities content at CMT-4 is obviously higher than the others. In order to know the effect of annealing conditions on the formation of Nb_3Al phase, Fig. 3(b and c) shows the XRD results of these Nb_3Al samples with heat-treatment of 800 °C/40 h and 950 °C/24 h, respectively. From the Fig. 3(b), it can be seen that when prolonging the annealing time, the Nb peaks are clearly weaken; however, there is no changes about the intensity of Nb_2Al peaks both in 24 at.% and 27 at.% Al samples. Fig. 3(c) shows the XRD patterns of PMT-1, PMT-2 and PMT-3, which suggest that the Nb_2Al impurities content largely enhance with the increase of the nominal Al at.% content. Obviously, the PMT-1 with 23 at.% Al has the least Nb_2Al impurities. It is consistent with the Nb–Al binary phase diagram due to the limited solid solubility of Al atoms at Nb matrix [2].

Therefore, the present results suggest that the content of Nb_2Al impurities mainly depends on the nominal Nb/Al stoichiometry.

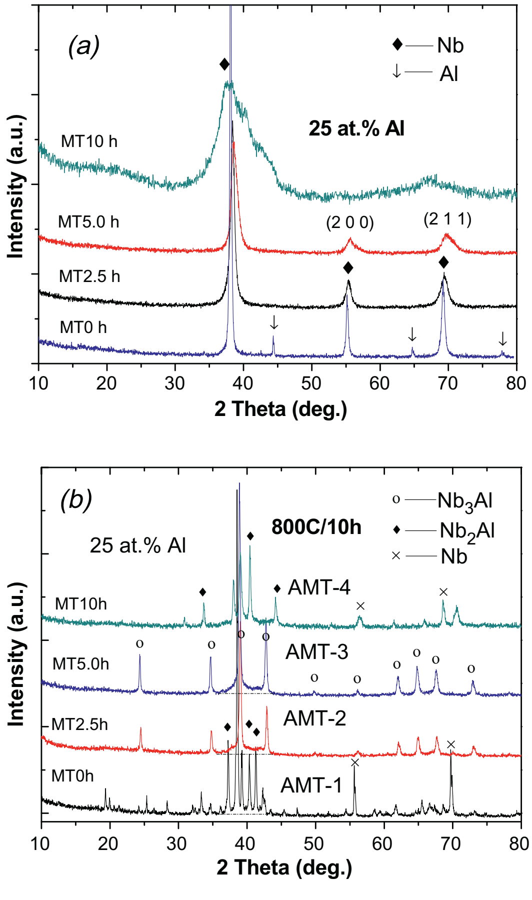

Fig. 2. XRD patterns of mechanically alloying Nb–Al powders and bulk Nb_3Al superconductors; (a) Nb–25 at.% Al powders with different milling times (0–10 h); and (b) AMT-1–AMT-4, Nb_3Al samples with 25 at.% Al content, annealing condition of 800 °C/10 h and different milling times (0–10 h).

Increasing the Nb–Al reaction kinetics conditions could reduce the Nb impurities content, but has few effects on limiting the formation of Nb_2Al impurities. According to the previous reports [2,3], the properties of Nb_3Al superconductor seriously depends on the actual Nb/Al stoichiometry and the contents of Nb_2Al and Nb impurities, so the purity of Nb_3Al superconductor prepared by mechanically alloying still need to be further enhanced. For the PMT-1 sample, it has fewer Nb_2Al impurities than the others, but the nominal 23 at.% Al content is too small and thus restricts its superconducting properties. The preparation conditions and T_c of these samples are shown in Table 1.

Fig. 4 shows the temperature dependence of magnetization of bulk MA Nb_3Al samples. From the Fig. 4(a), it can be seen that the onset T_c of Nb_3Al superconductors both depend on the nominal Nb/Al stoichiometry and the heat-treatment conditions. The T_c at the samples with a heat-treatment of 800 °C/10 h are clearly higher than those at the samples annealed at 950 °C/24 h. It is consistent with the XRD results that the later has much higher Nb_2Al impurities contents shown in Fig. 3. Furthermore, for these samples with same heat-treatment conditions, with the increase of the nominal Al at.% contents, the T_c of Nb_3Al improves and also the superconducting transition width becomes much sharper. For the A15-structure Nb_3Al superconductor, complete Nb_3Al phase

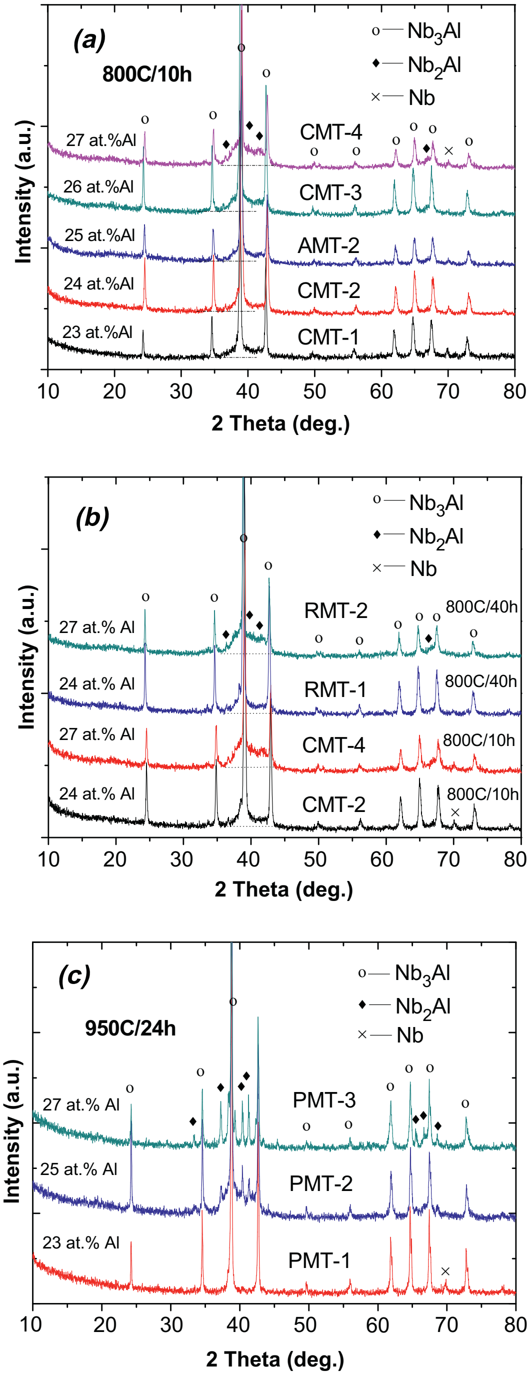

Fig. 3. XRD patterns of mechanically alloying Nb_3Al bulk superconductors; (a) CMT-1–CMT-4 and AMT-3, Nb_3Al samples with annealing condition of 800 °C/10 h and 23–27 at.% Al contents; (b) Nb_3Al samples with 24 at.% and 27 at.% Al contents, annealing condition of 800 °C/10 h and 800 °C/40 h and milling time of 2.5 h; and (c) PMT-1–PMT-3, Nb_3Al samples with 23, 25 and 27 at.% Al contents and annealing condition of 950 °C/24 h.

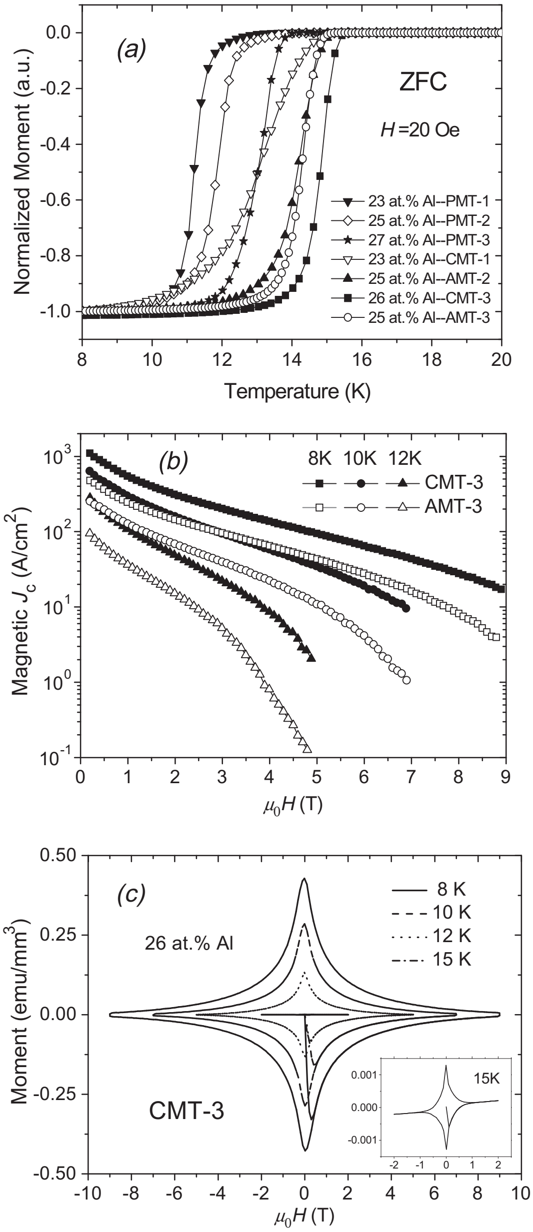

Fig. 4. Superconducting properties of different bulk Nb_3Al superconductors; (a) normalized moment vs. temperature curves of various Nb_3Al samples; the measurement was performed at an applied field of 20 Oe and with zero-field cooling (ZFC); (b) field dependence of magnetic J_c at 8 K, 10 K and 12 K for CMT-1 and AMT-3 samples; and (c) magnetization loop of CMT-1 sample at 8 K, 10 K, 12 K, and 15 K.

yields at the area of 17–22 at.% Al contents according to the Nb–Al binary phase diagram; but its superconducting properties strongly depend on the vacancy volume of Al atoms of Nb_3Al grains lattice [2]. While the actual Al contents is much closer to the 25 at.%, the T_c, J_c and H_{c2} of Nb_3Al are much higher. Therefore, it could be thought that at the PMT-3 and CMT-3 samples, the actual Al contents are more close to 25 at.%, which result in them with better T_c. However, it is noted that the PMT-3 sample is with the higher Nb_2Al impurities content than the CMT-3 sample (seen in Fig. 3(a)), so it will be invalid to the improvement of Nb_3Al superconducting properties by further increasing nominal Al at.% content. At the other hand, comparing the AMT-2 with the AMT-3 sample, it is interesting that although they possess almost same onset T_c, the superconducting transition width of AMT-3 is much smaller than that at the AMT-2, which may be attributing to the AMT-3 with less Nb_2Al impurities.

In order to further illuminate the properties of mechanically alloyed Nb_3Al superconductor, Fig. 4(b and c) shows the magnetic J_c–H curves of the AMT-3 and CMT-3 samples and magnetization hysteresis loops of CMT-3 at different temperatures. For the AMT-3 and CMT-3, they have same superconducting transition width and very similar impurities contents (see Figs. 2(b) and 3(c)), but the CMT-3 obviously has better onset T_c, up to 15.8 K. And for AMT-3, the T_c is 15.4 K. As shown in Fig. 4, the magnetic J_c of CMT-3 is much higher than that of AMT-3 at all the samples. Note that at the higher fields, the degradation of J_c at CMT-3 is much slower, which indicates that the CMT-3 sample has much better flux pinning property than the AMT-3. Since Nb_3Al is a typical grains-boundary pinning superconductor, the better flux pinning property at CMT-3 means it with much smaller grain size. Fig. 4(c) shows the complete M–H curves at 8 K, 10 K, 12 K and 15 K, which suggests that the MA Nb_3Al superconductors have very well low-field stability and without the existence of flux jump.

Fig. 5 shows the typical SEM pictures of the CMT-3 and AMT-3 samples. Both of them are annealed at 800 °C for 10 h. From this picture, it can be seen that at the times of 1000×, both of them are not packed and the grain particles at AMT-3 are much finer. It is attributing that the AMT-3 has much longer milling time. As shown in Fig. 1, the powders at milling time of 5.0 h are much less than that at milling time of 2.5 h. However, comparing to the AMT-3 in Fig. 5(f), the CMT-3 sample at Fig. 5(c) shows much smaller grain sizes, about 20–50 nm, which explains the reason that the CMT-3 possess better flux pinning performance than the AMT-3. The above results imply that while increasing the milling time of Nb–Al powders, the Nb_3Al grains are much easier to be grown. At the higher magnification scale, both of the AMT-3 and CMT-3 samples show a very well grains coupling.

After studying the intrinsic properties of mechanically alloying Nb_3Al superconductors, a series of Cu-matrix single filament Nb_3Al wires and tapes have been made by conventional PIT route. All of them have a thin Nb-barrier. Fig. 6(a) show the transport J_c vs. magnetic fields curves of Nb_3Al wires and tapes at 4.2 K. All the samples possessed nominal 25 at.% Al and were annealed at 800 °C for 10 h except the Φ2.0 mm wires, which were marked in the figure. From this figure, it can be seen that the J_c of Nb_3Al wires are clearly dependent on the diameter of wires and the annealing conditions. It may be attributing to the differences of fabrication process and the density of Nb_3Al superconducting wires. For the Φ1.0 mm wire, it has the optimal J_c–H property, and the transport J_c at 4.2 K and 12 T is up to 11,900 A/cm^2. At the annealing conditions of 850 °C/2 h and 800 °C/10 h, the J_c–H dependence of Φ2.0 mm wires is similar. However, by increasing the annealing time to 10 h at 850 °C, the J_c of Nb_3Al wires would significantly degrade. Fig. 6(b) shows the typical I–V curves of Φ1.3 mm wire at 4.2 K and different magnetic fields. Comparing with the RHQT Nb_3Al wires, the I_c of Nb_3Al wires by mechanically alloying still needs to be further improved at high fields.

In summary, Nb_3Al superconducting wires have been successfully fabricated by PIT method with the low-temperature annealing (800 °C for 10 h) in this work. Considering these Nb_3Al wires without complex RHQT heat-treatment, their transport J_c–H properties are significant. Especially noting that these Nb_3Al wires have a Cu-matrix, which is largely in favor of the thermal stability and practical use of Nb_3Al superconducting wires. At the other hand, comparing the Nb_2Al and Nb impurities contents between the MA and direct low-temperature diffusion Nb_3Al superconductors [16], the MA Nb_3Al is expected to have much better T_c, J_c and H_{c2} properties, due to it with much lower impurities contents. The PIT Nb_3Al superconducting wires will achieve the practical use of high-field superconducting magnets, by optimizing the mechani-

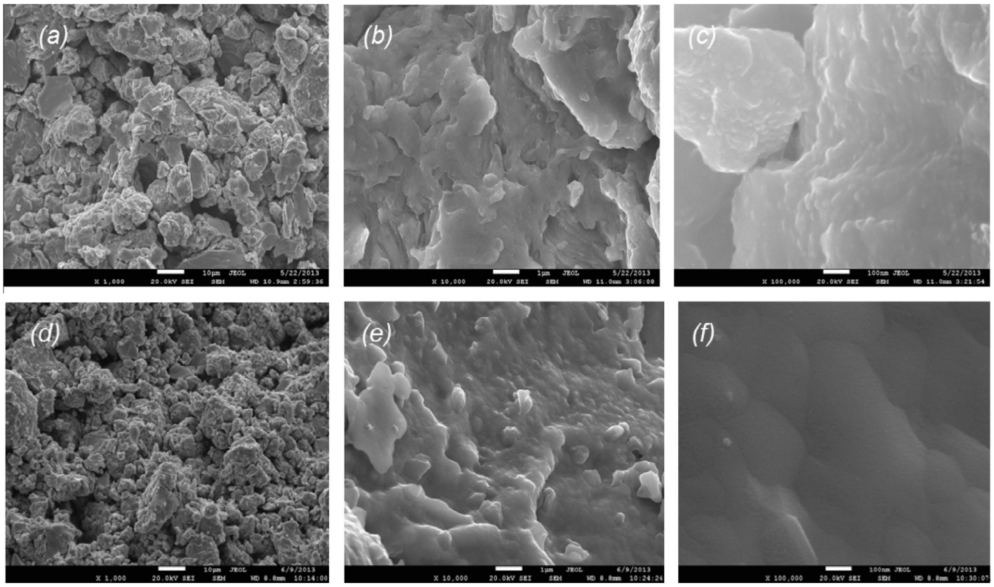

Fig. 5. Typical SEM images of the CMT-3 and AMT-3 samples with magnification times of 1000×, 10,000× and 100,000×; (a–c) are the CMT-3 sample, and (d–f) are the AMT-3 sample.

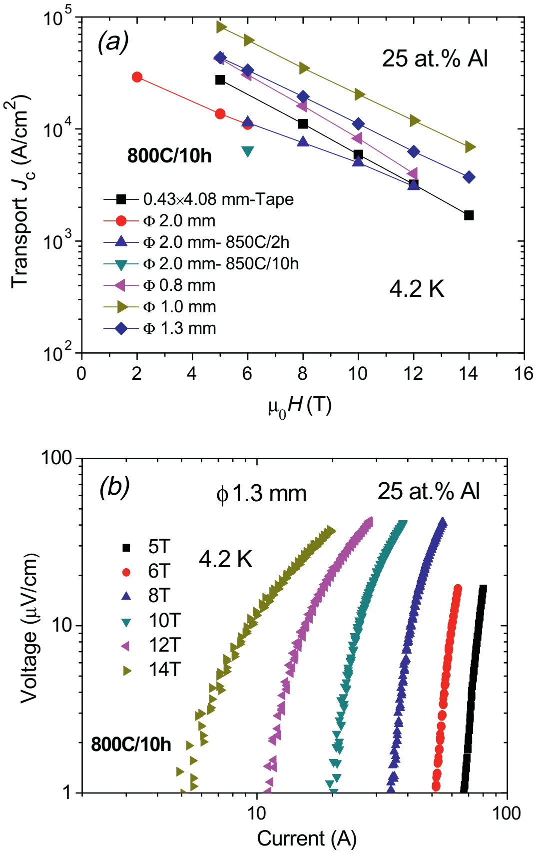

Fig. 6. Superconducting properties of Nb_3Al superconducting wires and tapes at 4.2 K and different applied fields; (a) transport J_c–H curves of the Nb_3Al wires and tape; (b) V–I curves of Nb_3Al wires with a diameter of ∅1.3 mm.

cally alloyed Nb–Al precursor powders and fabrication process of long wires.

4. Conclusions

In this paper, we have systematically studied the preparation of MA $Nb(Al)_{ss}$ supersaturated solid-solution and phase formation of Nb_3Al superconductor. The results suggest that comparing to the RHQT Nb_3Al superconductors, though a few of Nb_2Al impurities are still existed in the Nb_3Al superconductors, the onset T_c impressively reaches 15.8 K in our MA samples, which implies that the MA way is possible to prepare the high-quality Nb_3Al superconductor. At the other hand, the Cu-matrix PIT Nb_3Al superconducting wires have firstly been successfully prepared by using the $Nb(Al)_{ss}$ supersaturated solid-solution and a low-temperature annealing; and the transport J_c at 4.2 K and 12 T is up to 10,000 A/cm^2. By minimizing the content of Nb_2Al impurities and adding the alloying elements, e.g. Cu, Ag and Ge, it is expected to further enhance the J_c performance of the MA PIT Nb_3Al wires.

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