One of the key factors that limit the high-power applications for a type II superconductor is the irreversibility line $H_{irr}(T)$, which reflects the very boundary of resistive dissipation in the phase diagram of magnetic field versus temperature. In cuprate family, the Y-, Bi-, Hg-, and Tl-based systems have superconducting transition temperatures exceeding the liquid nitrogen boiling temperature ($\sim 77$ K). However, the toxic elements Hg and Tl in the latter two systems strongly constrain the applications. The best perspective so far is relying on the YBa$_2$Cu$_3$O$_{7-\delta}$ ($T_c = 90$ K) system, which is nontoxic and has a relatively high irreversibility magnetic field. We report the study of a nontoxic superconductor (Cu,C)Ba$_2$Ca$_3$Cu$_4$O$_{11+\delta}$ with $T_c = 116$ K. We found that the irreversibility magnetic field is extremely high, and it thus provides great potential for applications above the liquid nitrogen temperature.

INTRODUCTION

For a type II superconductor, the magnetic field will penetrate into the sample and form quantized vortices when the external magnetic field is beyond a threshold $H_{c1}$. This state, with the mixture of superconducting area and magnetic vortices, is called as the mixed state. The upper boundary of the mixed state is the upper critical magnetic field $H_{c2}$ at which the Cooper pairs are completely absent (within the Bardeen-Cooper-Schrieffer picture). However, for a superconductor to carry the nondissipative supercurrent, another boundary, namely, the irreversibility line $H_{irr}(T)$, is very crucial. This phase line actually separates the phase diagram into zero and finite resistive dissipation. In the cuprate family, some compounds of the Bi-based (1), Hg-based (2), and Tl-based (3) systems show superconducting transition temperatures beyond 100 K. Among all these materials, it was reported that the (Tl$_{0.5}$Pb$_{0.5}$)Sr$_2$Ca$_2$Cu$_3$O$_9$ [(Tl,Pb)-1223] (4, 5) exhibits a rather high irreversibility line with an irreversibility field of nearly 10 T at the liquid nitrogen boiling temperature ($\sim 77$ K), which is thought to be promising for applications. However, for Hg- and Tl-based systems, the toxic elements Hg and Tl strongly limit the high-power applications of these materials. The nontoxic Bi-based [Bi$_2$Sr$_2$Ca$_2$Cu$_3$O$_{10+\delta}$ (Bi-2223), $T_c \approx 110$ K] system also has a transition temperature exceeding 100 K, but the very layered structure and huge anisotropy do not allow a high irreversibility field at the liquid nitrogen temperature (6, 7). The irreversibility field and superconducting current density decrease rapidly with increasing temperature in the moderate temperature region. For the nontoxic Bi$_2$Sr$_2$CaCu$_2$O$_y$ system, the material can be made into round wire, and the irreversibility field is high at the liquid He temperature; therefore, it could lead to the high magnetic field applications at low temperatures (8). The Y-based YBa$_2$Cu$_3$O$_{7-\delta}$ (YBCO; $T_c \approx 90$ K), which is nontoxic and has a high irreversibility field (about 8 to 10 T at 77 K) (9, 10), is thought to be a promising material for applications. By irradiation, the first-order melting of vortex lattice in YBCO are turned into a second-order transition, but the irreversibility line does not change too much (11). Actually, on the basis of the material HgBa$_2$Ca$_3$Cu$_4$O$_{10+\delta}$ (Hg-1234) with $T_c$ of about 124 K (12), several nontoxic compounds in the formula of MBa$_2$Ca$_3$Cu$_4$O$_{11+\delta}$ with $T_c$ of about 117 K were fabricated, here, $M = (\text{Cu,C})$ or Cu (13–15), etc. On the basis of the model analysis, for example, by assuming the very small anisotropy $\gamma = \xi_{an}/\xi_{c} = 1.6$ in this system, Ihara (16) suggested a possibility of good performance of applications. Preliminary results of resistivity and magnetization were reported on this system (17–19), and the measured irreversibility fields at 77 K were much lower than the expected value (16). In present work, the synthesis and systematic measurements of resistivity and magnetization are reported in the nontoxic superconductor (Cu,C)Ba$_2$Ca$_3$Cu$_4$O$_{11+\delta}$ [(Cu,C)-1234] with $T_c = 116$ K, which exhibits, as far as we know, the highest irreversibility line so far among all superconductors in the liquid nitrogen temperature region in terms of practical applications. This statement is made on the basis of the comparison of $H_{irr}(T)$ between our polycrystalline samples of (Cu,C)-1234 and other systems (including polycrystalline samples, or thin films and crystals with $H||c$ axis). It is found that the irreversibility field $\mu_0H_{irr}$ of (Cu,C)-1234 made in this work reaches about 15 T at 86 K and 5 T at 98 K, providing a great chance of high-power applications in the temperature region of 100 K.

RESULTS

X-ray diffraction pattern, magnetization, and resistivity measurements of sample 1

The (Cu,C)-1234 bulk samples were made by solid-state reaction method under high pressure and high temperature. The details of synthesis and characterization are given in Materials and Methods and the Supplementary Materials. We conducted the resistivity and magnetization measurements with the Quantum Design instruments PPMS16T (physical property measurement system 16 T) and SQUID-VSM7T (superconducting quantum interference device–vibrating sample magnetometer 7 T), respectively. The powder x-ray diffraction (XRD) pattern of sample 1 can be fit quite well with the standard (Cu,C)-1234 XRD pattern (fig. S4). It shows a dominant tetragonal (Cu,C)-1234 phase with a space group $P4/mmm$ with parameters $a = 3.86$ Å and $c = 17.94$ Å. The volume fraction that resulted from this fitting was about 93%. The main impurity phase is CaCu$_2$O$_2$.
(<7%), which is marked with green vertical lines below the spectrum. Two asterisks indicate the unknown phase, which takes a very small fraction of the sample.

Figure 1A shows the temperature-dependent resistivity of sample 1 under different magnetic fields. One can see that the superconducting transition occurs at about 116 K with a rather narrow transition width (ΔTc ≈ 1 K) determined from the 10 to 90% normal-state resistivity. In the inset of Fig. 1A, we present the temperature dependence of magnetic susceptibility measured in zero-field–cooled (ZFC) and field-cooled (FC) modes for the as-synthesized sample 1 (Cu,C)-1234 under 10 Oe. A calculation of the magnetic screening volume using the ZFC data at 10 Oe is about 196%. The reason for the value larger than 100% is due to the demagnetization factor. Again, the diamagnetic susceptibility measurement shows a well-defined transition at about 116 K, being consistent with the resistivity measurements. It is important to emphasize that, without carbon involved in the last step of high-pressure and high-temperature sintering, we could not reach superconductivity. It was claimed in previous reports that even pure Cu (without using carbon) could lead to superconductivity under the pressure of 5 GPa (13, 15). It is worth mentioning that the carbon may not go to the Cu positions on the CuO2 planes. As revealed by neutron powder diffraction measurements and analysis, the carbon atoms in the form of CO3 may occupy the Cu position (which would be the Hg position in the Hg-1234) (20). It remains an interesting and open question how the carbon atoms are incorporated into the structure.

As shown in Fig. 1 (A and B), the superconducting transition becomes broad in the magnetic field, which is induced by the vortex motion. Since our sample is bulk, the crystallographic direction is random in different grains. It is reasonable to assume that the threshold of dissipation is determined by the grains with magnetic field parallel to c axis in some grains or the weak link areas. As we can see, the magnetic field–induced broadening is rather slow compared with the nontoxic Bi-2223 system. If a criterion of resistivity of 1%ρn (Tc) is chosen, as marked by the blue horizontal dashed line, then the determined irreversibility field is 15 T at about 82 K (Fig. 1B). With the same criterion, the irreversibility fields are about 3 T at 98.6 K and 5 T at 94.2 K. Actually, even higher values of irreversibility fields are found in another sample (sample 2). The irreversibility fields are 15 T at 86 K, 5 T at 98 K, and 3 T at 101.2 K (fig. S1). All these values suggest that the nontoxic system (Cu,C)-1234 may have the highest irreversibility line among all superconductors above the liquid N2 temperature.

**Magnetization hysteresis loops and critical current density of sample 1**

To evaluate the intrinsic critical current density and its behavior in a magnetic field, we crushed the sample into powder with an average grain size of about 5 μm and measured the magnetization. We measured the averaged grain size using a scanning electron microscope (SEM; Hitachi Co. Ltd.; fig. S3). Then, we used the hydrochloric acid to corrode the surface of bulk sample 1 for a few seconds to show the grains. We measured the magnetization hysteresis loops (MHLs) of sample 1 in powder state from 4.2 to 110 K for applied fields up to 7 T (Fig. 2A). It is clear that the magnetization is irreversible up to quite high temperatures and magnetic fields. For the powdered superconducting sample, the critical current density Jc was determined by using the Bean critical-state model Jc = 30ΔM/d (18, 21), where ΔM (in units of electromagnetic unit per cubic centimeter) is the width of the MHL, and d (in units of centimeter) is the averaged size of the grains. Using Bean critical-state model, we obtain the critical current density Jc versus magnetic field at different temperatures (Fig. 2B). It is clear that the Jc value can reach about 6 × 106 A/cm² at 4.2 K at Earth’s field. In addition, the critical current is quite robust in the presence of the magnetic field. Using a criterion of 1000 A/cm², we obtain an irreversibility field of about 2 T at 100 K, which is slightly lower than that determined from the resistive measurements because the criterion of Jc adopted here is rather high. At about 90 K, the critical current density Jc keeps flat versus magnetic field, indicating a much higher irreversibility magnetic field.

**DISCUSSION**

As shown above, the critical current density and the irreversibility line in the (Cu,C)-1234 system are both very high. Here, we
would like to give a comparison of the irreversibility lines in different cuprate systems. It is worth noting that the irreversibility line of sample 1 is obtained by using a resistivity criterion of $1\% \rho_n$ ($\rho_n \approx 0.09$ milliohm-cm), as shown by the dashed blue line in Fig. 1B, which is also adopted by other groups. Figure 3 shows the comparison of irreversibility lines for (Cu,C)-1234 (samples 1 and 2 in this work), YBCO (single crystals) (9, 10), Bi-2223 (thin film) (6), Bi-2223 (single crystal) (7), and (Tl,Pb)-1223 (5). It is clear that the non-toxic Bi-2223 has a very low irreversibility field at 77 K, while YBCO has a relatively high $\mu_0 H_{irr}$ (about 8 to 10 T at 77 K, $H||c$ axis) compared with Bi-2223. The material (Tl,Pb)-1223 shows a higher $T_c$ and irreversibility line than YBCO while it contains the toxic element Tl. However, the irreversibility field of the nontoxic samples (Cu,C)-1234 synthesized in this work shows a much steeper temperature dependence near $T_c$. In previous studies, the magnetizations were measured for samples of the same system (Cu,C)-1234 (17–19) or AuBa$_2$Ca$_3$Cu$_4$O$_{11}$ (Au-1234) (22), but the irreversibility lines reported there are not as high as our samples. This may be induced by the difference of the material morphologies inside the sample. We have gone through the literature that we could collect for the Hg- or TI-based systems, including that for Hg-1212 (23), Hg-1223 (24), Hg-1234 (25), and TI-1223 (26), and found that none of these materials has the irreversibility line beyond the present system (Cu,C)-1234. For films and crystals, the data when $H||c$ axis are used for comparison, since it is believed that the critical current density with magnetic field parallel to c axis or the weak links limit the irreversibility lines in the polycrystalline samples. From the literature, one can find that the irreversibility fields at 77 K are $\sim 4$ T (Hg-1212) (23), $\sim 6$ T (Hg-1223) (24), $\sim 2$ T (Hg-1234) (25), and $\sim 3$ T (TI-1223) (26), which are below the values of our present sample. We also checked the infinite layer system (Sr$_{1-x}$Ca$_x$)$_2$CuO$_2$ with $T_c \approx 110$ K (27), and there is quite little data about the irreversibility lines of this system perhaps because most of the samples have not been made to a perfect state. From the available data in one of the infinite layer system Sr$_{0.5}$La$_{0.1}$CuO$_2$ ($T_c = 43$ K) (28), one can see that the anisotropy is not very small and the irreversibility line is also quite low. As mentioned already, there is an irreversibility field of 15 T at 82 K for sample 1; it even shoots up to 15 T at 86 K in sample 2. Here, in Fig. 3, we use the highlighted area to indicate the region with finite superconducting (or zero/weak resistive) dissipation) of our sample as compared with YBCO. One can see that there is a large area beyond the irreversibility line of YBCO where the samples can carry nondissipative supercurrent, providing great potential for applications in the temperature region of 100 K. It should be emphasized that the present samples were made through the high-pressure synthesis. The present results just show very good intrinsic properties for application of the nontoxic material (Cu,C)-1234 and related systems. It is highly desired to try new methods with lower pressure or thin-film deposition to make the superconducting wire/tape based on this promising material. The presently used techniques for fabricating the coating conductor of
YBCO, such as ion bombardment–assisted deposition (29, 30), rolling-assisted biaxially textured substrates (31, 32), and metal-organic chemical vapor deposition, etc., may also be tried for this material. Comparing the critical current densities presented in Fig. 2 in the case of the grains and Fig. S2 in the case of the bulk crystalline sample, one can see a difference of two orders of magnitude. This implies that the connectivity between the grains of the polycrystalline bulk is strongly degraded and the currents cannot circulate very well over the whole bulk sample. If this weak link problem in the bulk polycrystalline can be improved or eliminated, then it will be promising to make round wires by using this material. The discovery reported in this work will stimulate new efforts in the practical applications based on this material system.

**SUPPLEMENTARY MATERIALS**

- **Fig. S1.** Resistivity and magnetization versus temperature of sample 2.
- **Fig. S2.** Magnetization and critical current density of sample 2 in bulk form.
- **Fig. S3.** SEM image of the surface after being treated by hydrochloric acid of sample 1.
- **Fig. S4.** Powder XRD pattern for sample 1.
- **Fig. S5.** SEM images and composition analysis on fresh surface of sample 1.

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