Mechanical milling of MgB_2

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Abstract

Changes in structure and magnetization of MgB_2 by mechanical milling have been examined by means of X-ray diffraction and magnetization measurements. Powder sample we used is commercially available and contains a small amount of MgO. Mechanical milling was done between 0h and 384h. The intensity of the Bragg peaks decreases and the width increases with milling time. This means that the crystallite size becomes smaller and the strain in the crystal structure increases. All samples show the superconducting transition at $T_c = 39$K and T_c does not change by milling. The diamagnetization caused by superconductivity decreases with milling time. This suggests that mechanical milling acts on only destroying a superconducting state in some part of MgB_2 without changing superconducting properties in the remaining part.

Key words: MgB_2; mechanical milling; X-ray diffraction; magnetization

Since the discovery of superconductivity in MgB_2 by Nagamatsu *et al.* [1], many researches for this material have been made. However, there are not many reports on the relations between the imperfection of the structure and the superconductivity, though such study is necessary and important to its applications. Serquis *et al.* [2] reported the influence of lattice strain and Mg vacancies in MgB_2 and found that high strain and the presence of Mg vacancies resulted in lowering T_c by only 2K. In this paper we have subjected MgB_2 to mechanical milling and have investigated the relationship between the superconductivity and the lattice strain introduced by milling.

Starting MgB_2 sample was a commercial powder (Soekawa Rikagaku, 99%). Mechanical milling was carried out in a hardened steel vial with one steel ball under a purified helium atmosphere [3]. The structure of all samples was examined from X-ray diffraction patterns obtained with Cu Kα radiation. A SQUID magnetometer (Quantum Design, MPMS2) was used to measure the temperature dependence of the magnetization in 100Oe and the field dependence of the magnetization at 10K.

In Fig. 1, the X-ray diffraction patterns of MgB_2 milled for several periods up to 384h are plotted together with the patterns of the starting MgB_2 powder. The present starting MgB_2 involves a small amount of some impurities; peak positions of MgO are shown with lines in this figure. The intensity of Bragg peak decreases and the width increases with milling time. The obvious decrease of the intensity and the increase of width are observed after 240h of milling. We could not separate only the contribution of MgB_2 from X-ray diffraction pattern and estimate crystallite size and lattice strain, since Bragg peaks of unspecified impurities were superposed on those of MgB_2. In many cases, crystallite size becomes almost constant after several ten hours of milling [3], [4]. Therefore, we can speculate that the breadth after 240h of milling is caused by the increase of lattice strain.

Figure 2 shows the field-cooled magnetization (FCM) and the zero-field-cooled magnetization (ZFCM) over a temperature range of 5 to 50K under an applied field of 100Oe. The starting powder has the onset superconducting transition temperature

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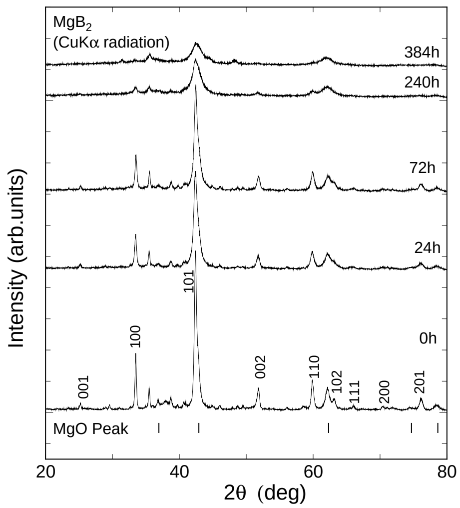

Fig. 1. X-ray diffraction patterns of mechanically milled MgB_2.

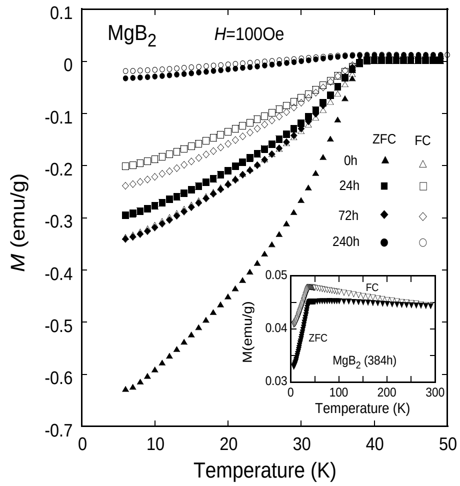

Fig. 2. Temperature dependence of field-cooled and zero-field-cooled magnetization of MgB_2 milled for 0h to 240h. The inset shows FCM and ZFCM of MgB_2 milled for 384h as a function of temperature.

of 39K (T_c) and the rate of FCM to ZFCM at 5K is 58%, though the diamagnetization caused by the superconductivity increases gradually below T_c with cooling. Takano *et al.* [5] reported the magnetization behavior of MgB_2 in powder and bulks prepared by high-pressure sintering. Our results are qualitatively consistent with their results, but the absolute values of FCM and ZFCM of the present sample at 5K are smaller than their results. After milling, the superconducting transition temperature T_c does not change at all within the error of the measurements. Though the magnetization of the sample milled for 384h is positive over the whole measurement temperature range, the decrease of the magnetization upon cooling is observed below 39K.

The field dependence of the magnetization of all samples at 10K is plotted in Fig. 3. $M-H$ curves with narrow hysterisis are observed for the present samples. The changes of the magnetization as a function of field become smaller with milling time. But the relative change of the magnetization scaled by the minimum value of the magnetization for each sample is almost the same among the samples up to 72h of milling. The change of $M - H$ curve for the sample milled for 240h is still smaller and its relative change is different from that for shorter period of milling. Moreover, the $M - H$ curve after 384h of milling is like ferromagnetic.

We can interpret above results as follows. The mechanical milling of MgB_2 up to 72h does not contribute to the change of T_c and has the effect of partially destroying the superconducting area. For longer period of milling, the superconducting fraction remains and the magnetic properties are affected by part of the area where the superconductivity is destroyed by milling.

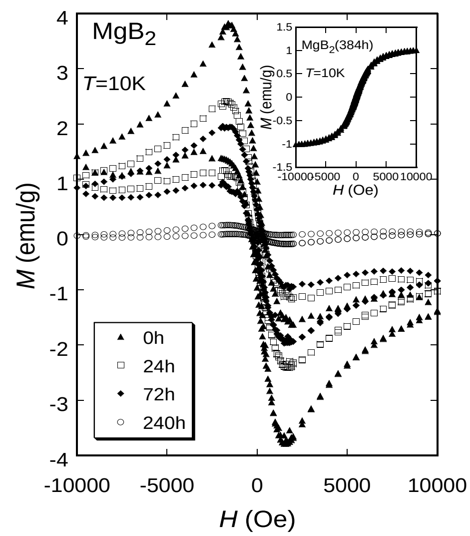

Fig. 3. Magnetization measured at 10K as a function of applied field for the mechanically milled MgB_2 at. The inset shows the $M - H$ curves of MgB_2 milled for 384h.

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