Electronic supplementary information (ESI)

Low-temperature Massive Production of Superconducting MgB_2 Nanofibers from $Mg(BH_4)_2$ decomposition and recombination

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Experimental Section

General consideration. Synthesis operations were carried out on the bench using Schlenk tube techniques in order to prevent productions from possible oxidation in air. The glassware was evacuated down to a pressure of 10 Pa and flushed with dry and oxygen-free nitrogen prior to use. Magnesium borohydride as precursor was put into a steel reactor and the system was then evacuated. The system was heated to desired temperature and maintained for 1h at 10^{-3} Pa. Solids were handled in an argon-filled glove box which the water and oxygen concentrations were kept below 1 ppm during operation. Diethyl ether was distilled over sodium before use. Sodium borohydride and magnesium chloride (both 99%, Sigma Aldrich) were used as received.

Sythesis of MgB_2 from $Mg(BH_4)_2$ precursor. The $NaBH_4$ mixed with $MgCl_2$ were mechanically milled for 10 h at 300 rpm under 5 bar hydrogen atmosphere by using a planetary ball milling apparatus (Pulverisette 5). The mole ratio of $NaBH_4$ and $MgCl_2$ was 2:1 and the weight ratio of ball-to-sample was around 20:1 in milling process. The milled fine white powder was transferred to a three-neck round bottom flask attached to a condenser. After the addition of diethyl ether, the suspension was vigorously stirred under refluxing for 12 h. The reaction mixture was filtered through a specially designed fine-grade round sintered discs for three times. The filtrate was vaporized and then dried under vacuum at 190 °C for 2 h. The yield was 90% with respect to $Mg(BH_4)_2$. This precursor was then put into a steel reactor and

heated under vacuum at 380 °C, 420 °C, 460 °C, and 500 °C for 1 h to form MgB_2. The yield of the transformation is up to 95%.

Characterization. The structural analysis of the samples was carried out by X-ray diffraction using an automated Rigaku X-ray diffractometer with monochromatic Cu Kα radiation. The differential scanning calorimetry was performed on a Netzsch DSC 204 HP calorimeter at a heating rate of 5 K/min under 2 bar argon from room temperature to 500 °C. The gas evolved was measured at 380 °C and a 0.8 bar hydrogen back-pressure using a PCT Pro-2000 Sievert's type pressure-composition-temperature apparatus from Hy-Energy to monitor the decomposition kinetics. Temperature and pressure of the sample and gas reservoirs were monitored by LabView-based control software. The size distribution and morphology of the samples were observed by field-emission scanning electron microscopy using a QUANTA 200 FEG at 10 kV. The magnetic properties of MgB_2 nanofibers, including *M-T* test and *M-H* test, were measured with the magnetic property measurement system (MPMS, Quantum Design).

Figures

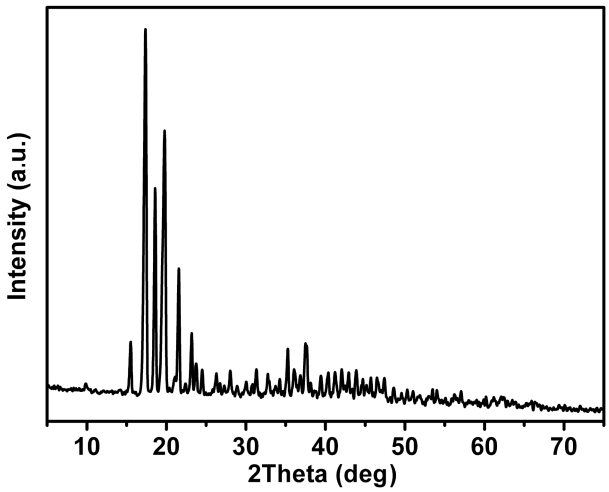

Fig. S1. XRD spectrum of the powder $Mg(BH_4)_2$ obtained through milling $NaBH_4$ and $MgCl_2$ followed by refluxing in diethyl ether.

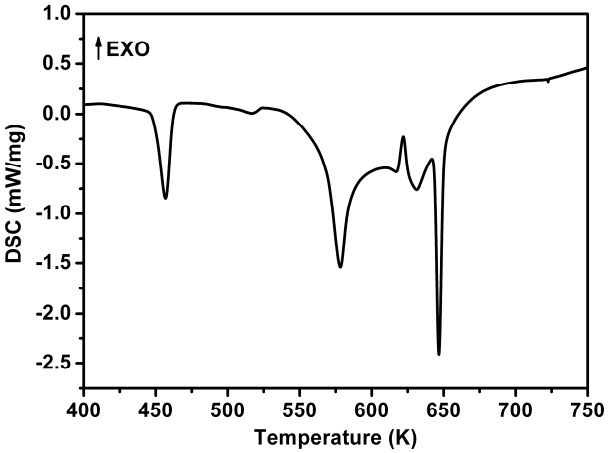

Fig. S2. DSC curve of $Mg(BH_4)_2$ decomposition at a heating rate of 5 K/min under 2 bar argon.

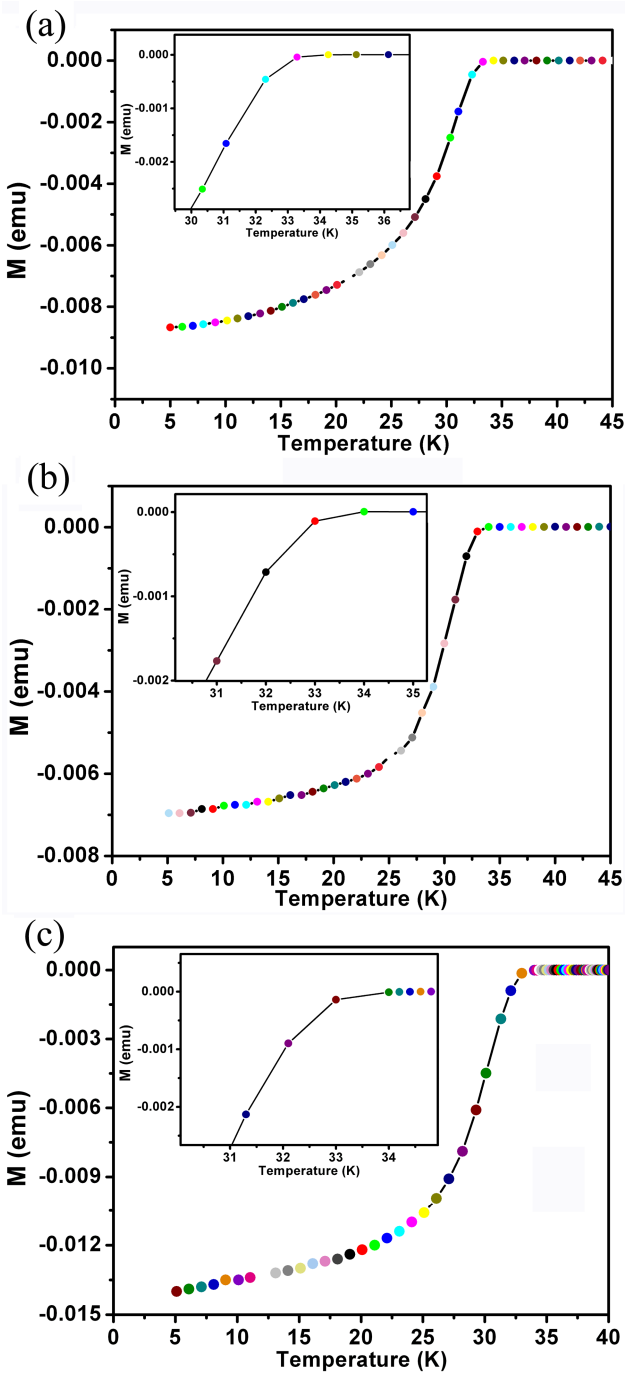

Fig. S3. *M-T* curves of the MgB_2 nanofibers measured under a magnetic field of 50 Oe. Reaction condition: annealing at (a) 380 °C (b) 420 °C (c) 460 °C for 1 h.

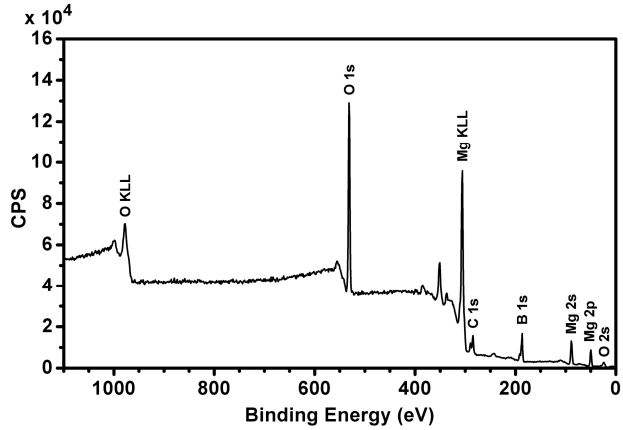

Fig. S4. XPS results of the superconducting MgB_2 obtained from $Mg(BH_4)_2$ decomposition and recombination

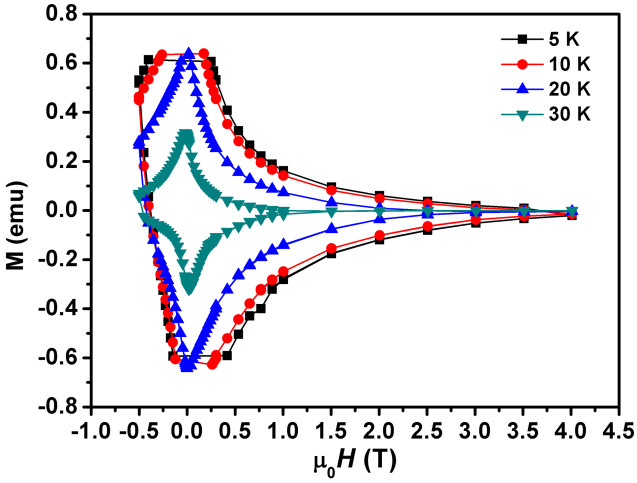

Fig. S5. The magnetization vs field plot for the MgB_2 nanofibers processed at 460 °C for 1 h.