Magneto-optical, microstructural and magnetization measurements of *in situ* Fe/MgB_2 conductors made from ball-milled precursor

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The effects of the precursor milling energy on the superconducting and microstructural homogeneity of Fe-sheathed MgB_2 monocore wires and tapes have been investigated. The conductors were produced by the powder-in-tube method using drawing and rolling deformation methods and in situ reaction. Combined magneto-optical, structural and magnetization measurements were performed on MgB_2 superconducting tapes prepared from differently milled precursors revealing hidden mechanisms controlling their critical current density. Local degradation of the superconducting properties observed in some analysed conductors has been identified and correlated with microstructural observations.

Preparation of MgB_2 wires and tapes

Monocore Fe-sheathed MgB_2 wires and tapes were prepared by the powder-in-tube (PIT) method using the *in situ* reaction and drawing and rolling techniques.

Some of these precursor powders were milled in Ar atmosphere using a planetary ball mill (Retsch PM 100).

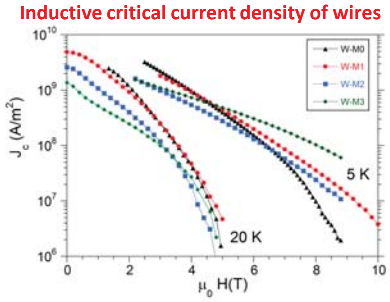
t is the effective ball milling time, ω_p is the mill rotation speed and E_t/m is the estimated energy per unit mass [1,2]. T_c and ΔT_c are the critical temperature and the transition width, respectively, as derived from $\chi_{ac}(T)$ measurements of the wires.

Name and manufacturing characteristics of the analysed samples

Wire	Tape	Ball milling			T_c	ΔT_c
		t (h)	ω_p (rpm)	E_t/m (J kg^{-1})	(K)	(K)
W-M0	T-M0	0	0	0	37.5	0.7
W-M1	T-M1	1.5	200	1.2×10^6	37.2	0.6
W-M2	T-M2	3.0	400	2.0×10^7	35.0	0.5
W-M3	T-M3	16.5	400	1.0×10^8	33.0	1.0

Critical current densities

Inductive critical current density of wires



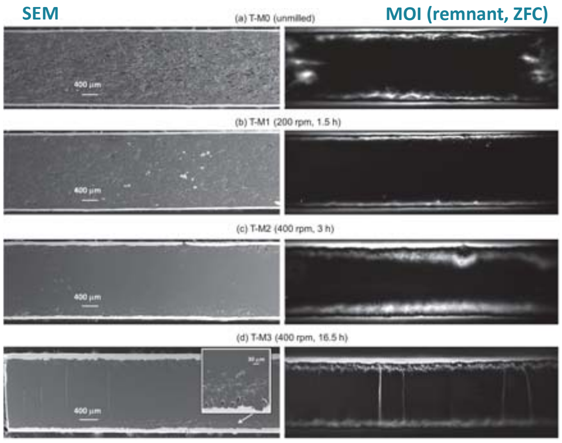
Higher energy milling enhances vortex pinning at high fields.

At low fields J_c decreases with increasing milling energy, which indicates poorer grain connectivity and/or larger amounts of microstructural defects than in unmilled samples.

Measurements of **transport J_c** of wire W-M1, gave values of 6×10^7 Am^{-2} at 4.2 K and 8 T.

Microstructural and Magneto-Optical imaging analysis of tapes

Comparison between SEM and MO images (ZFC)

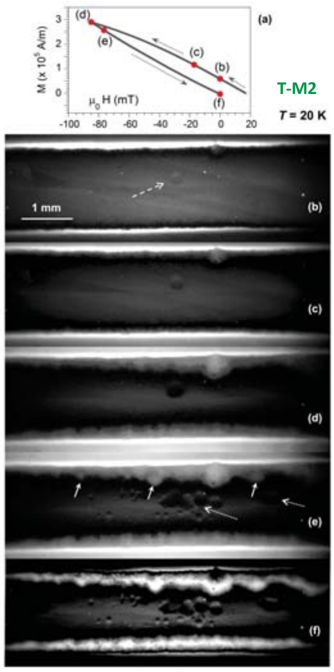


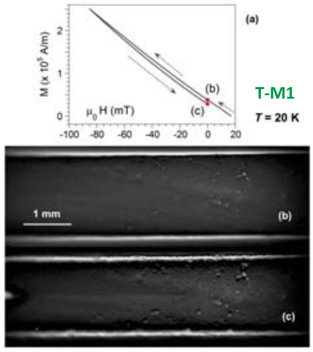
*SEM (secondary electrons detector) and MOI images (taken after **ZFC to 20 K**, then applying a field of **85 mT** perpendicular to the tape and finally removing the field) correspond to the same area, except in (d). The same magnification was used for both SEM and MO images.*

Flux front propagation depends on the milling energy.

- **Flux front of T-M0 (unmilled) reflects its porous structure**
- **T-M1 (lowest milling energy) shows the best behaviour**
- **T-M2 shows J_c deterioration in some areas**
- **T-M3 (hightest milling energy) presents transversal cracks**

MOI images after FC and during specific magnetic history



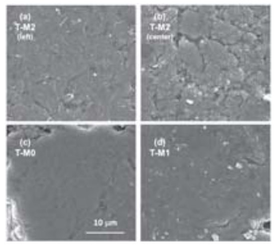


Magnetization values, M(H), and **MO images** of tapes **T-M2 (*left*)** and **T-M1 (*above*)** at different magnetic fields after FC in 17 mT to 20 K, then changing the field to -85 mT and finally removing the field. *The magnetic field is applied perpendicular to the tape.*

- **Very homogeneous magnetic flux profile in tape T-M1**
- **T-M2 presents magnetic inhomogeneities (see arrows). This suggests the existence of poor superconducting connectivity between regions in this part of the sample, which has been confirmed by SEM**

SEM analysis

of different areas of T-M1 and T-M2



SEM images (secondary electrons detector) of:

(a) **Left region of tape T-M2** (with better Jc)
(b) **Central area of T-M2** in the central area where "bubble like shape" magnetic inhomogeneities are seen.
(c) Image the tape **T-M0** of unmilled precursor
(d) Image of **T-M1** (with optimized superconducting properties).

Conclusions

Microstructural characteristics change strongly with milling energy of the precursor and affect significantly the magnetic flux front observed by MO imaging at zero-field-cooled and field-cooled conditions. Very homogeneous magnetic flux profile in tape T-M1 (in both ZFC and FC)

Magneto-optical imaging (MOI) performed in field-cooled samples, following a specific magnetic field sequence, highlights the distortions in the magnetic pattern so that it has been found very helpful to reveal inhomogeneities in the superconductor.

The combined analysis by MOI, SEM and magnetization measurements has revealed hidden mechanisms controlling their critical current density. Local degradation of the superconducting properties observed in some analysed conductors has been identified and correlated with microstructural observations.

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[2] C. Laliena et al, J. Alloys Compd. vol. 717, (2017) 164