

# The Influence of *Ex Situ* MgB<sub>2</sub> Barrier and HIP on the $I_c$ Anisotropy in Double Core MgB<sub>2</sub>/Cu Wires

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The effect of large  $I_c$  anisotropy reductions in MgB<sub>2</sub>/Cu sheathed wire is presented. The measurements of the critical current in rising magnetic field up to 14 T at 4.2 K were performed. Due to the application of the high isostatic pressure process to the copper sheathed MgB<sub>2</sub> wires with the *ex situ* MgB<sub>2</sub> homo barrier around the *in situ* core the anisotropy for such wires was very small and approached to less than 2% at magnetic field over 4 T. The presented geometry and technology show several advantages in their practical applications.

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## 1. Introduction

The majority of the MgB<sub>2</sub> [1] wires are performed with iron based or Cu–Ni alloys type clad, often with the chemical barrier made of expensive niobium or titanium. A disadvantage of such barriers is very high costs of its difficult plastic deformation as wires or tapes, and also considerable high electric resistance and low thermal conductivity of the sheath. Such problems have been resolved by use of copper composite cladding and the *ex situ* barrier (pat. US 12/098/475) [2]. However, the use of the copper type clad causes many technological problems. The strong reactivity of Mg with Cu causes the immediate formation of the Cu<sub>2</sub>Mg or CuMg<sub>2</sub> phases, even at low annealing temperature and also causes degradations of MgB<sub>2</sub> amount in a core. The rapid creation of these eutectic phases can considerably diminish the critical parameters of superconducting wires. High chemical reactivity of Cu especially with Mg can be strongly limited by using *ex situ* chemical barrier and high isostatic pressure (HIP) heat treatment [2, 3]. However, considerably small presence of low temperature eutectic phases causes substantial increase of the critical current in MgB<sub>2</sub> wires obtained by HIP at temperatures below the melting temperature of Mg. In addition, application of several nano tens average size grains obtained (by mechanical alloying, MA, or self-propagating high-temperature synthesis, SHS methods) increase critical current density and lower the annealing temperature (by HIP) eliminating the reaction between Cu and Mg. The dense MgB<sub>2</sub>

homo-barrier increases itself the active superconducting cross-section of the wire, which is not a case in conductors with Nb, Fe or Ti barrier.

## 2. Samples preparation

The wires were prepared from commercial grade of powders in the Institute of High Pressure Physics of Polish Academy of Sciences, by hot HIP (hot isostatic pressing) in high argon pressure medium [4]. The precursors of *in situ* powders were obtained by MA partially made in IFW Dresden and IHPP Warsaw and SHS method elaborated in IHPP Warsaw. The samples *A* and *B* consist of the *in situ* material Mg+2B+8at.%nSiC+5at.%MgH<sub>2</sub> and the *ex situ* barriers consist of MgB<sub>2</sub>+8%nD. Both materials were finally CIP-ed in the rubber mould at 0.3 GPa and the samples *A* and *B* were annealed at the same temperature 700 °C under Ar pressure of 0.33 GPa. The sample *A* was annealed 30 min and the sample *B* 15 min (Fig. 2).

## 3. Experimental

The critical current  $I_c$  of the wires was measured by a four-probe resistive method with 1  $\mu$ V/cm criterion used, for the samples of the length equal to about 25 mm. The wire diameter used for examination was 1.1 mm. All measurements were made at 4.2 K. Maximum measurement current was 150 A. Steady magnetic fields was generated by the Bitter-type magnet with maximum field up to 14 T. The critical transport current was measured by use of current change in constant magnetic field. The critical current measurements were made in the perpendicular

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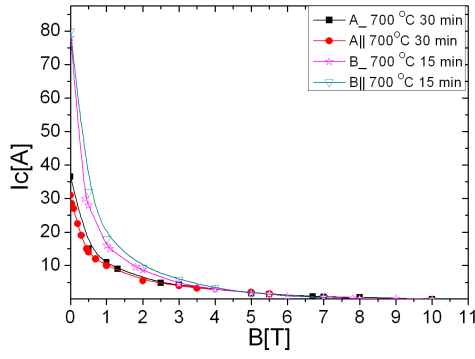


Fig. 1. The critical current  $I_c$  for all MgB<sub>2</sub> samples as function of external magnetic field at 4.2 K, symbol || means parallel magnetic field and symbol \_ means perpendicular magnetic field.

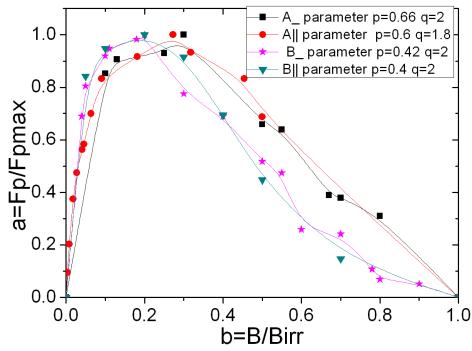


Fig. 2. Reduced pinning force  $f$  as a function of reduced field  $b$  for all MgB<sub>2</sub> samples as function of external magnetic field at 4.2 K, symbol || means parallel magnetic field and symbol \_ means perpendicular magnetic field.

magnetic field and the parallel magnetic field applied to the wire.

#### 4. Results and discussions

The  $I_c$  critical current anisotropy with increasing magnetic fields has been measured for the same sample in parallel magnetic fields applied to the wire axis and in perpendicular magnetic fields configuration. The anisotropy coefficient was determined as the percent of the difference of the measured  $I_c$  for both magnetic field directions to the wire axis. Such current anisotropy has been measured for two samples A and B in certain stabilized magnetic fields by steps reaching maximum field of 12 T. Generally, the anisotropy of the current distribution depends on the geometrical factor of the lids (e.g. wire or tape) and more important, from the point of view of application, of desired geometrical lids the structural anisotropy. The structural anisotropy depends mainly on the pinning center distribution in the wire core. Such structural anisotropy depends strongly on the methods of production of the wires: especially the grains size distri-

bution, its homogenization, the drawing process applied, the sintering conditions, developing of the texture, and of course of the natural tendency of the material to structural anisotropy.

The distribution of the pinning density is the main driven force for the  $I_c$  anisotropy of the lids. The pinning density distribution depends on the magnetic fields applied and the different kind of pinning (large grains boundary type — typical for MgB<sub>2</sub> or points type defects) has different effect on  $I_c$  depending of the magnetic fields applied. The MgB<sub>2</sub> material seems to be isotropic from the point of view of the grains size distributions, but certain technological procedures can be strongly involved on the pinning density (e.g. rolling, extrusion, HP etc.). The MgB<sub>2</sub> characterizes by considerably low anisotropy factor less than 10%.

In our HIP treated samples with *ex/in situ* configuration of the barrier and core, we found considerably lower anisotropy which is achieved by the specific homogeneous grain size distribution and especially high uniform density of the superconducting materials, with the best possible grains connectivity. The HIP process of such configuration permits to release the gradients of stresses between the core material and the same type of MgB<sub>2</sub> barrier themselves produces the homogeneous pinning force (especially that of the grains boundary type) which are particularly important in the middle magnetic fields application region of the MgB<sub>2</sub> wire uses.

The proposed *ex situ* MgB<sub>2</sub> barrier effectively eliminates the Mg–Cu phases development during sintering process. Moreover, the isomorphous to the *in situ* core *ex situ* barrier provides excellent grains contact, low stress during sintering process and further in application during the ramping cooling down to helium or other practical operating temperature. The high shrinking (of ca. 25 vol.%) [5] of *in situ* material during sintering is compensated by the analogous comprising of the *ex situ* barrier and soft copper by HIP-ing, so effective stress between the layers are minimized. Used Cu or Cu compound clad is the most effective method to eliminate the quench effect in wire at ca. 20 K — the best thermal and electric conductivity.

In our experiments we have used the commercial *ex situ* type of powders with considerably large grains size to see the worse conditions of the anisotropy arise, so the overall  $I_c$  level was also low, but the mechanism of anisotropy creating was minimized due to the isostatic conditions of HIP sintering and *ex/in situ* technology applied in copper alloys.

In Fig. 4 similar HIP effect on the specially prepared *in situ* and *ex situ* fine powder is presented. The uniformity and the high density of the HIP-ed material are presented (1 GPa argon gas medium, 720 °C, and 15 min). The highest known  $I_c$  results for such high dense homogeneous wires will be presented at ASC 2010 Conference, Washington.

The wires A and the B presented at this work have also low anisotropies from 2% to 4%.

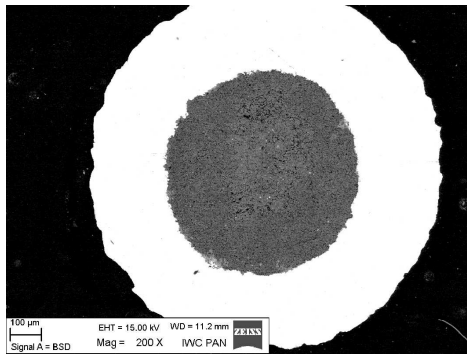


Fig. 3. The typical SEM image of the cross-section of the Cu/*ex situ*/*in situ* sample A and B after HIP-ing process.

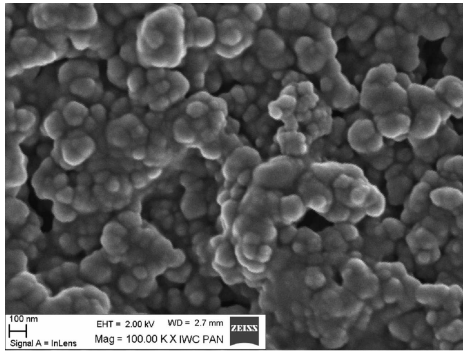


Fig. 4. The microstructure of very dense and homogeneous core section of the Cu sheathed wire sample after HIP process.

The authors in articles [6, 7] have described the physical properties and the chemical properties of the *ex situ*  $\text{MgB}_2$  barrier with the barrier prepared from commercial or coarse grains short time milled. They have not defined the anisotropy effect of these types of wires after HIP.

In earlier article [8] we had presented results of similar anisotropy level reduced in *in situ*  $\text{MgB}_2$  with *ex situ*  $\text{MgB}_2$  barrier in iron shield. This wire has had low anisotropy in a range from 3 to 5%. The scaled pinning force on magnetic field of our Cu shield wires determines dominant of pinning mechanism as the grain boundary mechanism [9, 10]. The evaluated from scaling process parameter  $p$  is close to 0.5 and appropriate  $q$  parameter is almost equal to 2 — all suggested the dominant grains boundary pinning (Fig. 2). On the basis of Fig. 3 one can certify that the structure of the HIP-ed wire is very homogeneous and dense. Any cracks and voids are formed in *in situ*  $\text{MgB}_2$  material, and the connection between *ex situ*  $\text{MgB}_2$  barrier and *in situ*  $\text{MgB}_2$  core is very good.

## 5. Conclusion

Our investigations proved that *in situ*  $\text{MgB}_2$ /Cu wires with even commercial grade grains size *ex situ*  $\text{MgB}_2$  barrier, which were HIP-ed at severe pressure conditions result in  $\text{MgB}_2$  highly homogeneous dense and stress free material. This allowed creating the uniform  $\text{MgB}_2$  homogeneous material in the whole cross-section of the wire and making possible to obtain homogeneous distribution on grain boundary pinning. The homogeneous distribution of pinning in grains boundary allowed to obtain very low anisotropy.

Further work must be performed in optimization of the *ex situ* barrier grains size distribution and the method of densification to avoid the local grains condensation caused by the different surfaces properties of grains.

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