New - hot high pressure technologies for high performance MgB2 conductors in application perspectives.

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See also the poster presentation concerning: HIP – positive influence on pure MgB_2 wires..

Presented by :

- - **Daniel Gajda** **S-P-326** (May 1st) **poster**

Content of the presentation

- Principal attributes of high pressure HIP applicated for MgB_2 wires sintering
- Surprisingly high Jc of MgB_2 *in/ex-situ* wires obtained by HIP in 2005
- The stress effect & rapid degradation of Jc in wires over certain strain (1,3 GPa)
- Solid state reaction and the **pressure dependence of Mg melting by DTA**
- The kinds of PIT technology for high Jc classic and new (ex/in) wires technology
- Morphology of the HIP-ed wires from both techniques (in/ex and HyperTech)
- The Jc and Fp (pining force) for HIP-ed in **solid state and liquid phase** wires
- Proposal of the next investigations and conclusions

Mains attributes of HIP (Hot Isostatic Pressing) applied to MgB_2 structure

- HIP is high densification process enabled to obtain the best grains connectivity during sintering by simultaneous pressure and temperature action at isostatic conditions. Typically for the grains dimensions as low as tens nano meters a few GPa pressure is required, to get highest ceramic density without cracks.

- High pressure can substantially modify the liquid – solid line
of the Mg melt temperature (Tm), which change the phase diagram.
Due to the higher melting temperature of Mg under high pressure the solid state reaction of the substrates is occurred and the grains growth is strongly restricted and limited.

- The plasticity of the wire core obtained by the solid state reaction of the nano grains, is much better than that obtained with liquid Mg or other liquid phases as : Mg_2Si, Mg-Cu etc.

- The stress applied to the sheath material by HIP can be transmitted directly to the in situ core and is constant during all HIP process! Contrary to the CIP all other cold deformation processes, which act only on the beginning of the sintering/ synthesis, due to the high shrinkage of the Mg+2B sintering and softening of any of the sheaths metal at high temperatures - so CP process is much less effective than HIP in densification of nano grains structure.
- The HIP easily avoid the grains growth and effectively block the growth of grain at the last part of sintering process especially (where the applied temperature is usually made higher) due to the high nano dispersion of the final MgB_2 hard grains- self reinforcement.
- The high pressure powder technology can be used for powder mixing ,cleaning and pulverize of the agglomerates e.g. US-HP

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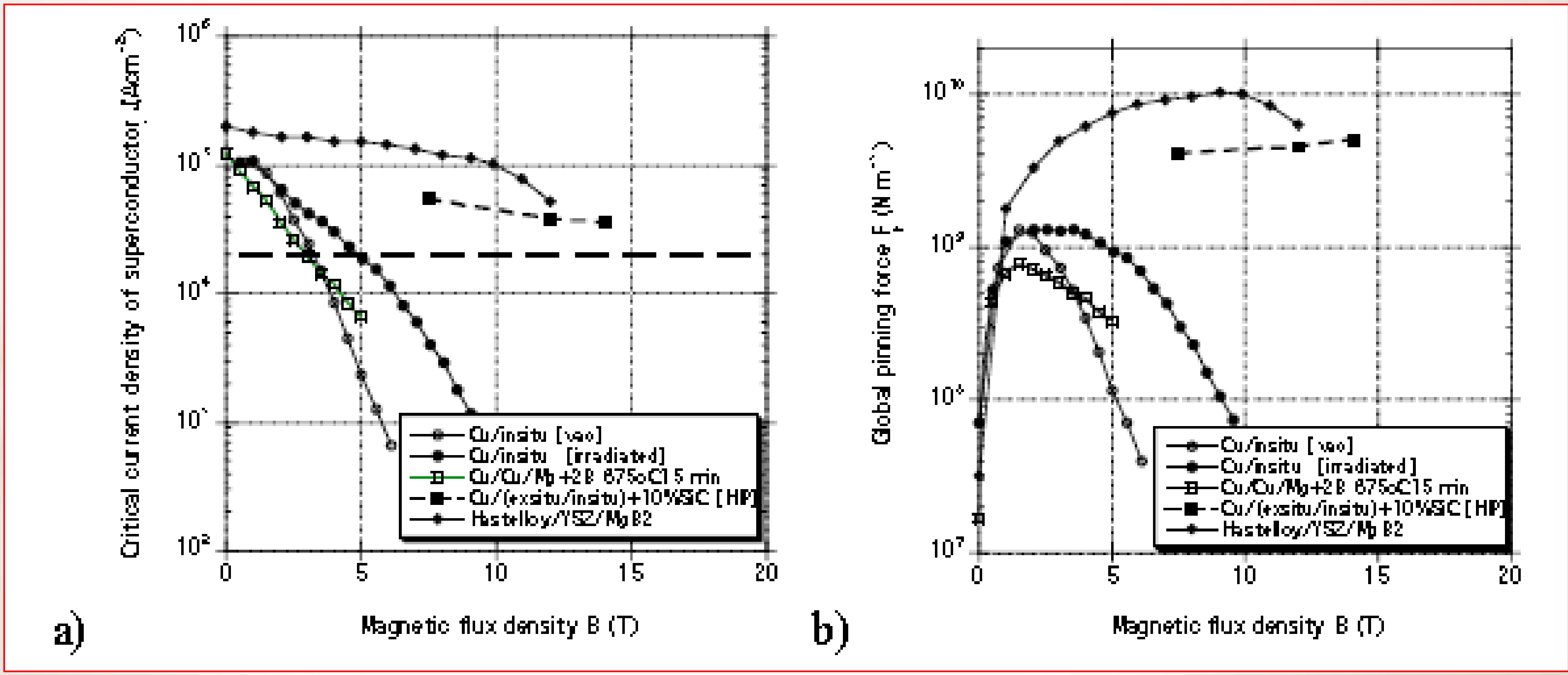
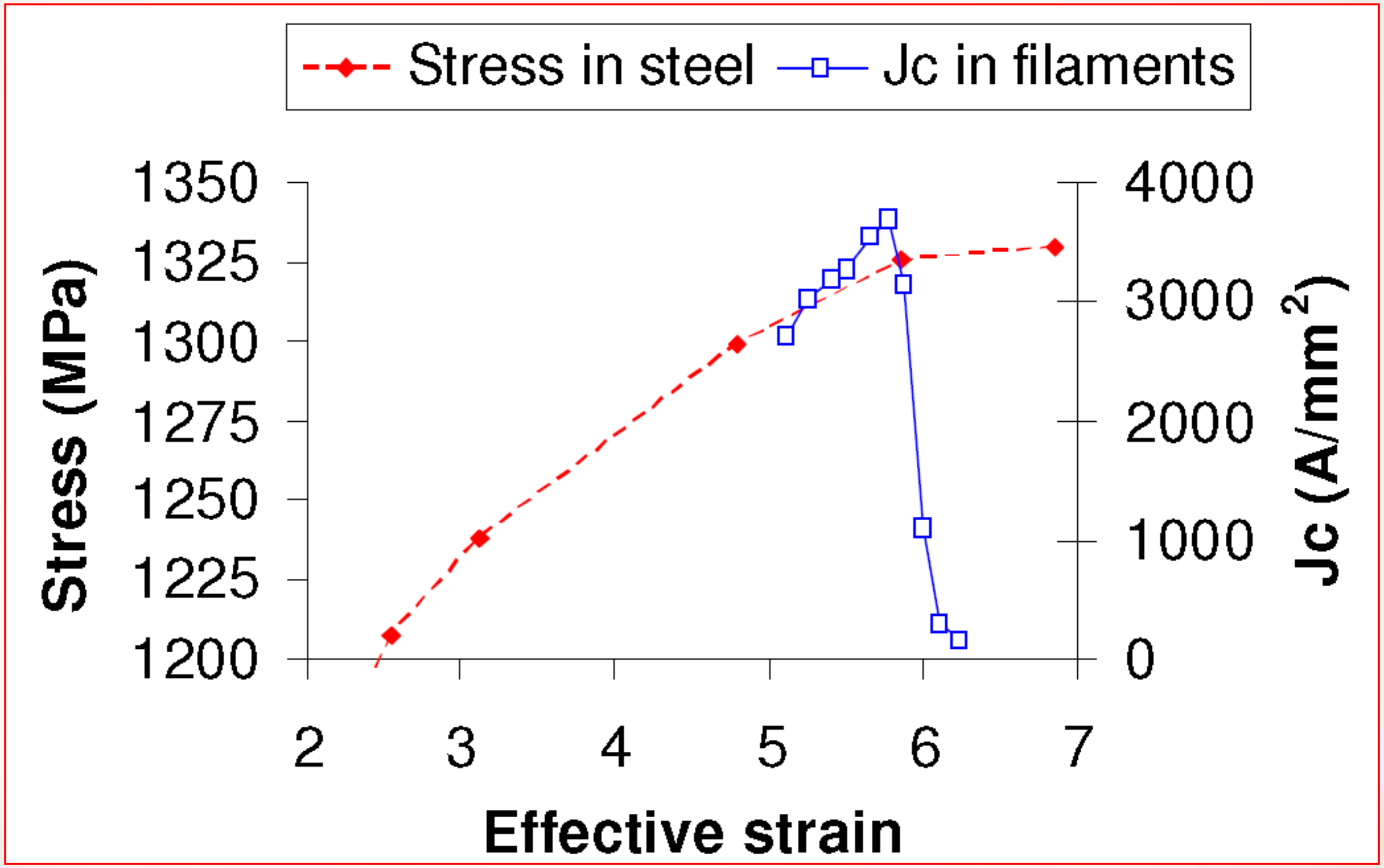
Why such high Jc at high magnetic field after the HIP treatment at 1 GPa made on Cu/MgB_2 sheathed wires was found by us at 2005? ; presented at MT-20

High Jc (a) and Fp (pining) (b) was found to be the effect of the **solid state reaction** performed by the HIP for an infiltration process in *in situ* composite wires with Cu stabilizer (without any expensive Nb barrier) **due to the solid state process and further high pressure infiltration in the obtained MgB_2 nano grains . The high densification, and the best possible connectivity with limited grains growth was performed at 1 GPa isostatic pressure** see; (A.Morawski B. Głowacki, - MT20 & Hipermag EU project reports).



Where is a limit of the stress apllied to the MgB_2 ceramic at ambient temperature?



Stress in the steel matrix of a multifilamentary conductor during manufacture. **The critical current drops rapidly above the marked strain**, corresponding to a wire diameter of around 0.9 mm. – **the core break effect** (B.Głowacki)

The High Pressure (HIP) and (HP) Processes adapted for MgB_2 wires and tapes preparation in these investigations:

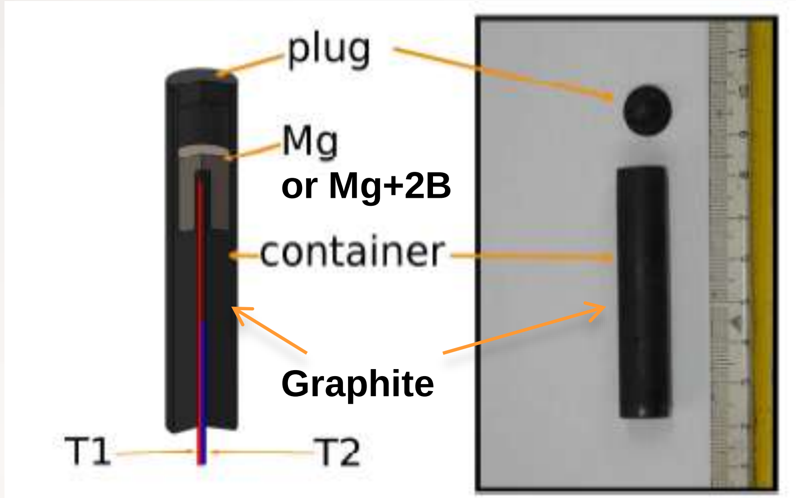
- HIP up to 1.6 GPa in argon gas medium, Temp up to 1200 ^{o}C
- HIP up to 1 GPa in liquid medium KCl+ NaCl, silicon oil (450 -700 ^{o}C)
- HP up to 0.3 GPa in solid - plastic medium 0.3 GPa (BN, graphite)

Hot Isostatic Pressing – HIP- laboratory gas medium equipment

High Pressure HIP — (Hot Isostatic Pressing) chamber up to **1.6 GPa** used for MgB_2

Pressure – up to 1.6 GPa (argon, nitrogen, helium & oxygen up to 0.2 GPa),
Temperature (max 2100 deg C) – here from 550 to 1200 ^{o}C, typically 700 ^{o}C
Duration of the process (longest 120 hours) – here 5...1600 min

The components of the furnace set for HIP & DTA crucible construction



Furnace for HIP or DTA experiment

High pressure plug

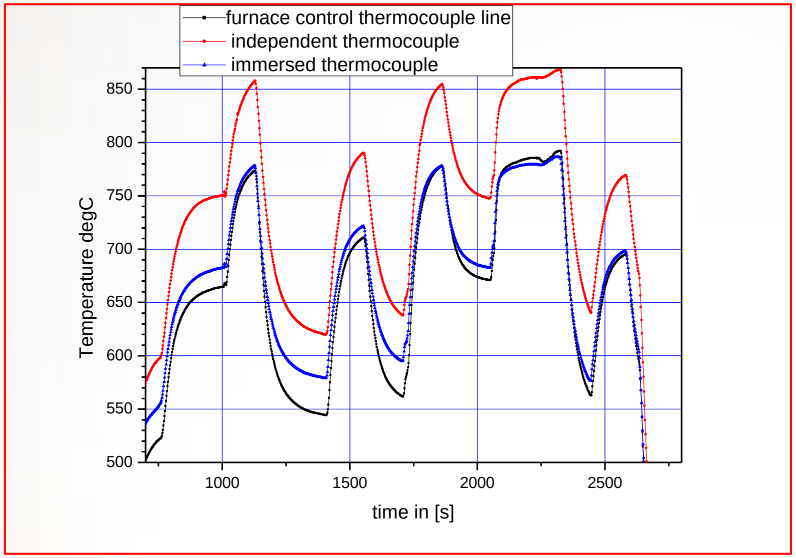
Fit trough and reference thermocoupl
type T

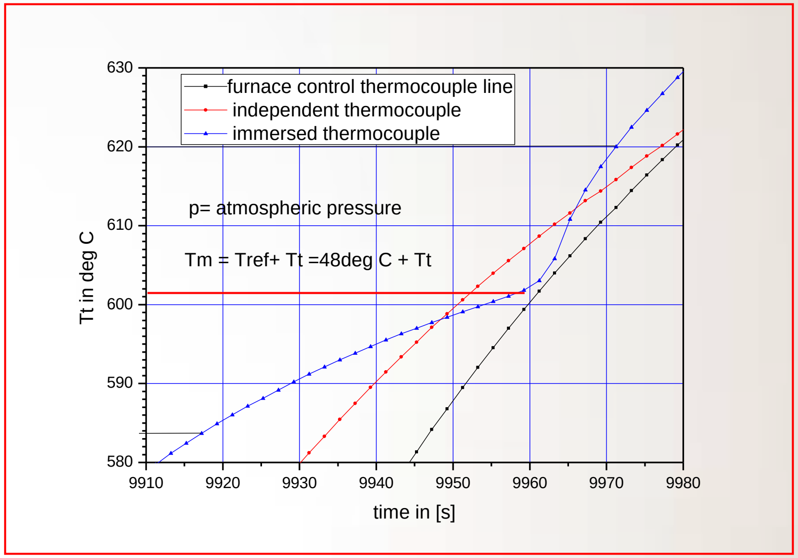
Outside and inside thermocouples
type K

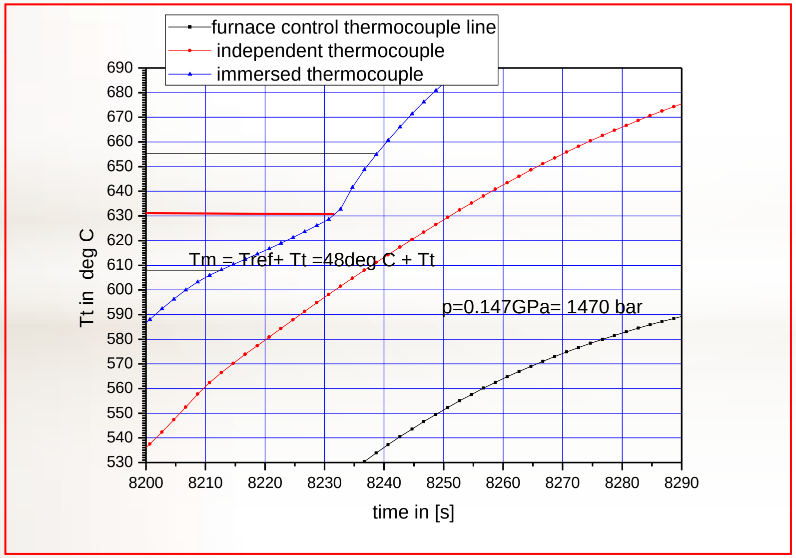
Cu tube like conteiner

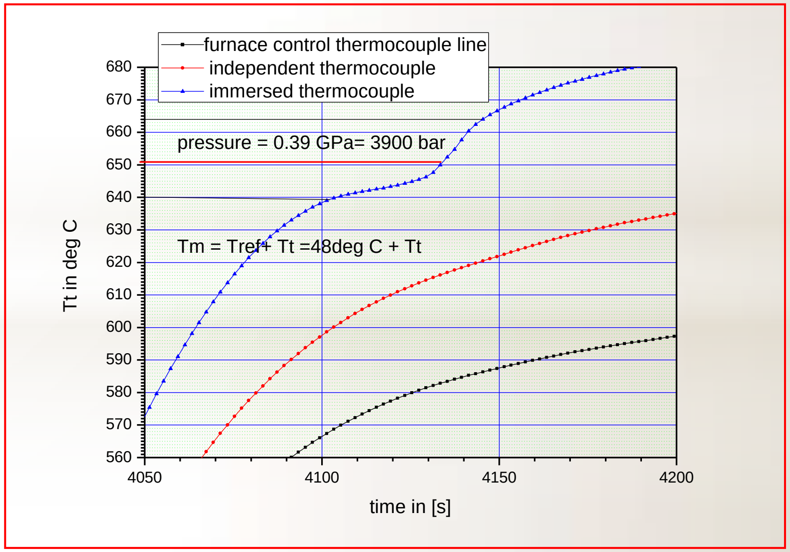
Alumina oxide tube & ampules

DTA profiles taken at various argon pressures for Mg melting experiment

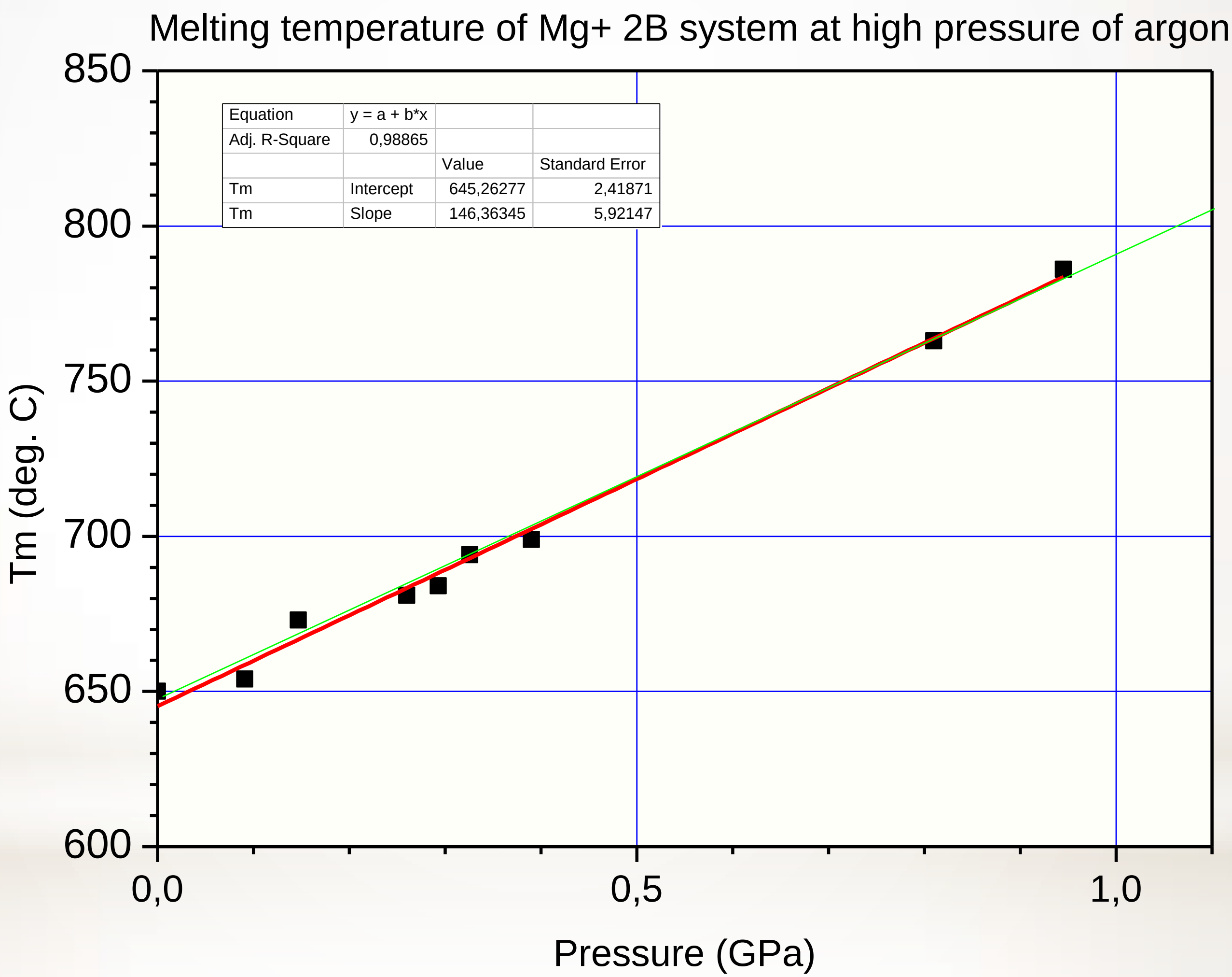




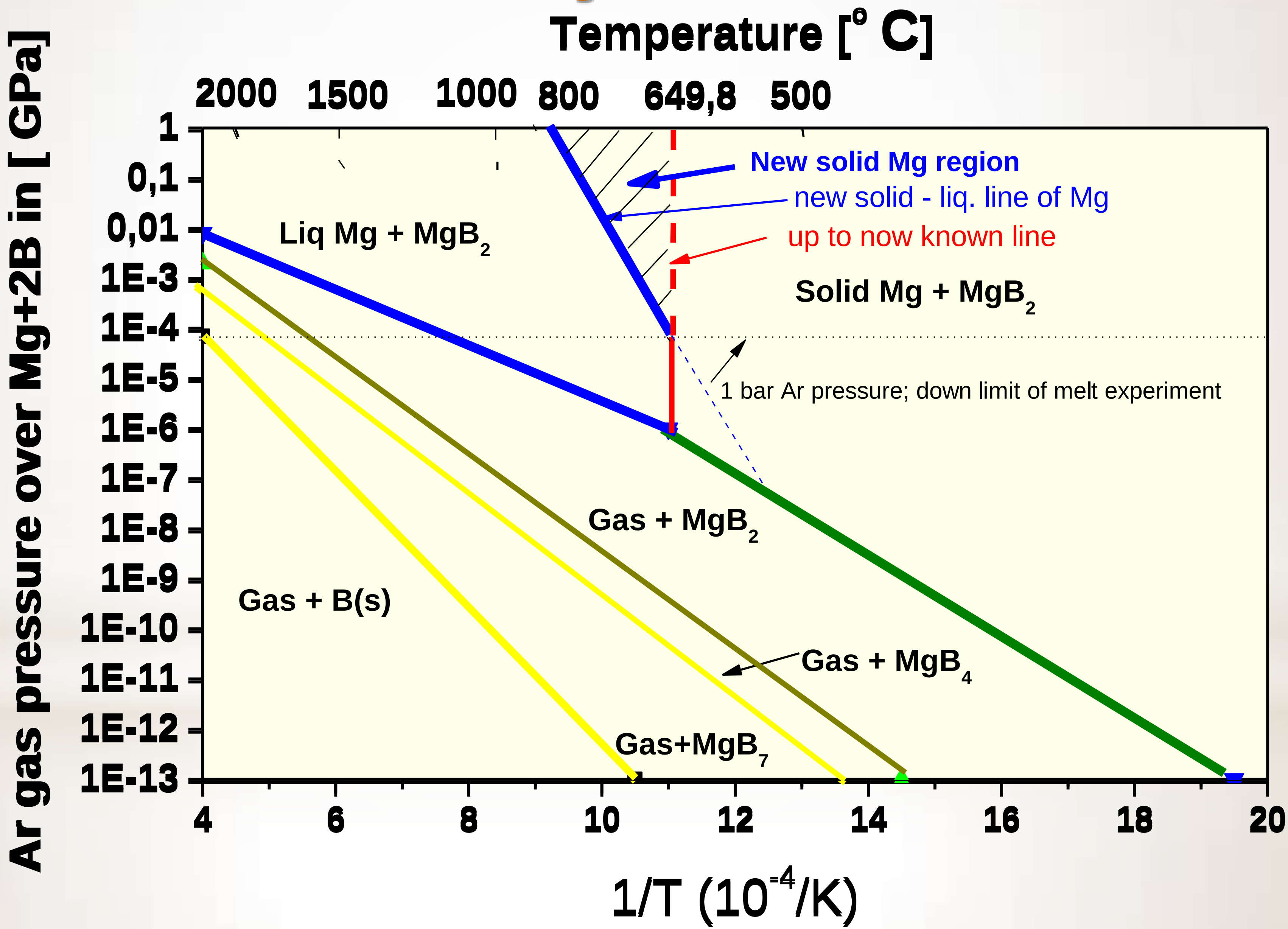




Melting temperature of pure Mg +2B at various Ar pressure (plot and fit)

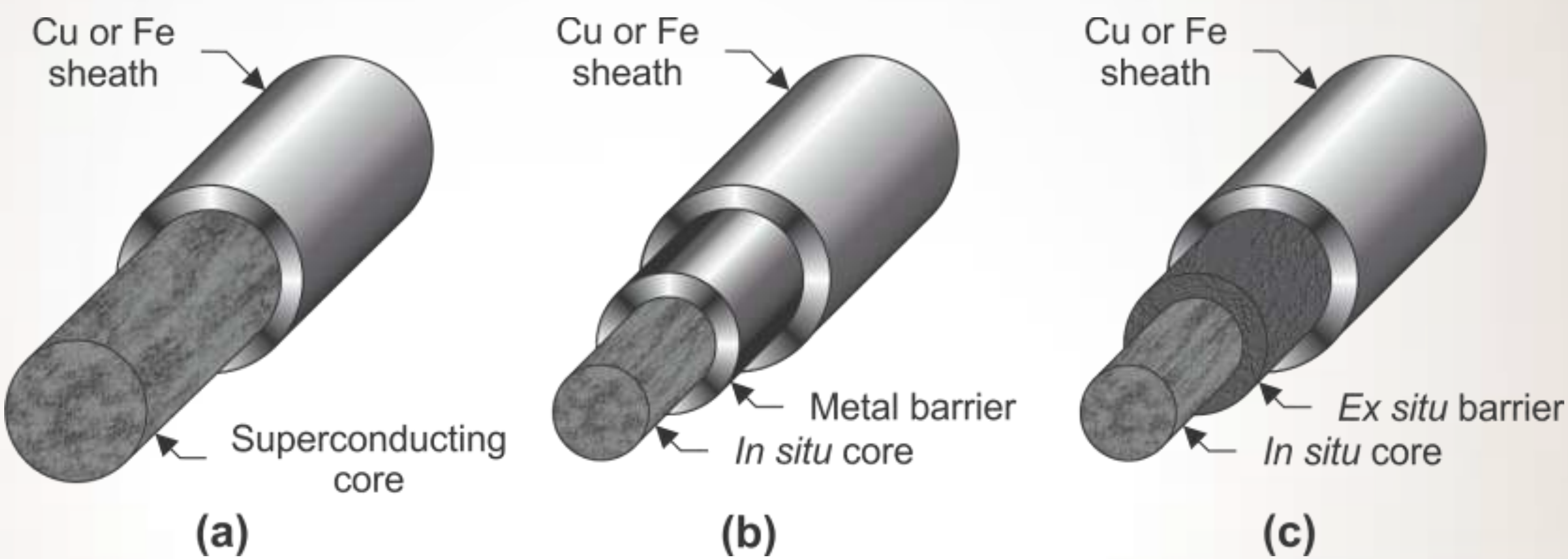


Experimentally modified Mg-B phase diagram after high gas pressure investigations



PIT concept technology

FF~0.15 FF~0.50



(a)Traditional PIT concept; (b) PIT with metal **Nb** barrier; typical for e.g. **HyperTech** (c) The new concept of PIT The ***in/ex situ***/ ***Cu***-stabilizer; **patent**

Title:	Composite Electrical Conductors and Method for Their Manufacture
Inventors:	Dr Bartek Glowacki and Dr Andrzej Morawski
Filing Date:	10 April 2007
Filing No:	GB 0706919.8
Country:	Great Britain
Title:	Composite Electrical Conductors and Method for Their Manufacture
Inventors:	Dr Bartek Glowacki and Dr Andrzej Morawski
Filing Date:	10 April 2007
Filing No:	60/907590
Country:	USA

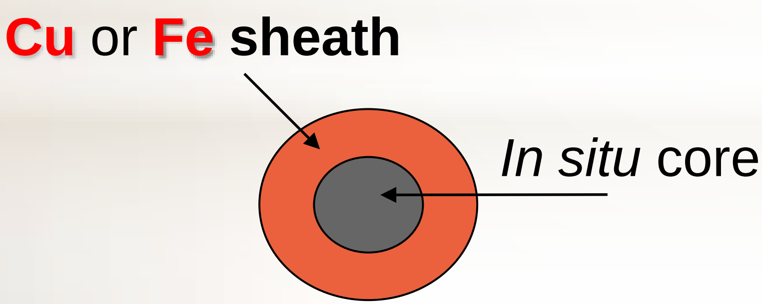
Sheathed composite superconducting MgB_2 wires

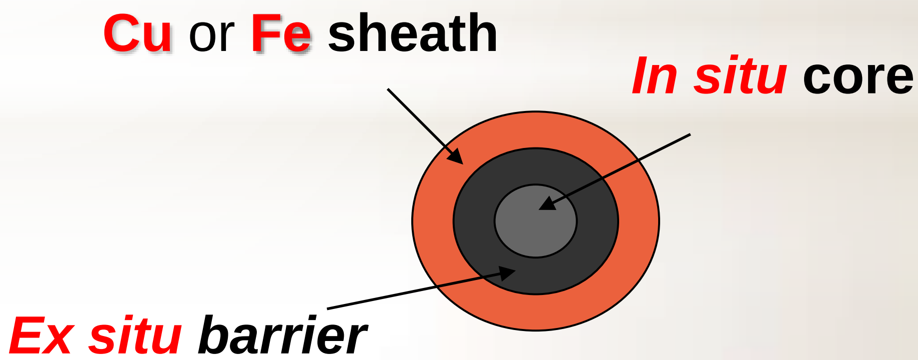
Sheathed MgB_2 superconducting wires — Cu- and Fe-
Easy to produce long tape or wire

Advantages of Cu. Why Cu copmposite sheath?
Presence of Cu as stabiliser for cryogenic stability. High thermal conductivity of Cu. Minimum quantity of stabilizer is required. Chemical stability. Mechanical stability, good mechanical properties. Low material cost. Length of composite wires over few km

Problems of Cu-sheathed wires developemnt
Reaction between inner in situ and outer Fe or Cu sheath or expensive Nb barrier uses.

«Know how» - *ex situ* barrier in Fe or Cu /MgB_2 superconducting wires
Ex situ *as a* ***barrier*** *between inner in situ and outer Fe/Cu layers*

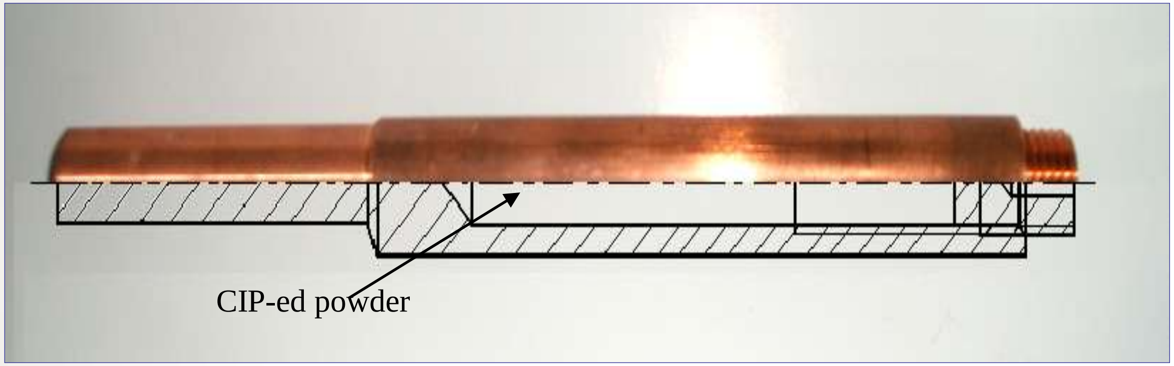




Preparation of the Cu-sheathed *ex/in* wires

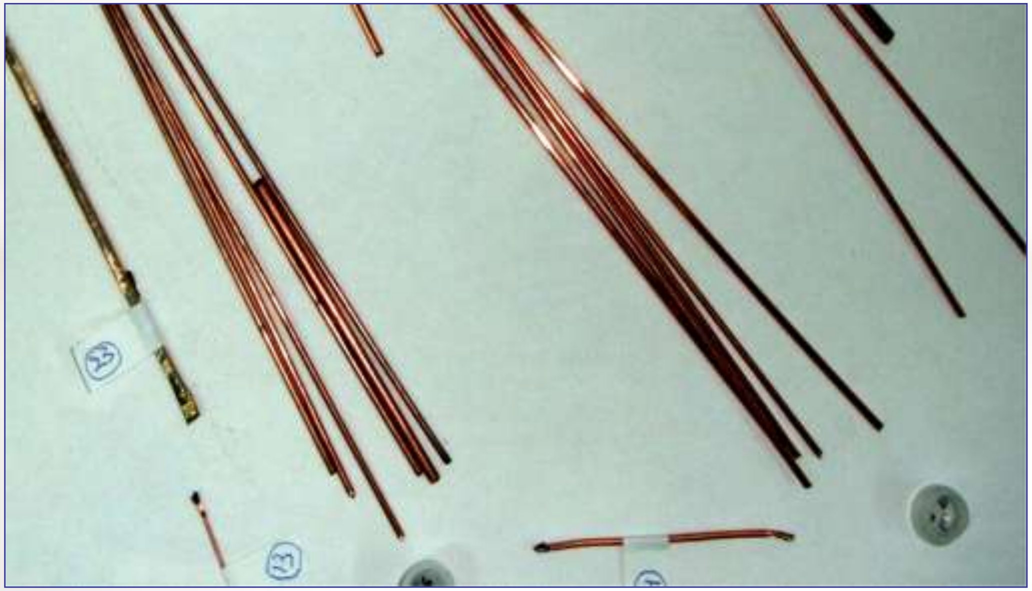
1. Powder compacting by CIP-in argon gas pressure medium from 0.8...1.0 GPa in rubber mould

2. Billet for drawing:



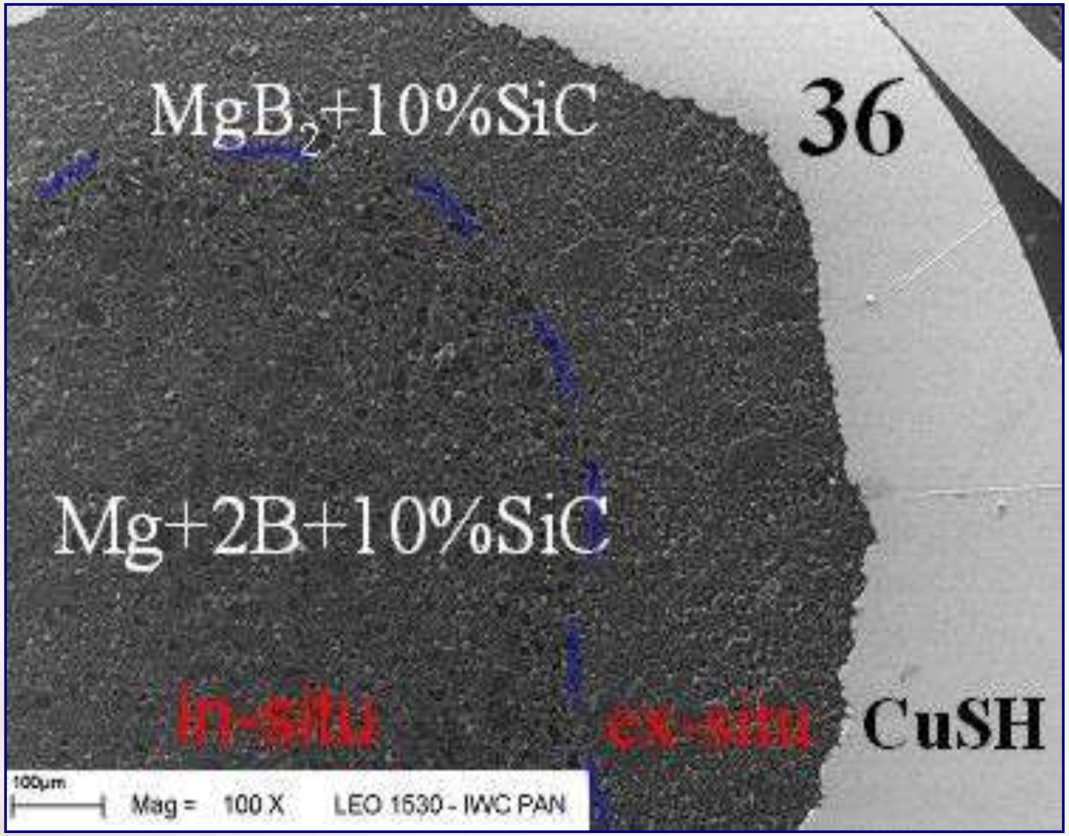
Length 120-620 mm
OD diameter – up to 38 mm
ID diameter – up to 24 mm

3. After drawing:



Length up to 500 m
Diameters 0.5...1.4 mm

The Cu/ex/in-situ technology and other high pressure powder processing designed and manufactured in IHPP-UNIPRESS since 2005

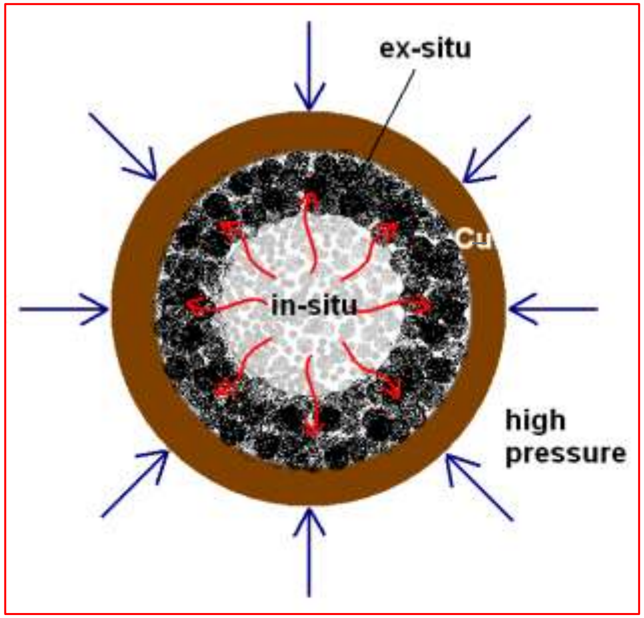


SEM crossection micrograph of the composite (ex/in) superconducting MgB_2 wire in the Cu sheath – before HIP

High Pressures Methods for cleaning and mixing of the nano and micro metric powders, ultrasonic high pressure US-HP and ultrasonic activated vacuum sublimation US-VS, have been worked-out and checked in practice in the specially made devices

The superconducting wire with Ø ca 1, mm diameter was made with the SCIP (Sequential Cold Isostatic Pressing) method, the own technological achievement of the group.

The high pressure infiltration process, presented by Unipress in HIPERMAG project meeting at 2005 & at MT-20 conference in Philadelphia 2006, followed by HIP-ing of the *ex/in situ* wires or tapes at high argon pressure



- The **highly stressed (over 1.3 GPa) nano-grains of dense MgB_2 net is produced by solid state reaction of *in situ* core ,below Tm temp. of (Mg +2B) mixture**

- **The self densification & infiltration is provided by migration / diffusion process which take place (belowe, but close to Tm;e.g. 570 -680 ^{0}C of (Mg +2B), under high pressure)**

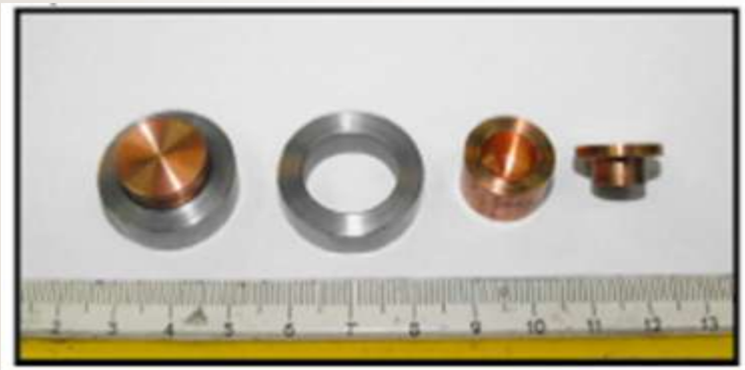
- **The effective diffusion barrier for Mg and Cu by *ex situ* hard micro grains & dense material is produced**
  (the apropiate quantity of nano blocker to the grains boundary is supplied)

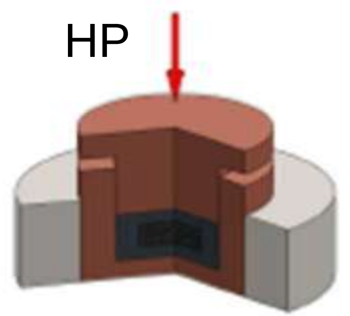
- **Simultanously the effective self-reinforcement by *ex situ* hard composite ceramic sheath is applied at elevated temp. at the second step of short HIP process (at temperature over 780^{o}C)**

- **Final HIP results in:**
  high densification of **highly sterssed , well connected** nano part of grains and aglomerates (of primary *in situ- actually ex situ) core* and the final high densifications and **sintering of both unificated *ex situ* cores**.

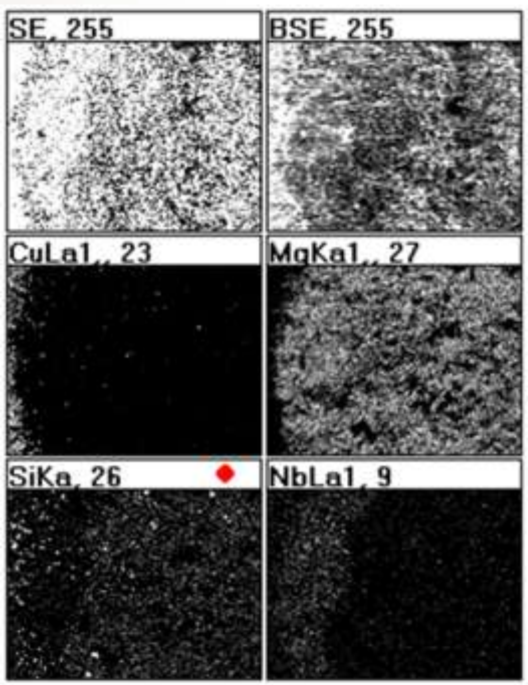
New composition of the *ex/in* barrier of the Unipress wires

HP - *ex situ* barrier investigations with nano additives doping

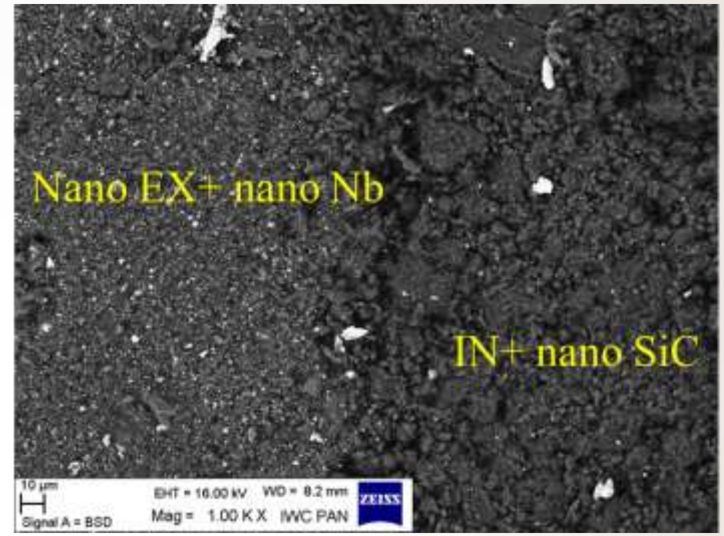




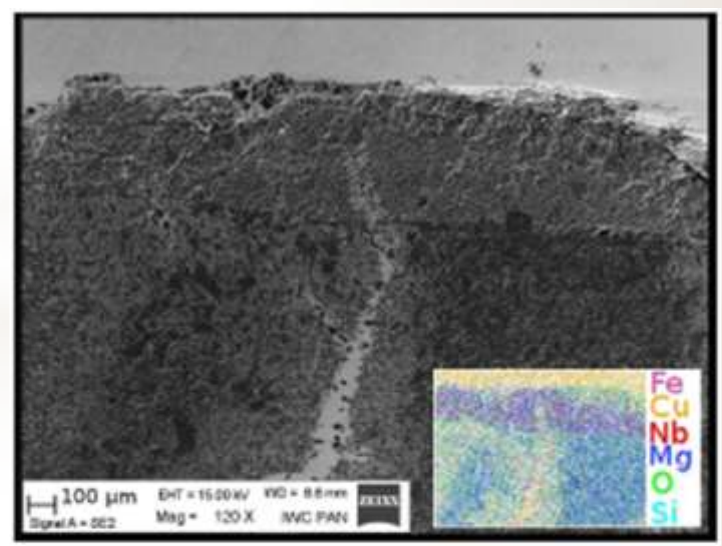
Cu/EX+nano SiC+nano Nb/In+nanoSiC



The cross section of the Cu / ex / in situ wire with nano additives



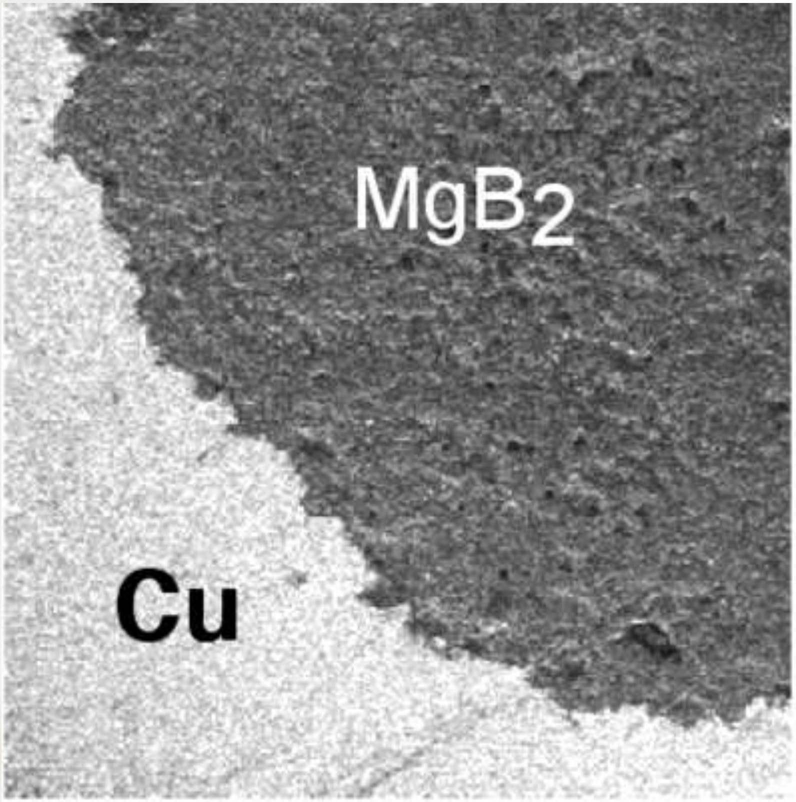
Before HP annealing



After HP annealing

What is the difference after HIP annealing?

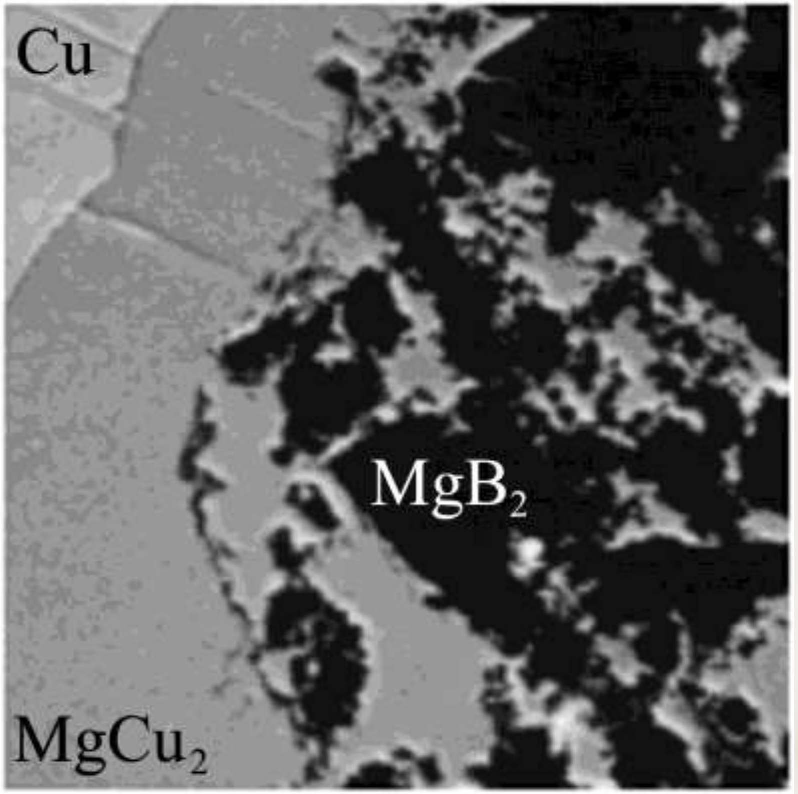
The Cu/ex/in concept of PIT



(a)

(a) The SEM micrograph of the coaxial *in situ* / *ex situ* wire after final HIP process: the *in situ* - *ex situ* interface can not be resolved, and there is any of a Cu-Mg layer at the copper-MgB_2 border

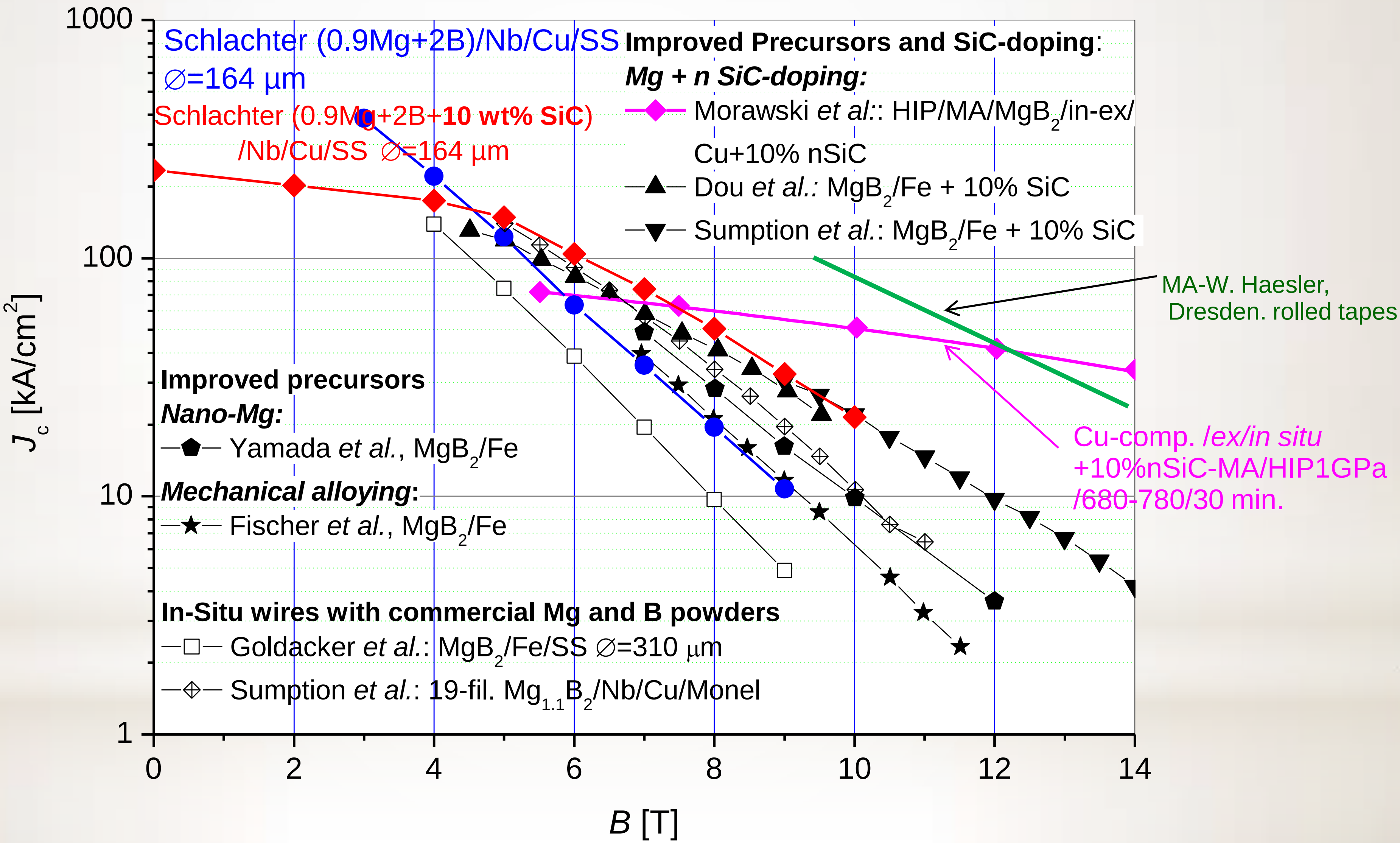
The traditional PIT in Cu



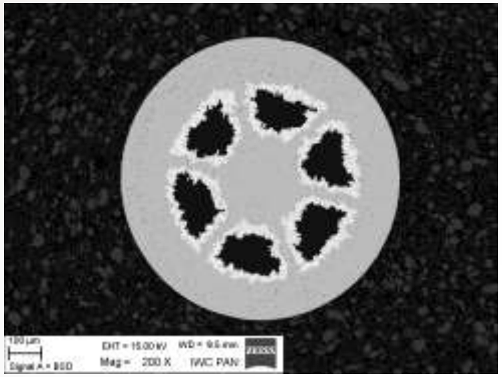
(b)

(b) The SEM micrograph of a conventional copper-matrix *in situ* MgB_2 wire for comparison: a large thick layer of $MgCu_2$ and Mg_2Cu exists at the copper MgB_2 border

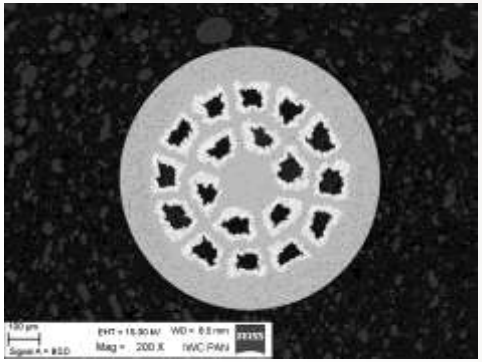
The critical current density vs. magnetic fields at 4.2 K - comparison



The Hypertech Inc. Columbus , Ohio, USA wires samples used for HIP investigation



Stand 1019-30



Stand 1019-43

OD of 0.63 and
OD of 0.83 mm multicore wires were investigated

Strand #	# Mon o	Barrier	Mono sheath	Multi sheath	B source	Mg:B	Additive	dia (mm)	% powder
961-03	6	Nb	Cu	Monel	SMI	1:2	C	0.83	17.7
961-18	6	Nb	Cu	Cu	99B	1.10:2	SiC	0.83	14.9
961-22	18	Fe	Cu	Glidcop	Ts	1:2	$C_4H_6O_3$	0.83	13.9
1019-30	**6**	**Nb**	**Cu**	**Monel**	**99B**	**1.10:2**	**SiC**	**0.83/0.63**	**15**
1019-43	**18**	**Nb**	**Cu**	**Monel**	**99B**	**1.10:2**		**0.83/0.63**	**15**
961-70	1	Fe	Cu		Ts	1:2	$C_4H_6O_3$	0.83	28.7
961-76	6	Nb	Cu	Glidcop	99B	1.10:2	SiC	0.83	16.6
961-92	6	Nb	Cu	Cu	99B	1.10:2		0.83	19.4

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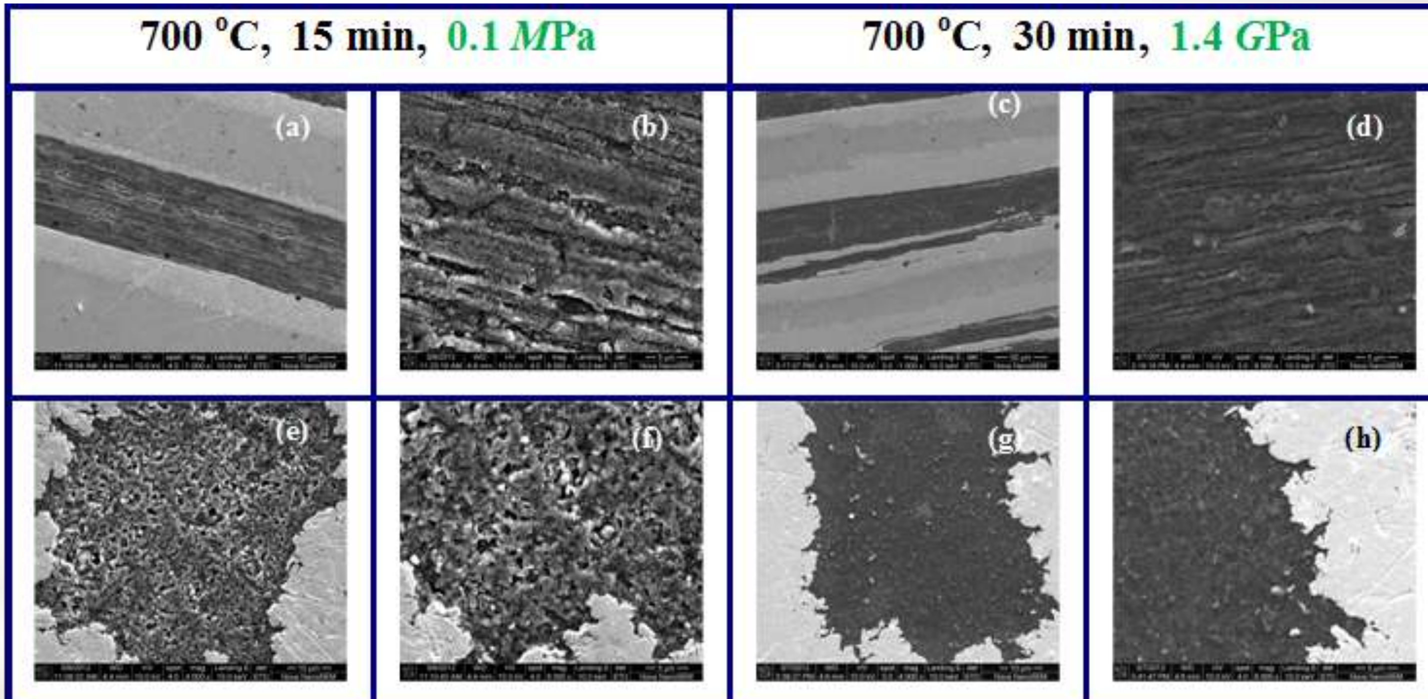
The microstructural SEM comparison of the pressure effect on the standard ambiante pressure annealing and high pressure HIP of pure MgB_2 samples

*Results for thicker **1019-43** wires: of 0.83 mm in diameter*

The standard ambient argon pressure annealing was performed at 700 ^oC/15 min. and the highest HIP applied pressure was the 1.4 GPa at 700 ^oC / 30 min.

The pressure influences on the microstructure and density of the cross section and longitudinal views is shown.

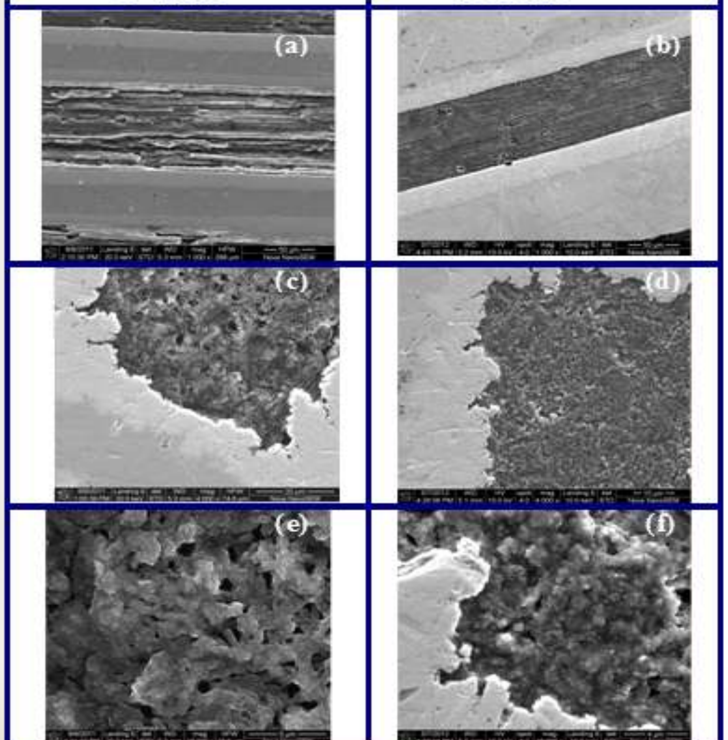


The effects of **pressure** on the microstructure of eighteen filament undoped MgB_2 wires, with Nb barrier, copper matrix and monel outer sheath, with 0.83 mm diameter. The SEM images of the microstructure were made in SE mode. The (a) – (d) longitudinal section, and (e) – (h) cross-section.

See considerably higher density of the 1.4 GPa HIP made sample.

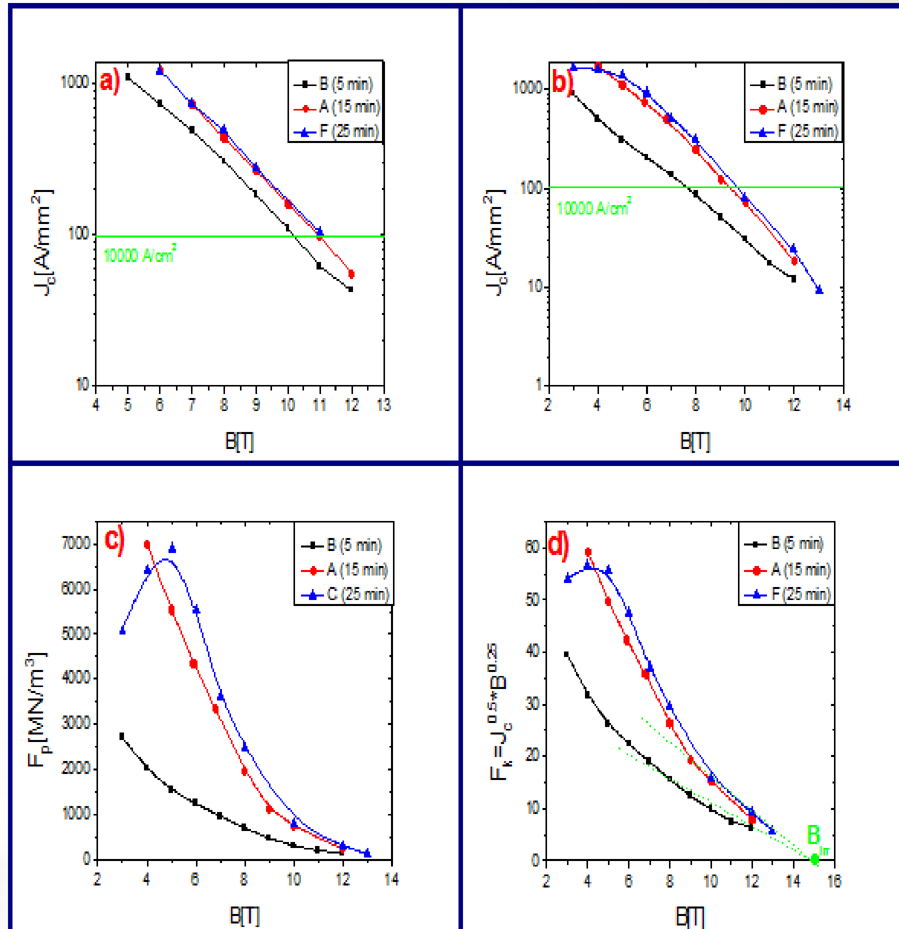
The 1 GPa constant pressure HIP- annealing time dependence on the Jc, F_p and microstructure of the core

700 °C, 15 min 1 GPa	700 °C, 25 min 1 GPa



The effects of annealing time on the microstructure of eighteen filaments undoped MgB_2 wires with Nb barrier, copper matrix and monel outer sheath with 0.83 mm diameter. The SEM images of the microstructure were taken in SE mode. The (a) – (d) longitudinal section, and (e) – (f) cross-section.

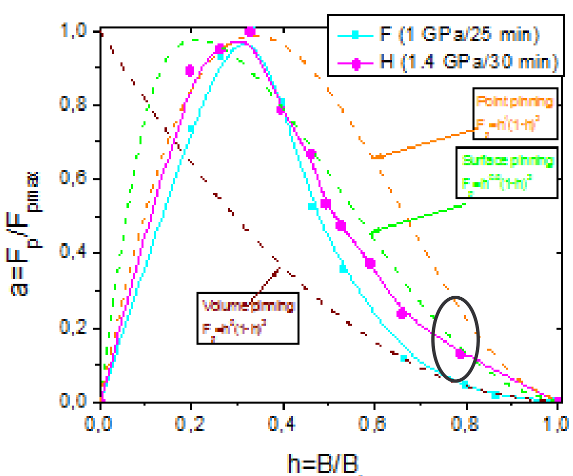
*Results for thicker **1019-43** wires: of 0.83 mm diameter*



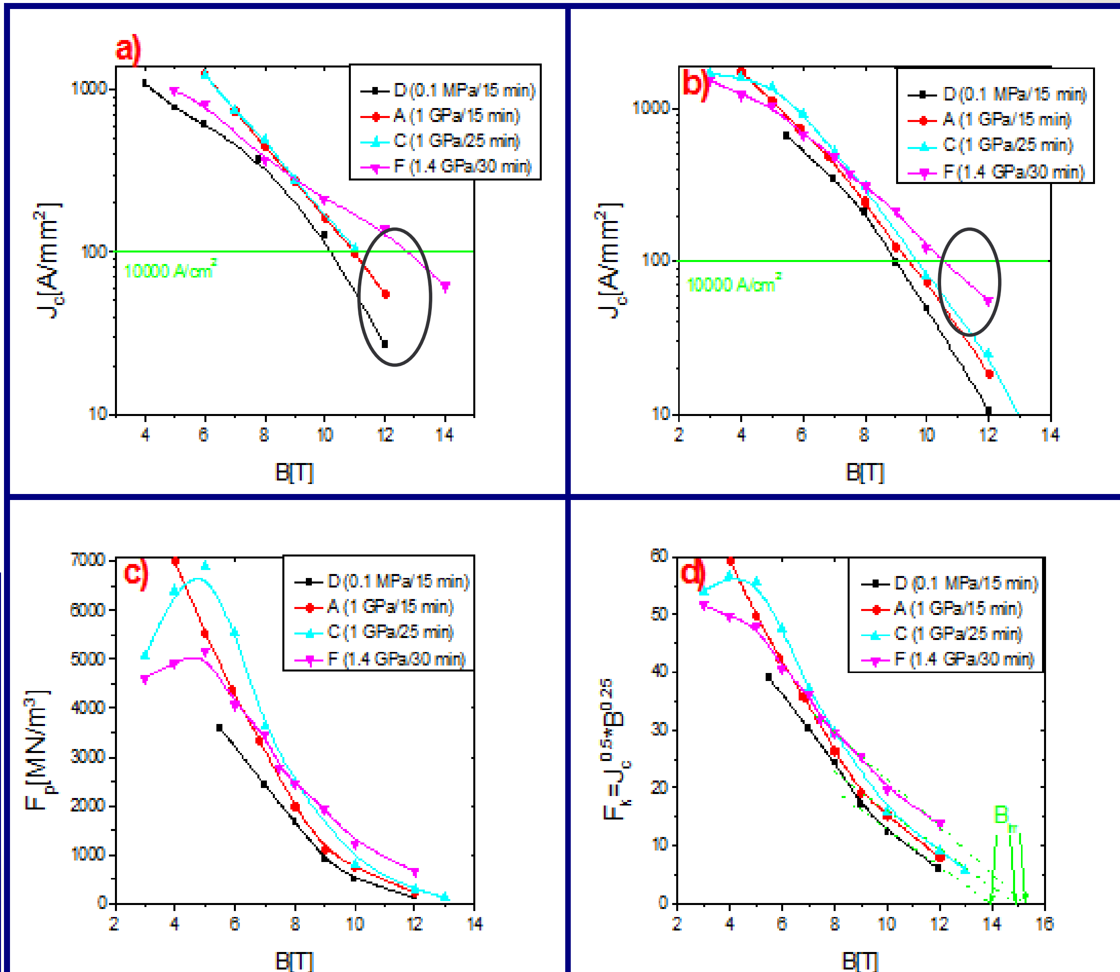
(a) The transport J_c-B curves for undoped MgB_2 wires in paralle to the axis magnetic field, (b): The transport J_c-B curves for undoped MgB_2 wires in perpendicular to the axis magnetic field; (c) The pinning force density F_p as function of perpendicular magnetic field B; (d) The change of Kramer's plot $J^{0.5}B^{0.25}$ as a function of perpendicular, to the wires axis, magnetic field B.

The **high pressure** HIP effect on J_c and F_p rises at high magnetic fields, especially in comparison to the standard ambient pressure annealing parameters at 700°C.

*Results for thicker **1019-43** wires: of 0.83 mm in diameter*



The reduced pinning force density as a function of perpendicular to the wire axis magnetic field.

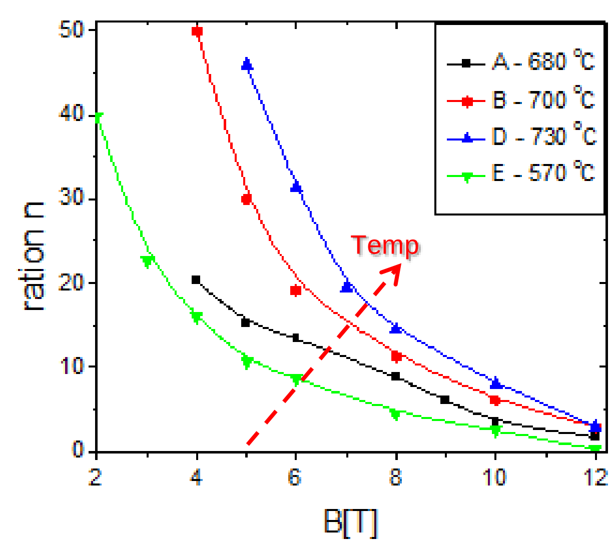


(a) The transport J_c-B curves for undoped MgB_2 wires in paralle to the wire axis magnetic field, (b): The transport J_c-B curves for undoped MgB_2 wires in perpendicular to the wire axis magnetic field; (c) The pinning force density F_p as function of perpendicular to the wire axis magnetic field B; (d) The change of $J^{0.5}B^{0.25}$ plot as a function of perpendicular, to the wire axis, magnetic field B.

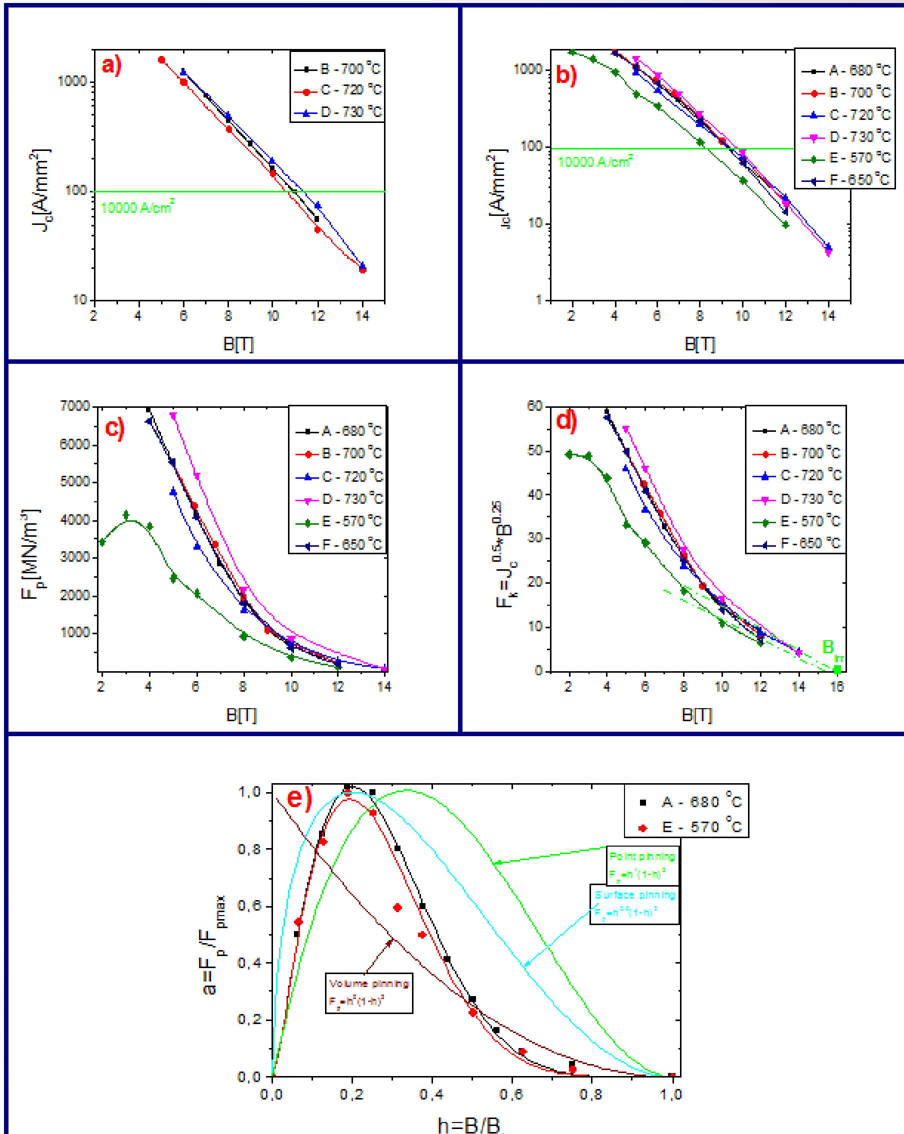
The critical transport parameters and n value of wires HIP-ed at equal 1GPa pressure and equal time of annealing but at **various temperatures.**

The J_c and F_p did not changed substantially but the **n-value parameter increase efficiently**, for the wires of **0. 83 mm** diameter with rising temperature.

*Results for thicker **1019-43** wires: of 0.83 mm in diameter*



The n - value dependence for perpendicular magnetic field orientation in undoped MgB_2 wires.



(a) The transport J_c-B curves for undoped MgB_2 wires in paralle magnetic field, (b): Transport J_c-B curves for undoped MgB_2 wires in perpendicular magnetic field; (c) The pinning force density F_p as function of perpendicular magnetic field B; (d) Change of $J^{0.5}B^{0.25}$ as function of perpendicular magnetic field B (e) The reduced pinning force density as function of perpendicular magnetic field.

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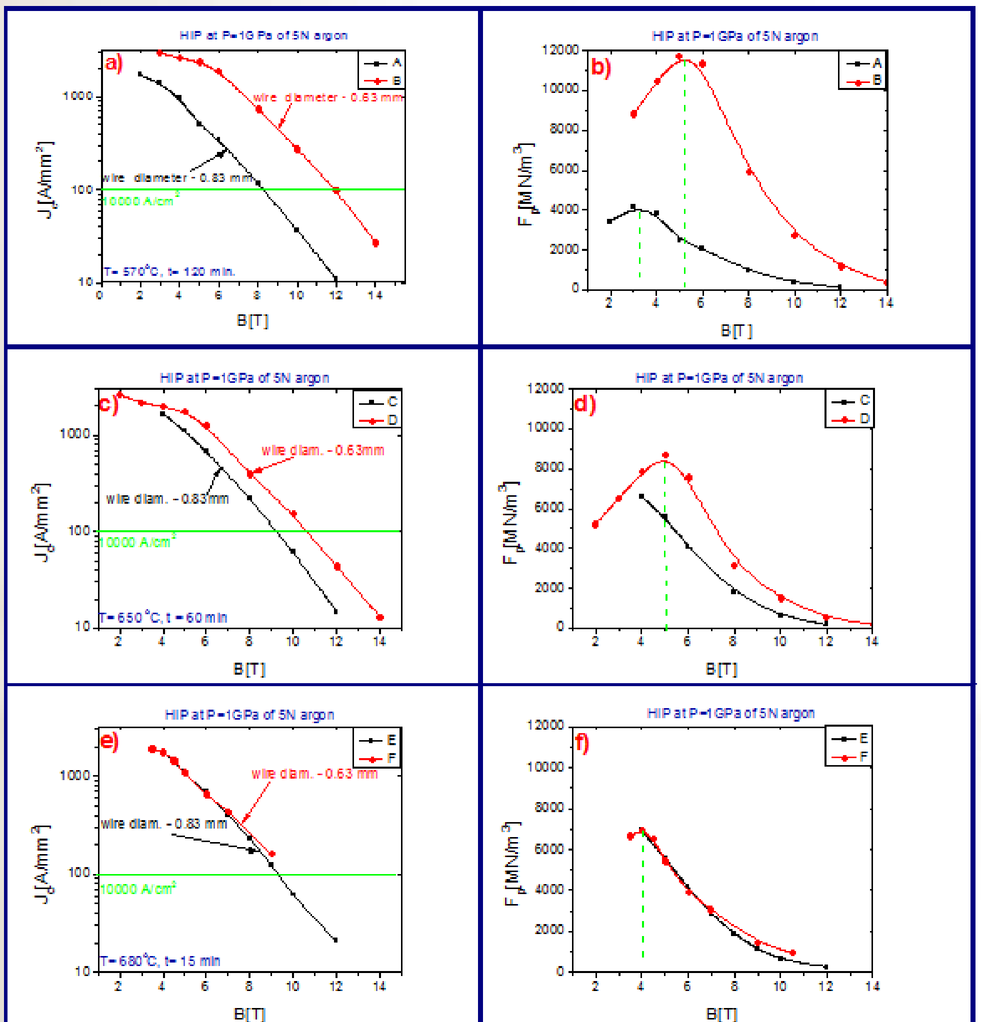
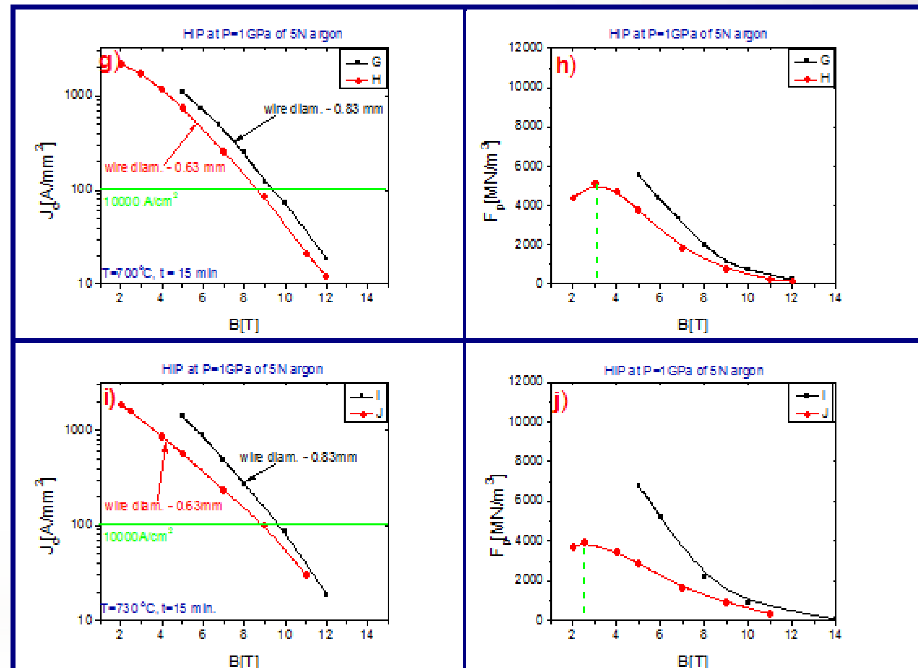
The J_c and F_p comparison of two different dimension of the wires obtained at exactly the same HIP experiment (e.g. pressure. temperature, and time of annealing).

See the evolution of the J_c & F_p properties of thin 0.63 mm and thick 0.83 mm wires, with increasing temperature of each HIP experiment.





(a), (c), (e), (g) and (i) Transport J_c-B curves for undoped MgB_2 wires in perpendicular magnetic field, (b), (d), (f), (h) and (j) The pinning force density F_p as function of perpendicular magnetic field for undoped MgB_2 wires.

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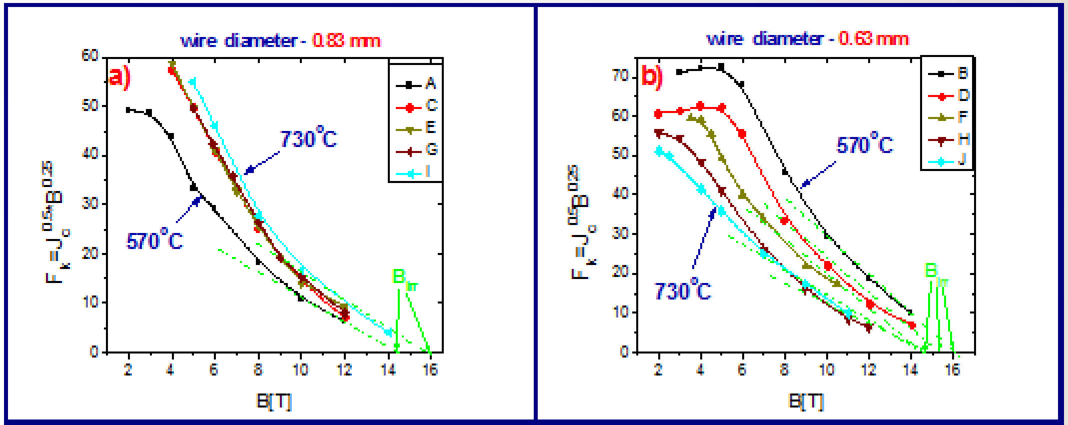
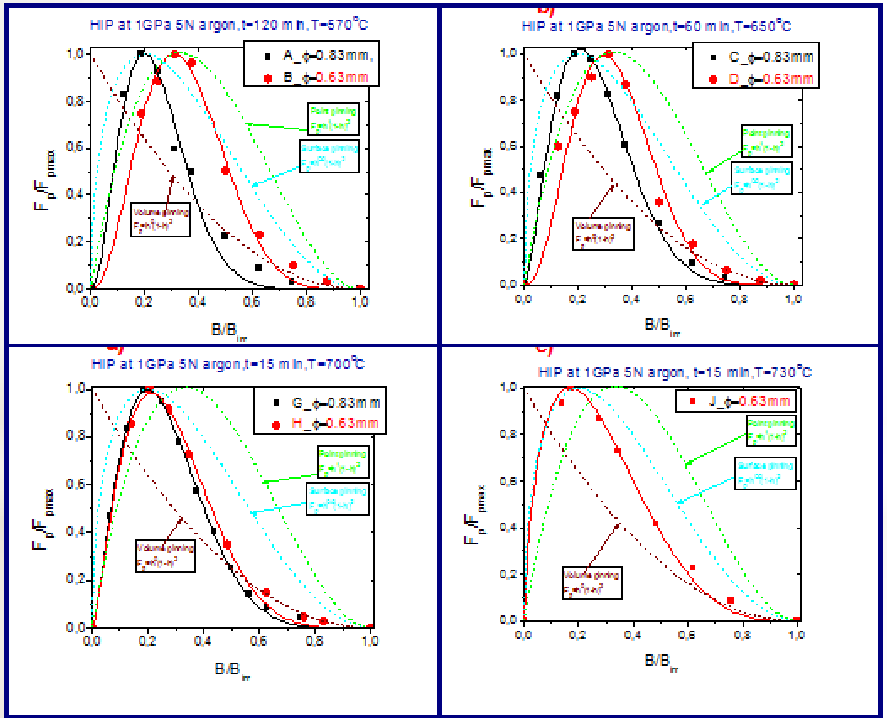
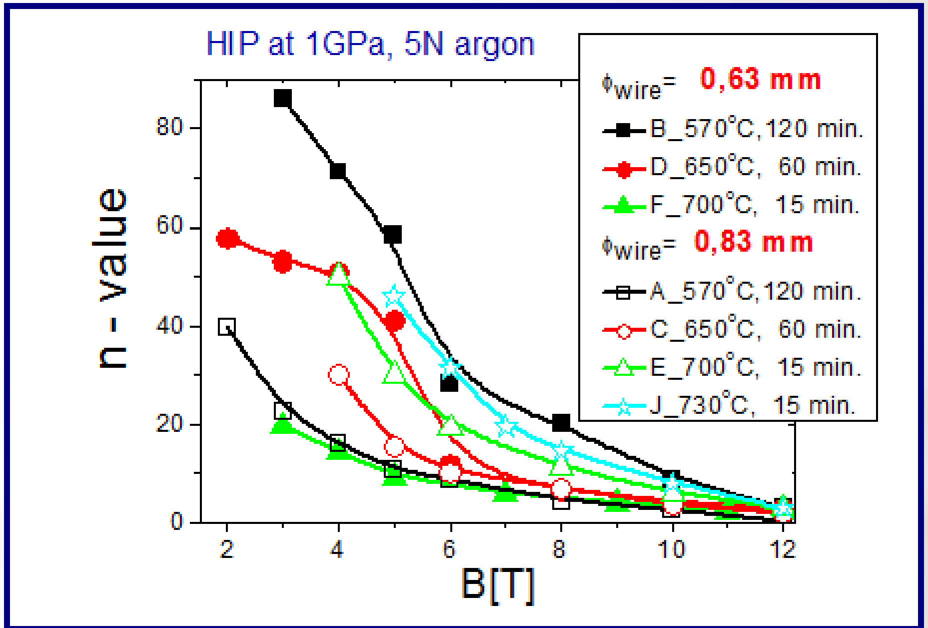
The Kramer's plots, reduced F_p and n- value dependence on the wires diameters and specific high pressure and temperature conditions.



(a) and (b) The change of $J^{0.5}B^{0.25}$ as a function of perpendicular to the wire axis magnetic field, for the 0.83mm and 0.63 mm wires diameter.



(a), (b), (c), and (d) The reduced pinning force density as a function of perpendicular to the wires axis magnetic field, for undoped MgB_2 wires for several (p,T,t) conditions and two specific deferent wire diameters.



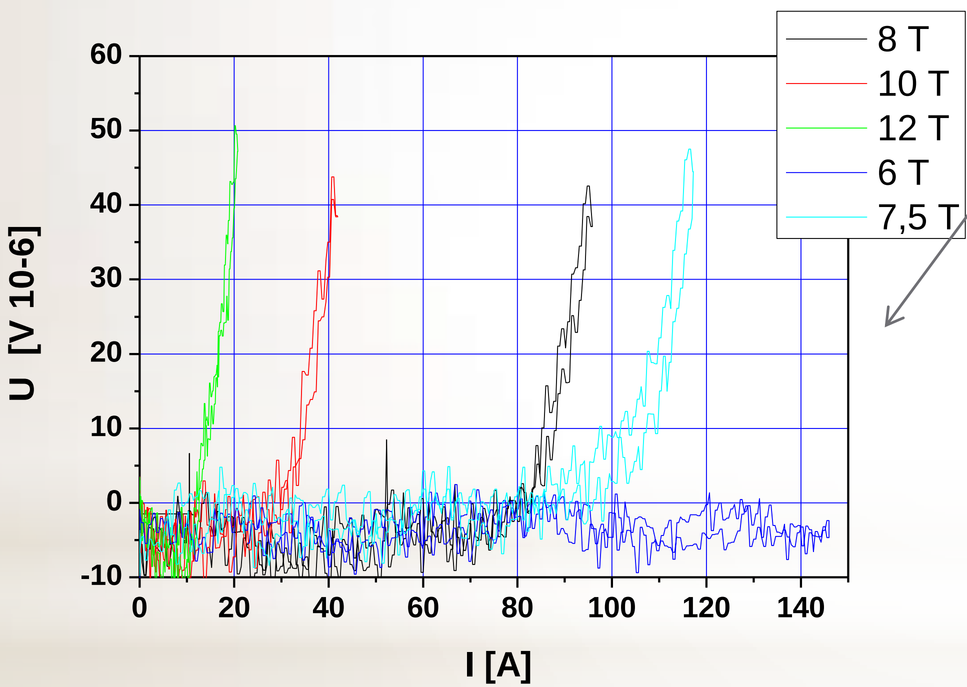
The n – value dependence of the two specified wires diameters obtained for the perpendicular to the wires axis magnetic field in undoped MgB_2 wires obtained at various temperatures and time annealing at the same 1 GPa pressure.

HyperTech Inc.- Unipress HIP-ed wires

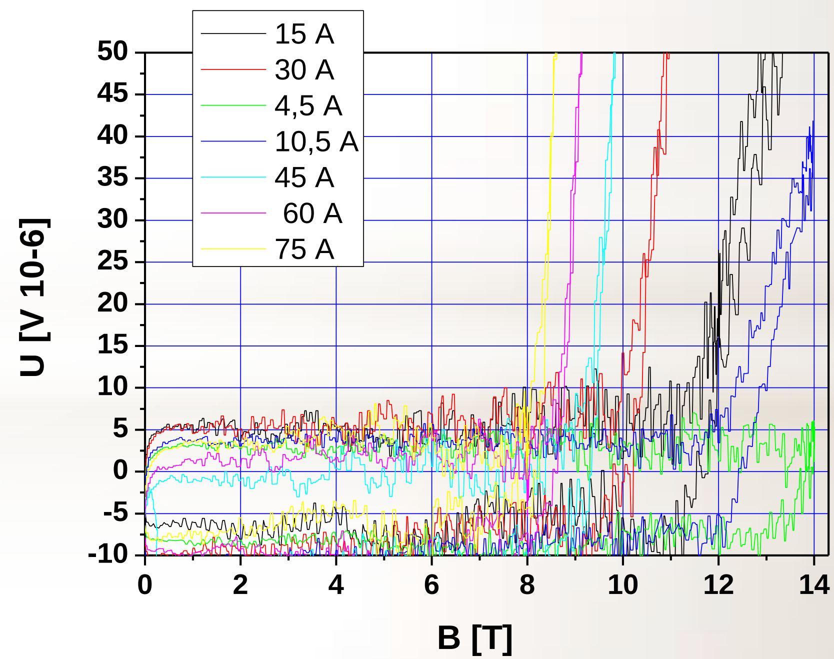
HU-1019-43 after 1 GPa / 710 ^{o}C ramp to 780 ^{o}C / 22 min. HIP in Ar

Pure MgB_2 any additives, amorphous boron, Nb barrier in Cu and Monel

The transition from superconducting to the normal state measured by resistive method at Bitter type magnet in current rise and magnetic field rise mode



Constant magnetic field (clasic)

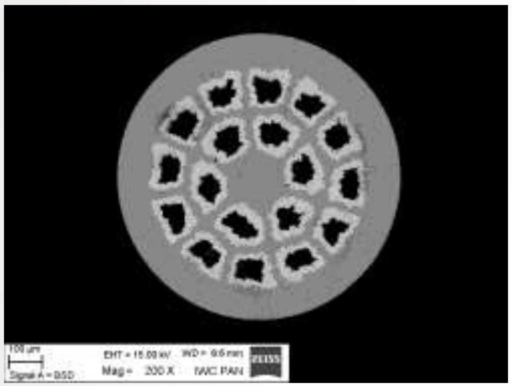
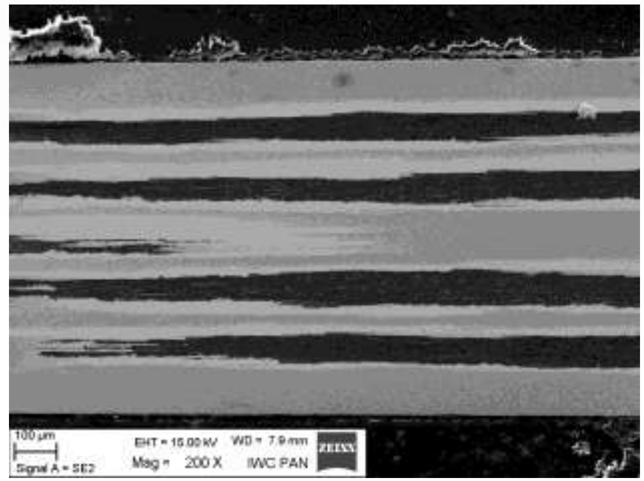
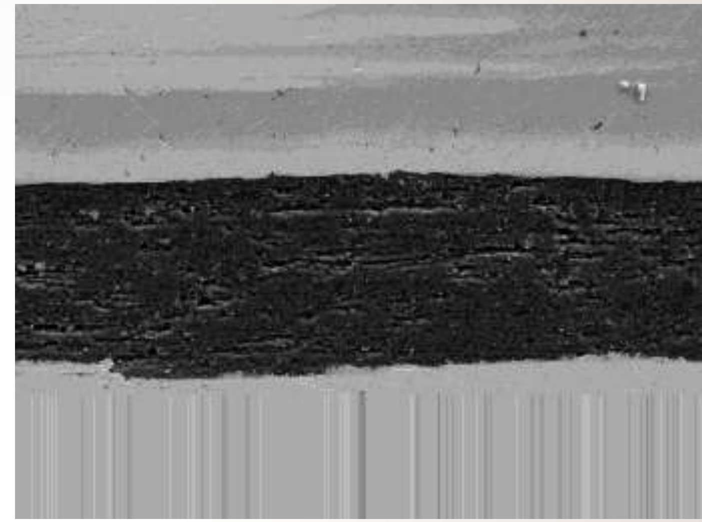


Constant current measurements

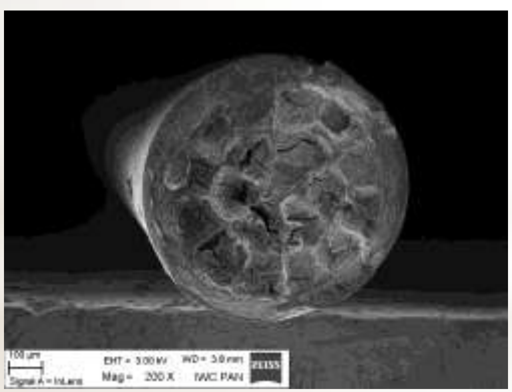
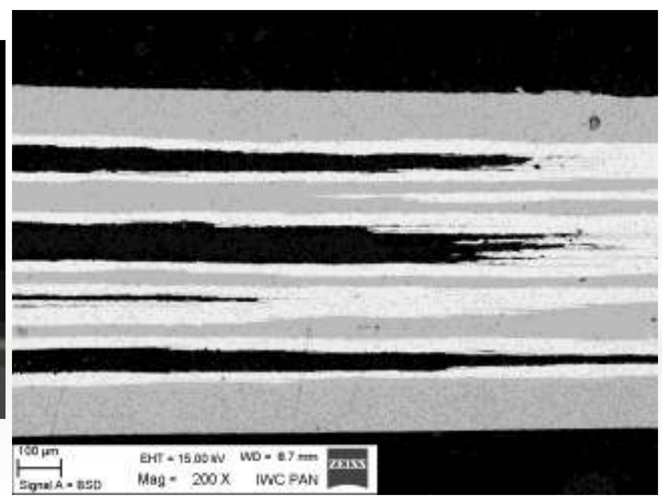
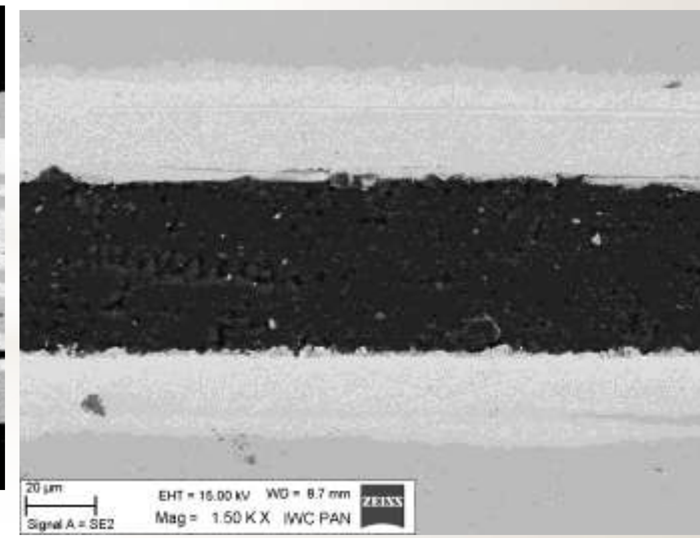
HU-1019-43 after 1 bar (upper line) & 1 GPa (lower line) / 710 °C ramp to 780 °C /in t=22 min
Pure MgB_2- any additives, amorphous boron, Nb barrier in Cu and Monel

HyperTech Inc.- Unipress HIP-ed wires

1 bar Ar

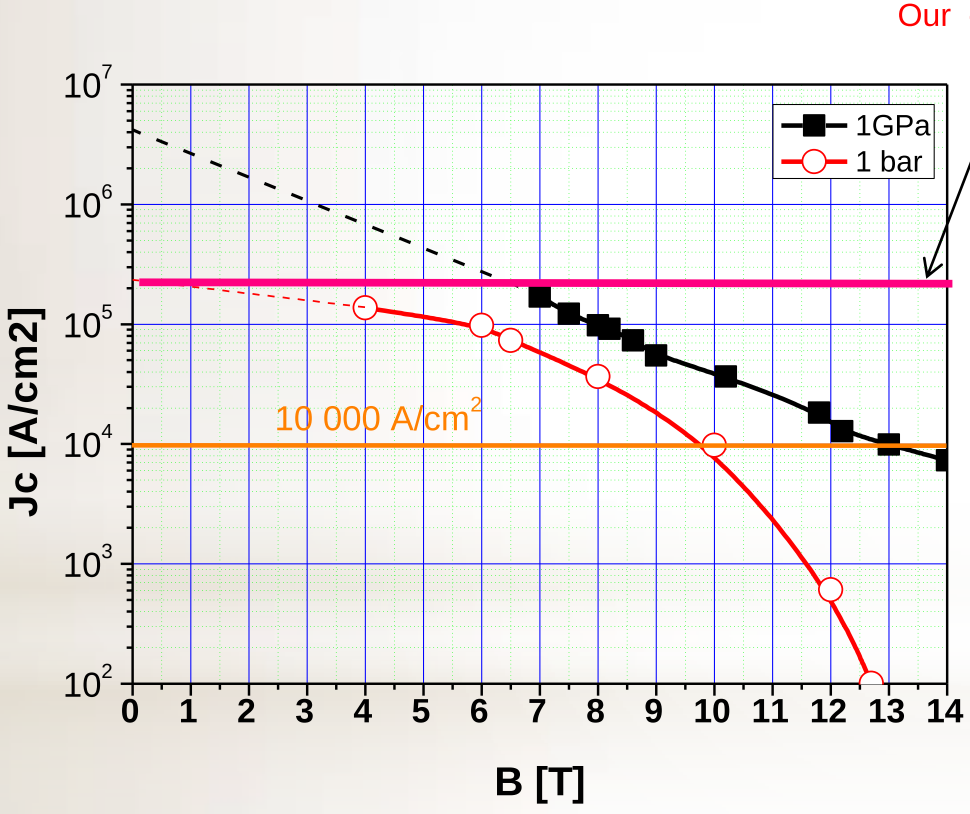




1 GPa Ar

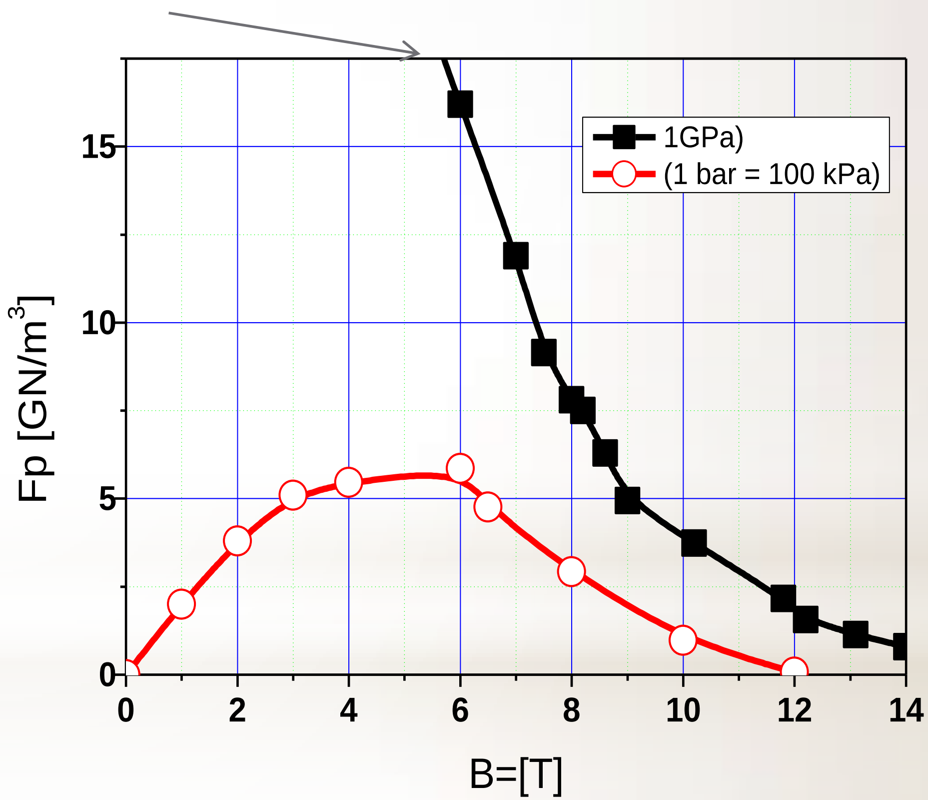




HU # 1019- 43 after 1 GPa / 710 °C ramp to 780 °C / 22 min. HIP in Ar
Pure MgB_2- any additives, amorphous boron, Nb barrier in Cu and Monel

HyperTech Inc.- Unipress HIP-ed wires



Critical current density at 4,2 K , I_ field



Pining force vs. magnetic I_ field

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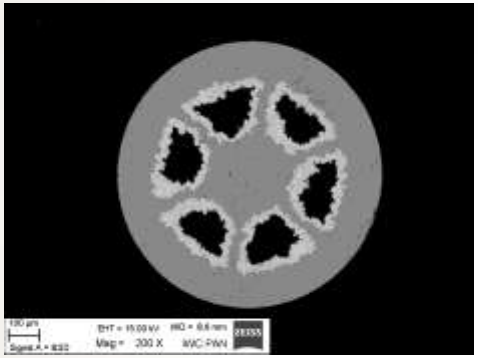
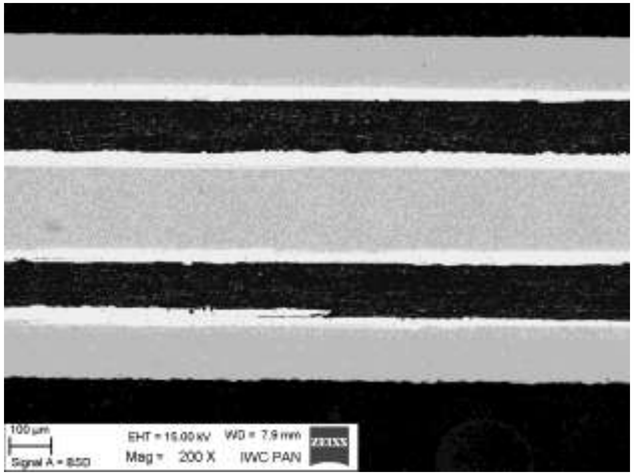
HU-1019-30 after 1 bar (upper line) & 1 GPa (down line) / 710 °C ramp to 780 °C/30 min
Nano SiC additives, amorphous boron, Nb barrier in Cu and Monel

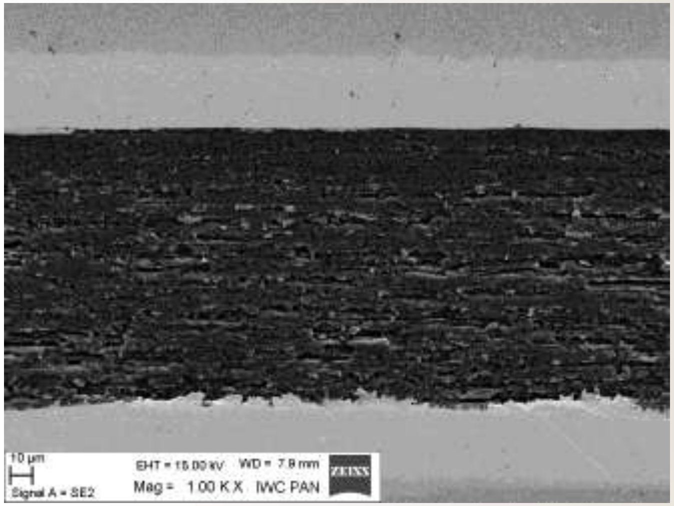
HyperTech - Unipress HIP-ed wires

Perpendicular and longitudinal cross section

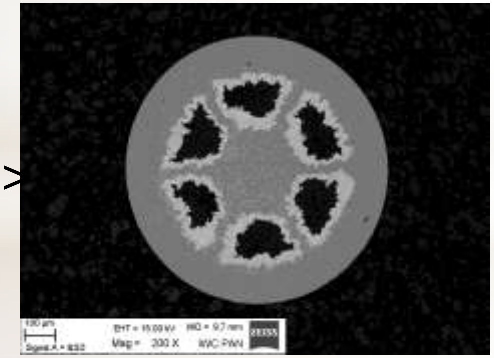
1 bar Ar =>

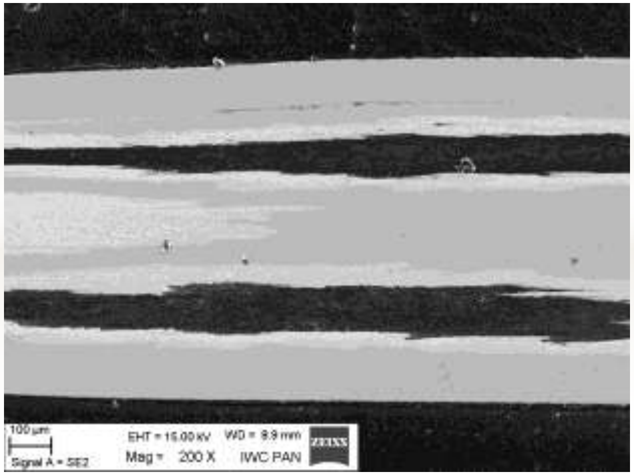


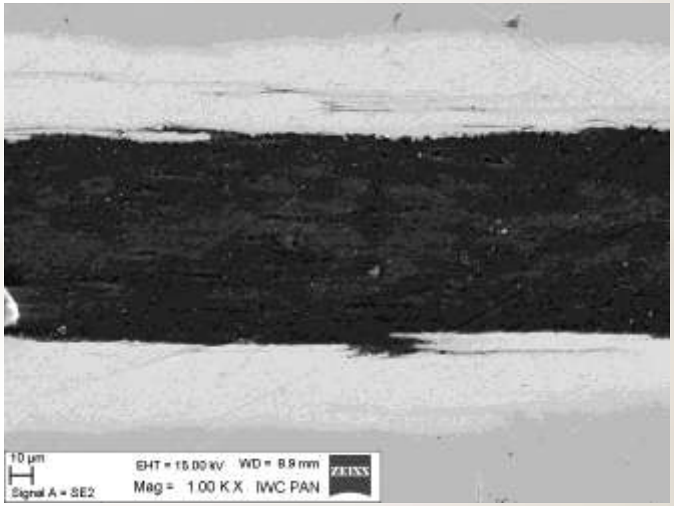




1 GPa Ar =>







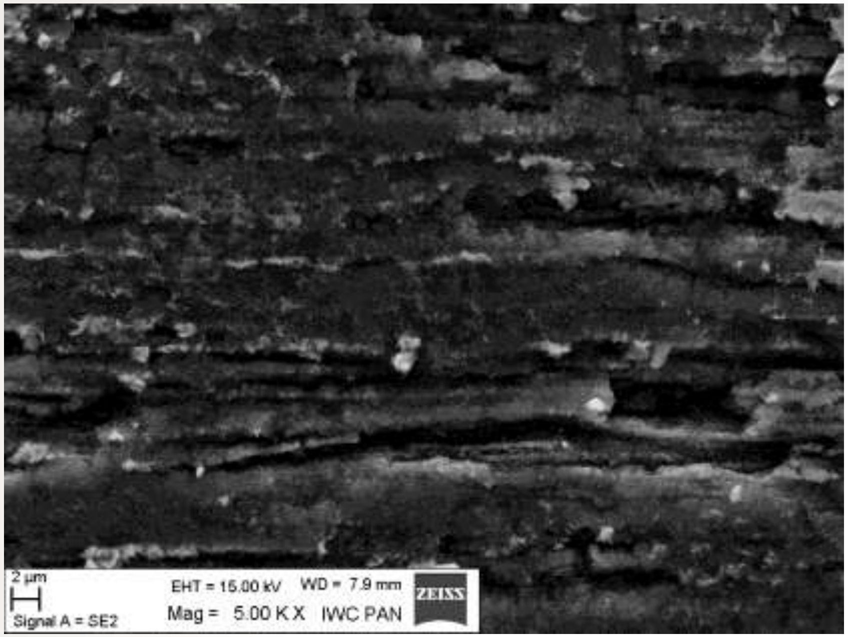
HU-1019-30 after 1 bar (upper line) & 1 GPa (down line) / 710 ^{o}C ramp to 770 ^{o}C /30 min

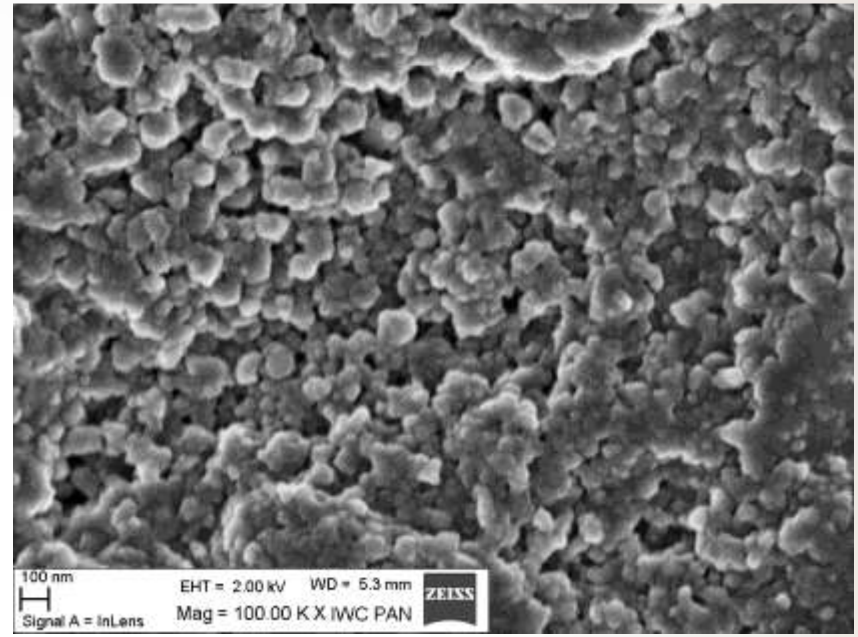
Nano SiC additives, amorphous boron, Nb barrier in Cu and Monel

Longitudinal cross section

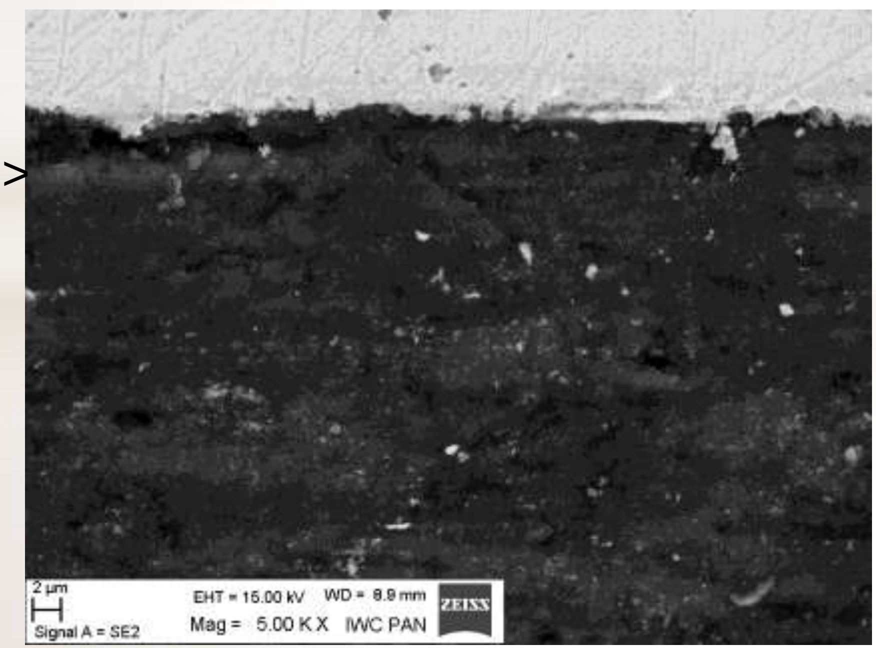
Broken wire, powder morfology

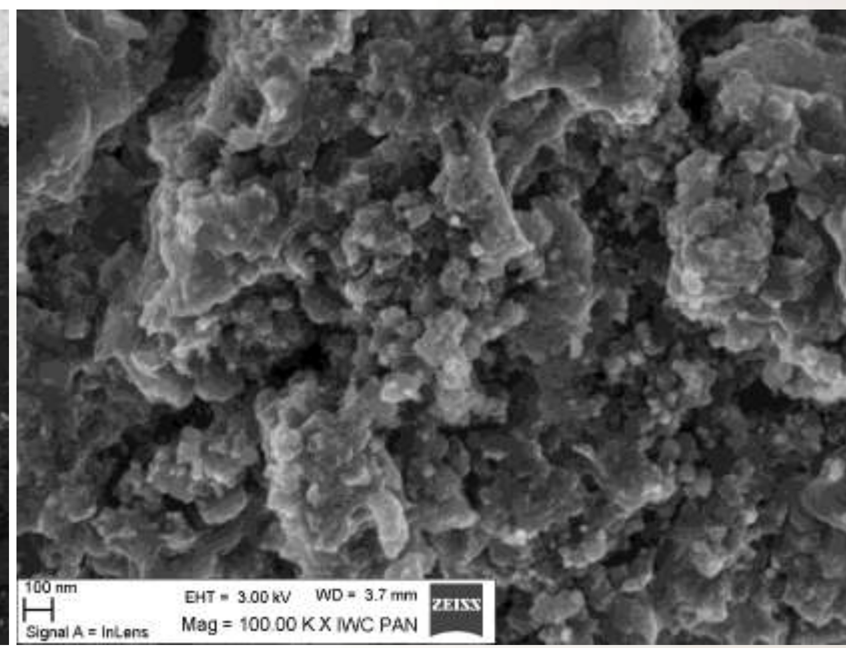
1 bar Ar =>





1 GPa Ar =>





The MgB_2 coils for neutron flux experiment destinated for HIP

The coils of 24 mm diam. of # 30 & # 43 wires after HIP and further neutron flux investigations in Wiena.

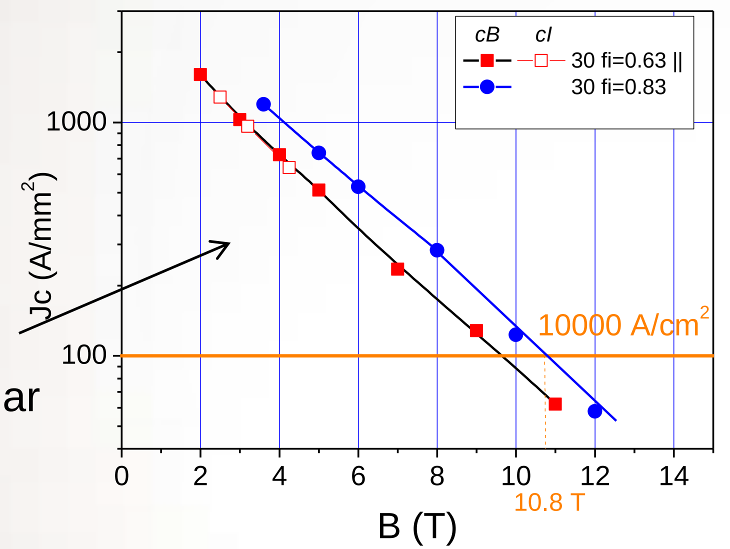
The inox-steel spools with Cu connectors of 12, 14 and 24 mm diameters for winding the HyperTech „green body" wires, destined later for HIP process and ITER regarding the neutrons flux examinations of Jc.

Hypertech - Unipress HIP-ed wires and 14 mm diam. coils

Nano SiC additives, amorphous boron, Nb barrier in Cu and Monel;

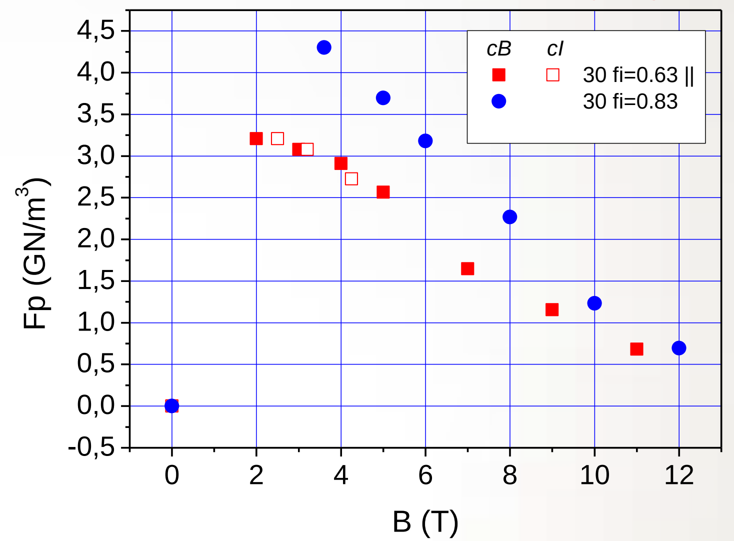
HU # 1019- 30 after HIP at various p,T, t , const. Temp. conditions

P= 1GPa; T=700^{0}C; 15 min - const. annealing temperature



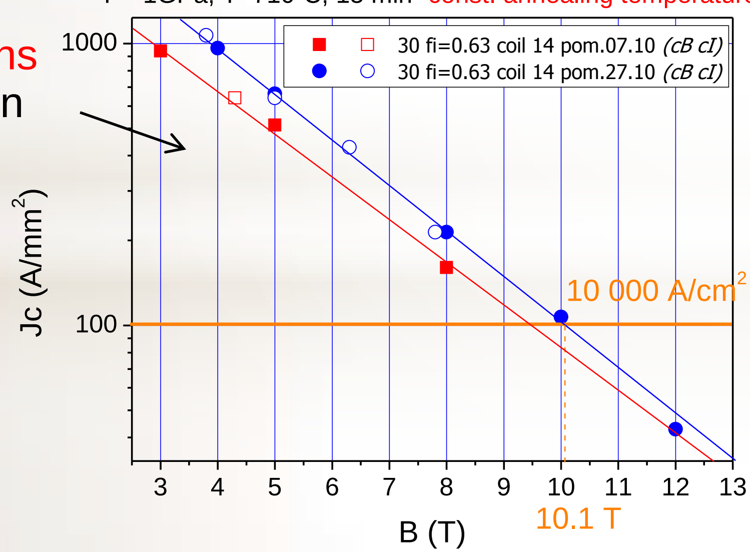
Wires from similar HIP process

P= 1GPa; T=700^{0}C; 15 min- const. annealing temperature

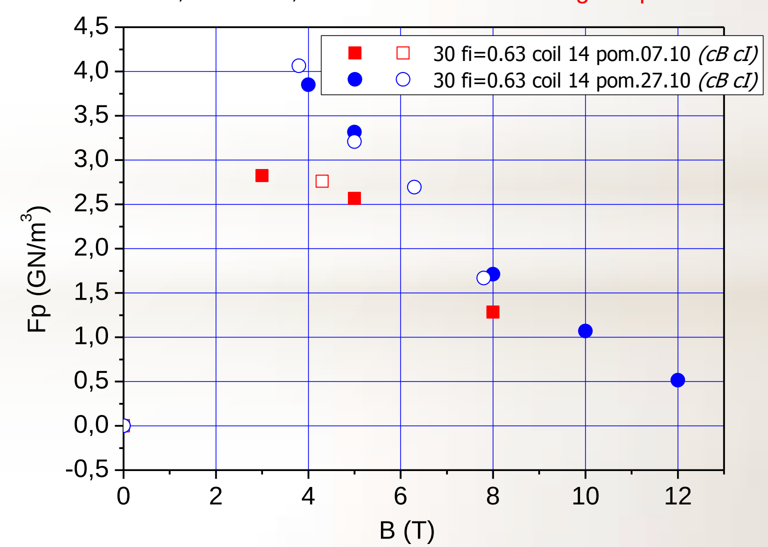


P= 1GPa; T=710^{0}C; 15 min- const. annealing temperature

Coils for neutrons flux examination

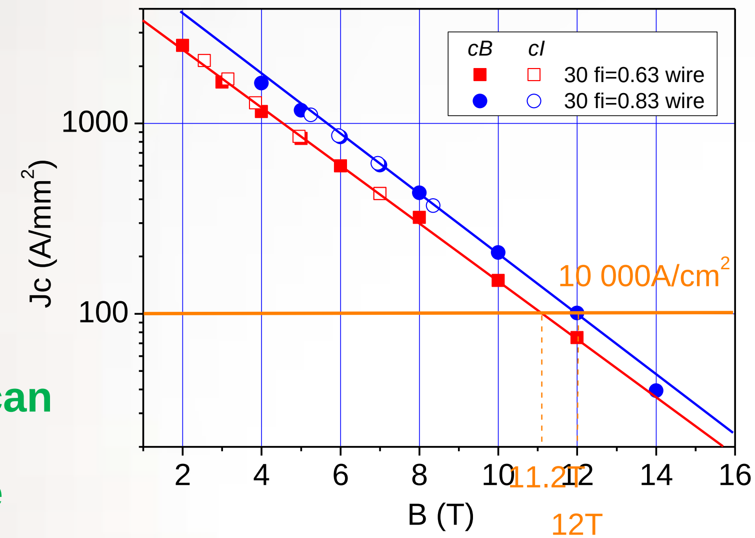


P= 1GPa; T=710^{0}C; 15 min.- const. annealing temperature

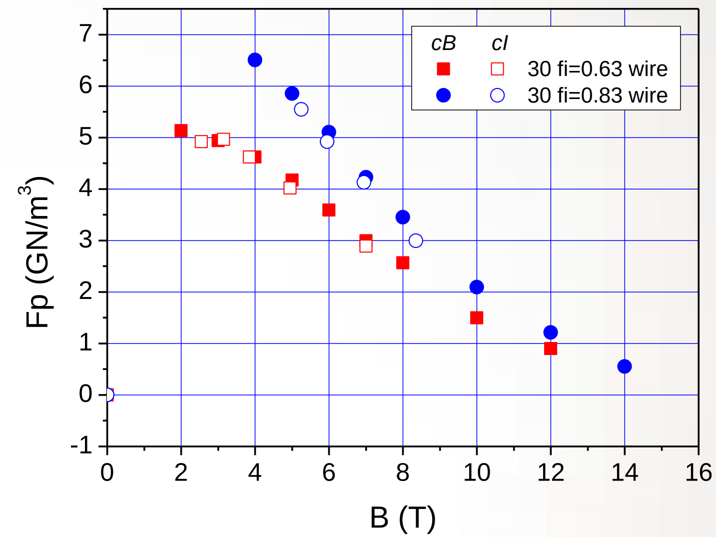


Hypertech - Unipress HIP-ed wires;
Nano SiC additives, amorphous boron, Nb barrier in Cu and Monel;
HU # 1019- 30 after HIP at various p,T, t , ramp conditions

P= 1.0GPa; T=660-705^{0}C; 15 min. - ramping annealing temperature

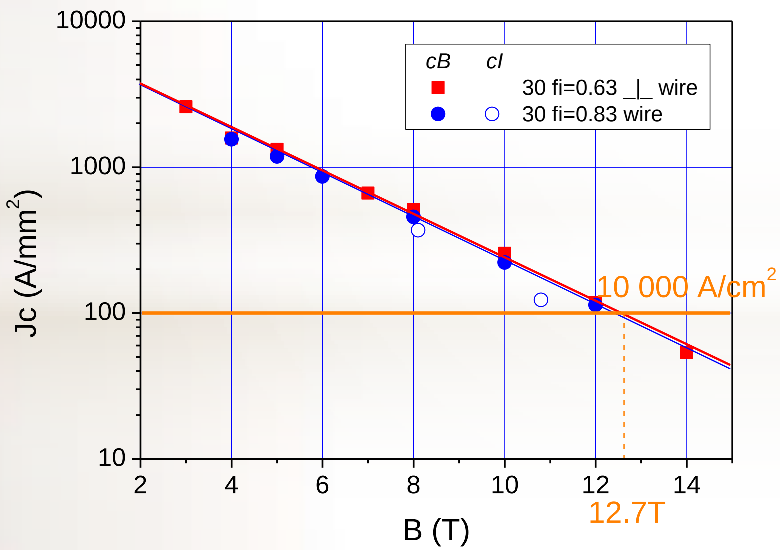


P= 1.0GPa; T=660-705^{0}C; 15 min- ramping annealing temperature

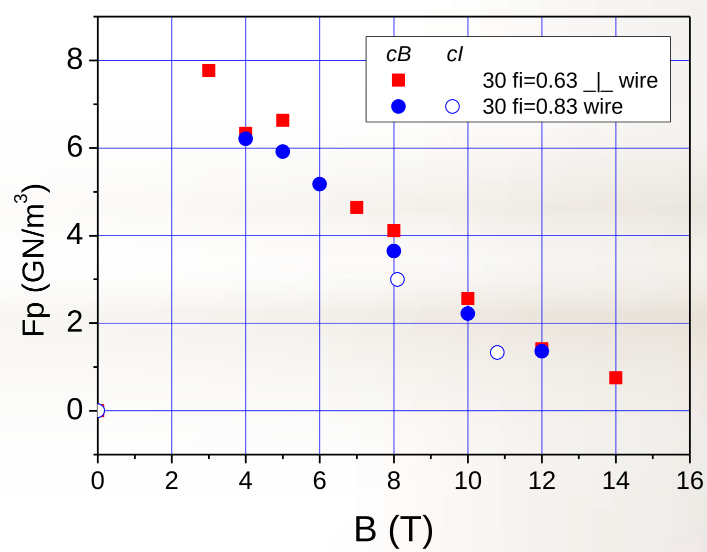


Ramping type of annealing can protect a little leaking of the Nb barrier!

P= 1.0GPa; T=700-745^{0}C; 15 min.- ramping annealing temperature



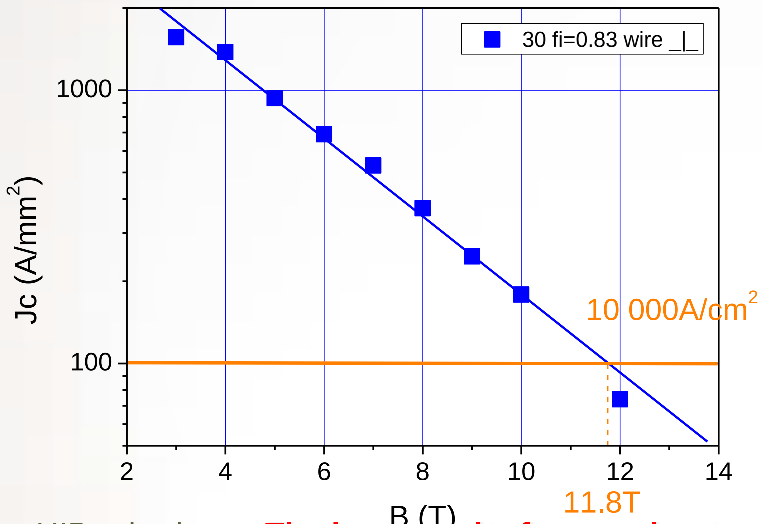
P= 1.0GPa; T=700-745^{0}C; 15 min.- ramping annealing temperature



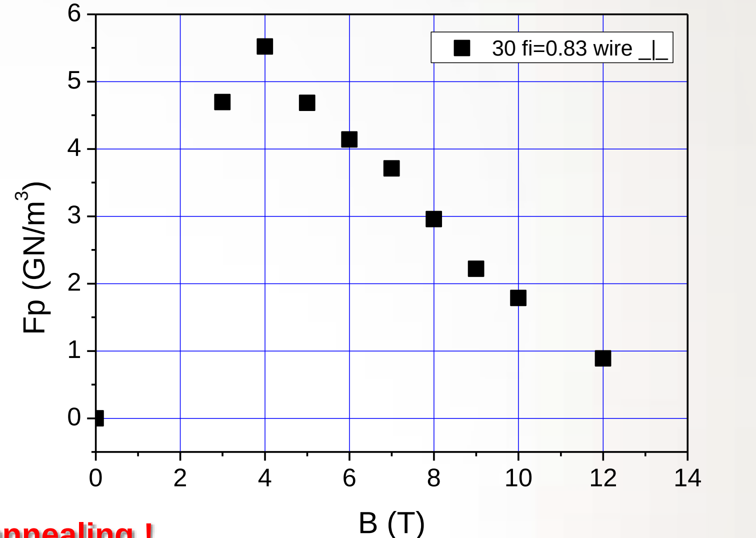
Hyper Tech Research, Inc.

Hypertech - Unipress HIP-ed wires;
Nano SiC additives, amorphous boron, Nb barrier in Cu and Monel;
HU # 1019- 30 after HIP at various p,T, t , ramp conditions

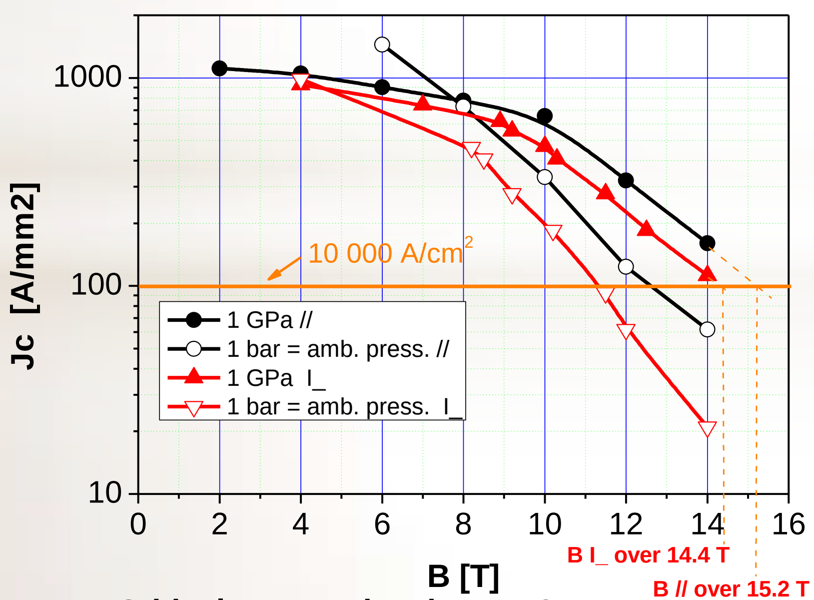
P= 1.0GPa; T=735-780^{0}C; 15 min. -ramping annealing temperature



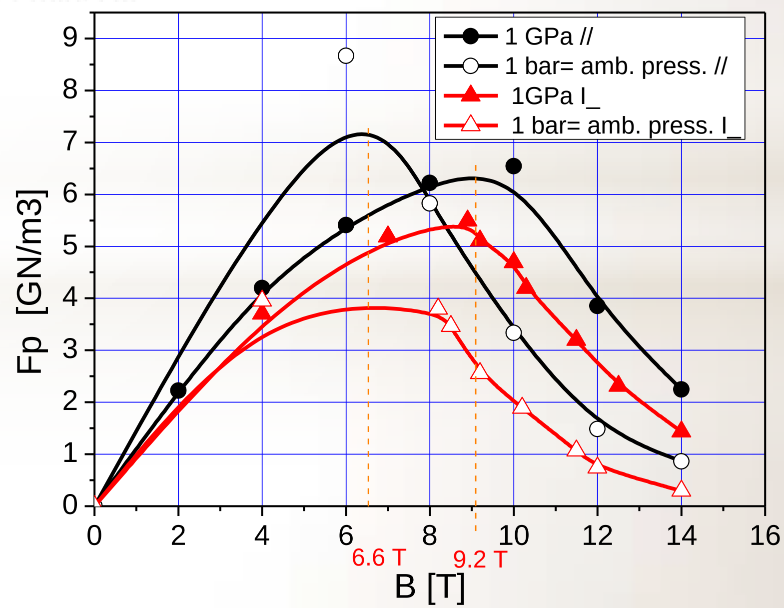
P= 1.0GPa; T=735-780^{0}C; 15 min. -ramping annealing temperature



Hypertech - Unipress HIP-ed wires - **The best results for ramping type annealing !**
Nano SiC additives, amorphous boron, Nb barrier in Cu and Monel;
HU # 1019- 30 after clasic 1 bar 700ºC & 1 GPa / 710-ramp to 770ºC/ in t= 34 min. HIP

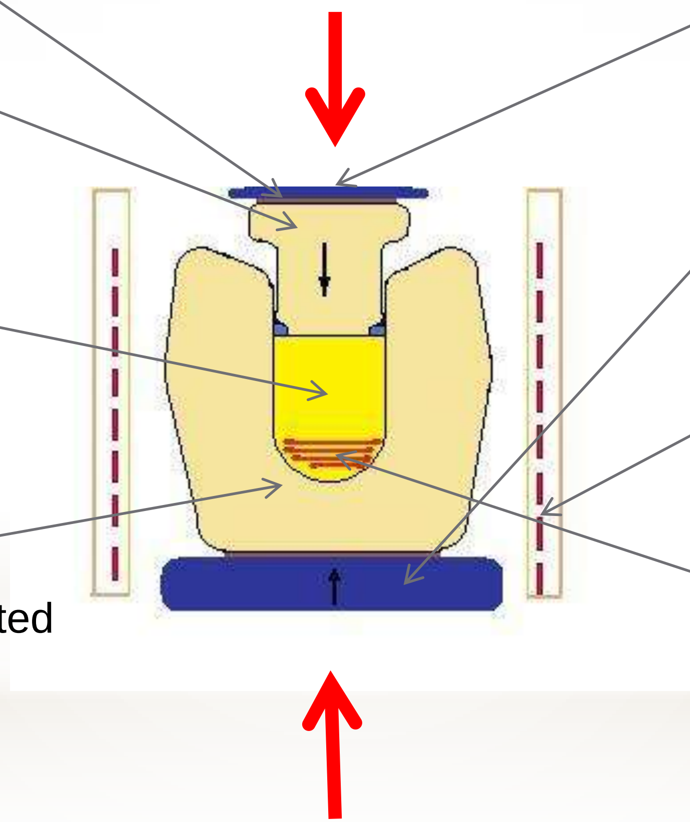


Critical current density at 4,2 K



Pining force vs. magnetic field

The scheme of the **liquid medium HIP apparatus** for examination of short wires samples at pressure up to 1.0 GPa and temperature up to 700 ^oC



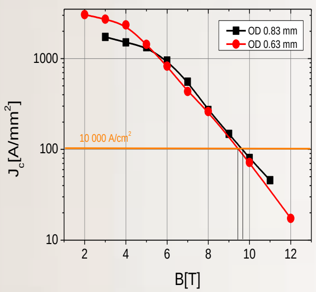
1019 # 43 wires

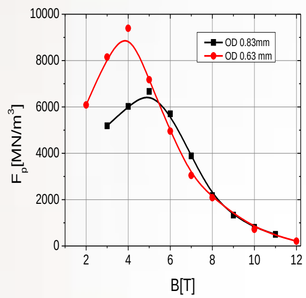
HT-UN #43	t=5 min (up 60, down 90)	T= 700 ⁰C	P= 0.3 GPa	OD 0.83 mm
HT-UN #43	t=5 min (up 60, down 90)	T= 700 ⁰C	P= 0.3 GPa	OD 0.63 mm

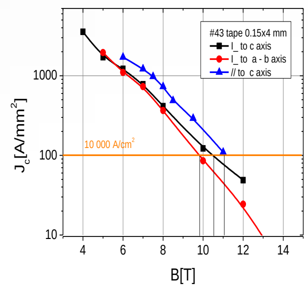
HIP in liquid; 1019 # 43 tape 0.15 X 4 mm

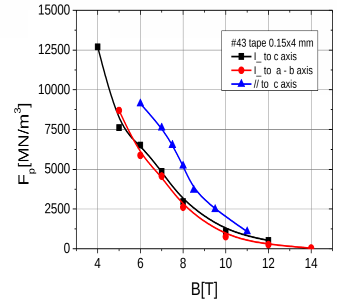
1019 # 30 wires

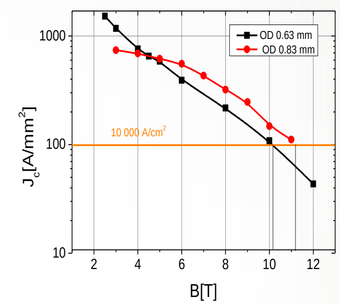
HT-UN #30	T= 700 ⁰C	P= 0.3 GPa	t=5min. (up 60, down 90)	OD 0.63 mm
HT-UN #30	T= 700 ⁰C	P= 0.3 GPa	t=5min. (up 60, down 90)	OD 0.83 mm

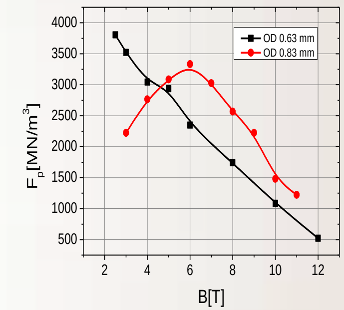


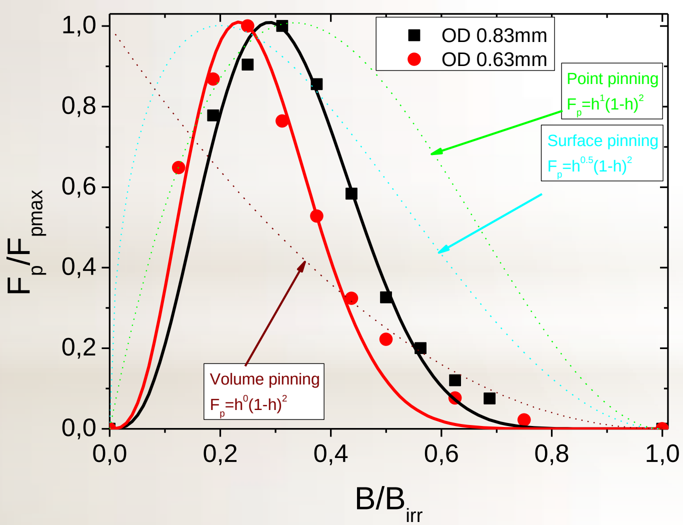


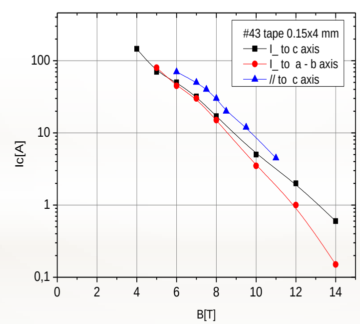


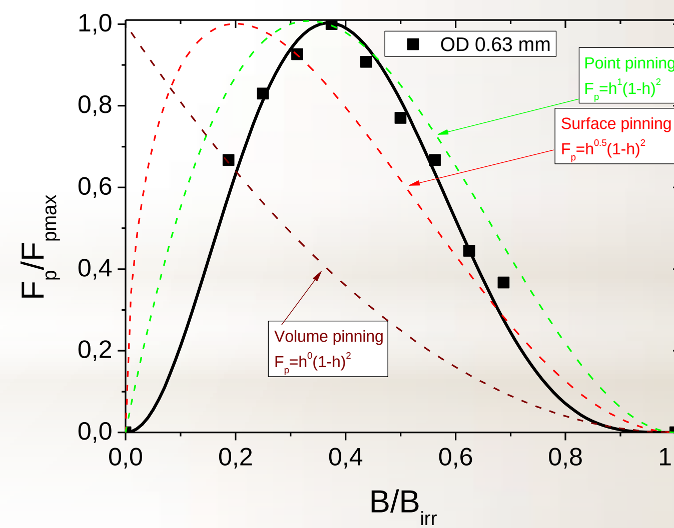






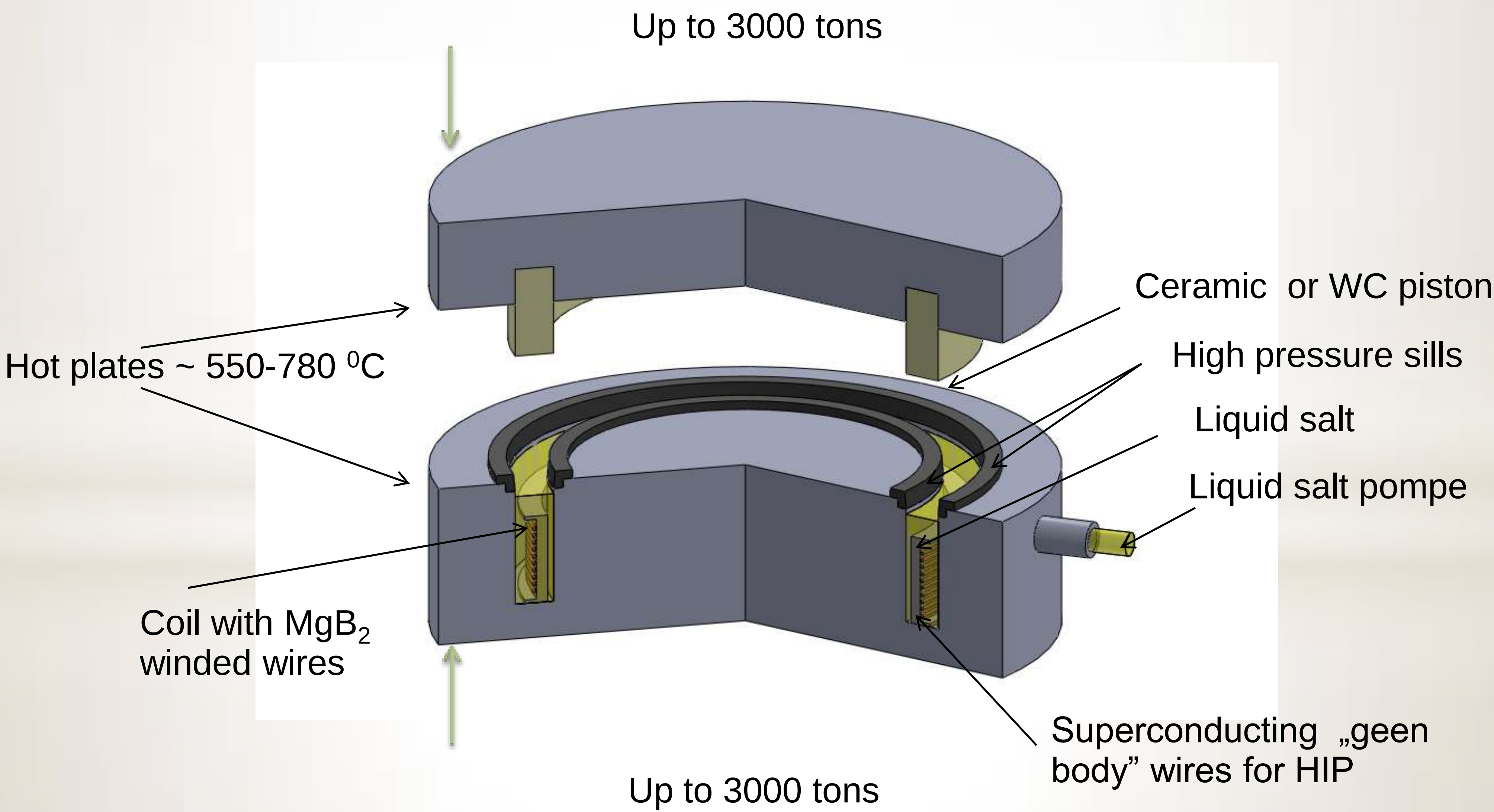






Hot Isostatic Presssed samples of wires and tapes in liquid the medium

The pictorial design of the first prototype of liquid salt / graphite , HIP machine designated for MgB_2 MRI, Ind. Heaters, ITER, etc., coils up to 1000 mm of diameters or/and for „wind and react" long wires productions by the HIP method in liquid medium at pressure up to 1.4GPa and temperatures up to 780 ^{0}C, as well as for rewinding wires from „react and wind". Actually accepted for the new technology program at the New Technology Investment Park in Celstynów, Poland



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Temporary used for testing the **press machine** up to 1000 tons at Unipress with new 350 mm toroidal chamber and high pressure generator designated for HIP experiments with operating annealing temperature up to 750 ^{0}C.

Summary& Conclusions

- The homo barrier, made of *ex situ* MgB_2 is effective against the Cu and Mg inter reaction process as well as for B reaction with metals Cu, Fe, Ni alloys. It selves can conduct very high current see PhD of A. Kario - Dresden,2011 (increase FF at last 3 times) Posses very low normal state resistivity. The long wire is easy for preparation by CP or CIP, extrusion or drawing.

- All HyperTech HIP-ed wires had significant increase of Jc nad Fp . The tightness /quality of Nb barrier is the most important factor of Jc increasing receiving by the HIP process

- Any kind of cold deformation (CP) is „not sufficient" for high state densification of the conductor contrary to the HIP process which permit to compensate the 25 % shrinking of *in situ* material, continuously during all HIP process and the stress on the core is independent of the any sheaths softening at elevated temperature of sintering.

- The melting temperature of the Mg or Mg + B substrates can be easily adjusted by the pressure – its permits to make the solid state reaction in relatively short time with absence of the liquid growth- it results in very dens nano grains of the final composite. It reflect directly by the high Jc increases at elevated magnetic fields especially.

- The tightness of the *ex situ* barrier is a function of:
  - the nano granulates of *ex situ* MgB_2 and nano additives added, purity of all components
  - thickness of the barrier,
  - final annealing temperature regime and outside stress pressure, applied during the wire/tape sintering preparation- e.g. HIP.

- *The Jc of all investigated kind of HIP-ed wires increases and gets the 10^4 A/cm^2 at 4.2 K already at around 14 T and even for undoped core materials reach 13 T. The Nb free barrier can carry the same current at such high fields, but the costs of wire productions is much smaller.*

Thank you for your attention