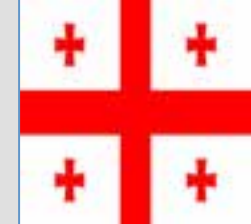




**Ilia Vekua Sukhumi Institute of Physics and Technology**  
7 Mindeli Str., 0186, Tbilisi, Georgia, E-mail: [sipt@sipt.org](mailto:sipt@sipt.org)



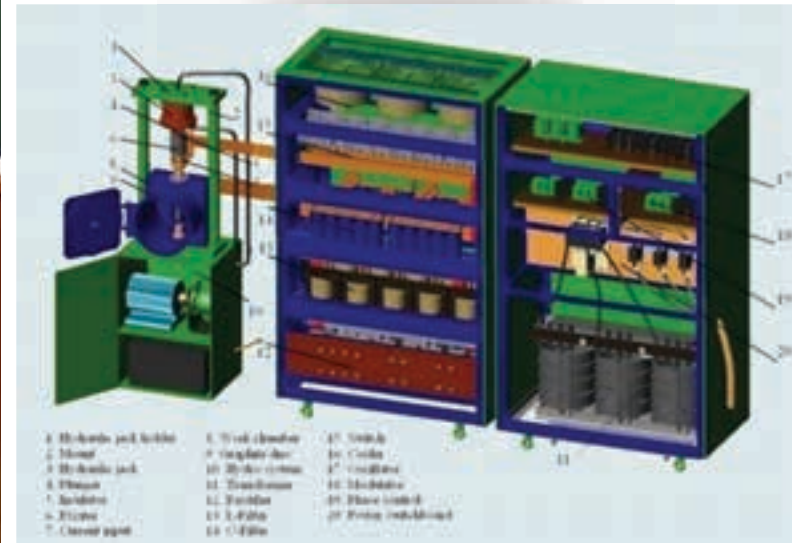
## Obtaining of Bulk Targets of Magnesium Diboride

**Presenter - Ekaterine Sanaia**



**8th Workshop on Mechanical and Electromagnetic Properties of Composite  
Superconductors**  
**MEM 2016**

# Spark Plasma Sintering Device

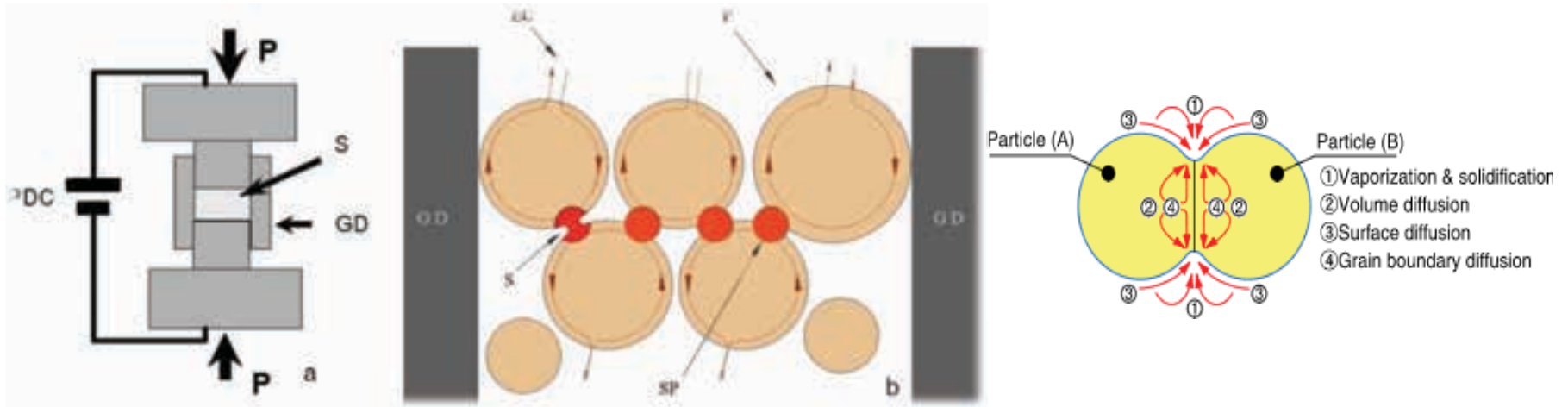


Sintering temperature (max 2500°C)

Current (max 5000 A)

Applied pressure (max 100 MPa)

# Spark plasma between powder particle

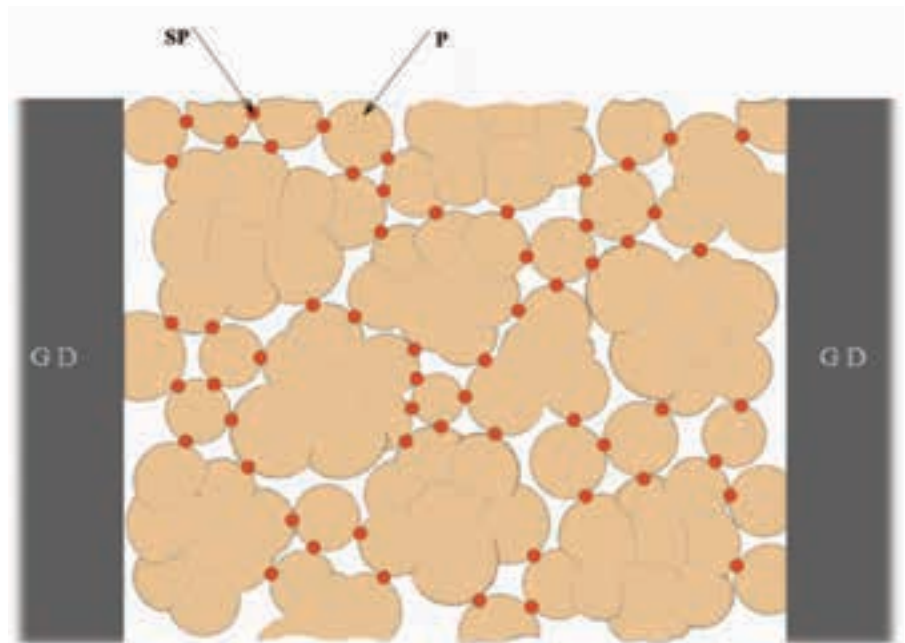


a)

b)

**Scheme of SPS device (a) and of the process of sintering(b);**

PDC- pulsed DC, GD- graphite die, S-Powder sample, P-pressure loading, EC- electric current, s-spark, sp-spark plasma and p-poder particles



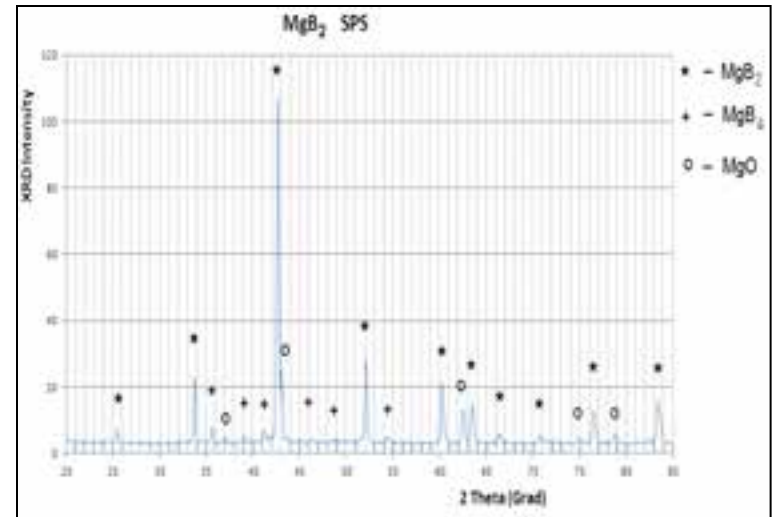
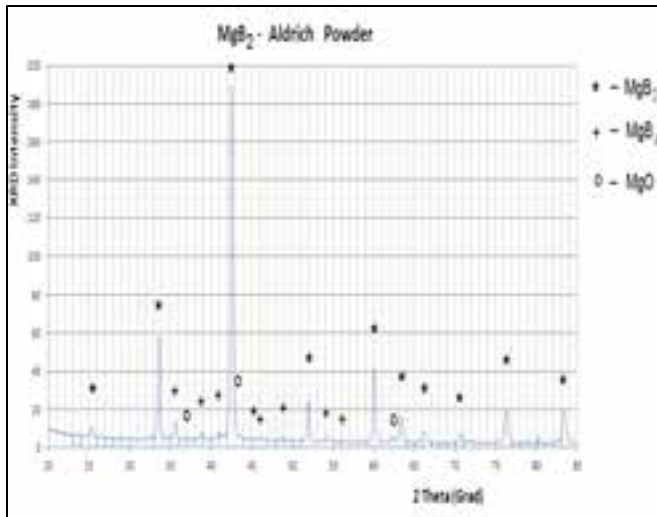
## EXPERIMENTS

Simultaneous synthesis and consolidation of magnesium diboride were been performed by **spark-plasma synthesis** and hot pressing method in an **induction furnace**.

Homogenization and activation of the mixture of magnesium and boron powders (molar ratio Mg:B=1:2) were conducted in a planetary-type nanomill by WC balls (*Pulverisette 7 Premium Line*, Fritsch) during 30 min. A mixture of Mg and B powders, or tablets made from this mixture, preliminarily pressed in the die under 200 MPa, were put in the die. During sintering In spark-plasma devices to produce samples in graphite dies lined with **graflex plates** from sides, top and bottom of the punches. For the purpose of easy removal of consolidated  $MgB_2$  from the die, a graflex plate (0.5 mm thick) was inserted in the graphite die coated with a BN layer. The BN layer is formed using boron nitride aerosol lubricoat (Manufacturer ZYP coatings Inc.). During spark-plasma synthesis the graphite die is heated in vacuum in the following conditions: current 2000-4000 A, voltage 4-6 V, sintering duration 5-15 min.

The spark-plasma method allows successful synthesis and sintering of magnesium boride for 10 mm samples.

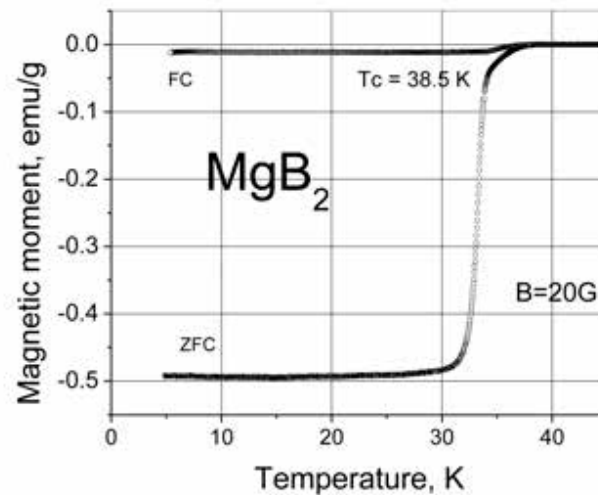




a)

b)

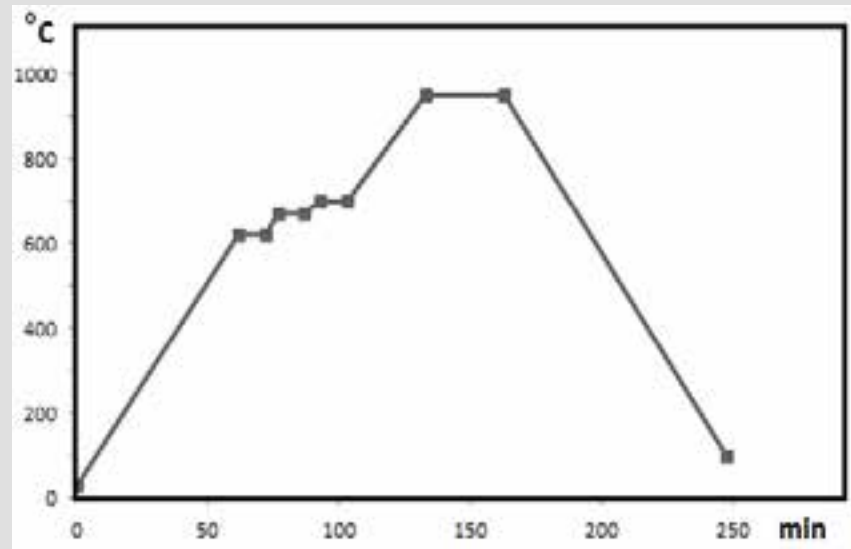
XRD of commercial magnesium diboride powder (Aldrich, >99%) (a) and  $MgB_2$  powder by spark-plasma synthesis method (b).



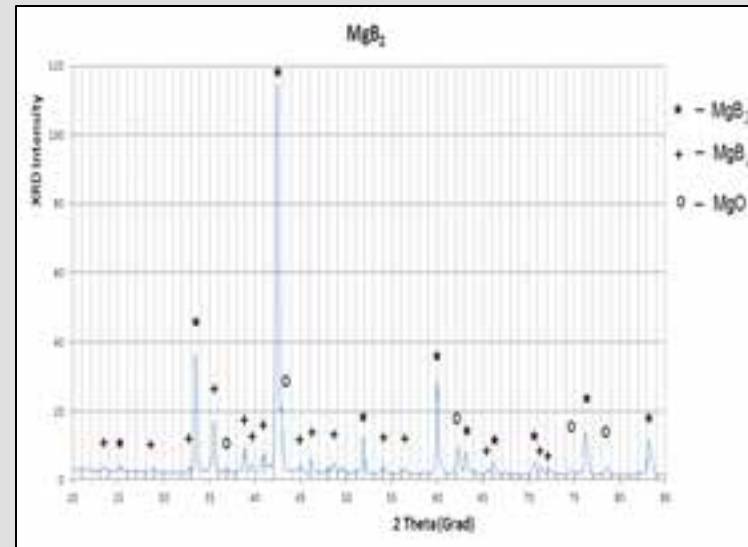
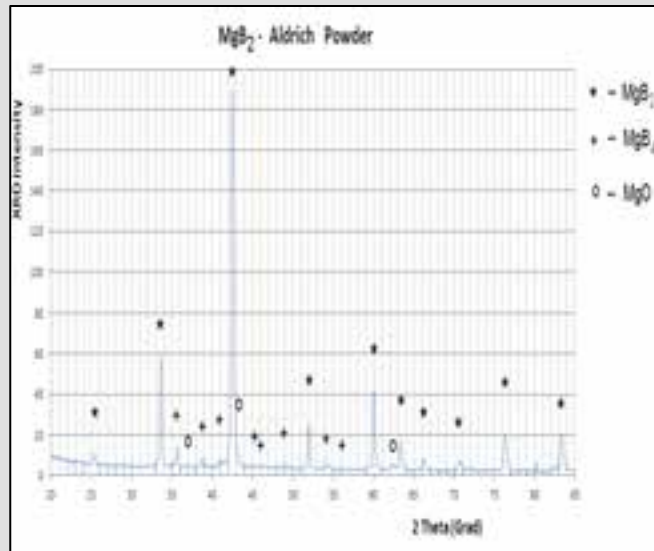
**Obtaining of samples:** Magnesium diboride bulk cylinders ( $d=52$  mm) were obtained in the induction furnace. Therefore both synthesis and sintering were performed in the induction furnace equipped with a vacuum system and press. Sharp increase of pressure during this interval is not acceptable, because melt magnesium comes out of the die and Mg:B ratio is changed, pressing is increased according to temperature ratio increase till 600-1000°C during 80-100 min and pressure 25 MPa. Bulk samples were obtained in optimal condition at 600-1000°C in induction furnace.



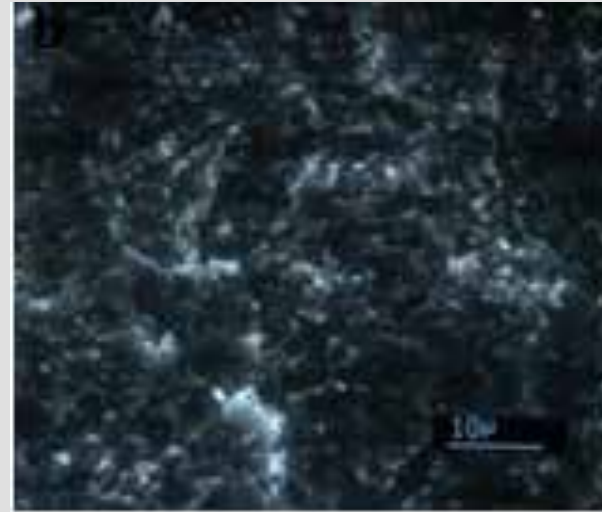
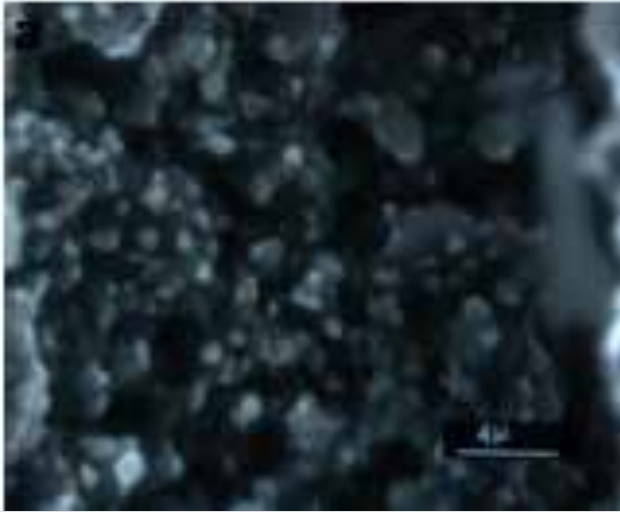
MgB<sub>2</sub> sample synthesized and consolidated in the induction furnace within 600-1000°C



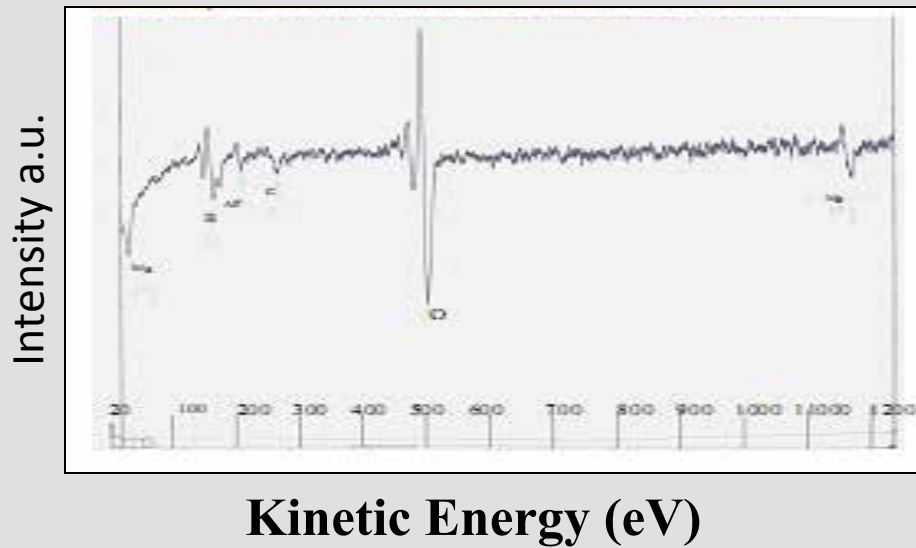
Temperature regimes for synthesis and consolidation of MgB<sub>2</sub> non-cracked samples by induction furnace



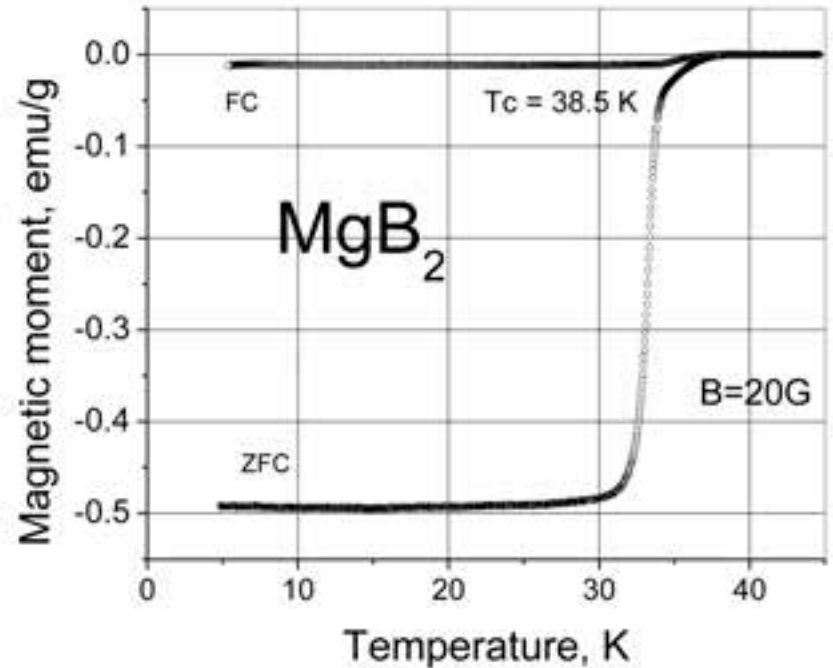
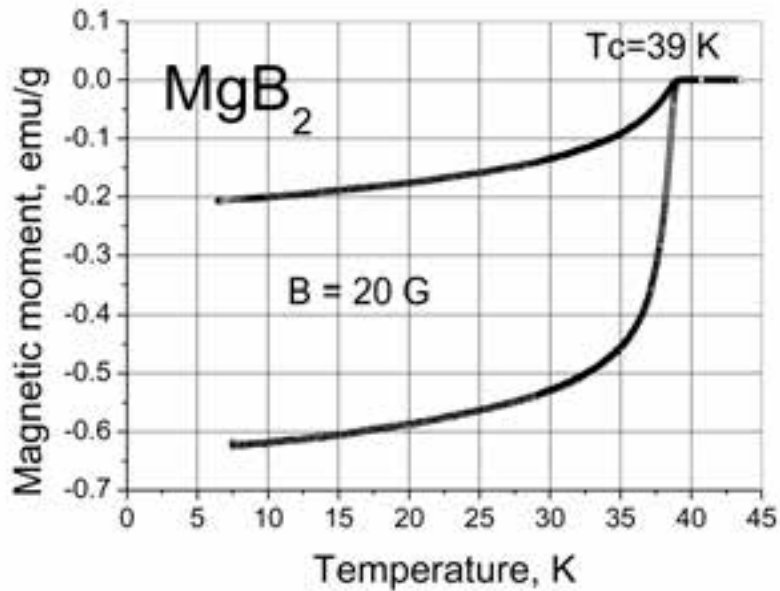
XRD of commercial magnesium diboride powder (Aldrich, >99%) (a); obtained consolidated sample (b) and MgB<sub>2</sub> powder by induction furnace has small amount of MgO and MgB<sub>4</sub> phases (b).



SEM micrograph of bulk MgB<sub>2</sub> samples.







Temperature dependence of magnetization under an applied field of 20G for commercial  $\text{MgB}_2$  powders (a) and for sample obtained by simultaneous synthesis and consolidation of Mg+B mixture in a spark-plasma device (b).

## Conclusions

- ✓ Magnum diboride bulk superconductor samples (targets) with a diameter 10-52 mm has been obtained in an **induction furnace and spark-plasma synthesis methods**.
- ✓ It is established, that production of large-diameter samples from the magnesium and boron powders by the hot pressing method is accompanied by several peculiarities: samples' fragility, pyrophoricity, porosity, etc.
- ✓ Bulk superconductor non-cracked samples with a diameter 52 mm are obtained in an **induction furnace** by using the hot pressing. Heating of Mg+B mixture in 600-1000°C is optimal condition, when pressure was gradually increased up to 25 MPa. Sharp increase of pressure during this interval is not acceptable, because melt magnesium comes out of the die and Mg:B ratio is changed.
- ✓ The onset of superconductivity is found to be ~38.5-39K with sharp transition

*Thank you  
for your attention*

*Looking forward for collaboration*