Growth Of MgB_2 Films On Cu For SRF Cavities

Wenura Withanage

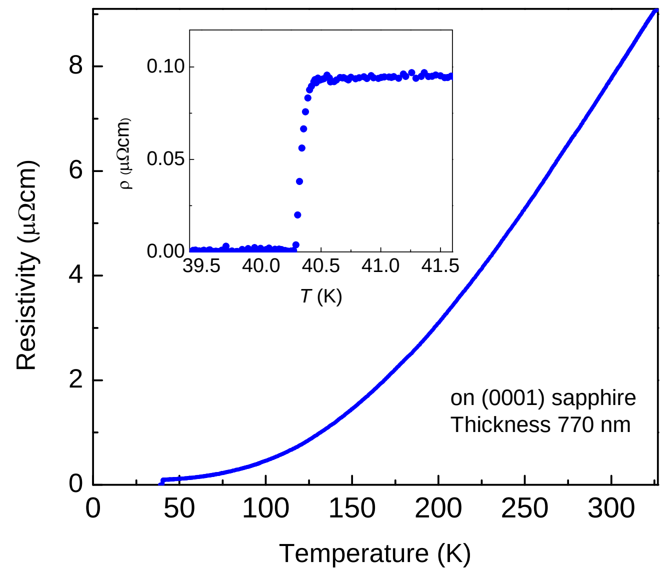
Advisor – Prof. Xiaoxing Xi

Outline

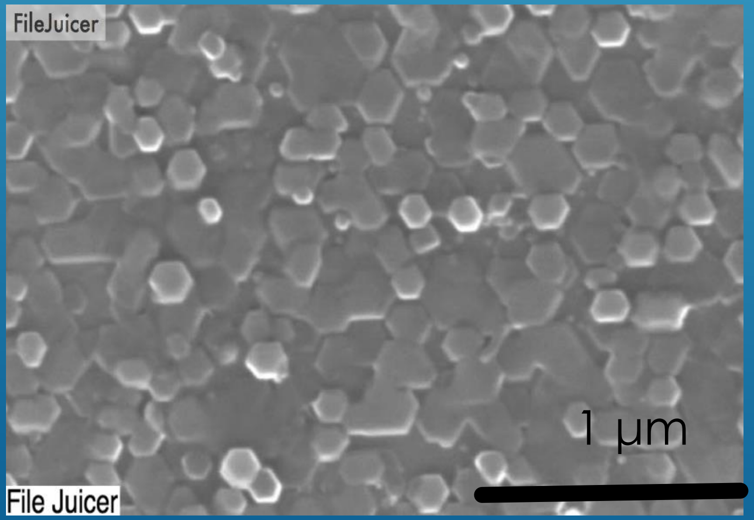
- Superconducting MgB_2
- Motivation & Goal
- Hybrid physical chemical vapor deposition (HPCVD) of MgB_2 at Temple University
- MgB_2 growth on metal substrates
- MgB_2 growth on 2” sapphire wafers
- MgB_2 deposition on 2” Cu discs and characterizations
- Coating of MgB_2 on inner walls of Cu tubes with the diameter similar to the diameter of beam tube of a 3 GHz RF cavity
- Summary and Next steps
- Publications and acknowledgement

Superconductivity in MgB_2

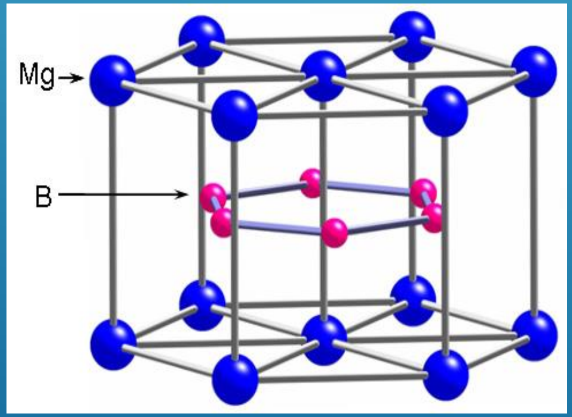
- Discovered in 2001
- High transition temperature(Tc) ~39 K
- Absence of weak links at grain boundaries
- High thermodynamic critical field
- Has a hexagonal structure



Xi *et al*, Physica C 456, 22 (2007)



Zeng et.al., Nature Materials, Vol.1, 2002



Hexagonal structure

Motivation & Goals

Motivations

- MgB_2 is one of the most promising superconducting material for SRF applications
- MgB_2 coated Cu cavities will have high thermal conductivity
- Less expensive alternate for commonly used Nb bulk cavities

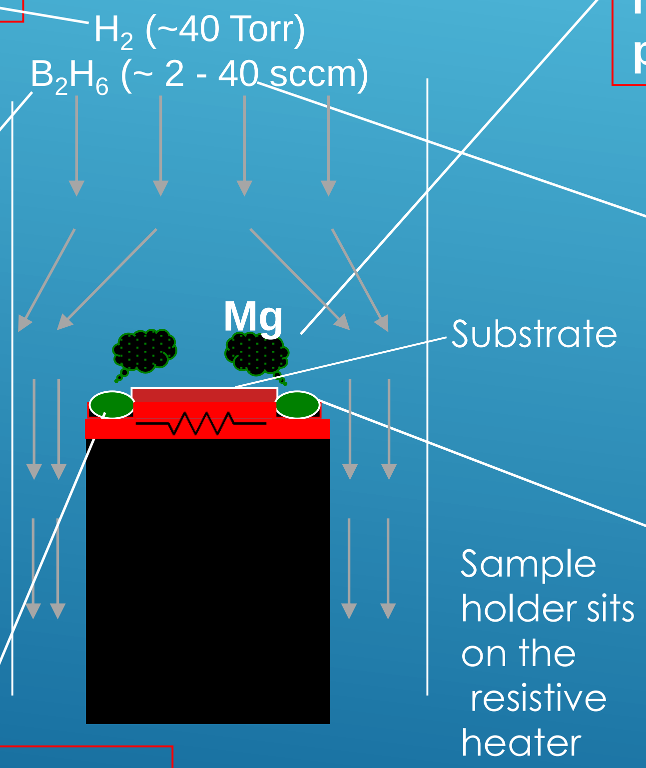
Goals

- Developing a recipe to grow MgB_2 on large area Cu discs ☑
- RF characterization of the MgB_2 coated Cu discs ☑
- Coating MgB_2 on the inner walls of a 3 GHz Cu cavity
  - ❑ Coating of Cu tubes with diameter close to ☑ beam pipe of a 3 GHz cavity
  - ❑ Characterization of the coated tubes ☑
  - ❑ Evaluation and modification to the coating setup
  - ❑ Coating of 3 GHz Cu cavity with MgB_2

HPCVD of MgB_2 at Temple University

get rid of oxygen
prevent oxidation

pure source of B



high enough *T*
for epitaxy ~ 730 C

make high Mg pressure possible

B supply (B_2H_6 flow rate) controls growth rate

Pure source of Mg

Pressure – Temperature phase diagram for Mg – B system

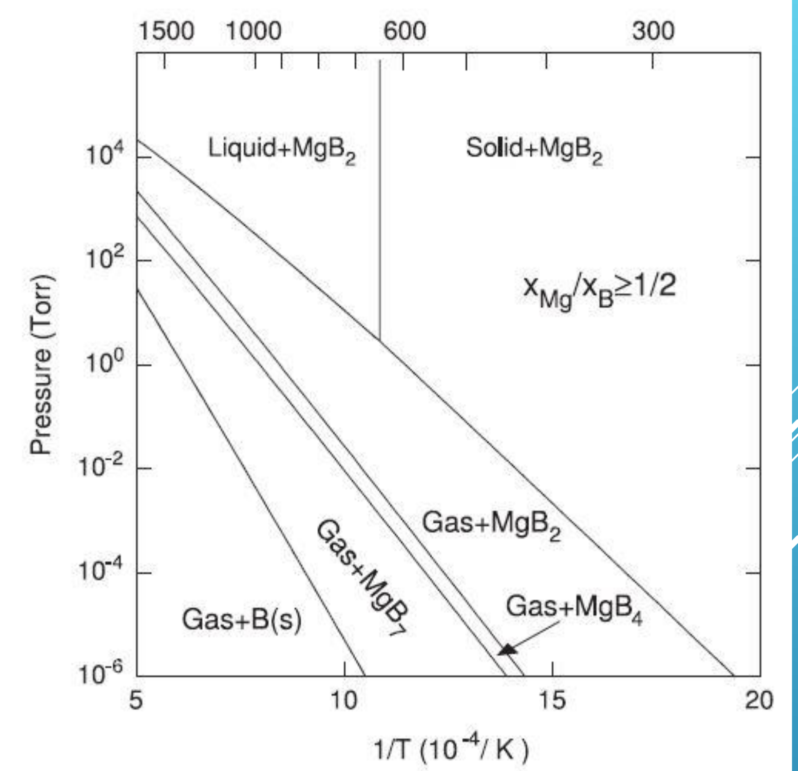


Figure 1. The pressure–temperature phase diagram for the Mg:B atomic ratio $x_{Mg}/x_B \geqslant 1/2$. The region marked by 'Gas + MgB_2' represents the growth window for MgB_2 films (from [2]).

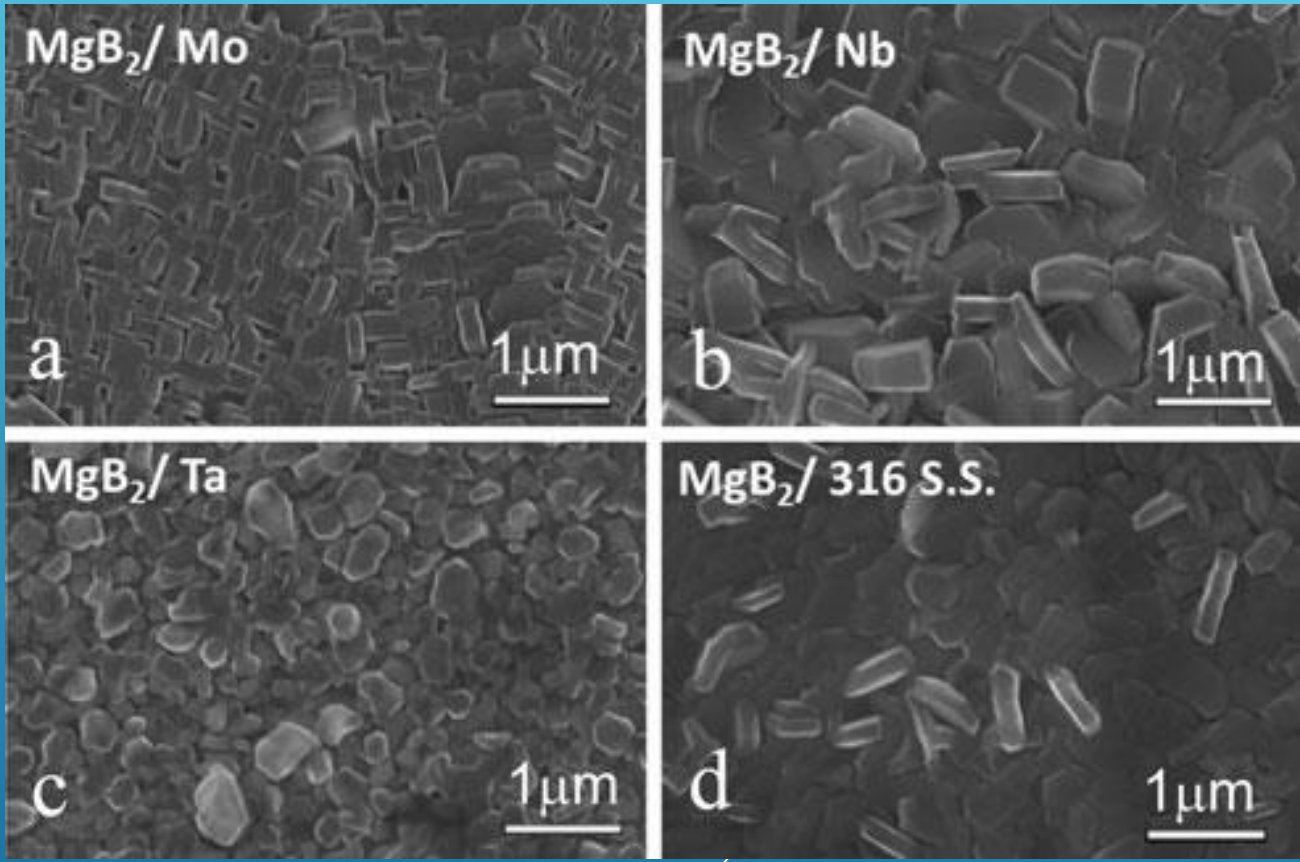
Liu Z K, Schlom D G, Li Q and Xi X X 2001 Thermodynamics of the Mg–B system: implications for the deposition of MgB2 thin films *Appl. Phys. Lett.*

MgB_2 growth on metal substrates using HPCVD

- MgB_2 was grown on metal substrates
- Deposition temperature ~ 700 C
- 300 nm MgB_2 were grown
- Tc ~ 39 K

Mg readily reacts with Cu at high temperature
$MgCu_2$ and Mg_2Cu

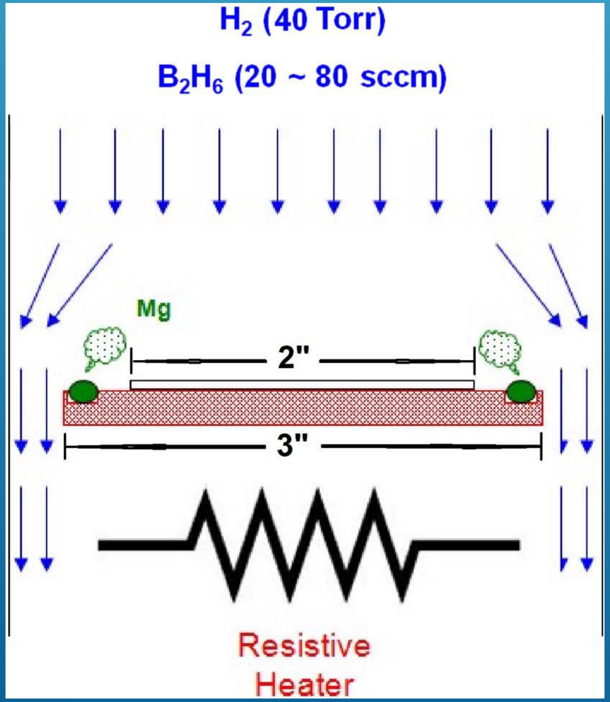
Materials	Reaction with Mg	Melting point
Niobium [8]	Stable	2477
Molybdenum [8]	Stable	2623
Tantalum [8]	Stable	3017
316 stainless steel [10]	Stable	1375–1400

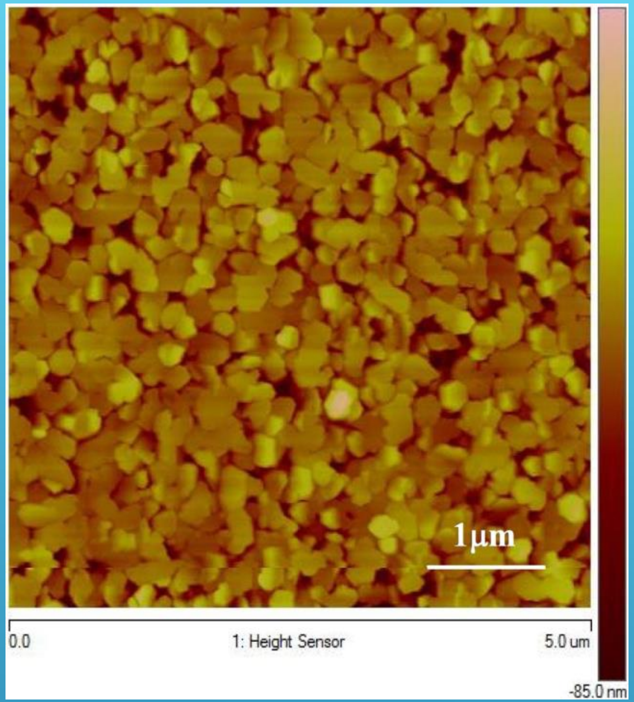


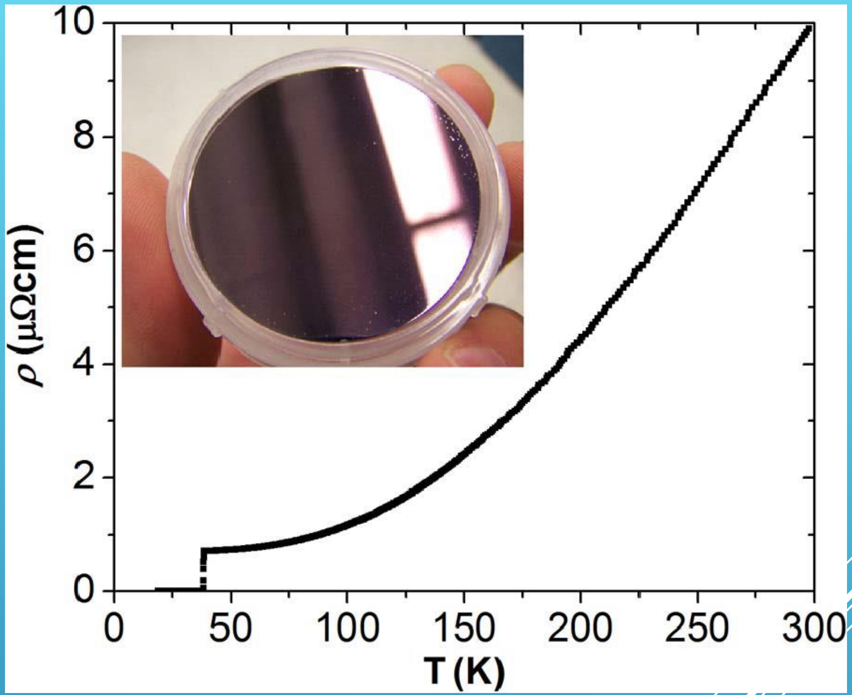
Chenggang Zhuang · Teng Tan · Alex Krick · Qingyu Lei ·Ke Chen · X.X. Xi
J Supercond Nov Magn (2013) 26:1563–1568

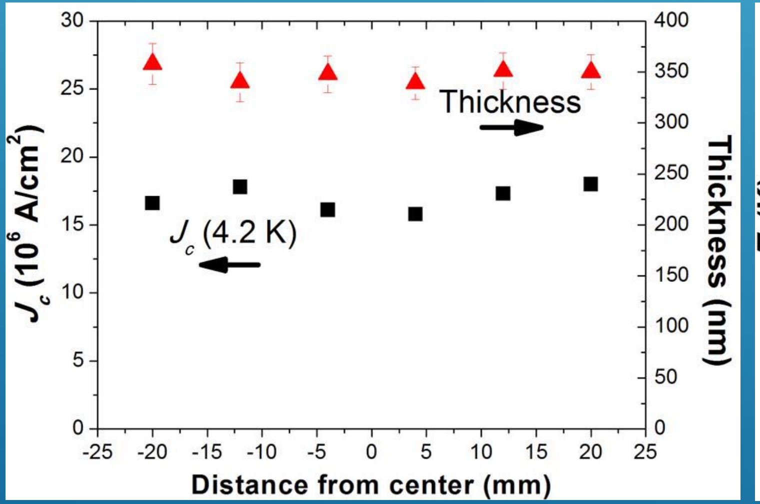
MgB_2 film growth on 2" ceramic substrates

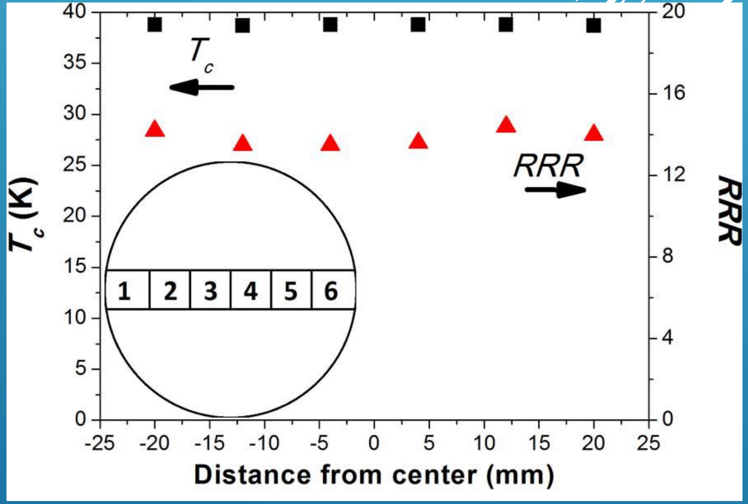
- HPCVD setup was scaled up to accommodate 2" substrates
- 2" sapphire discs were coated successfully
- Tc ~ 39 K





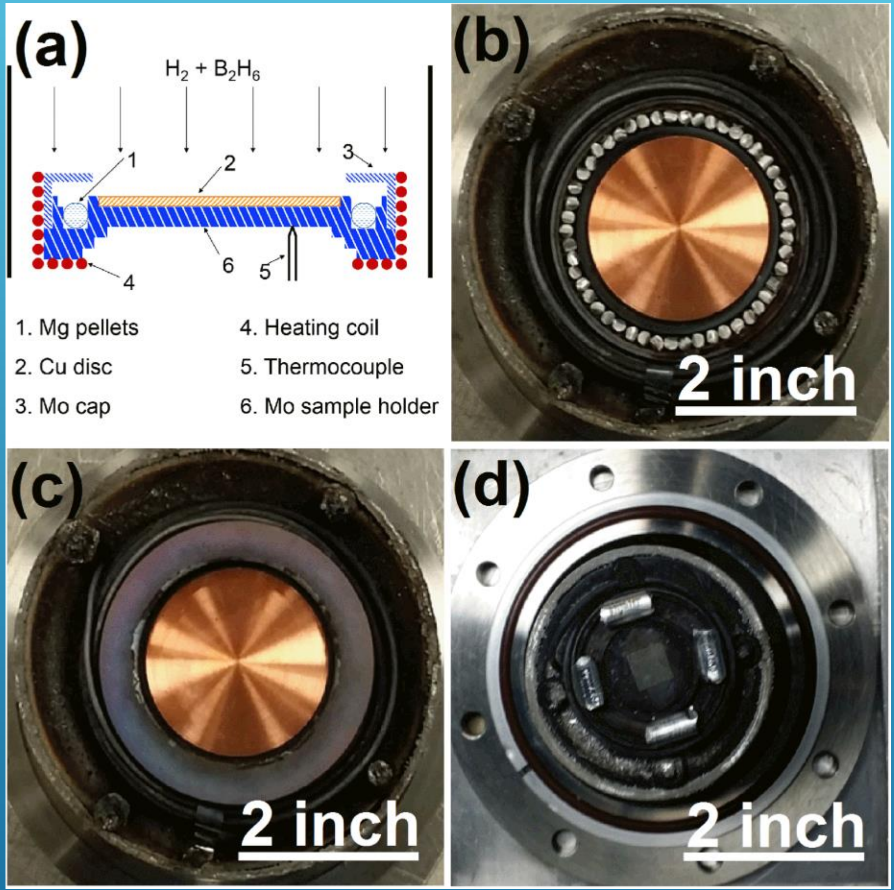






Teng Tan, Chenggang Zhuang, Alex Krick, Ke Chen, and X. X. Xi
IEEE TRANSACTIONS ON APPLIED SUPERCONDUCTIVITY, VOL. 23, NO. 3, JUNE 2013

MgB_2 deposition on 2" C u discs



(a) Schematic diagram of the modified HPCVD setup to accommodate 2 inch diameter Cu discs. (b) Photograph of the sample holder containing 2 inch Cu disc and Mg pellets placed on the heating element. (c) Photograph of the sample holder containing the Mg pellets and Cu disc with the Mo cap. (d) Photograph of a regular HPCVD setup for small substrates.

Challenges in growing MgB_2 on Cu

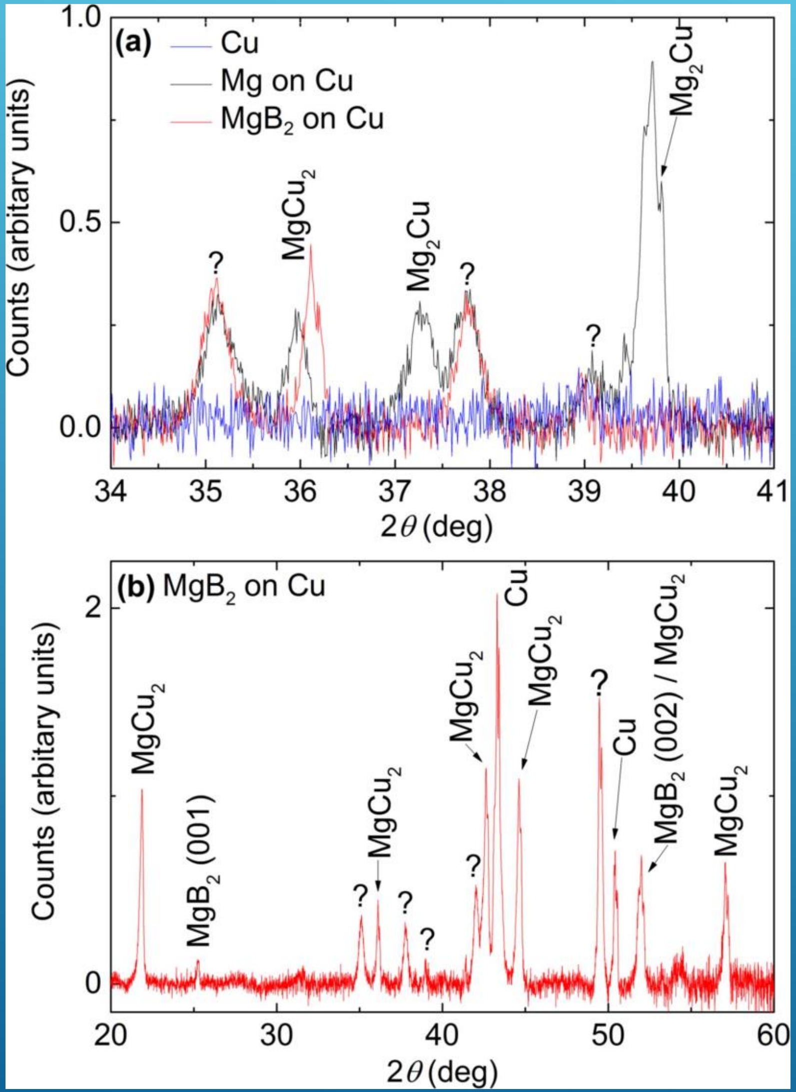
- Mg vapor reacts and forms alloy with Cu starting around 450 C at 10 Torr (Copper surface color changes to silver)
- Mg vapor pressure is low at low temperatures
- Coating whole 2" area uniformly challenging

Scaling up for 2" Cu discs and reducing the growth temperature

- HPCVD system is modified to accommodate thick 2" Cu disc
- Deposition conditions were optimized for Cu substrates

Withanage *et al.*, *Supercond. Sci. Technol.* 30, 0450016 (2017)

Role of Mg-Cu alloy on the growth of MgB_2 on Cu



(a) XRD θ–2θ scans for Cu, Mg on Cu and MgB_2 film on Cu for 2θ from 34° to 41°. (b) XRD θ–2θ scan for a MgB_2 film on Cu for 2θ from 20° to 60°.

Types of Mg-Cu alloy

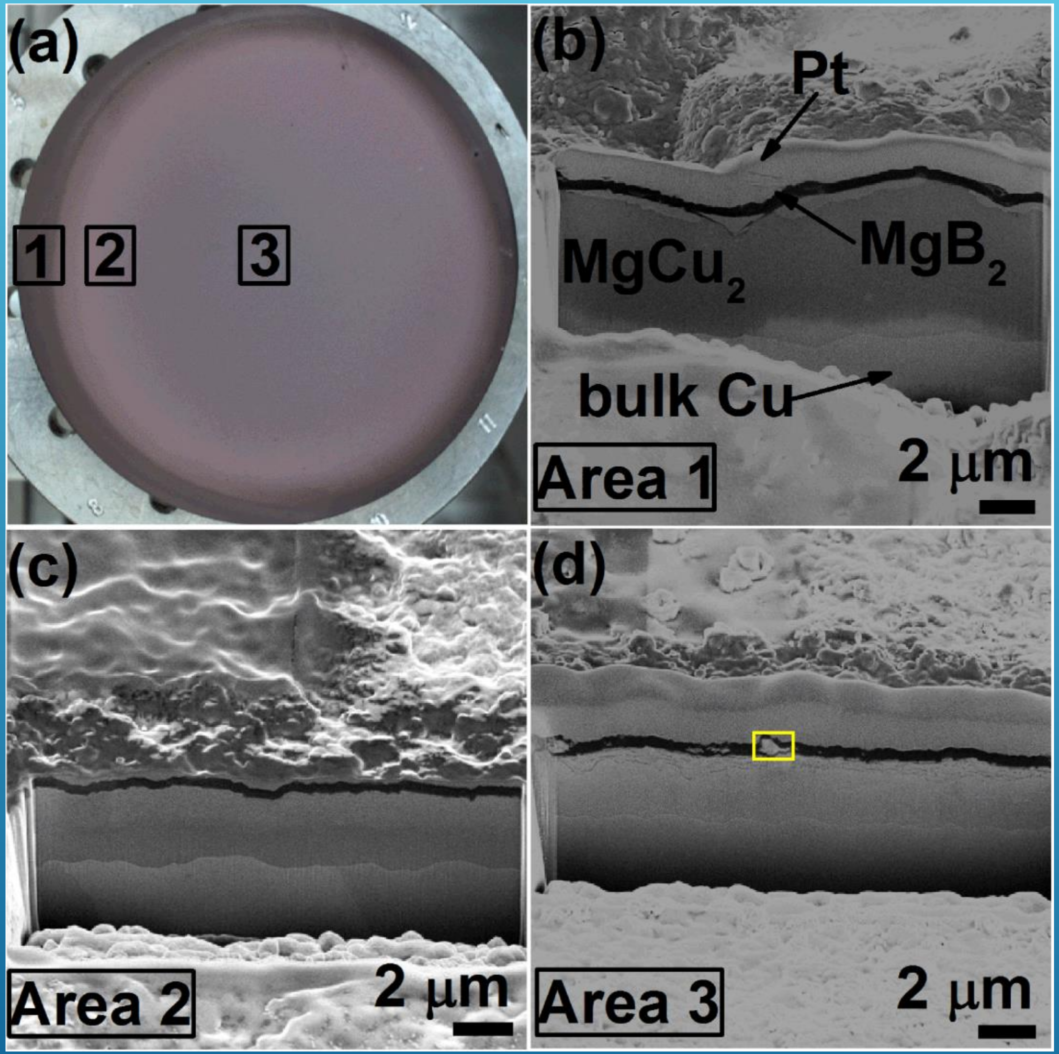
- Mg_2Cu – Promotes MgB_2 growth at low temperature
- $MgCu_2$

Helpful nature of Mg2Cu at low temperature

- Mg_2Cu has been used as the Mg source for MgB_2 growth in powder in tube method in early studies of MgB_2-Cu mixed wires
- Same behavior observed in MgB_2-Cu tape research

Withanage *et al.*, *Supercond. Sci. Technol.* 30, 0450016 (2017)

Cross-sectional studies of MgB_2 coated 2" Cu discs



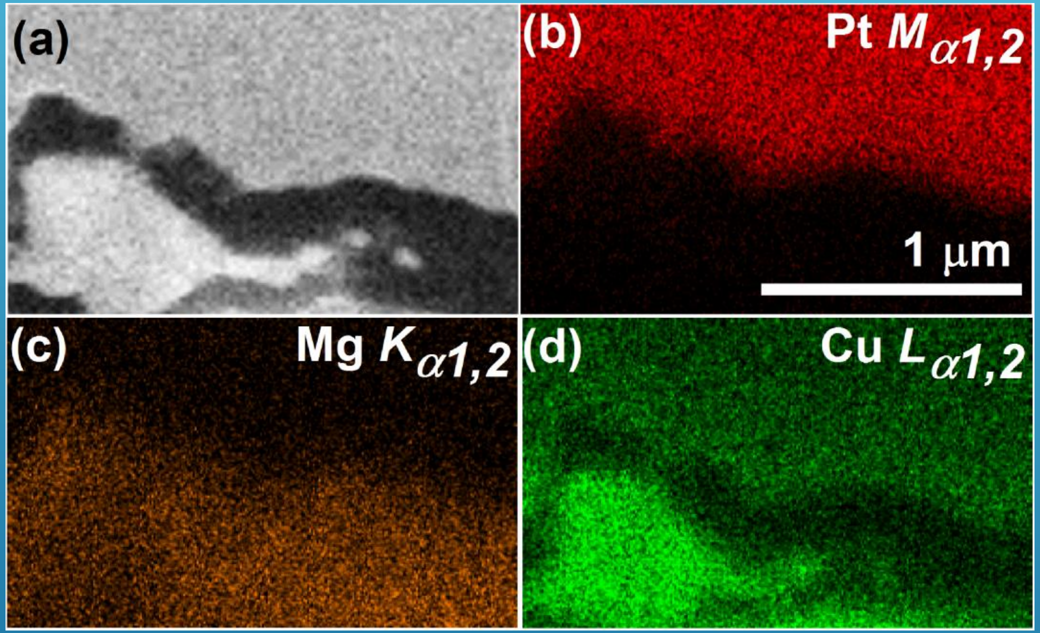
(a) Optical image of a MgB_2 film on a Cu disc. (b), (c) and (d) SEM images of the FIB fabricated cross sections from three different areas. The area inside the yellow color box in (d) was used for the EDS elemental mapping shown in figure 4.

MgB_2 coating on Cu

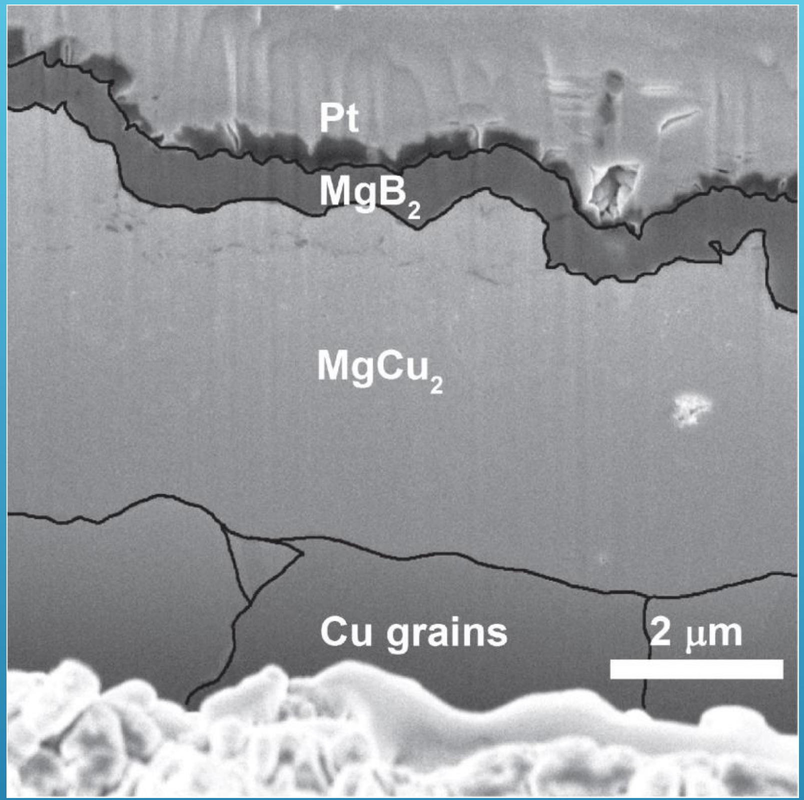
- Fairly uniform coating
- Conformal coating
- Thick (~ 3 – 6 μm) alloy layer underneath the MgB_2 layer
- Mg-Cu alloy incursion into the MgB_2 layer observed in area 3

Withanage *et al.*, *Supercond. Sci. Technol.* 30, 0450016 (2017)

Cross-sectional and elemental studies of MgB_2 coated 2" Cu discs



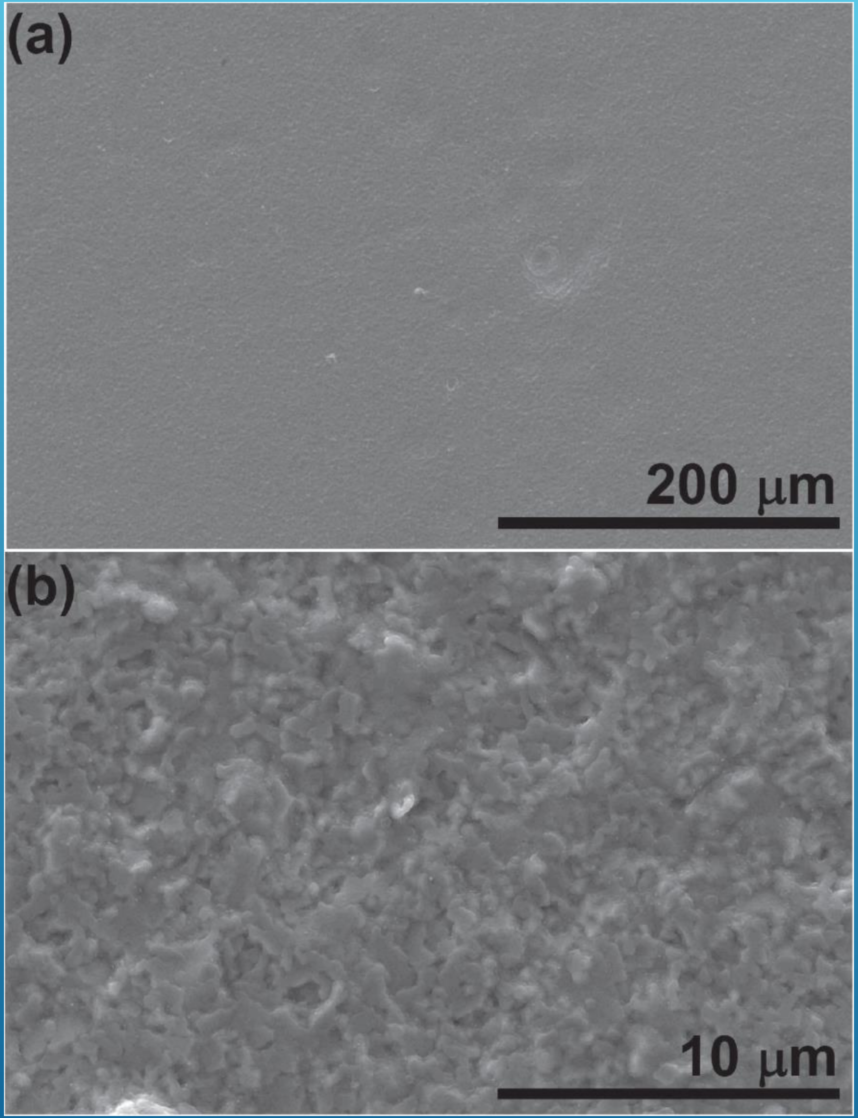
(a) SEM image of the EDS elemental mapping area; (b) EDS signal from the Pt Mα1,2 line; (c) EDS signal from the Mg Kα1,2 line; and (d) EDS signal from the Cu Lα1,2 line.



SEM image of the FIB fabricated cross section of a MgB_2 film grown on a Cu disc with Pt layer, MgB_2 layer, $MgCu_2$ alloy layer and bulk Cu. The black lines were drawn along the interfaces for illustration purposes.

Withanage *et al.*, *Supercond. Sci. Technol.* 30, 0450016 (2017)

Surface morphology of MgB2 films grown on Cu discs

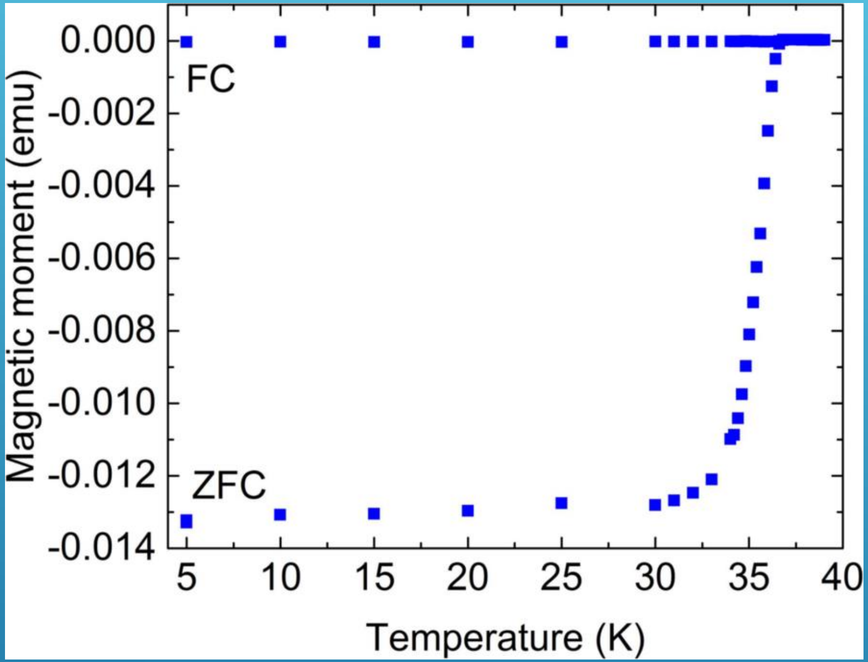


- Dense coating
- No pinholes

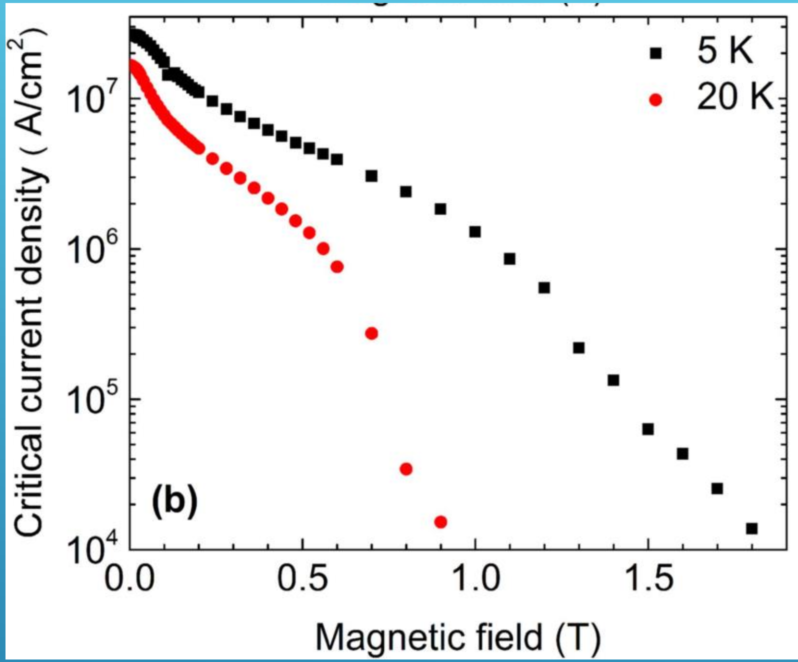
Withanage *et al.*, *Supercond. Sci. Technol.* 30, 0450016 (2017)

(a) SEM image of the MgB_2 film surface on a Cu disc. (b) Zoomed in SEM image.

DC superconducting properties



Magnetic moment versus temperature curve of a 650 nm thick MgB_2 film on a Cu disc.

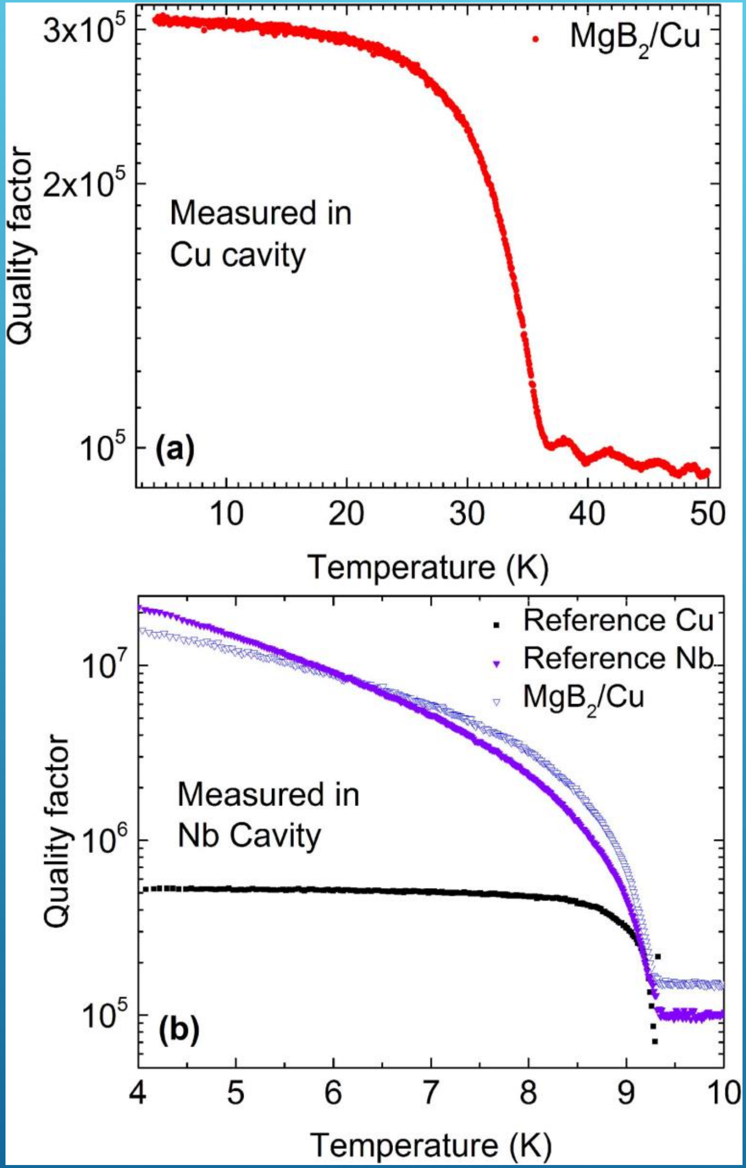


Critical current density versus applied magnetic field curves of a 650 nm thick MgB_2 film on a Cu disc at 5 and 20 K. The magnetic field was applied perpendicular to the film surface.

- High T_c ~ 37 K
- High J_c ~ 10^7 A cm^{-2}

Withanage *et al.*, *Supercond. Sci. Technol.* 30, 0450016 (2017)

RF properties

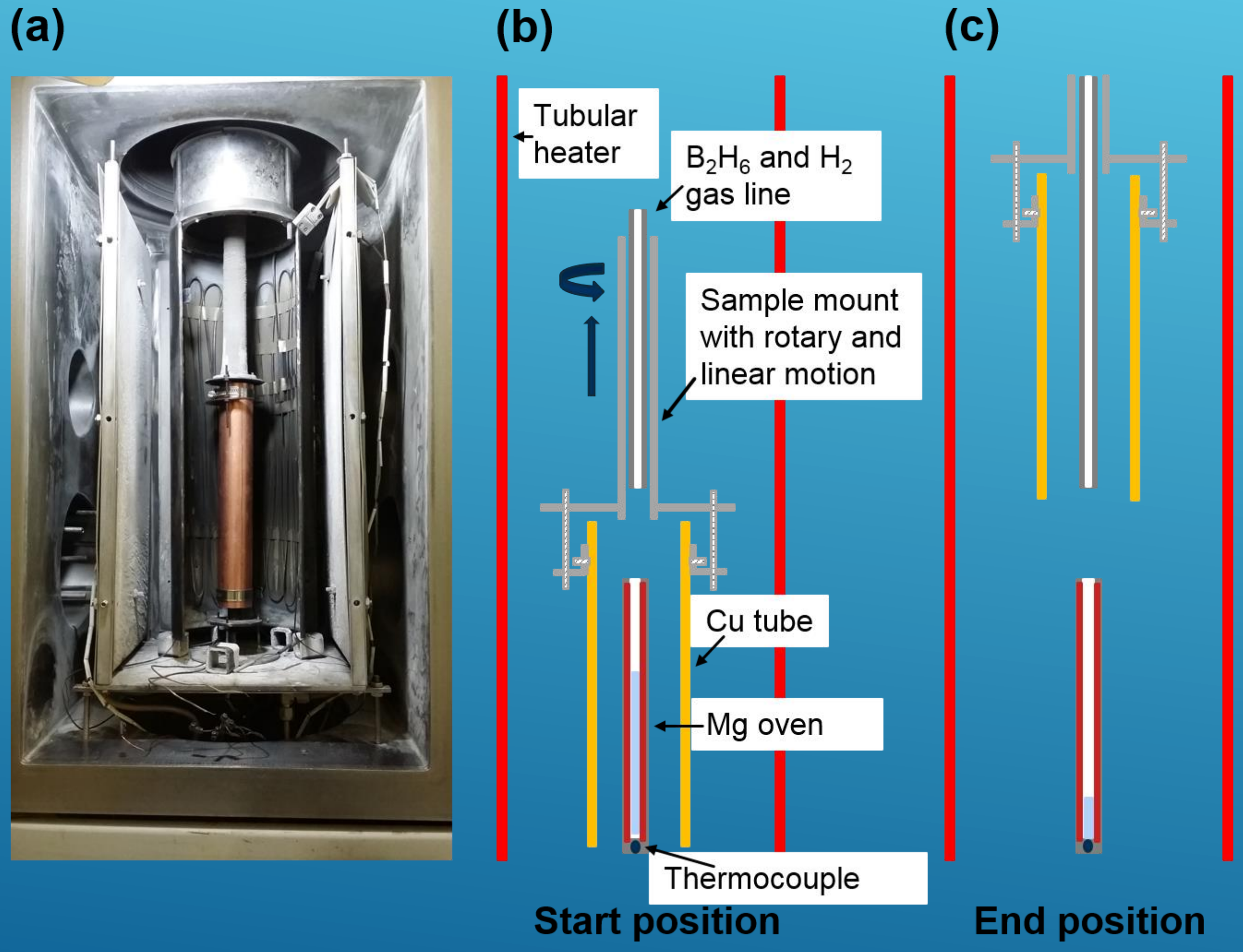


- RF characterizations were carried out using a cryogenic high-Q hemispheric cavity with a TE032-like mode at 11.4 GHz at SLAC National Accelerator Laboratory
- T_c of the MgB_2 films measured ~ 37 K
- Samples showed Q close to the reference Nb disc
- Q of MgB_2 and Nb cross-over at 6.2 K observed.

Withanage *et al.*, *Supercond. Sci. Technol.* 30, 0450016 (2017)

(a) Quality factor versus temperature curve of a MgB_2 coated Cu disc measured in a Cu cavity. (b) Quality factor versus temperature curves for a MgB_2 coated Cu disc, a Nb reference sample, and a Cu reference sample, measured in a Nb cavity.

MgB_2 coating on inner wall of Cu tubes with diameter similar to a 3 GHz cavity



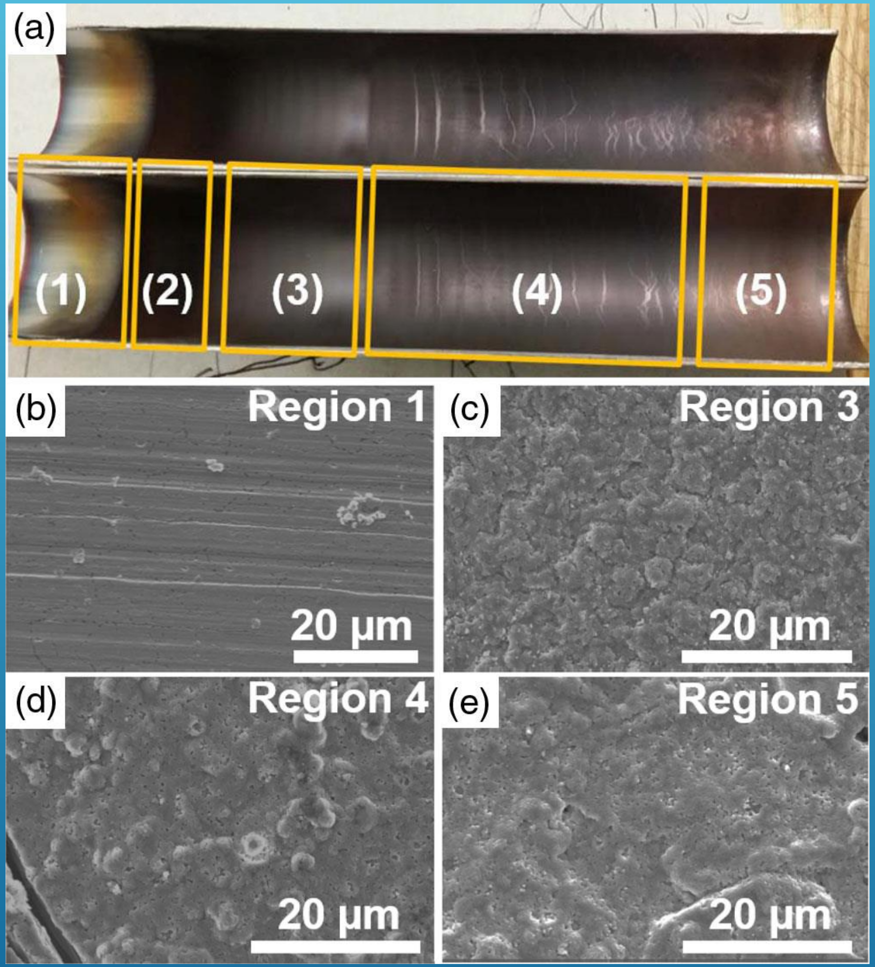
(a) A photo of the HPCVD system for cavity coating is shown here. (b) The schematic of the system with the Cu tube at the starting position. (c) Schematic of the system with the Cu tube at the end position.

- Cu tube ID – 1.495" OD – 1.625"
- Thermally insulated, water cooled B_2H_6 gas line from the top (T < 300 °C)
- Mg oven in the center ~ 600 °C
- Outside tubular heater ~ 660 °C
- Deposition pressure – 5 Torr
- B_2H_6 flow rate – 20 sccm

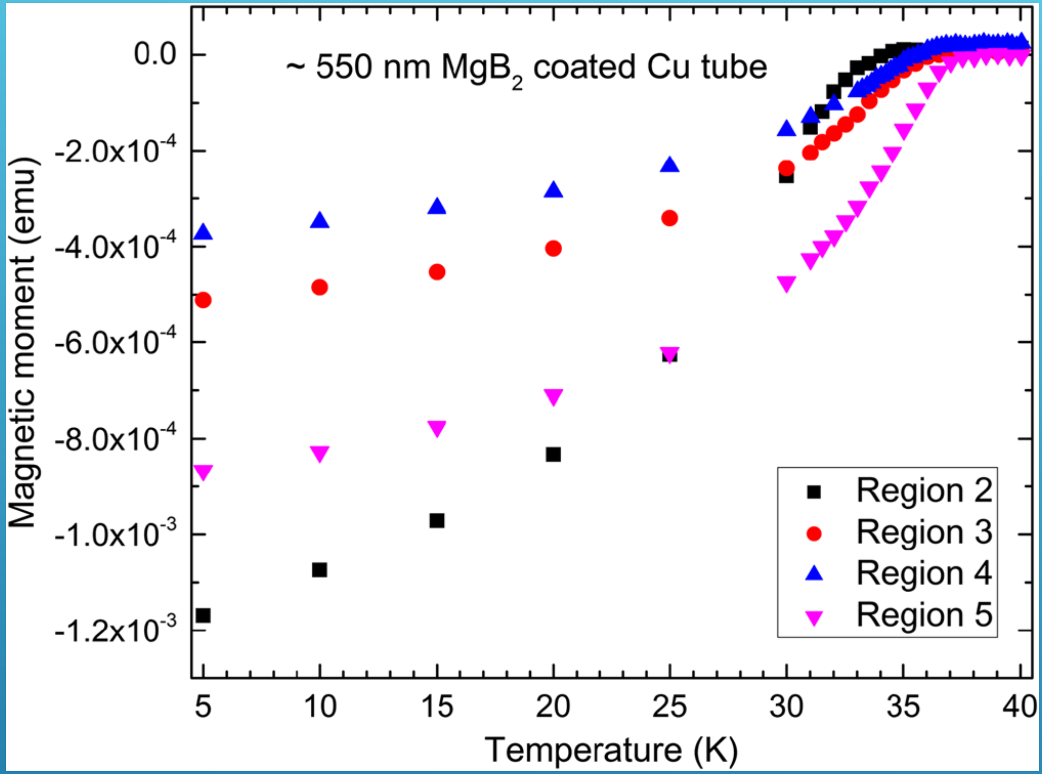
Withanage et al.,Phys. Rev. Accel. Beams, 20.102002 (2017)

Characterizations of MgB_2 coated Cu tubes

550 nm thick MgB_2 coating



(a) A photo of ~550 nm thick MgB_2 coated Cu tube halves. (b)–(e) SEM images of MgB_2 film surface on Cu samples cut out from regions 1, 3, 4 and 5, respectively.



Magnetic moment vs temperature curves of samples from regions 2, 3, 4 and 5 of a ~550 nm thick MgB_2 coated Cu tube.

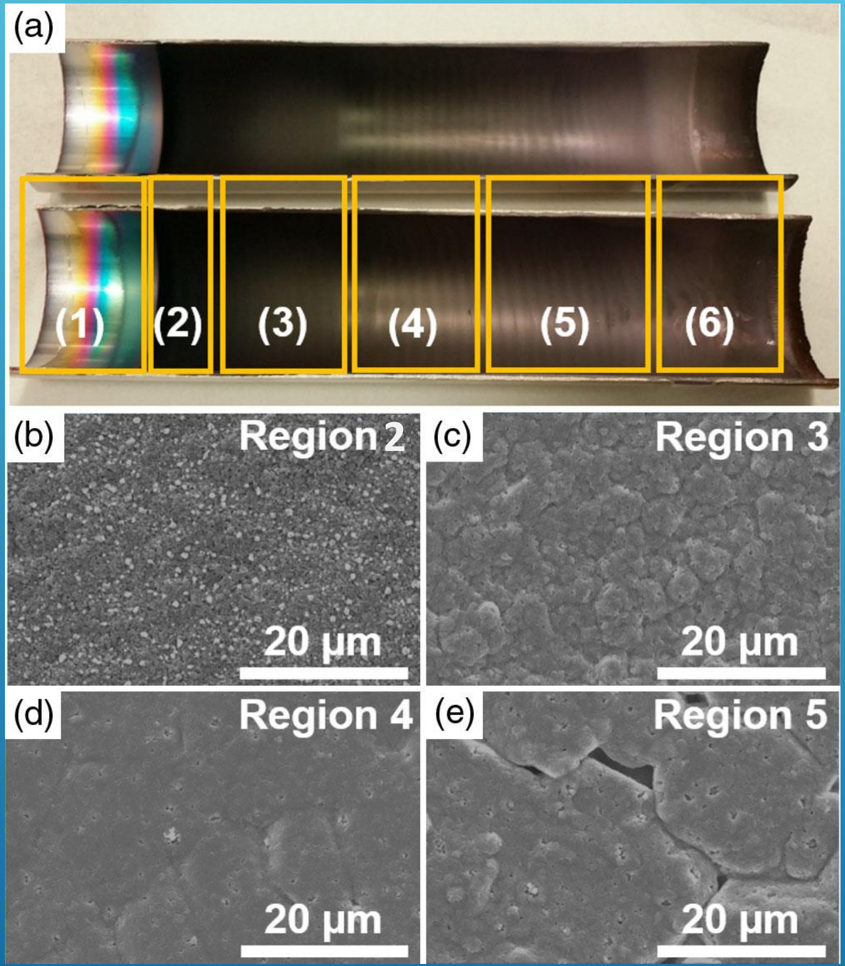
** Magnetic moment values cannot be compared due to the irregular shapes of the samples used in the measurement

- Coating length ~ 6 inch
- Uneven coating along the length
- T_c 35 K top to 37 K bottom

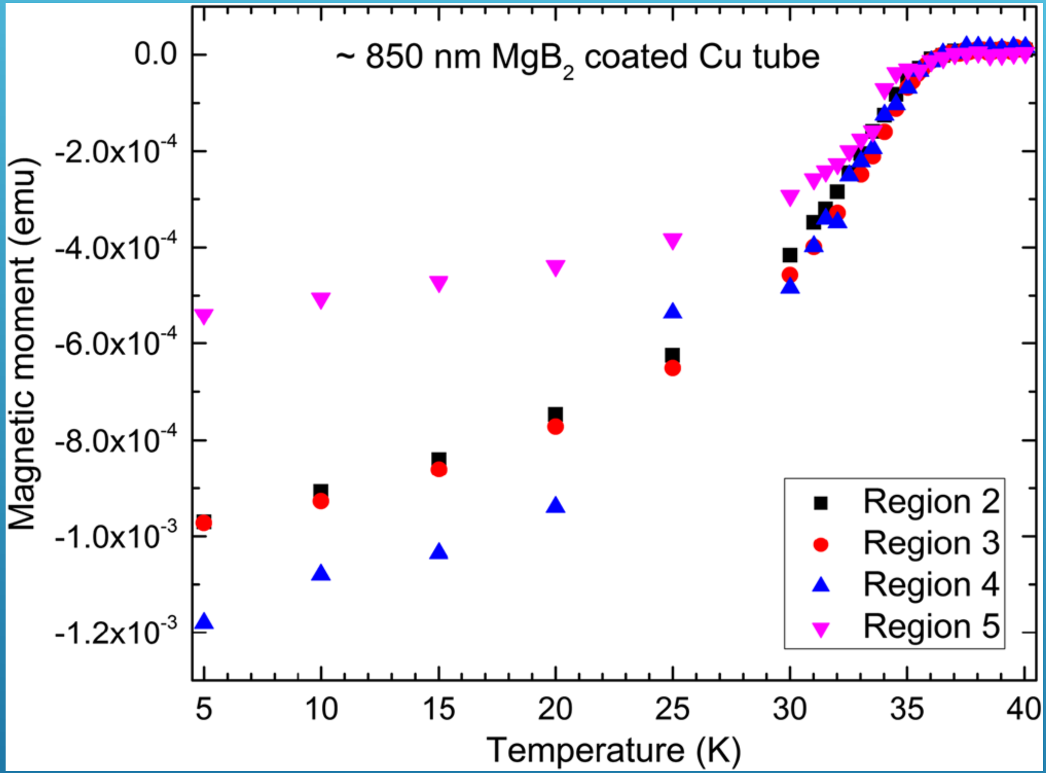
Withanage et al.,Phys. Rev. Accel. Beams, 20.102002 (2017)

Characterizations of MgB_2 coated Cu tubes

850 nm thick MgB_2 coating



(a) Photo of ~850 nm thick MgB_2 coated Cu tube halves. (b)–(e) SEM images of MgB_2 film surface on Cu sample cut out from regions 2, 3, 4 and 5, respectively.

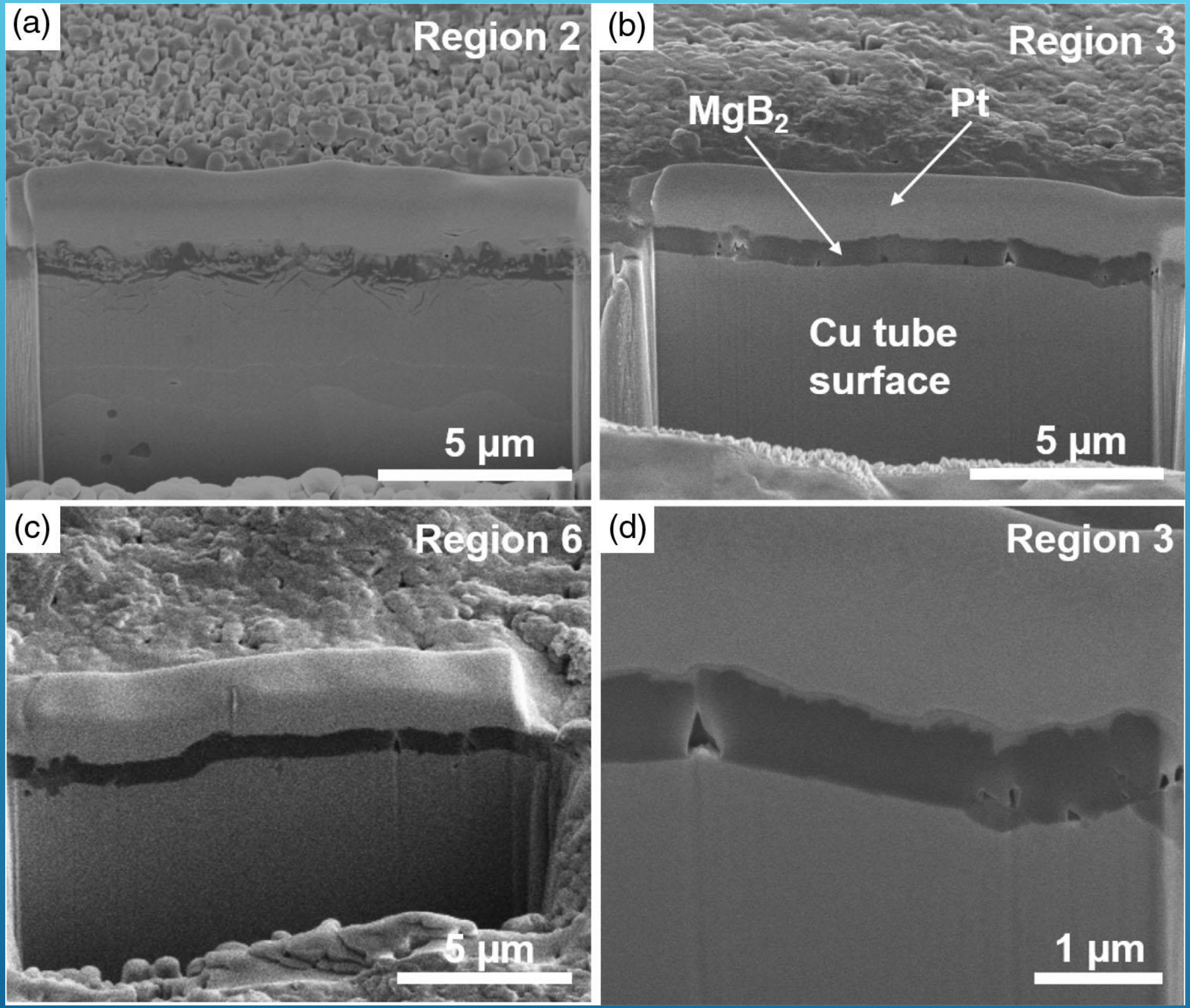


Magnetic moment vs temperature curves of samples from regions 2, 3, 4 and 5 of a ~850 nm thick MgB_2 coated Cu tube.

- Coating length ~ 6 inch
- Uneven coating along the length
- Uniform T_c of 37 K

Withanage et al.,Phys. Rev. Accel. Beams, 20.102002 (2017)

Cross-sectional studies

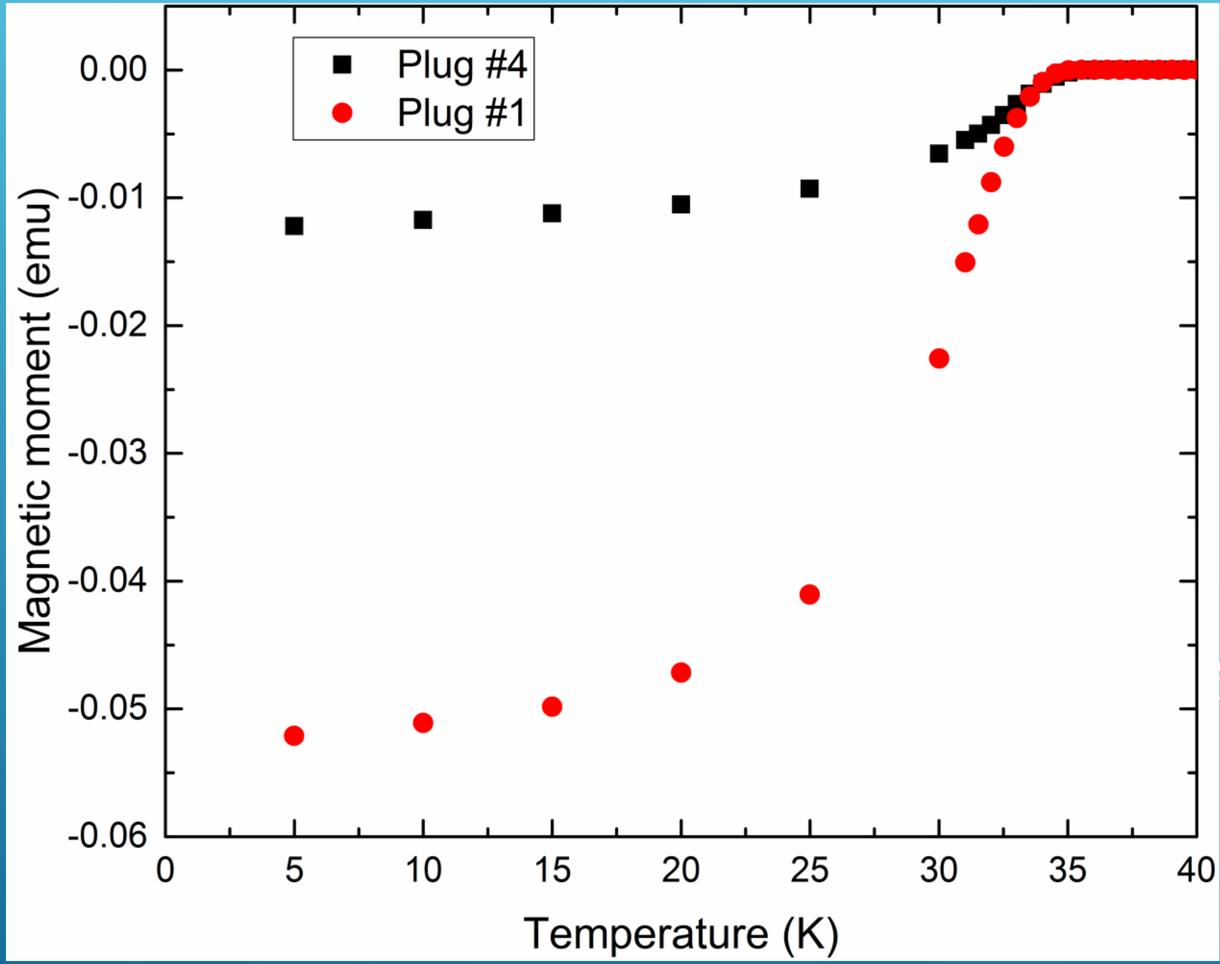


(a)–(c) Cross section images of samples cut out from regions 2, 3 and 6, respectively, of a ~850 nm thick MgB_2 coated tube. (d) Zoomed in cross section image from region 3.

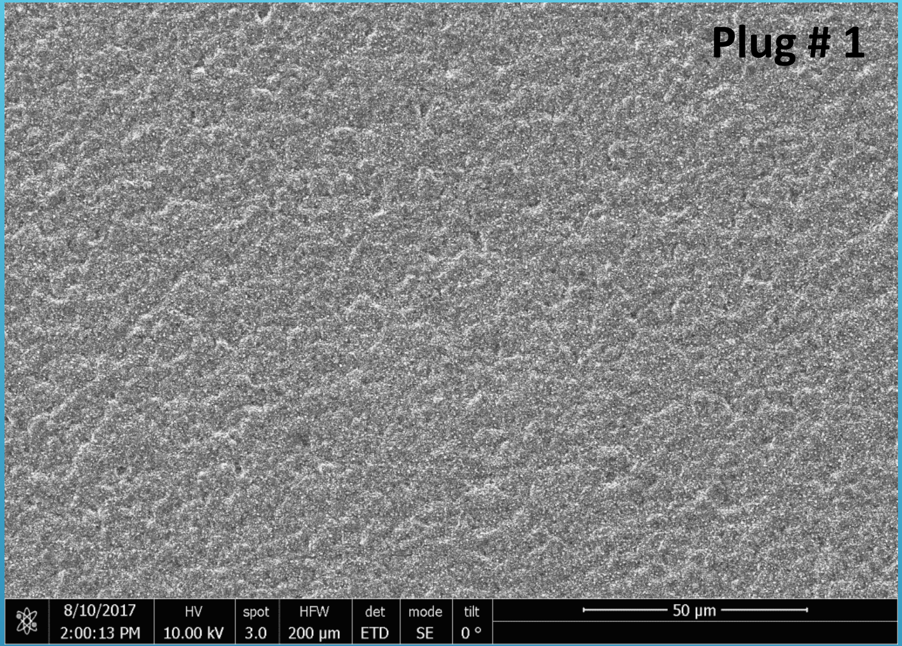
- Poor coating in the region 2
  - Similar coating was observed in the center of the 2-inch discs
  - Indication of Mg deficiency
- Region 3 to 6, fairly uniform coating with occasional voids
  thickness ~ 850 nm
- These voids can be due to the unpolished Cu surface or overlapping of large MgB_2 grains

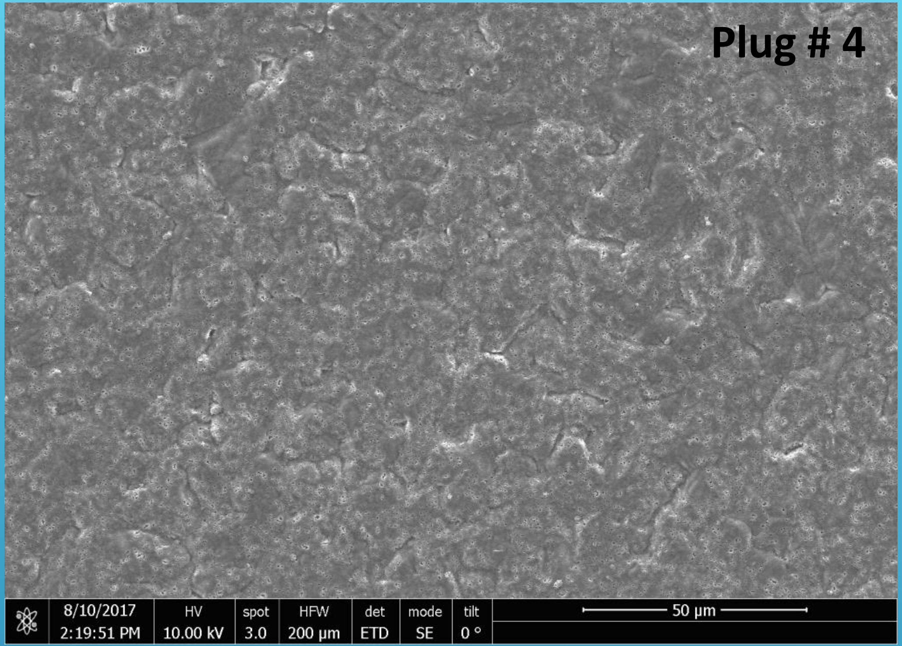
Withanage et al.,Phys. Rev. Accel. Beams, 20.102002 (2017)

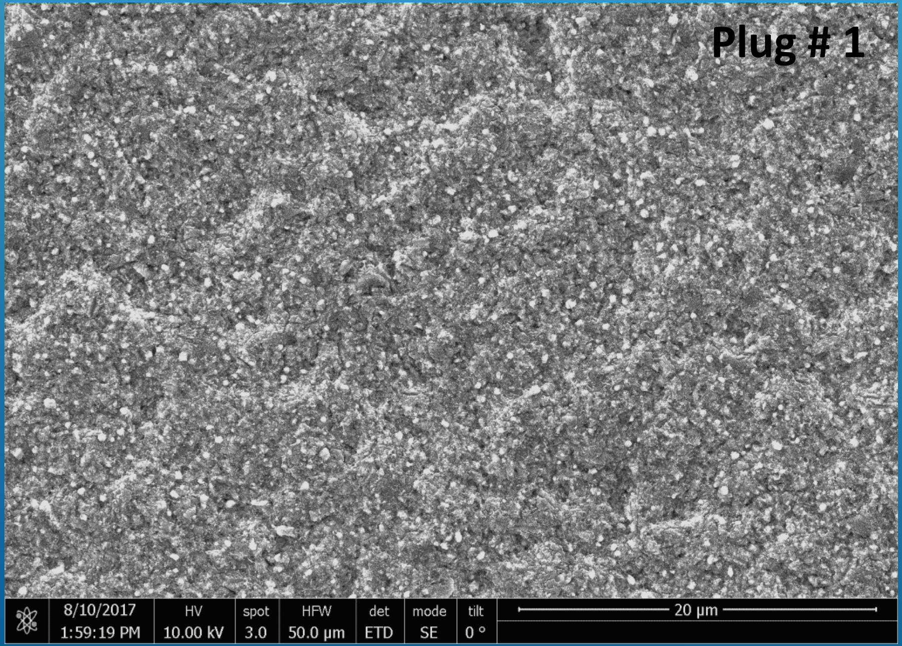
MgB_2 on polished Cu plugs attached to the Cu tube

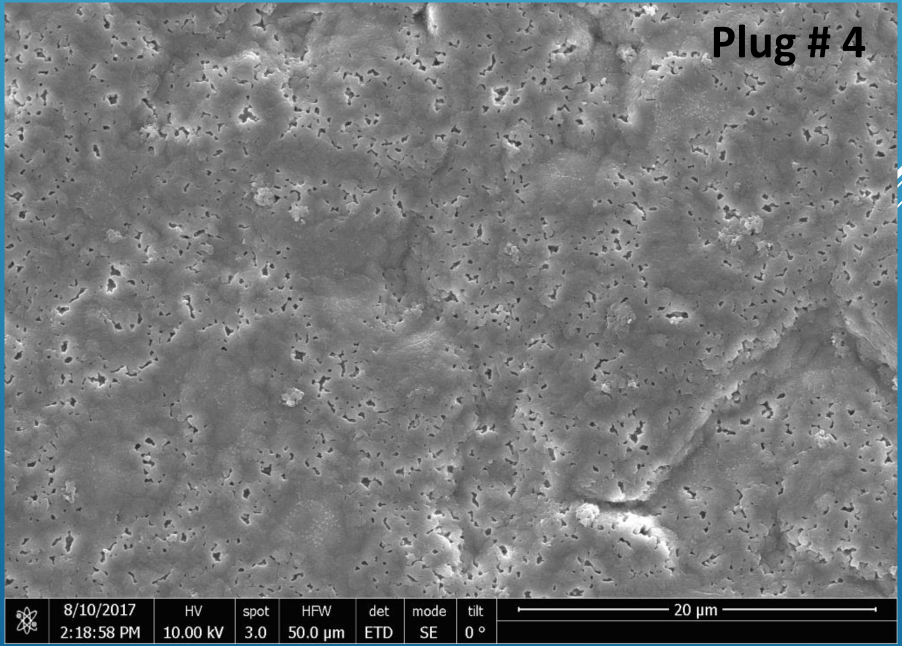


MgB_2 on polished Cu plugs attached to the Cu tube









SUMMARY

- We have successfully developed a MgB_2 growth process on Cu without using buffer layers at low temperature
- MgB_2 coated Cu discs showed excellent DC superconducting properties with high T_c (~37 K) & high J_c (10^7 A cm^{-2}).
- MgB_2 coated Cu discs showed high Q close to the reference Nb discs.
- The growth process was scaled up to accommodate 3 GHz cavity and coating of the inner wall of Cu tubes with diameter similar 3 GHz RF cavity was tested
- MgB_2 coated Cu tubes showed Tc close to 37 K in thick films and the coating was uneven along the length of the tube.

FUTURE WORK

- Modification of the coating system to reduce the temperature variation along the length
- Coating Cu tubes with diameter similar to the equator of a 3 GHz RF cavity

PUBLICATIONS

1. Growth of magnesium diboride films on 2 inch diameter copper discs by hybrid physical–chemical vapor deposition
Withanage *et al., Supercond. Sci. Technol.* 30, 0450016 (2017)
2. Magnesium diboride on inner wall of copper tube: a test case for superconducting radio frequency cavities
Withanage et al.,Phys. Rev. Accel. Beams, 20.102002 (2017)

ACKNOWLEDGEMENT

1. X. X. Xi, N. H. Lee, T. Tan and M. A. Wolak at Temple University
2. A. Nassiri at Argonne
3. P. B. Welander at SLAC
4. G. Eremeev, F. Hannon and Robert Rimmer at JLab