Synthesis of Nanoscale Magnesium Diboride Powder

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Collaborator – Made Pure Boron

• James V. Marzik of Specialty Materials Inc. developed the plasma synthesis method to make high purity boron nanopowders having particle size in the range of 20 nm to 200 nm.
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Plasma Synthesis rf Argon Torch
Gas feed into the rf torch

- $\text{BCl}_2 + \text{H}_2 \rightarrow \text{B} + 2 \text{HCl}$
- Add methane, $\text{CH}_4$, for carbon doping
- Add titanium tetrachloride, $\text{TiCl}_4$, for $\text{TiB}_2$ precipitates
- In this work, we use either undoped boron or boron 2% carbon.
- The point of adding carbon is to raise $H_{c2}$ of the resulting $\text{MgB}_2$ to 35 Tesla
SEM of undoped boron powder from Specialty Materials Inc.
Boron contains both amorphous and crystallized boron – TEM micrograph
TEM of mix of amorphous & crystalline boron particles
Amorphous boron particles
Selective Area Diffraction (SAD)
TEM -- SAD shot
TEM to get particle size distribution
SEM of starting boron powder
Boron powder particle size

Particle size distribution higher power

- SM1 062211
- SM1 041912
Broad descriptors of boron powder

• Fluffy & often electrostatically charged.
• Lacy agglomeration – easily can flatten with spatula.
• Difficult to pack evenly.
• Traces of chlorine in EDS/SEM analysis ~0.2%
• We always handle this powder in dry N₂ in glove box or transport it in Mason jars.
Convert \textbf{B powder to MgB}_2 \textbf{powder}

- Use 3 mm size Mg chunks
- Place Mg & B in Mason jar under N_2 gas and mix by rolling for 1 min. [50 g batch]
- Transfer mixed powders to Nb reaction vessel 150 mm long by 28 mm OD by 1 mm wall.
- Evacuate the vessel and e-beam weld shut.
- React Nb vessel under Ar gas at 830\textdegree C for 24 h in horizontal furnace. Open under N_2 in glove box. Temperature & Time can be adjusted.
SEM of MgB$_2$ Powder 830$^\circ$C, 24 h
SEM of MgB$_2$ Powder 830$^\circ$C 8h
Grain size for 3 batches $830^\circ\text{C} - 24\ h$
Grain size at 830°C for 8h, 12h, 24h
Grain size at 830°C for 24h, 12h & 8h

![Graph showing average grain size MgB₂ reacted at 830°C]
Particles may have several grains

Look at particle size

Particle size distribution for undoped B at 830°C
Change in particle size 830°C for 24h, 12h & 8h
Average particle size for 48 h

![Graph showing average length of largest grains at different reaction temperatures.](image)
Average particle size vs time 830°C
X-ray spectrum for MgB$_2$ powder
Conclusions

• 1) 830°C for 24h gives particle size under a micron.
• 2) average grain sizes tend to run about $\frac{1}{2}$ the average particle size
• 3) reducing the temperature to 800°C and/or the time to 8h substantially reduces particle and grain size.
Errors of Judgment
Things we did wrong

• 1) Use an Fe reaction vessel
• 2) Seal the Nb reaction vessel under Ar and use arc welding.
Mistakes that we made.

Problem #1 – Fe depresses $T_c$

- Fe reaction vessel suppresses $T_c$ about three degrees Kelvin.
- Nb give the same result as Ta so we used Nb for cost reasons.
Problem # 2, Fe promotes a platy growth, often 10 \( \mu m \) across

- In Fe reaction vessel we saw many large hexagonal plates, many in the 5 micrometers to 10 micrometers across
Problem #3, must evacuate the Nb reaction vessel

• We initially sealed the Nb reaction vessels under \( \frac{3}{4} \) atmospheres of Ar which gives about 2 atmospheres of Ar at 830\(^\circ\)C.
• Reaction is slow and not reproducible
• The Ar impedes the diffusion of Mg vapor through the packed boron powder.
• The Ar slows the conversion of boron to MgB\(_2\)