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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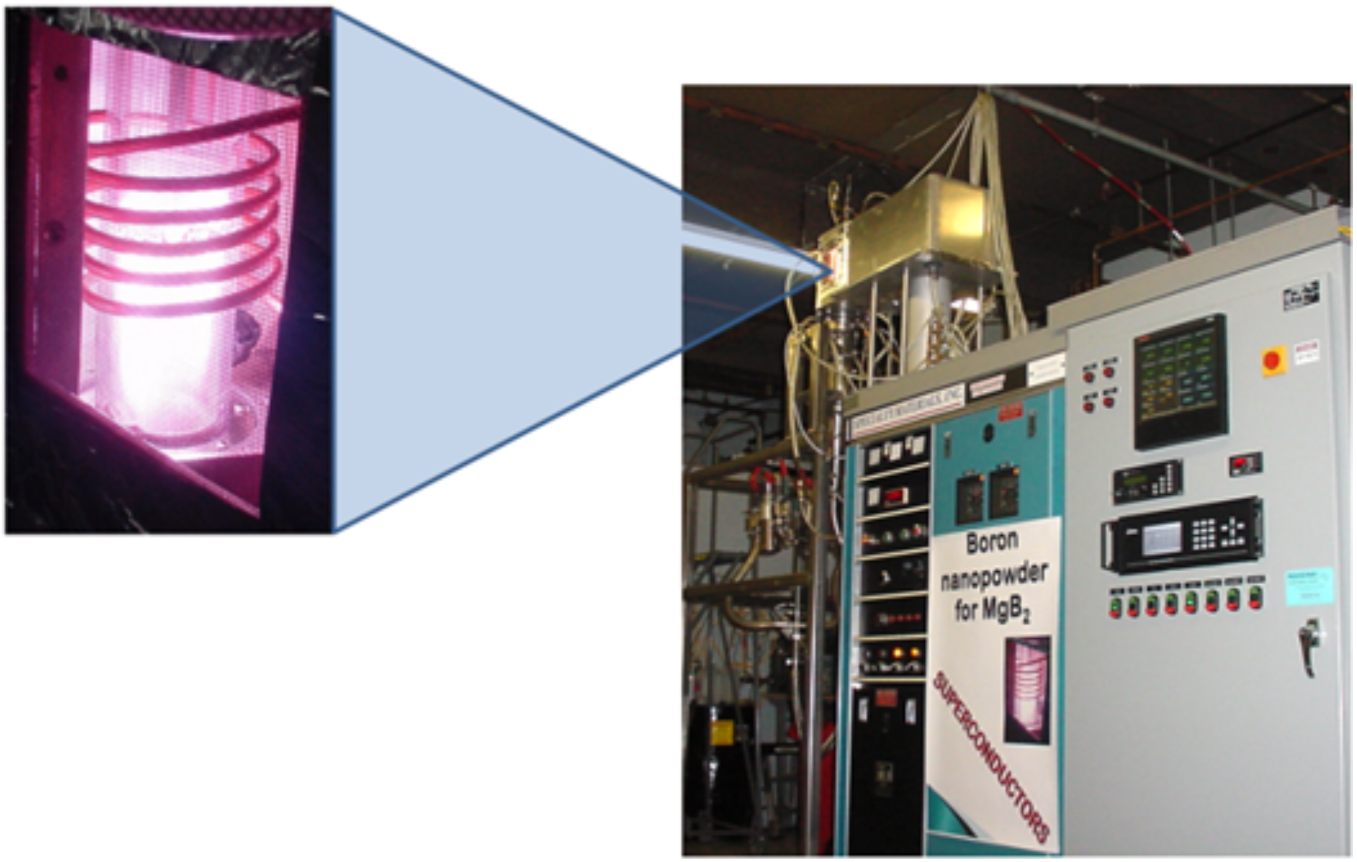
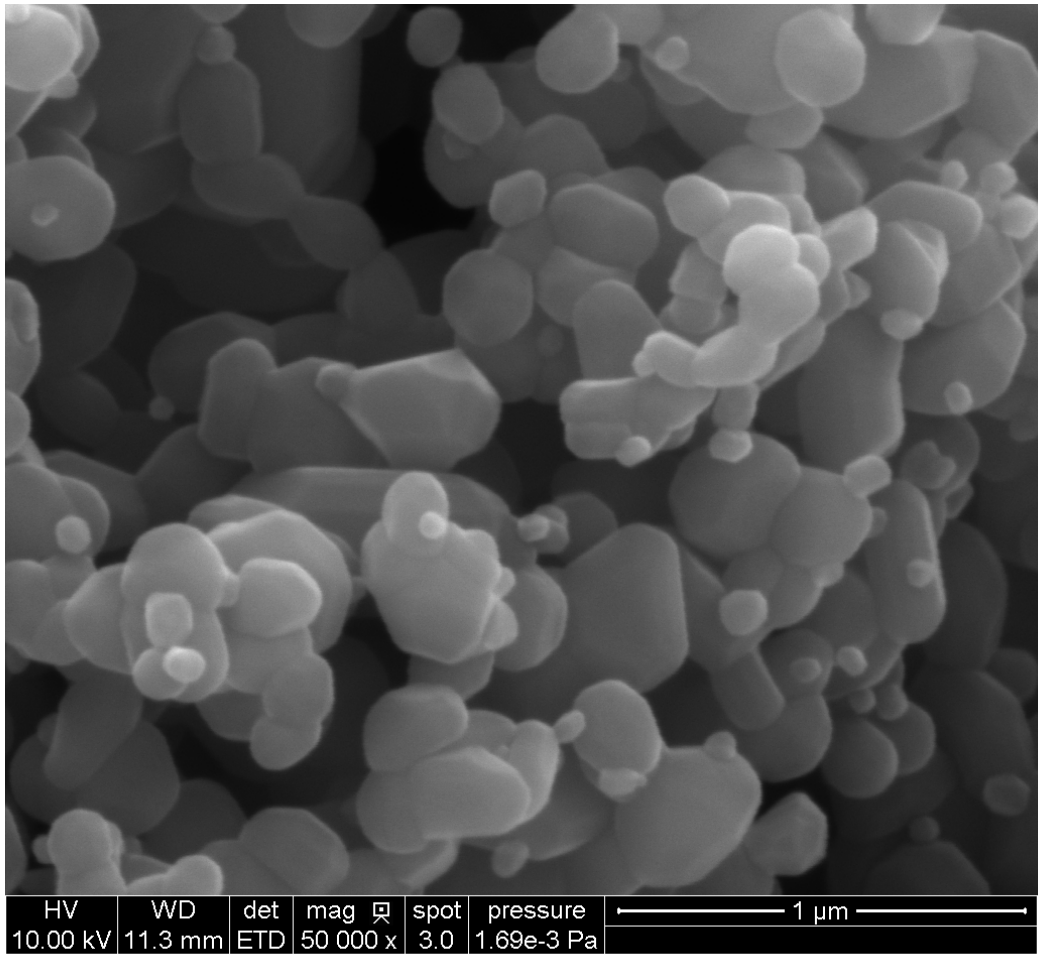
Plasma Synthesis rf Argon Torch



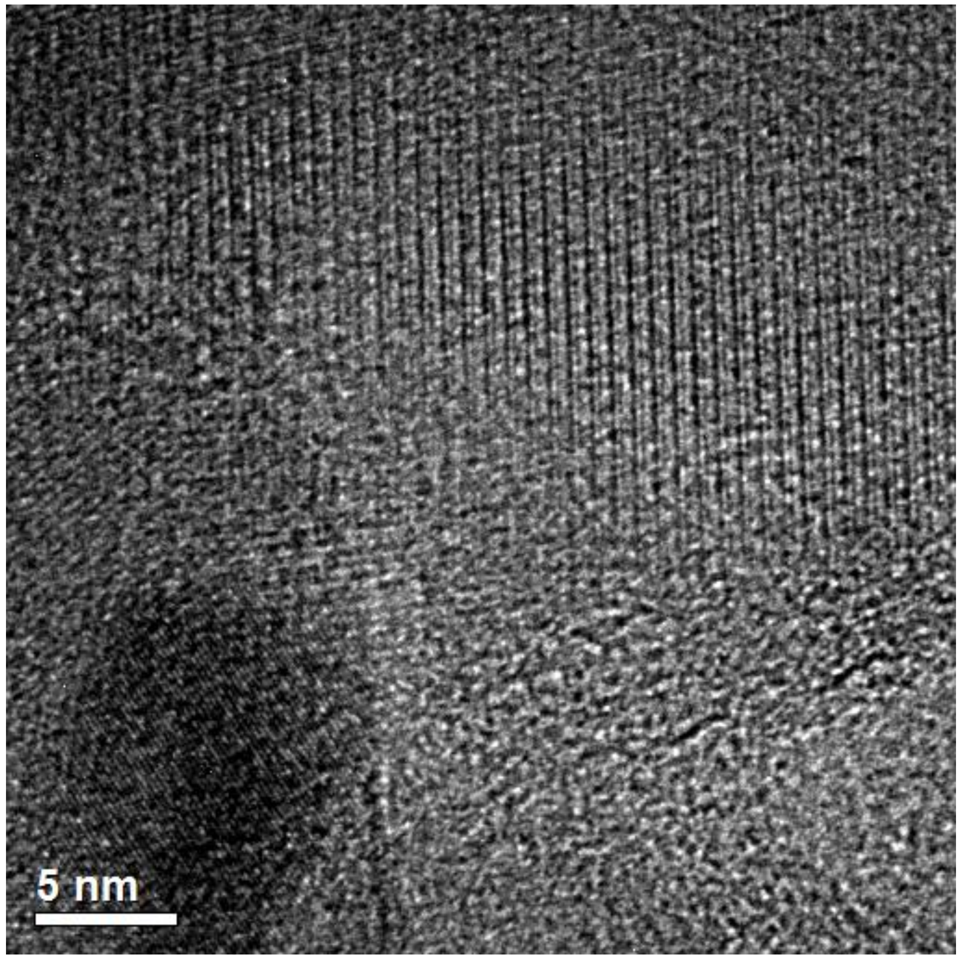
Gas feed into the rf torch

- $BCl_2 + H_2 \rightarrow B + 2\ HCl$
- Add methane, CH_4, for carbon doping
- Add titanium tetrachloride, $TiCl_4$, for 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 MgB_2 to 35 Tesla

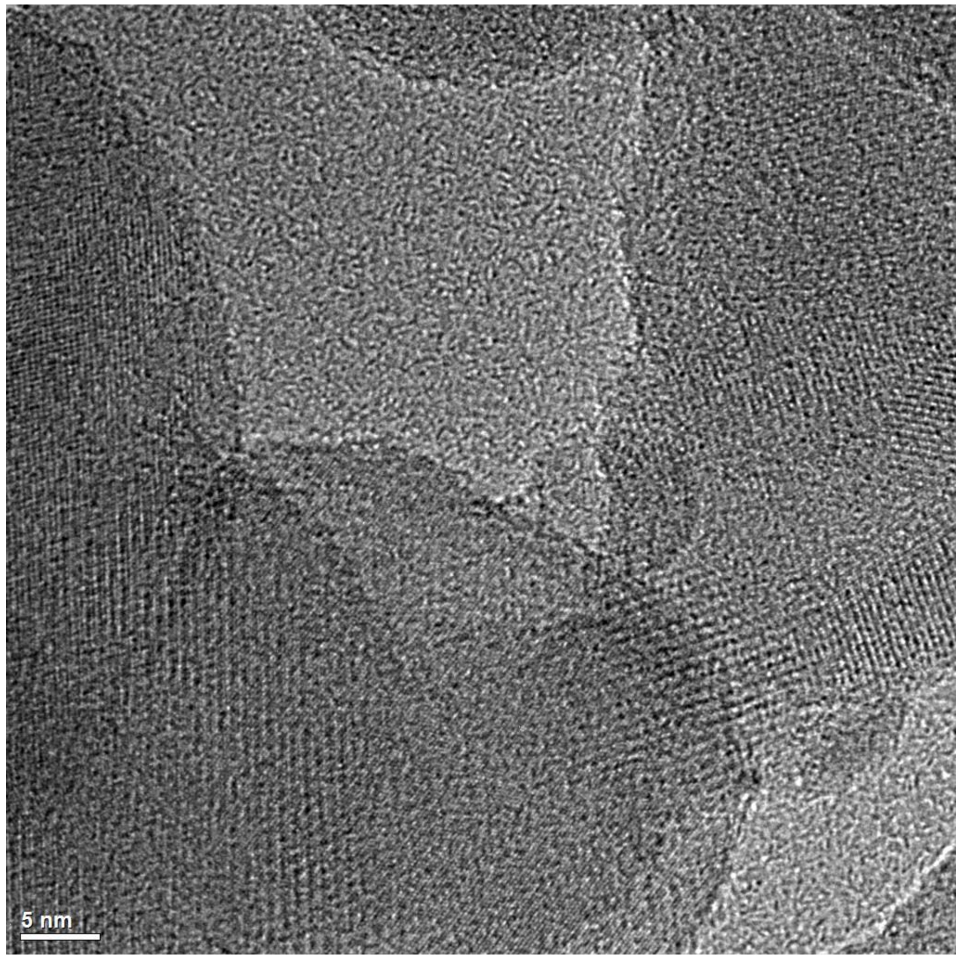
SEM of undoped boron powder from Specialty Materials Inc.



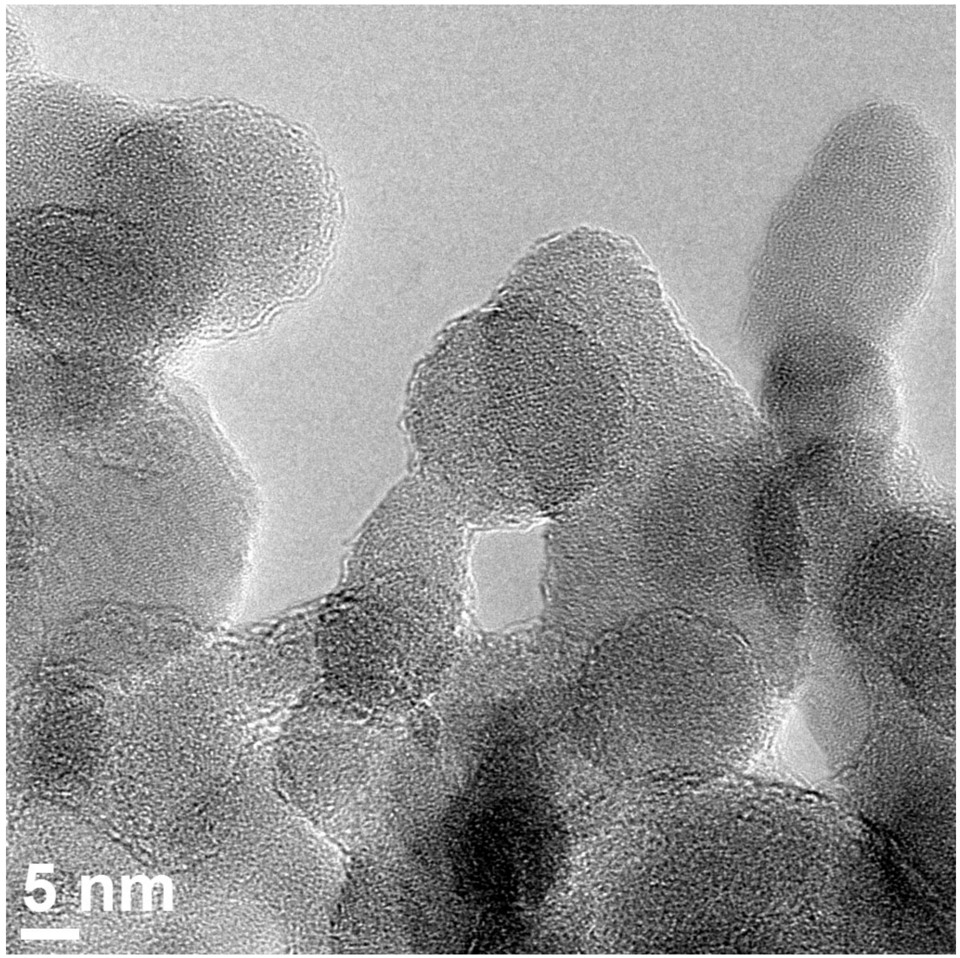
Boron contains both amorphous and crystallized boron – TEM micrograph



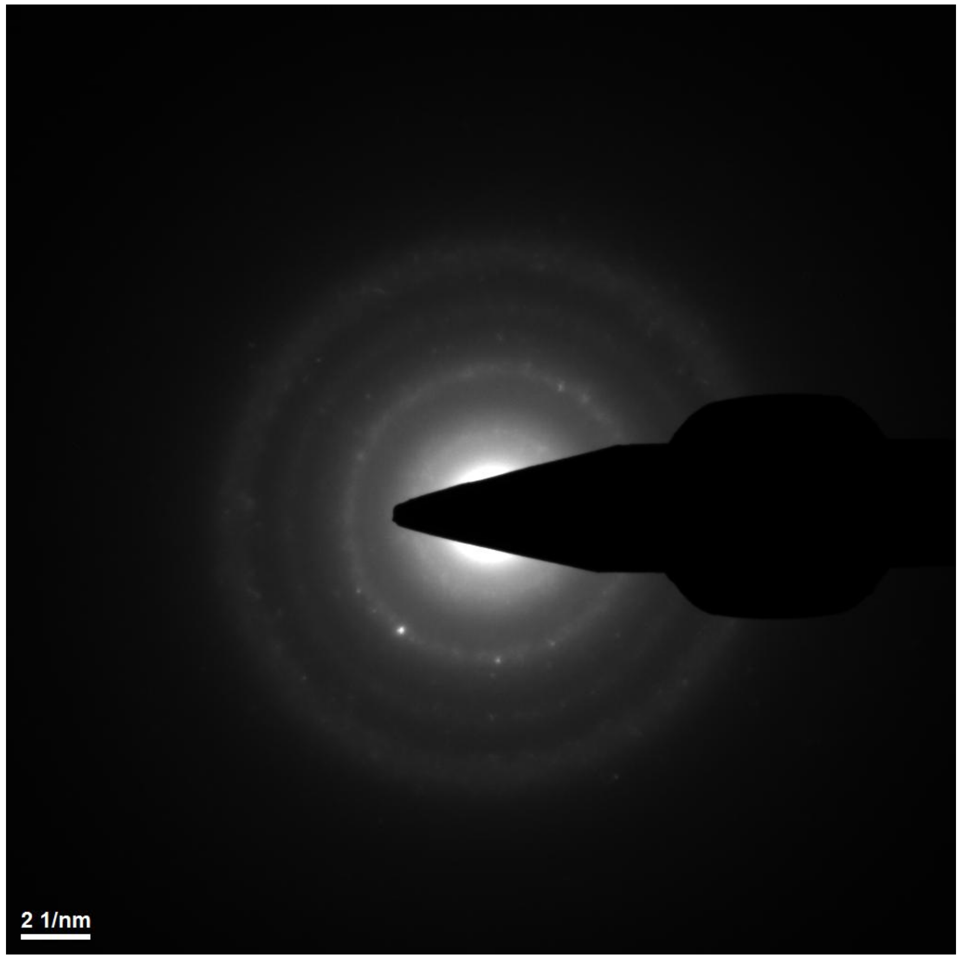
TEM of mix of amorphous & crystalline boron particles



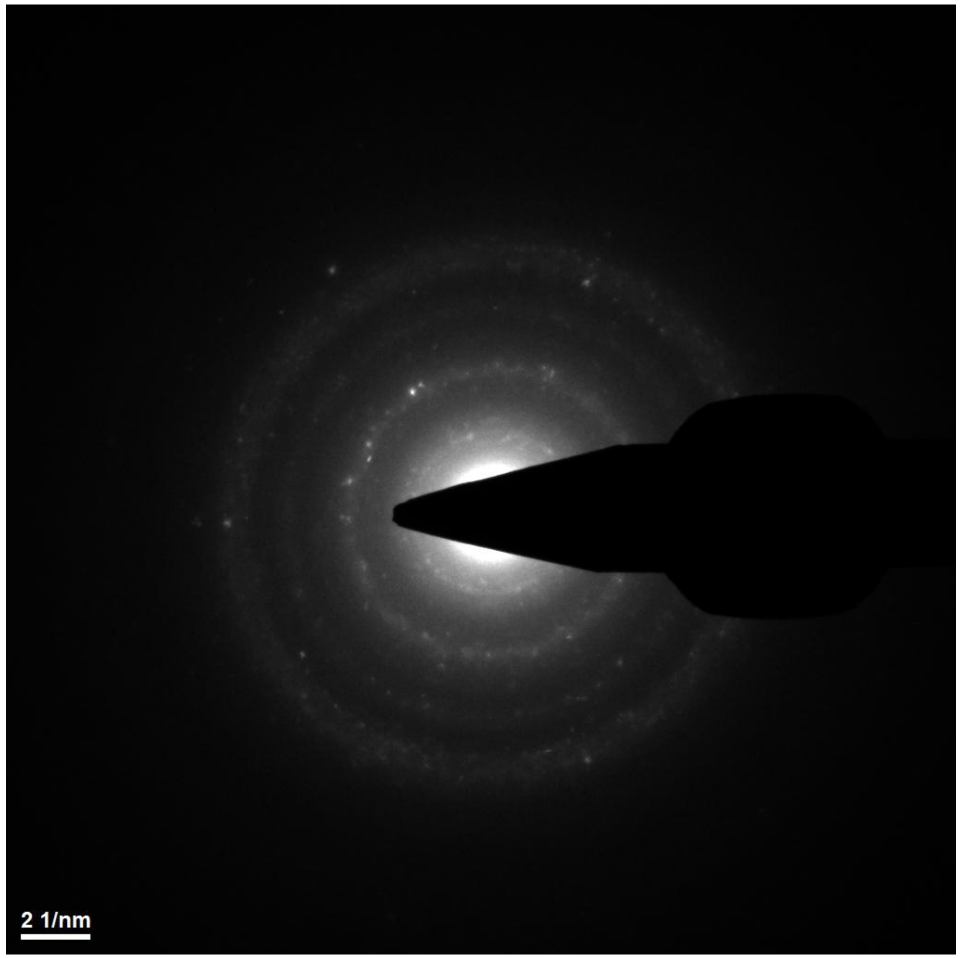
Amorphous boron particles



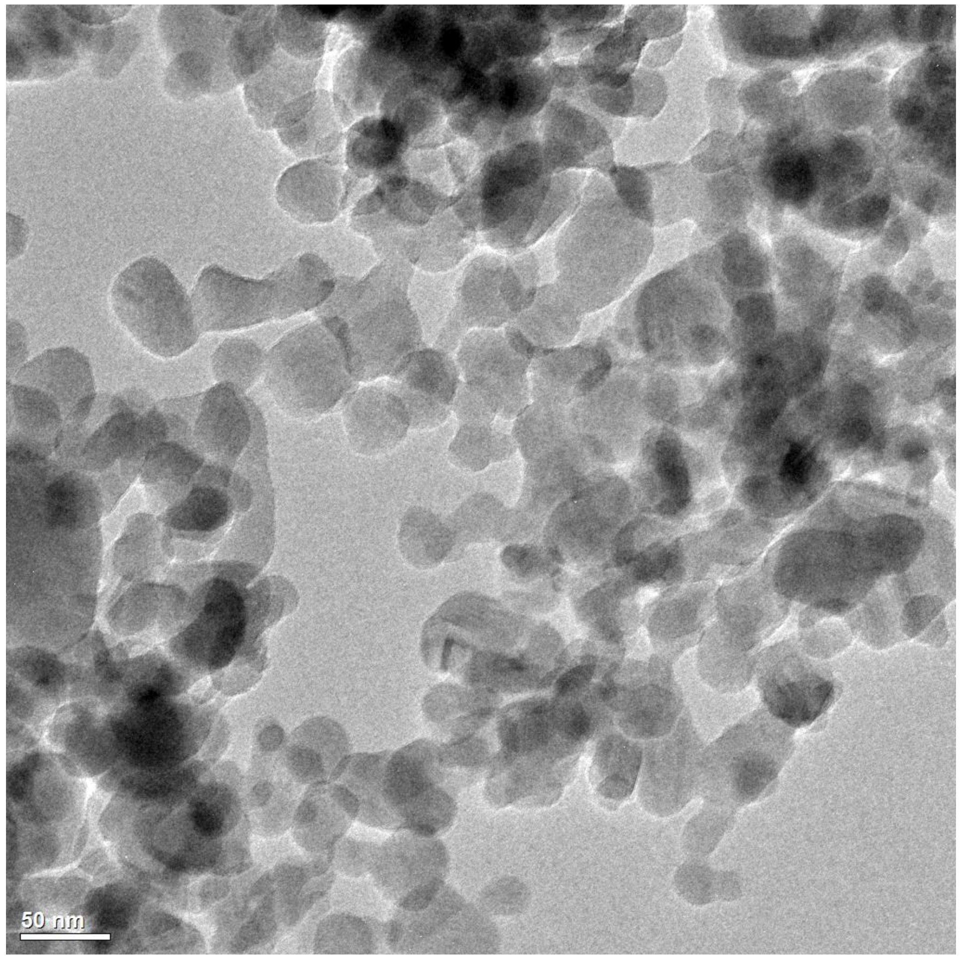
Selective Area Diffraction (SAD)



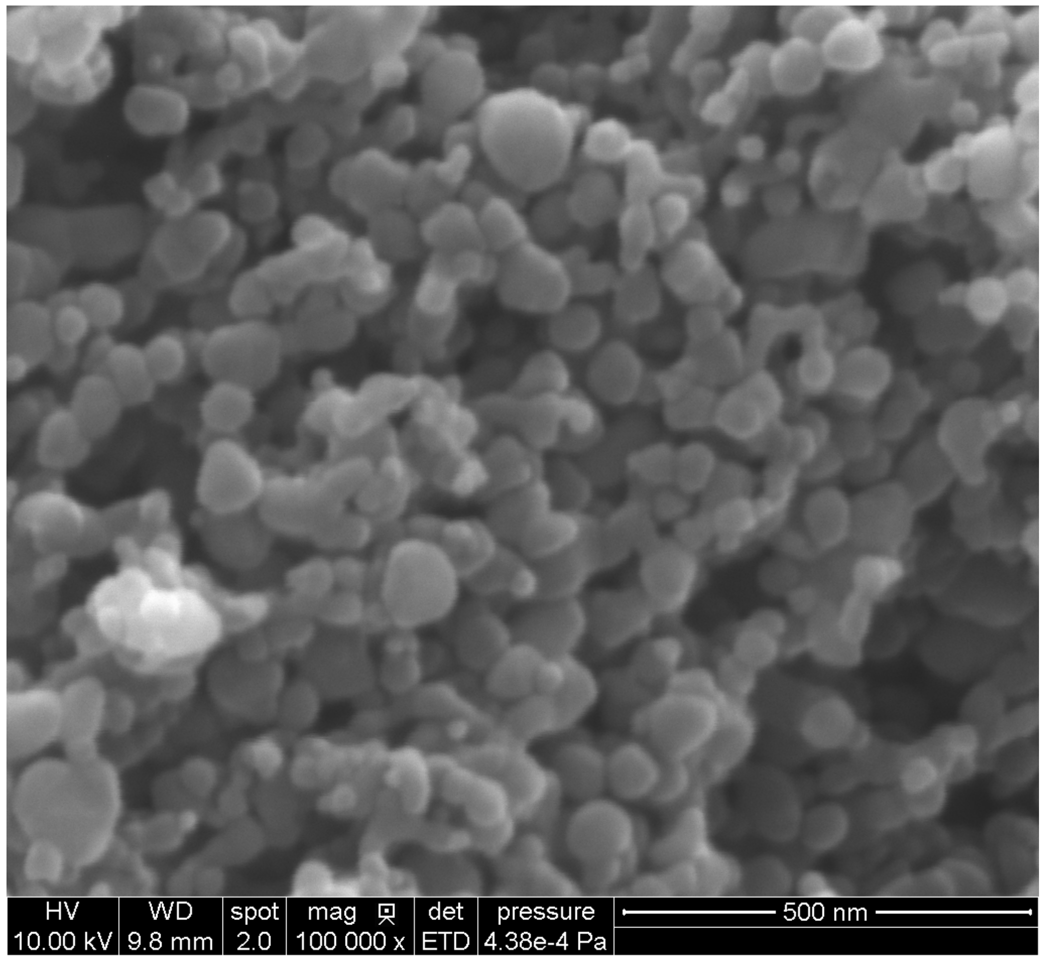
TEM -- SAD shot



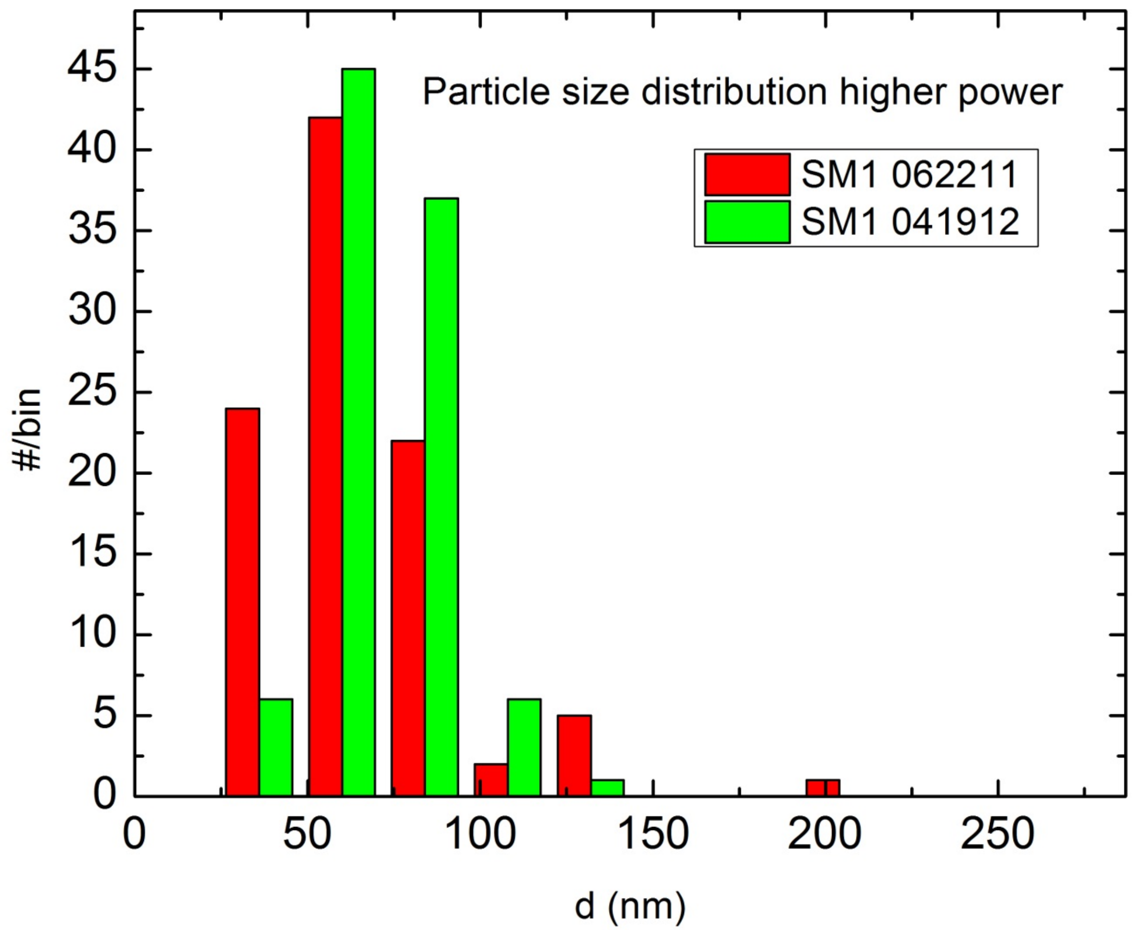
TEM to get particle size distribution



SEM of starting boron powder



Boron powder particle size



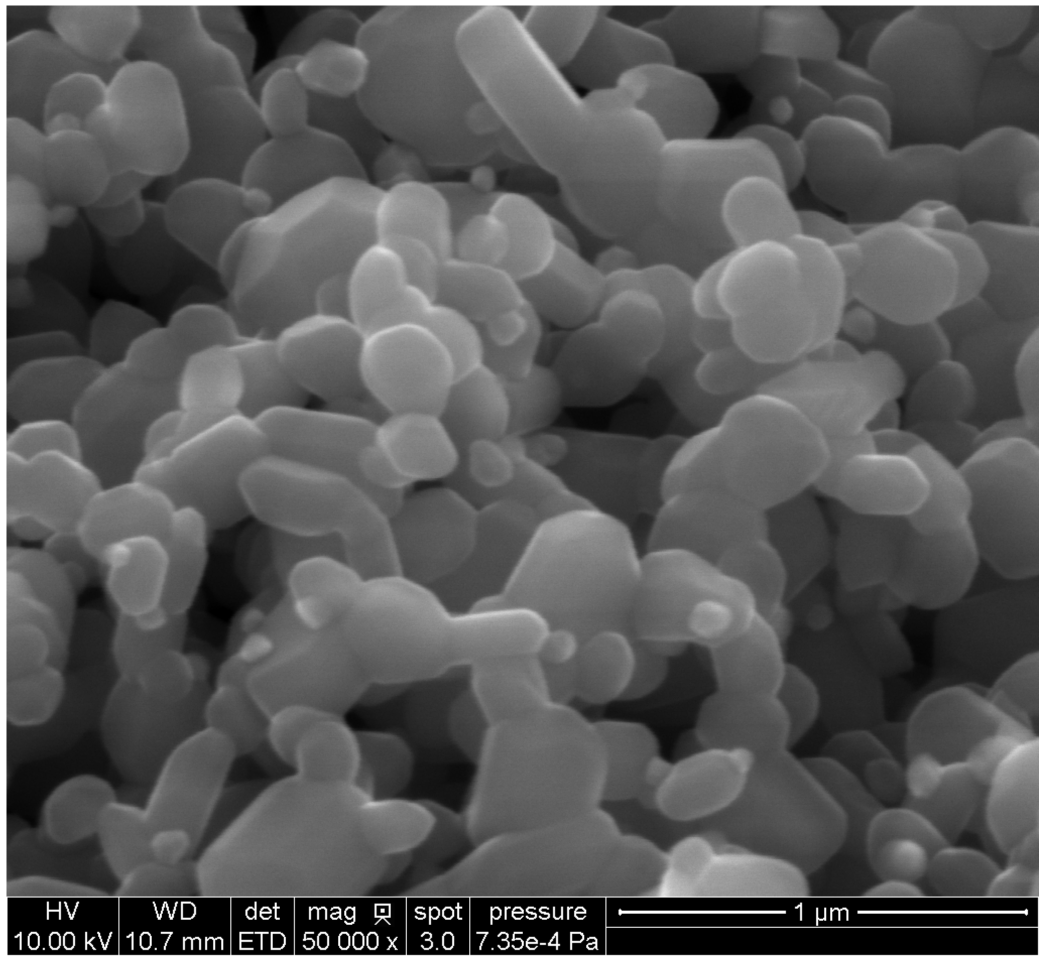
Broad descriptors of boron powder

- 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 N_2 in glove box or transport it in Mason jars.

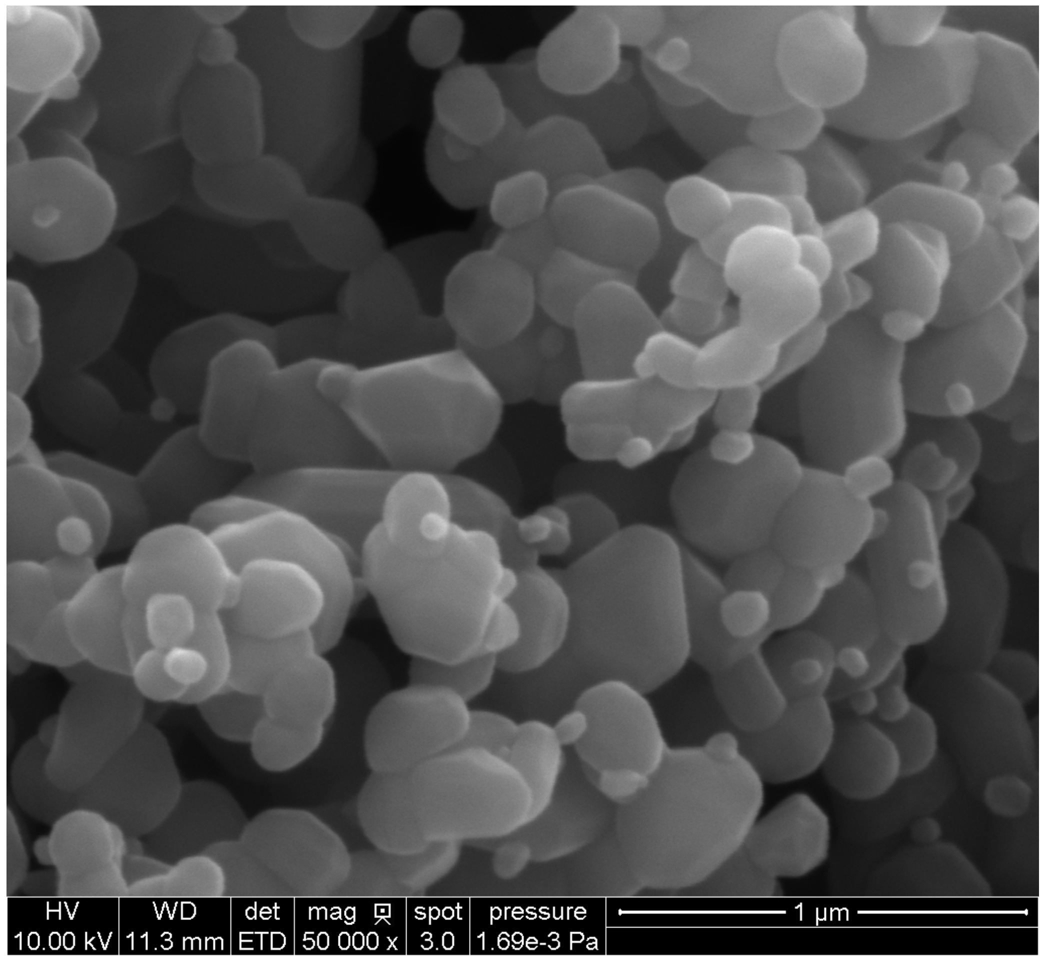
Convert B powder to MgB_2 powder

- 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^{o}C for 24 h in horizontal furnace. Open under N_2 in glove box. Temperature & Time can be adjusted.

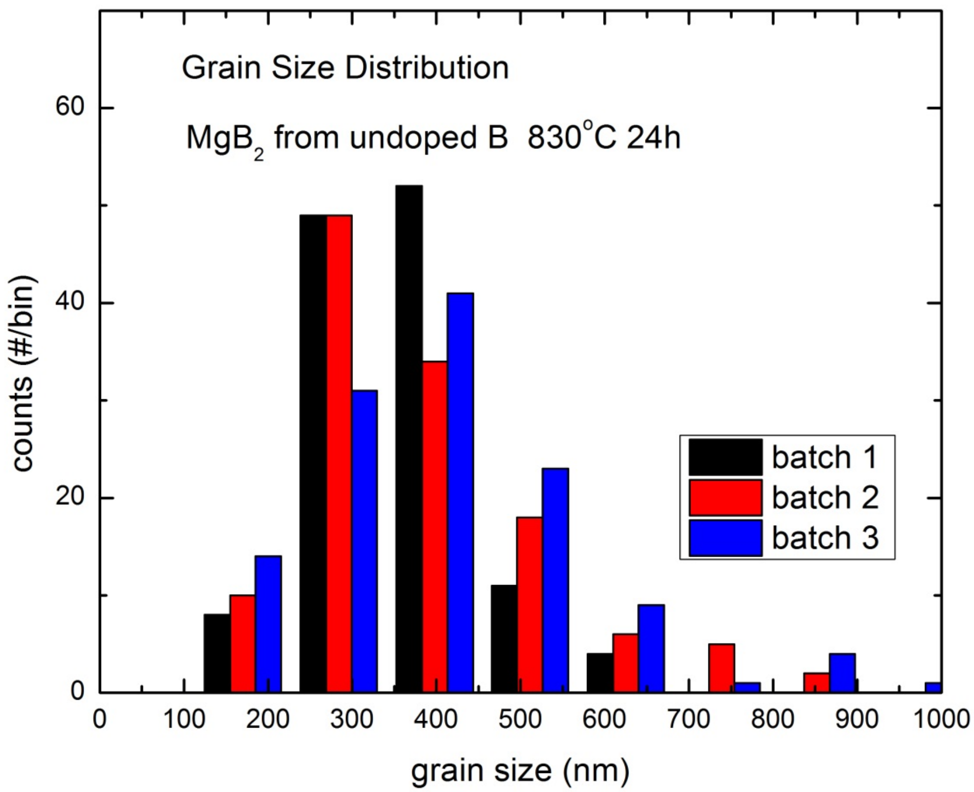
SEM of MgB_2 Powder 830ºC, 24 h



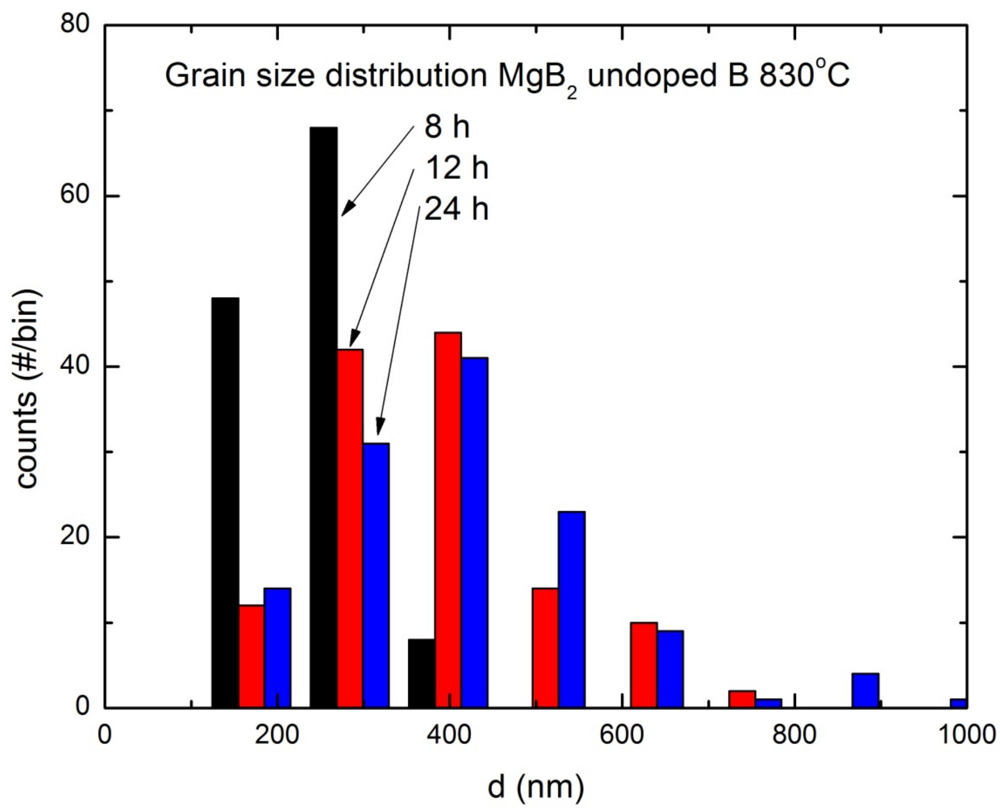
SEM of MgB_2 Powder 830^{o}C 8h



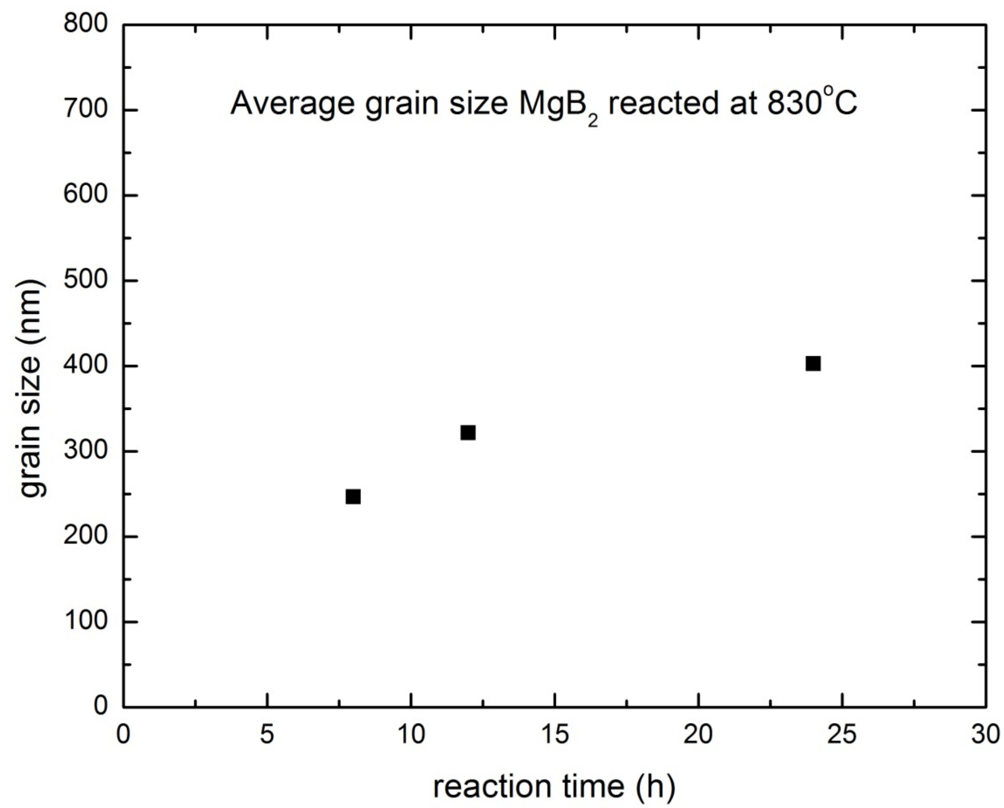
Grain size for 3 batches 830ºC – 24 h



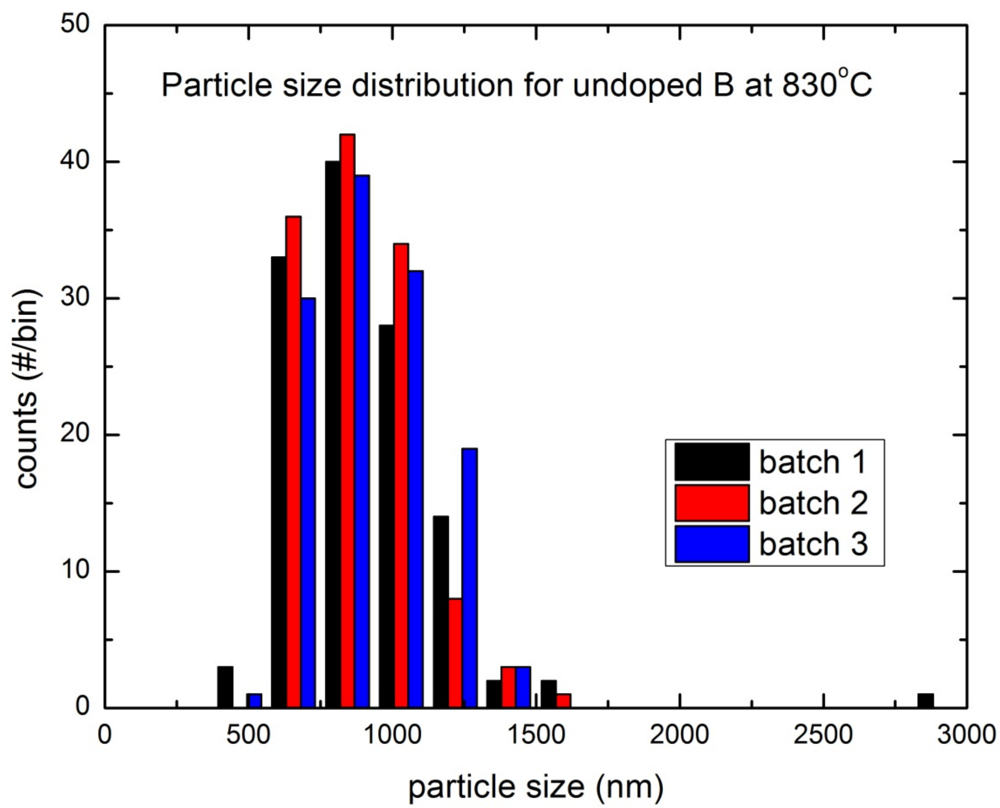
Grain size at 830^{o}C for 8h, 12h, 24h



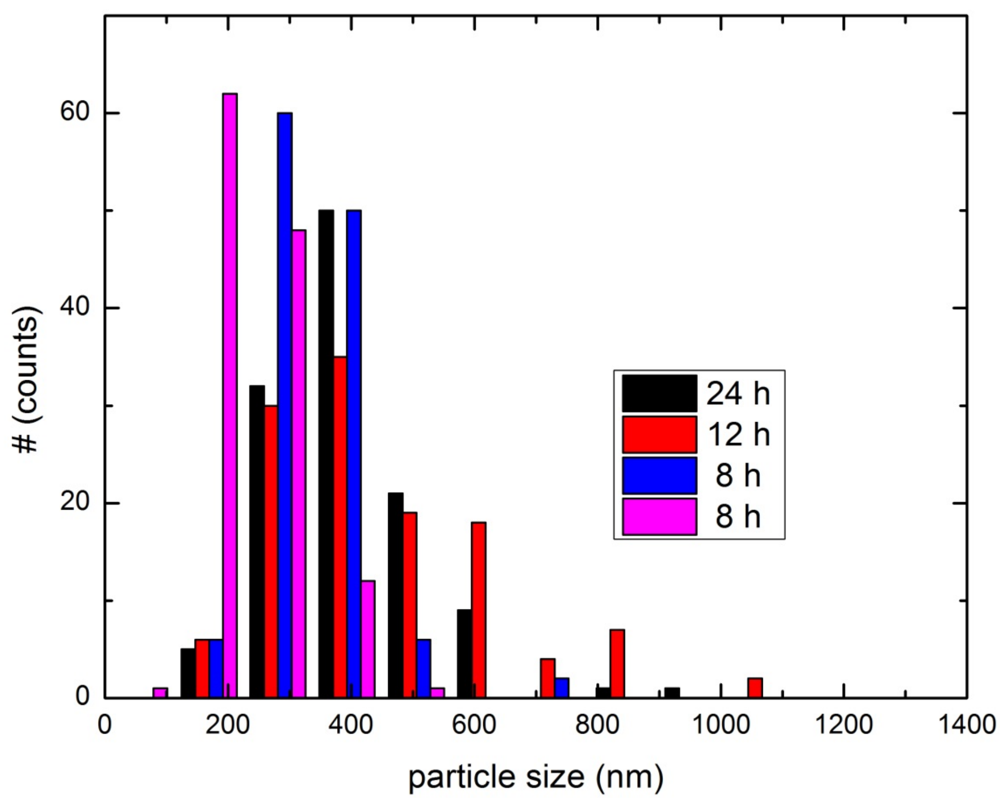
Grain size at 830^{o}C for 24h, 12h & 8h



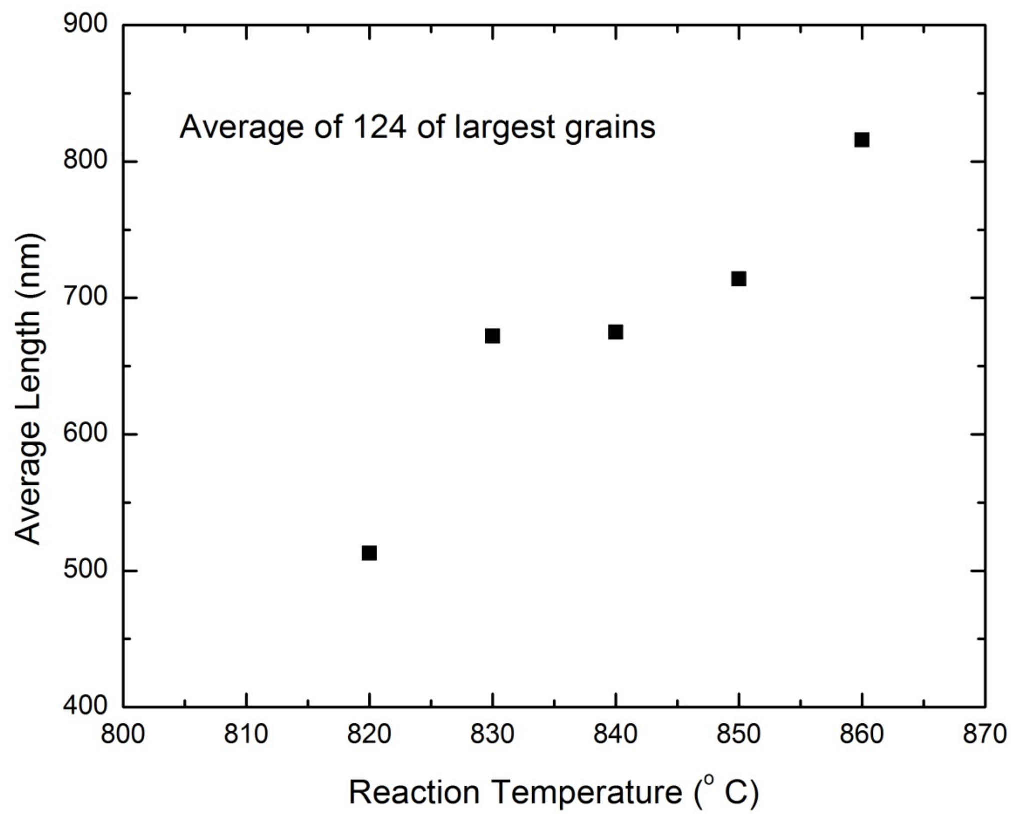
Particles may have several grains
Look at particle size



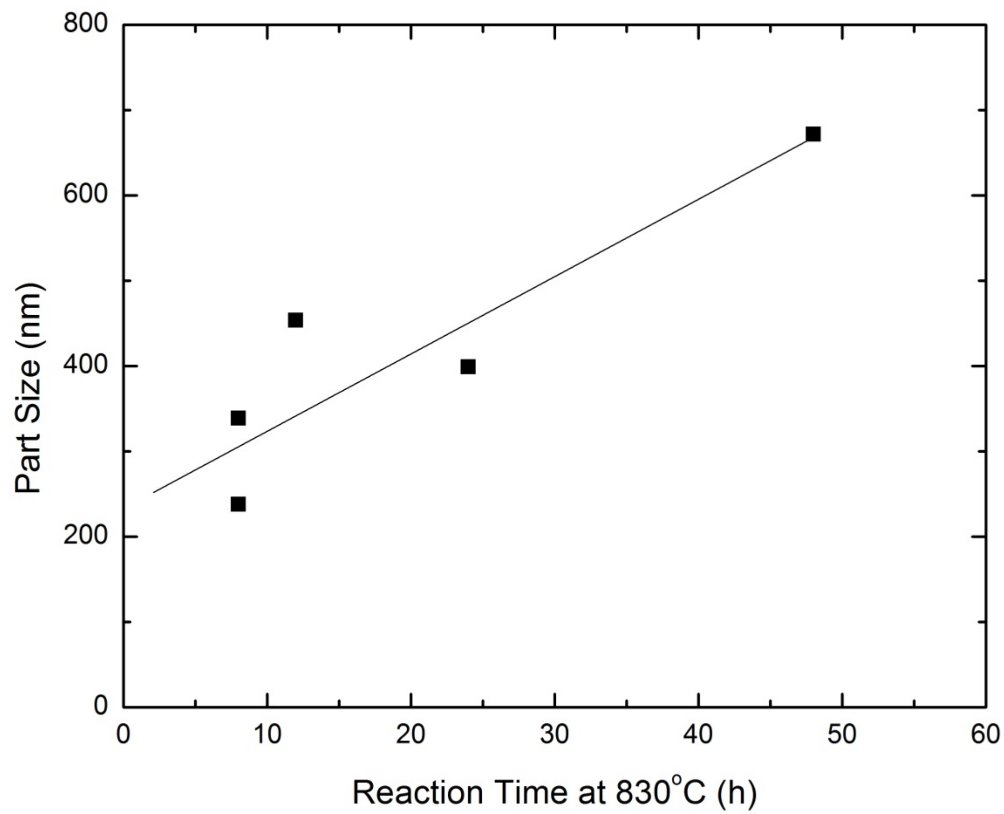
Change in particle size 830^{o}C for 24h, 12h & 8h



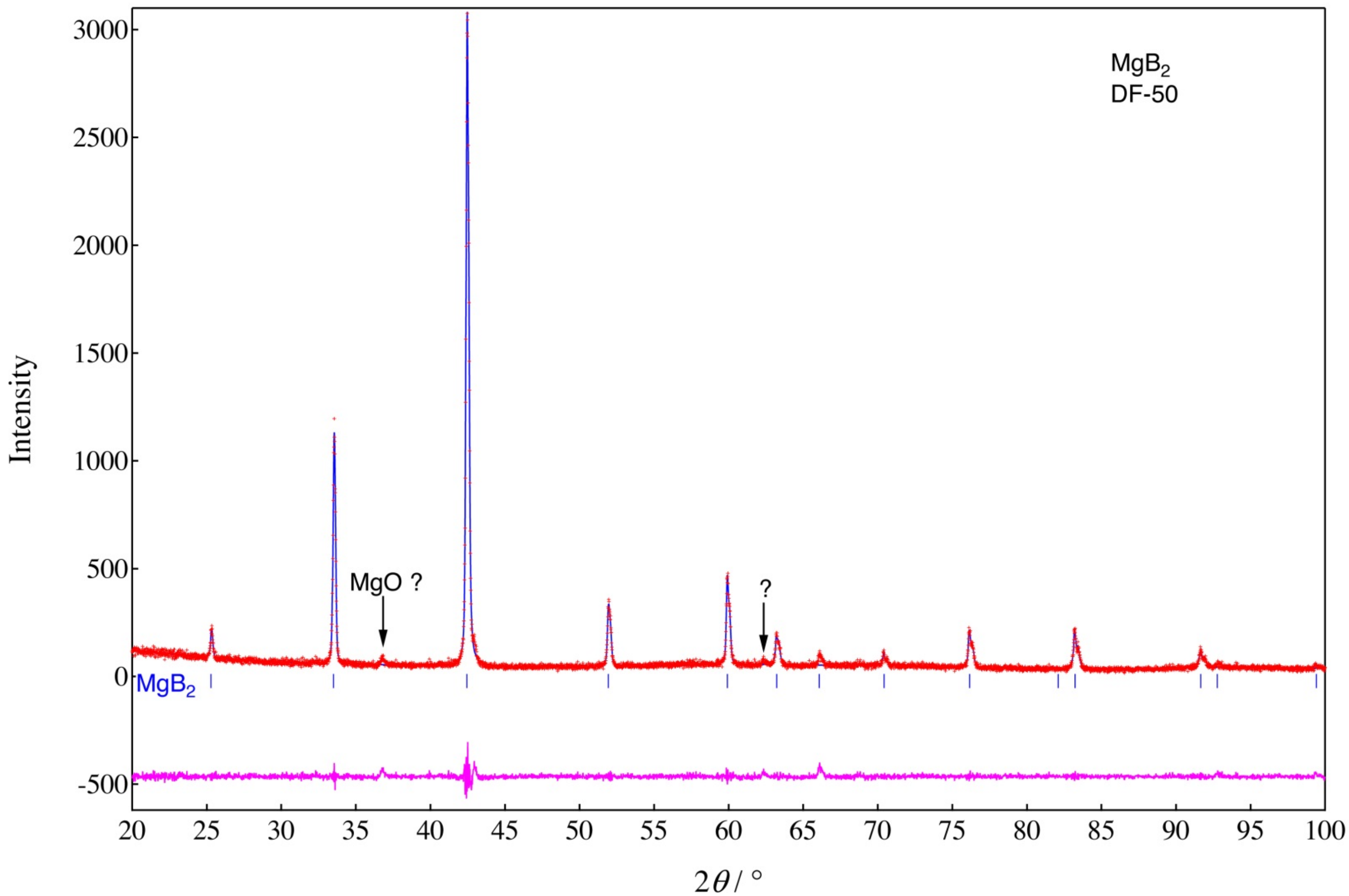
Average particle size for 48 h



Average particle size vs time 830°C



X-ray spectrum for MgB_2 powder



Conclusions

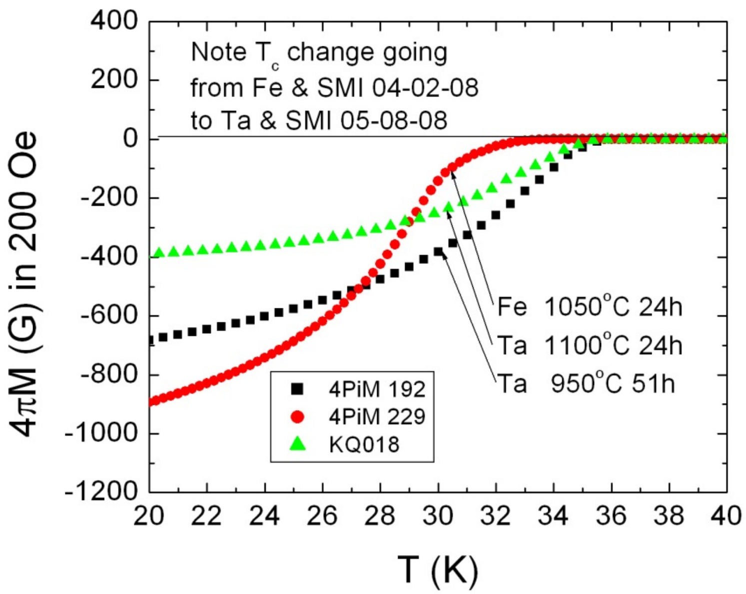
- 1) 830^{o}C for 24h gives particle size under a micron.
- 2) average grain sizes tend to run about ½ the average particle size
- 3) reducing the temperature to 800^{o}C and/or the time to 8h substantially reduces particle and grain size.

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.

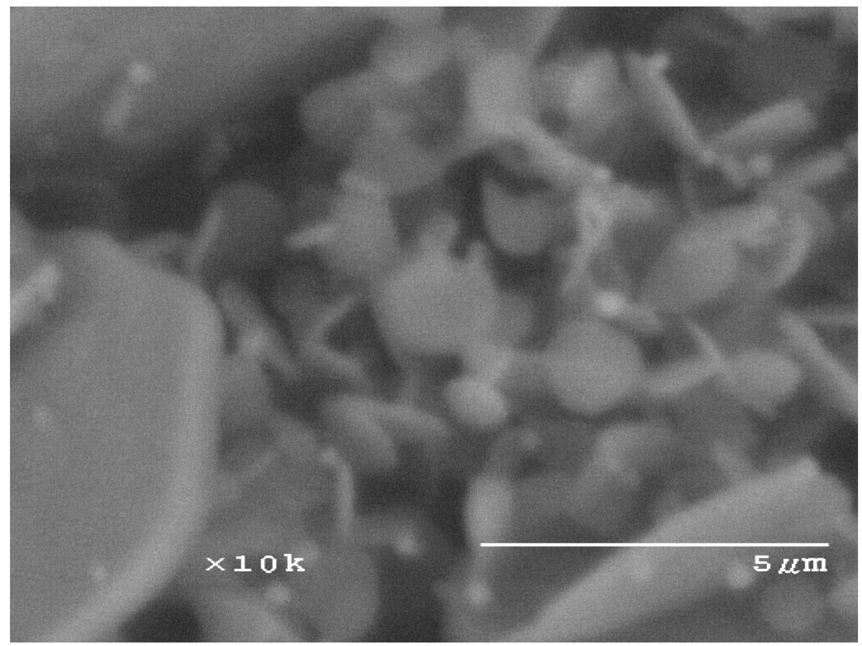
Mistakes that we made. Problem #1 – Fe depresses T_c

- 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 μm *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 ¾ atmospheres of Ar which gives about 2 atmospheres of Ar at 830^{o}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