Hot Isostatic Pressing C-Doped Magnesium Diboride

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Goals:

- Change the superconducting properties of MgB_2 with carbon doping ($Mg(B_{1-x}C_x)_2$)
- Increase carbon doping in MgB_2 by heating at 1500 °C
- Develop method to heat MgB_2 at 1500 °C

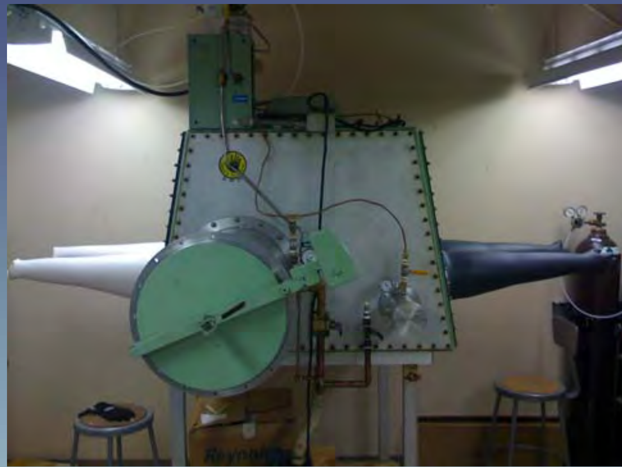
Summary:

- Refurbished glovebox so it removed O_2 and H_20 to handle MgB_2 powder
- Succeeded in heat treating MgB_2 + C at 1500 °C
  - Eliminated high pressure gas leak in Hot Isostatic Press (HIP)
  - Developed method to weld Nb tube
  - Modified method to wrap MgB_2 in Nb foil
  - Altered orientation of sample in the Nb tube
- Graduate student is determining if C content increases at 1500 °C
- Observed MgO nanorods in unsuccessful HIP run

Procedure:

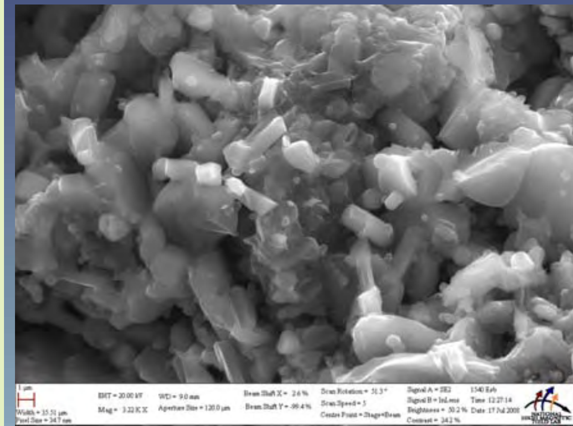
- Prepared MgB_2 + C in powder form by ball milling powders of MgB_2 and C in SPEX Mill inside the glovebox
- Densified the MgB_2 powder into a pellet by applying Cold Isostatic Pressing (CIP)
- Welded the end of the Nb tube to create a capsule for the MgB_2 pellet
- Wrapped MgB_2 pellet with Nb foil and inserted pellet into the Nb capsule
- Welded other end of Nb capsule to hermetically seal MgB_2 pellet in Nb capsule
- Placed Nb capsule with MgB_2 pellet in Hot Isostatic Press (HIP)
- HIPped at 1500 ˚C, 200 MPa (= ~2000 Atm = ~30,000 psi) for 3 hours

Note: Needed to use HIP to keep Mg from evaporating from MgB_2 at elevated temperatures



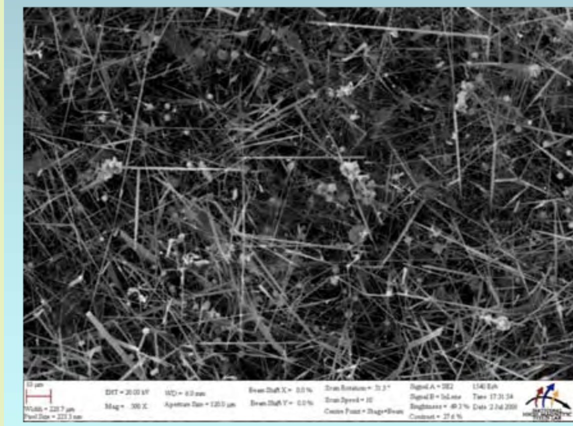
End view of an inert atmosphere glovebox. This glovebox is filled with Ar. The glovebox is designed to remove O_2 and H_2O from the Ar. This allows samples that are sensitive to O_2 and/or H_2O to be handled relatively easily in the glovebox without being affected by O_2 or H_2O. The circular green plate is the end port of the antechamber through which samples are put into or taken out of the glovebox.

View of the hot isostatic press (HIP) unit. The maximum capabilities are ~2500 ˚C and 200 MPa (= ~2000 Atm = ~ 30,000 psi). Ar gas is used to achieve the high pressure. The high temperature is achieved using a C or Mo heating element. This HIP is a cold-wall system, which means the pressure vessel is heated on the inside and the walls are “cold”. A hot-wall system has the heater on the outside of the pressure vessel and the walls are hot.



Scanning electron microscope (SEM) image of an MgB_2 + C sample that was HIPped at 1500 ˚C, 200 MPa for 3 h. The sample started as powder whose grain size was just a few tens of nm. After the 1500 ˚C heat treatment the sample is very dense and hard, and the grains have grown to several µm in size.

Note: The 1 µm length marker is 1/100 the thickness of a piece of copy paper



SEM image of debris on the surface of the Nb tube after a failed HIP run at 1500 ˚C. Energy dispersive spectroscopy (EDS) on the SEM identified the nanorods as MgO. These nanorods formed because the Nb tube cracked during the HIP run allowing Mg gas to escape from the tube. The Mg gas reacted with O in the system forming the MgO nanorods.