

Sintered Boron as High-Strength, Lightweight Structural Material for Aerospace Vehicles¹

HY-Tech Research Corporation



Presented by
C.C. Klepper



at the

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In Collaboration with

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and the

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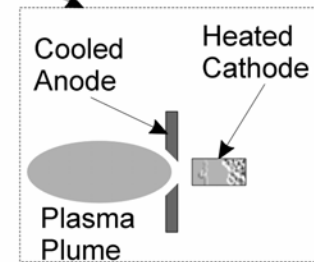
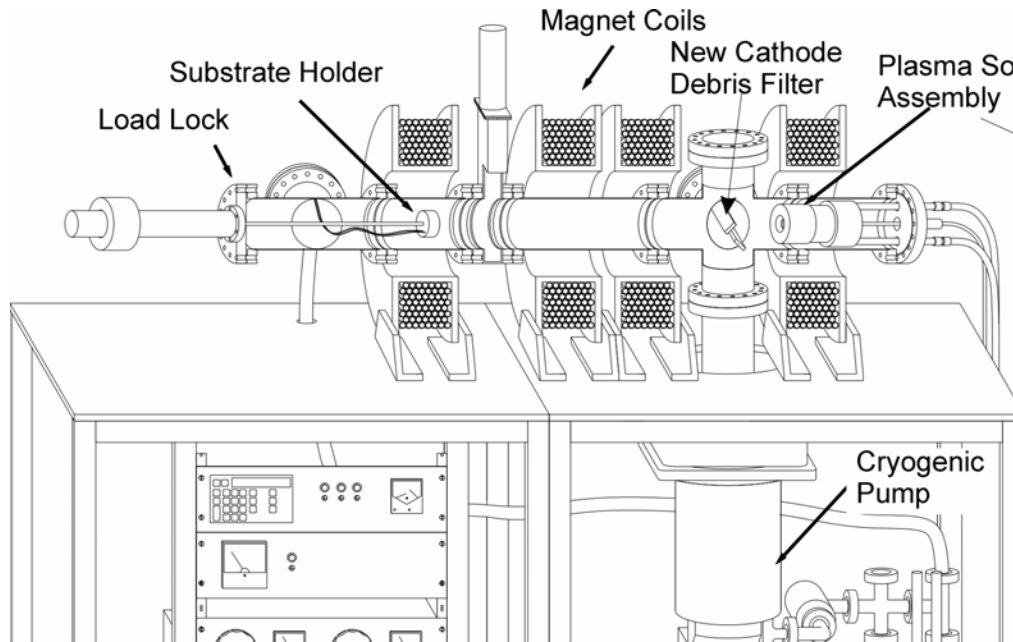
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Introduction

- The low density, high stiffness and low thermal expansion characteristics of B₄C and B make them attractive Be/Be alloy replacement candidates.
- Telescope applications require the parameter $\alpha = (\rho^3/E)^{1/2}$ to be as small as possible.
 - **The value of α for B₄C exceeds that of Be and SiC. For B it is comparable to that of Be:**
 - $\alpha(\text{B}_4\text{C}) / \alpha(\text{Be}) \sim 0.85$
 - $\alpha(\text{B}_4\text{C}) / \alpha(\text{SiC}) \sim 0.68$
 - $\alpha(\text{B}) / \alpha(\text{Be}) \sim 1$
- The thermal expansion coefficients of B and B₄C are less than half that of Be.
- **New powder processing and sintering technologies can economically produce near net shapes.**
- Porous materials can be made without reducing α

Robust Cathodes: The original “market”



Developed for HY-Tech’s vacuum arc-based coater which uses **non-metal cathodes**.

No commercial boron or boron carbide cathode survives the process.

Only specific recipes that include microwave processing have survived the process.

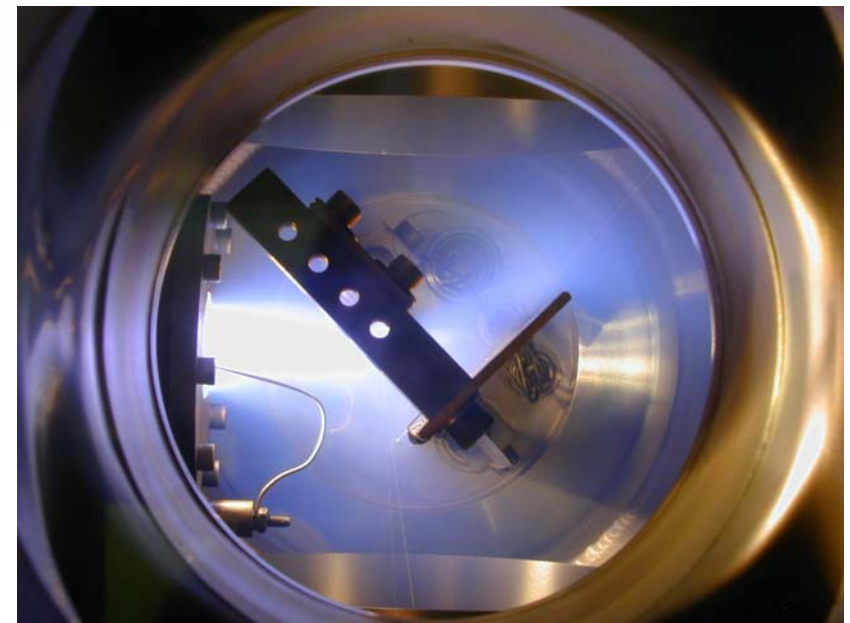


Table 1. Comparison of properties of light materials

Material / Property	Be	Boron (B) -ideal-	B sintered	SiC	B₄C	Graphite ATJ	Pyrolytic normal	Pyrolytic parallel
Density (g/cm³)	1.85	2.46	1.7	3.16	2.50	1.85	1.85	1.85
Melting Point (C)	1287	2076	2000	Sub-limes	2350	-----	-----	-----
Boiling Point (C)	2196	3927	3927	-----	>3200	-----	-----	-----
Heat Conductivity (W/cm-K) at 300K	2.0	0.27	0.19*	0.1 to 4.0	0.23	1.1	0.1	20
Thermal expansion coef. @ 300K (10⁻⁶)	11.3	4.7	4.7	3.4	4.5	3.6	23.1	-0.6
Hardness (H) (GPa)	1.67	33	22.8*	18 to 24	20 to 23	0.3	-----	-----
Tensile strength (GPa) ~1/3 H [9]	0.55	11	7.6	7	7	0.1	-----	-----
Bulk Modulus (GPa)	130	310	214*	410	440	33	-----	-----

- *Estimated (projected values, as contained in the proposal)

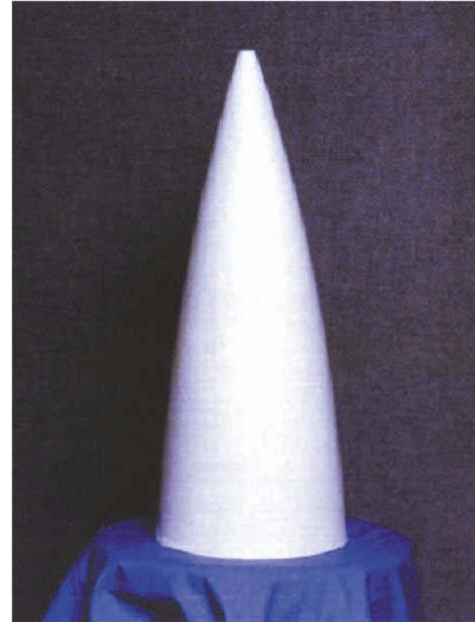
For isotropic materials that do not strain harden much, the tensile strength may be estimated as about 1/3 of the hardness. George E. Dieter, *Mechanical Metallurgy, Second Edition*, McGraw-Hill, New York (1976) page 395.

Versatility & Cost Effectiveness of Demonstrated & Indicated Fabrication Techniques.

- HY-Tech, in collaboration with ORNL scientists, has developed **techniques to fabricate complex components** from these refractory materials.
- These techniques are significantly **less costly** than the state of the art for both ceramic components and Be alloys.
- These include:
 - **Microwave sintering techniques, which are 50-90% more efficient than standard hot pressing or sintering in conventional furnaces.**
 - **Gel casting techniques to form machinable green bodies, which sinter with minimal distortion (~1%) to near net shape components.**
 - **Machining of sintered material and brazing to similar materials to form structures.**

Gel Casting Advantages

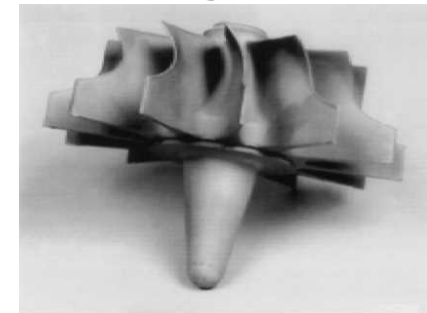
- **Gel casting** allows these ceramic materials to be **engineered for specific applications**.
- **Allows for complex shapes with minimal distortion with sintering**.
- Reduces costs
 - molds can be generally reused
 - green bodies easy to machine (if needed)
 - minimal machining after sintering
- **HY-Tech**: Only company pursuing gel casting of borides for commercial applications.



e.g. SiAlON Radome
(ORNL/Hughes)

M.A. Janney et al. 2004

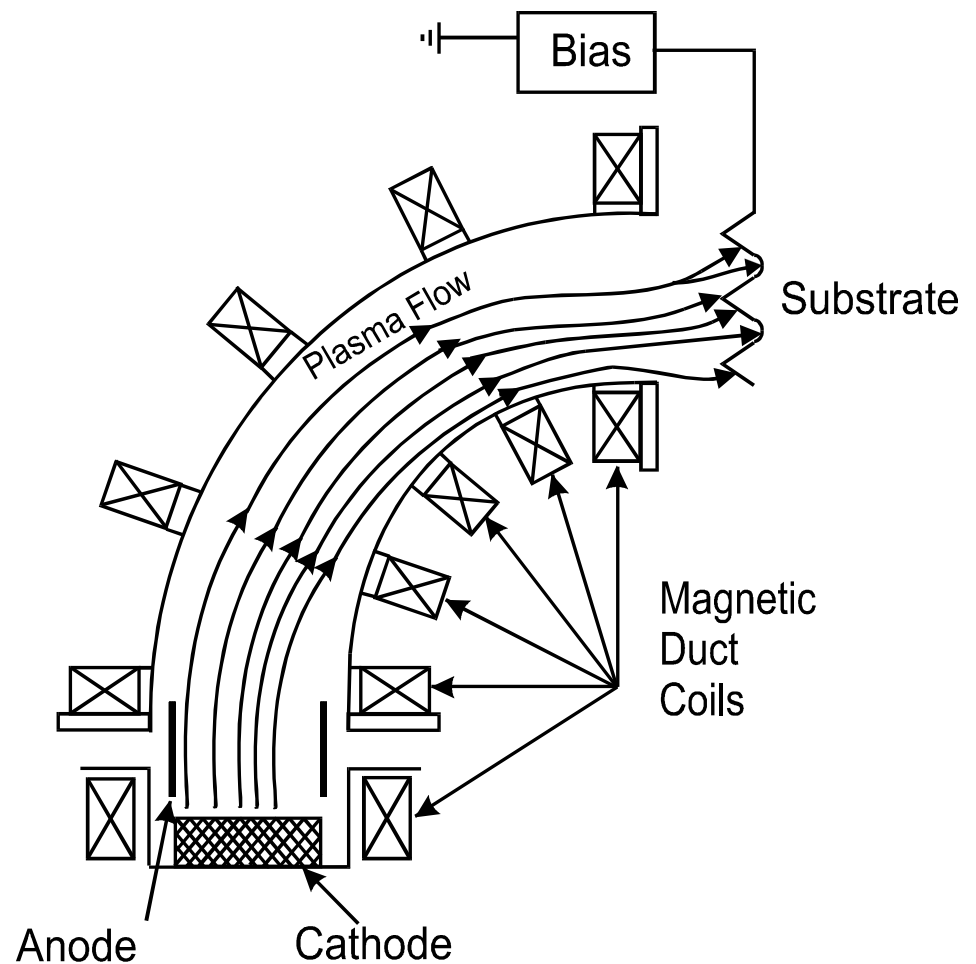
Full reference in
www.ceramicbulletin.org



e.g. gel cast turbine

Coating Capability

- HY-Tech has also demonstrated efficient coating techniques for the deposition of amorphous B and B₄C coatings.
 - **Technique uses vacuum cathodic arc evaporation (from sintered cathodes) to deposit with high deposition rates and great economy of scale.**
 - **For mirror applications, this could be used to coat the sintered components to improve polishing characteristics of mirror surfaces.**



MDA Phase I SBIR Project

- Current MDA/GMD Phase I project is allowing a 1st evaluation of these materials for **structure applications**.
 - The emphasis is on pure B, which has been sintered for the first time to 70% using a patented mm-wave processing technique.
 - Mechanical and thermal properties measured on these materials have been extrapolated to predict values for fully densified materials.
 - No limitation on the ultimate densification achievable has been encountered.
 - Results suggest that B₄C and B could be used for structural telescope members and also mirrors.

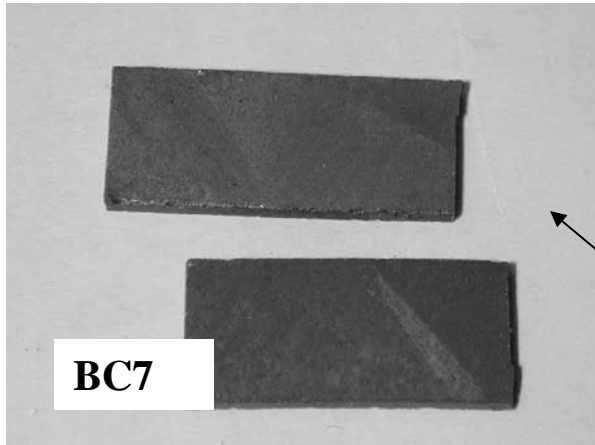
Sample Availability

- The Phase I project relied on the study of existing samples, remaining from the cathode development projects. These included:
 - cold-pressed, then mm-wave sintered B (to 70% TD), which worked well as cathode material.
 - Gel cast B_4C that was sintered to 80% TD with 2.45 GHz microwaves, but did very poorly as cathodes.
- This MDA-funded project provided the first opportunity to carry out a microstructural characterization of these materials and an attempt to determine mechanical properties.
- Most of the processing of the existing samples was carried out by national lab collaborators; this SBIR is allowing HY-Tech to bring the whole technology home.

Metalization Techniques

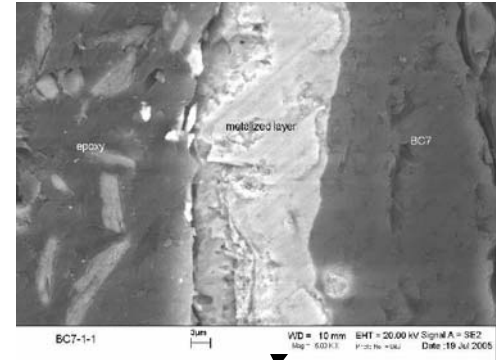
- Surface metalization of sintered B and B₄C are being explored for a number of reasons:
 - Possibility of using in joining with known brazing techniques for metals.
 - To provide high electrical conductivity to surface of components (e.g. to prevent charging).
 - To allow for possible transition to metal reflective surfaces in mirror applications.
- **At least one metalization technique has been demonstrated: Microwave-enhance, chromium diffusion.**
- **Brazing directly to B and B₄C has also been shown.**

Machining, Metalization and Joining



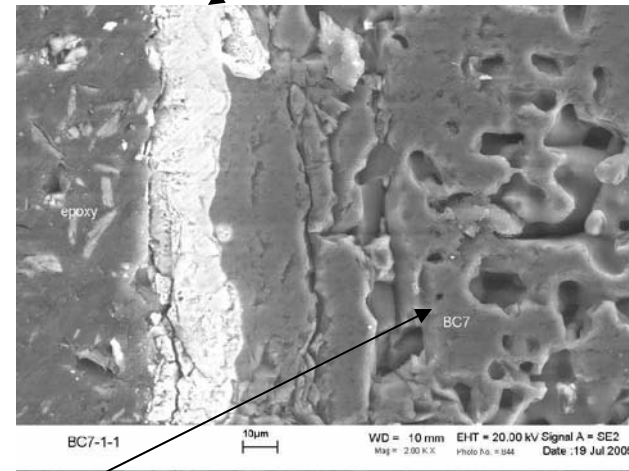
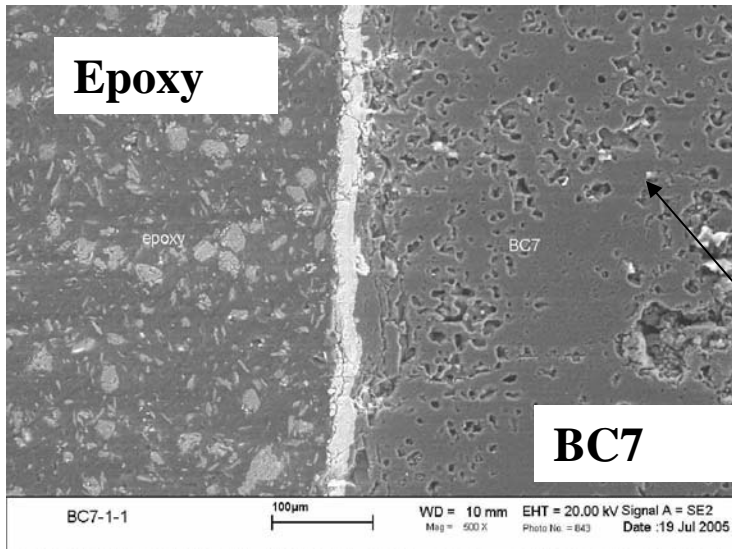
BC7 = B₄C, pressureless casting, followed by microwave-sintering to 80% of theoretical density.

Thin slice cut with diamond saw from 1.6" dia. Disk; then cut again into 2 samples and metalized



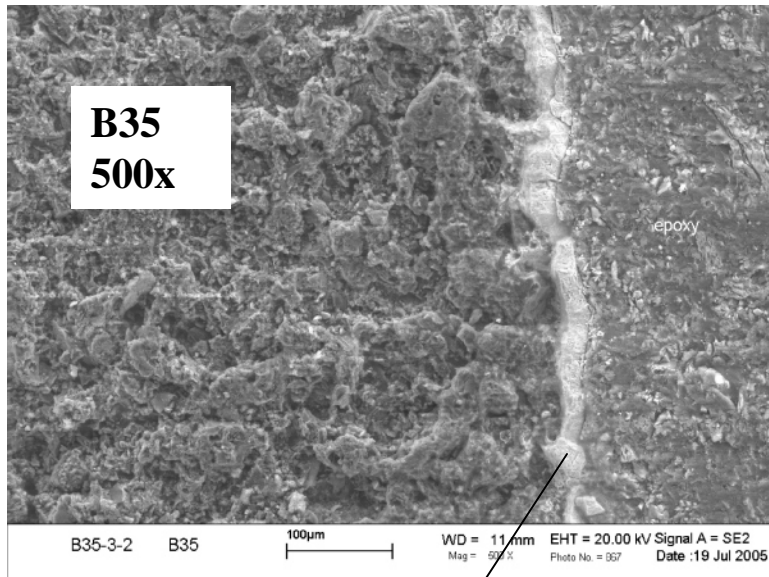
SEM 5k

Metalization; deep (~20micron diffusion layer)

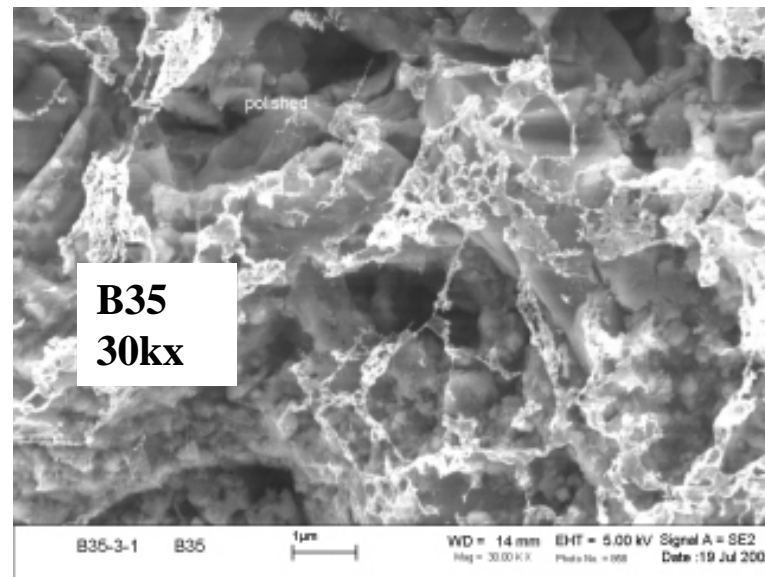


SEM (BC7) center region) 2000x Magnif.

Closed pores; average porosity ~20%

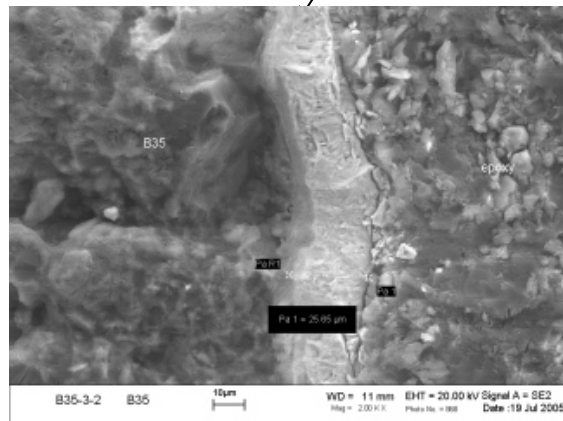


Open-pore microstructure appears to be typical of sintered compacts that do well as arc discharge cathodes (high thermal stress environment).



**B35
2kx**

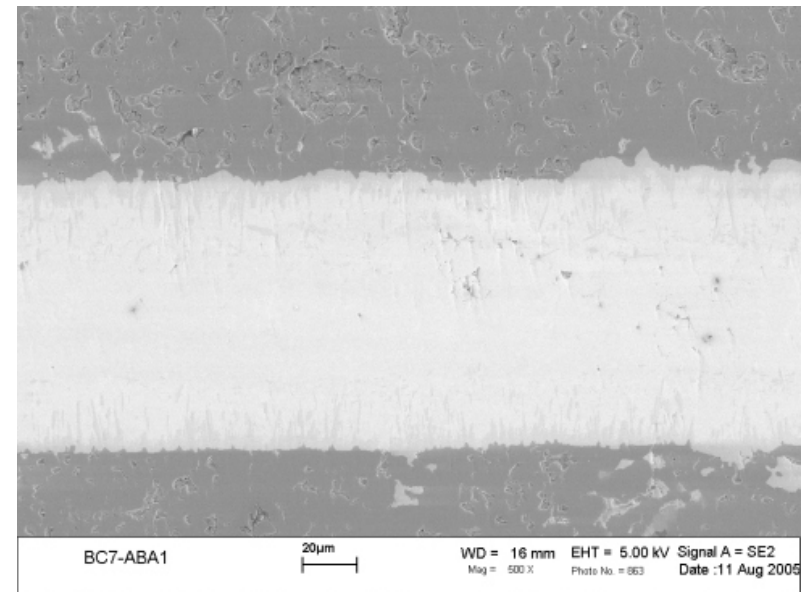
**Metalization
still works**



B35 = Pure B, cold-pressed, mm-wave-sintered to 70% of theor. density

Brazing Results

- It was found that the simplest, most economical approach is to **braze directly to the boron-based ceramics.**
- **At least one Active Brazing Alloy (ABA) was found to work well between similar components.**
- Since most ABAs are Ti-based, they would **allow brazing to Ti-alloys as well.**



SEM of successful braze (500x)

Characterization Results: Indentation Hardness and Modulus

- Both micro- and nano- indentation results for hardness and modulus gave values comparable to fully dense material:
 - e.g. $H \sim 30$ GPa, $E \sim 450$ GPa for the open-cell porous B compacts
- Indentation-based fracture toughness studies were found difficult to do for porous materials, but still being tried for the B_4C samples.
- For the **BULK MODULUS**, Resonant Ultrasound Spectroscopy (**RUS**) is being used.

Characterization Results: Bulk Modulus

- For porous material, the results of micro- and nano-indentation reflect properties of the grains.
- RUS was used to determine the bulk modulus:
 - $E \sim 100$ GPa for the open-cell porous B compacts
 - $E \sim 200$ GPa for the closed-cell porous B_4C compacts
- The value of E for our B_4C samples is consistent with models for the reduction of E as a function of the porosity.
- However, for both B and B_4C , $\alpha = (\rho^3/E)^{1/2}$ is still about equal to that for fully dense material, \rightarrow still ideal for mirror structures.

Thermal Conductivity

- Use **laser flash technique** according to *M.V. Krishnaiah et al., Rev. Sci. Instrum. 73 (9) 3353-3357, 2002* to get **thermal diffusivity** from laser pulse propagation through sample.
- Multiply by density and heat capacity (e.g. for B, $\rho = \rho_o \times .7 = 1640 \text{ Kg m}^{-3}$ and $C = 1300 \text{ J Kg}^{-1}\text{K}^{-1}$) to get **thermal conductivity**
- **Result: $6 \text{ W m}^{-1} \text{ K}^{-1}$ for our B samples**
- **Result: $20 \text{ W m}^{-1} \text{ K}^{-1}$ for our B₄C samples**
 - Compare to $\sim 200 \text{ W m}^{-1} \text{ K}^{-1}$ for Be
 - Compare to $\sim 20 \text{ W m}^{-1} \text{ K}^{-1}$ for steel
 - Compare to $\sim 30 \text{ W m}^{-1} \text{ K}^{-1}$ for solid B₄C
- The result for our sintered B is not unreasonable, given the open pore structure. May be useful as a thermal barrier.
- Customizing this property for both B and B₄C looks quite feasible!

Gel Casting Results

- Early ORNL recipes for B₄C have been successfully reproduced at HY-Tech.
 - **Objects of complex shape have been now produced at HY-Tech.**
- First (ever) successful gel cast B compact has been produced at HY-Tech in the shape of a disk. Work is continuing.
- **The refinement and scale-up of the process will be an important focus of the next Phase.**

Content -- Summary

1. The low density of boron rich ceramics makes them attractive candidates for Be replacement materials; however, their refractory properties make the processing of these materials challenging.
2. **Innovative processing technologies developed by HY-Tech to satisfy its needs for robust cathodes in its coating systems can lead to economic means for processing these materials for structural applications as well.**
3. Current (Phase I) SBIR program, sponsored by the MDA, is allowing a first evaluation of these materials for structures.
4. **Initial results show feasibility for machining of sintered material, brazing to similar material to form structures, as well as the potential for near-net shape casting of green bodies before sintering.**
5. The emphasis has been on pure B, which has already been sintered to 70% MF, using a patented mm-wave processing technique.