Powder processing of high temperature cermets and carbides Marshall Space Flight Center,

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Abstract:

The Materials and Processing Laboratory at NASA Marshall Space Flight Center is developing Powder Metallurgy (PM) processing techniques for high temperature cermet and carbide material consolidation. These new group of materials would be utilized in the nuclear core for Nuclear Thermal Rockets (NTR). Cermet materials offer several advantages for NTR such as retention of fission products and fuels, better thermal shock resistance, hydrogen compatibility, high thermal conductivity, and high strength. Carbide materials offer the highest operating temperatures but are sensitive to thermal stresses and are difficult to process. To support the effort, a new facility has been setup to process refractory metal, ceramic, carbides and depleted uranium-based powders. The facility includes inert atmosphere glove boxes for the handling of reactive powders, a high temperature furnace, and powder processing equipment used for blending, milling, and sieving. The effort is focused on basic research to identify the most promising compositions and processing techniques. Several PM processing methods including Cold and Hot Isostatic Pressing are being evaluated to fabricate samples for characterization and hot hydrogen testing.

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Program's Objectives

- Establish a laboratory at MSFC for processing of refractory cermets and carbides for possible use in NTP systems.
- Procure feedstock necessary for the development of prototypic materials.
- Produce a consistent article in a cost effective, safe and timely fashion.

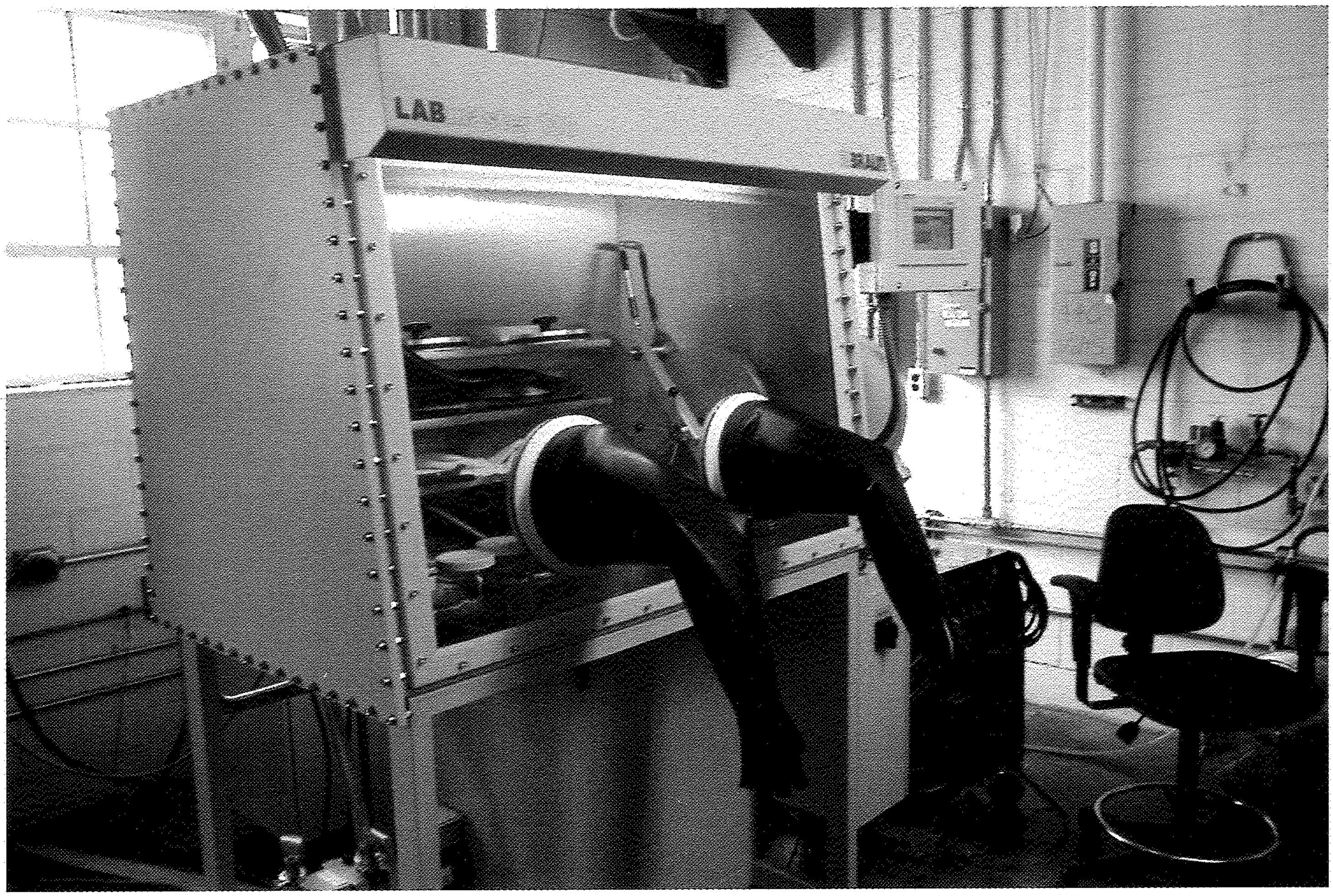
Developmental Approach

- Research lessons learned from previous work of the NERVA, ANL, UF/INSPI, Russia et.al.
- Investigate both conventional and alternate processing norms.
- Characterize compositions, phases, microstructure and compatibility to hot hydrogen.
- Optimize candidate materials and processes with prototypical test articles.
- Form cooperative Partnership with interested government, collegiate and/or corporate agencies.

Establishing the Lab

- Inert gloveboxes for handling and processing of reactive materials
- New inert/vacuum high temp furnace (3000C)
- Powder blending, milling, sieving, pressing
- Metallographic station for materials preparation and analysis of finished product.
- Systems must be adaptable to NRC compliance with the introduction of DU or other alternate materials.

Inert Glovebox



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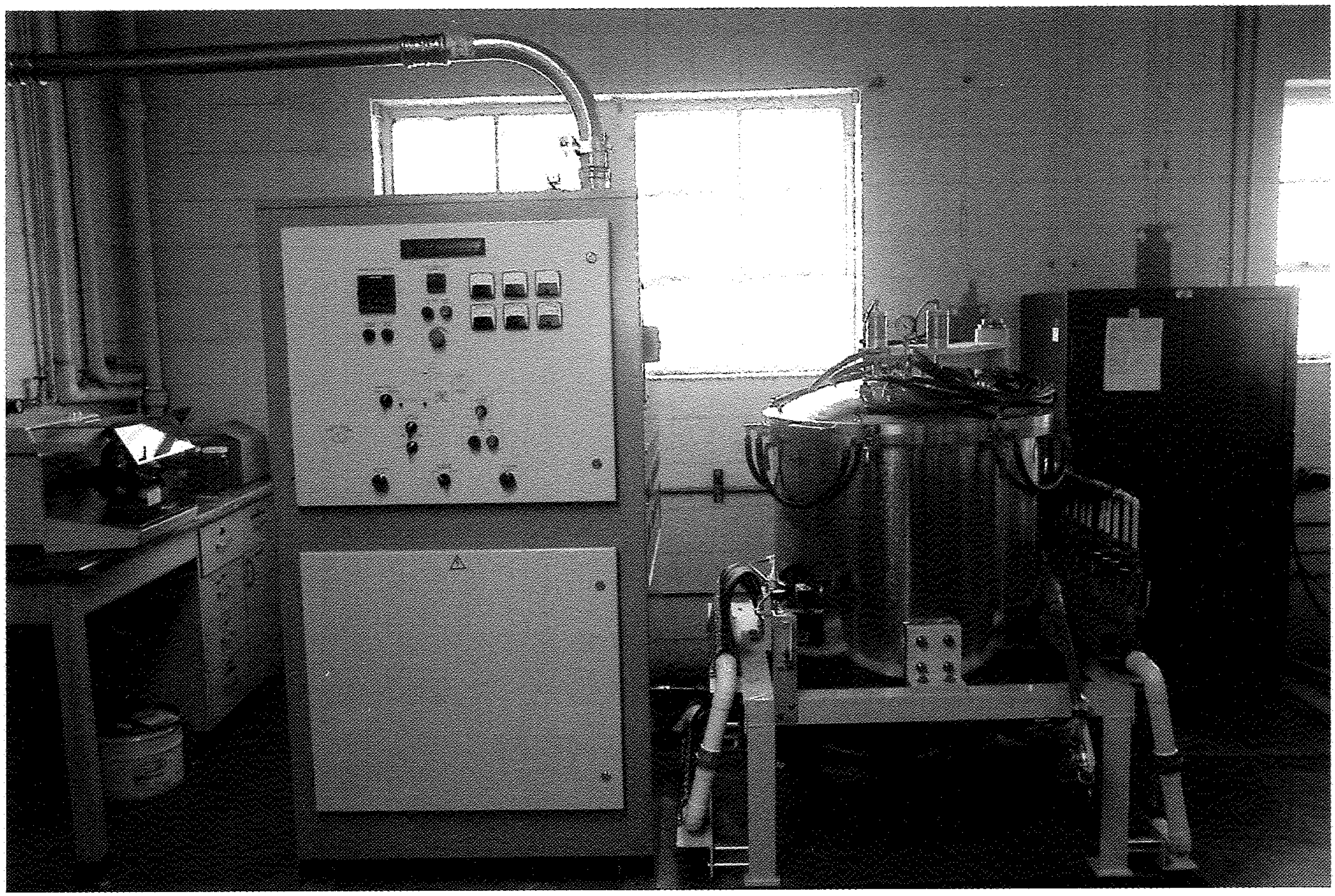
Inert Glovebox Capabilities

- Allows for ultra clean conditions with less than 1 ppm moisture and oxygen in an argon atmosphere.
- Allows for the heated vacuum cycling of samples and the TIG assembly and final closure of canisters.
- Meets or exceeds NRC guidelines for handling of DU powders.

High Temperature Furnace



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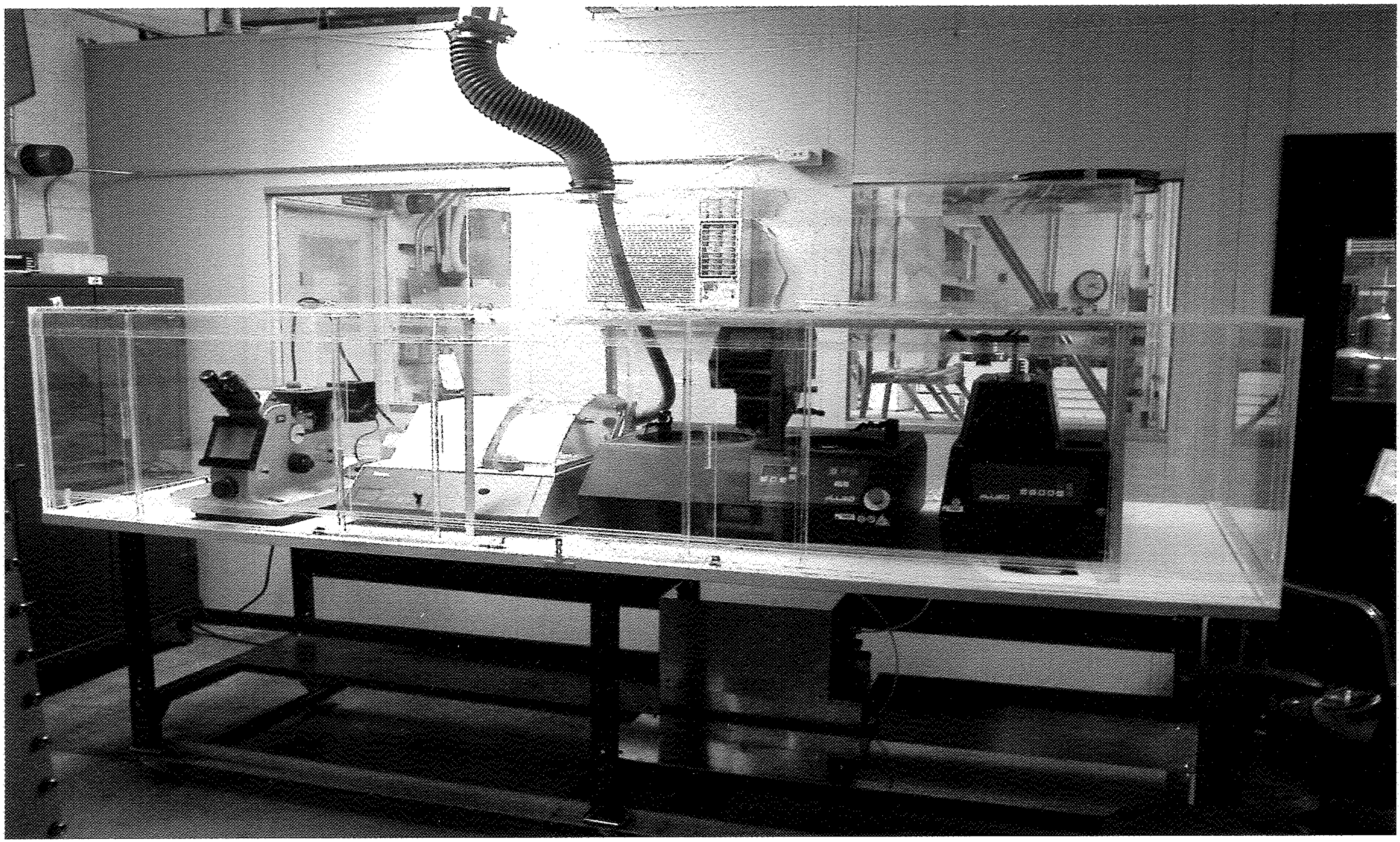
Furnace Capabilities

- Allows for ultra high temperature of ~ 3000 ^{0} C processing in argon with a uniformity of +/- 10^0 C in gas above ^{0} C .
- Computer controlled and logging ability.
- Chamber size of 18" Dia. X 12" allows for multiple or larger samples.
- Temperature controlled by two type "C" thermocouples below 1600^0 C, and with an optical pyrometer at higher ranges.

Sample Analysis Preparation



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Sample Analysis Equipment

- Struers *Secotom -10* cut off saw for sectioning materials.
- Allied *Techpress-2* mounting system.
- Allied *Duelprep-3* sample polishing platform.
- Nikon *EPIPHOT* microscope with a Sony model DXC-S500 color digital camera attached.

Auxiliary Glovebox

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Powder Preparation and Blending

- Labconco glovebox provides an atmosphere of ~ 1% moisture and oxygen for powder storage and sample weighing and filling.
- *Turbula* blender works the powders into a homogeneous matrix with it's three-dimensional motion.

Candidate Materials

Cermets

- W-5Re/40 volume % HfN with -150/+75 micron HfN
- W-5Re/40 volume % HfN with -75/+45 micron HfN
- W-5Re/40 volume % HfN with -45/+25 micron HfN
- W-5Re/40 volume % HfN with -25/+20 micron HfN

Carbides

- ZrC:NbC:Mo2C (60:30:10 ratios in weight) all -325 mesh
- ZrC:TaC:Mo2C (70:20:10 ratios in weight) all - 325 mesh
- ZrC:NbC:W2C (60:30:10 ratios in weight) all in up to 5 micron rang

Candidate Materials, Continued

- Feedstock developed from high purity refractory metal and ceramic powders and purchased from commercial vendors and participating agencies.
- For the cermets, HfN particle size was varied to investigate the affect on particle distribution, clustering, density, and microstructure. Prior to mixing of the cermets, the elemental tungsten and rhenium powders were blended and sintered in hydrogen at 700-800°C to remove surface oxides.
- For the carbides, similar methods were used with ZrC as the base forming the solid solution product.

Fabrication

- The HIP cans fabricated using niobium and tantalum tubing and plate materials.
- The materials were TIG welded in the inert glovebox < 1 ppm O^2 and H^2O.
- All parts were chemically cleaned prior to welding to remove oxides scales and other contaminants.
- After welding, the cans are helium leak checked to $<10^{-9}$ sccm to verify integrity of the welds.

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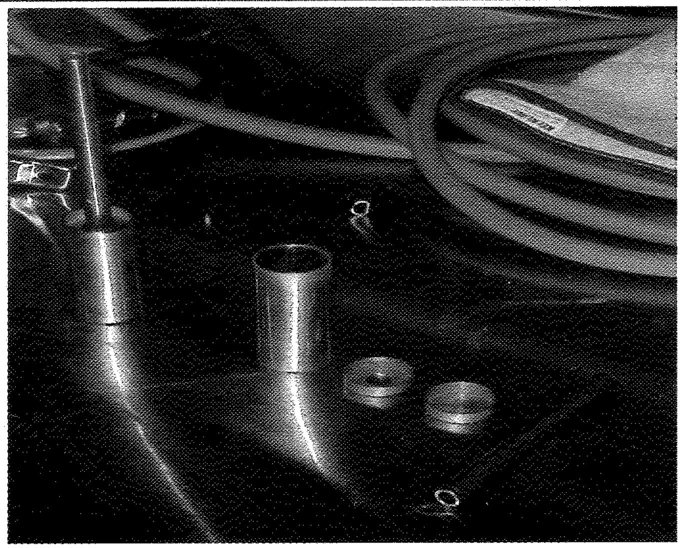
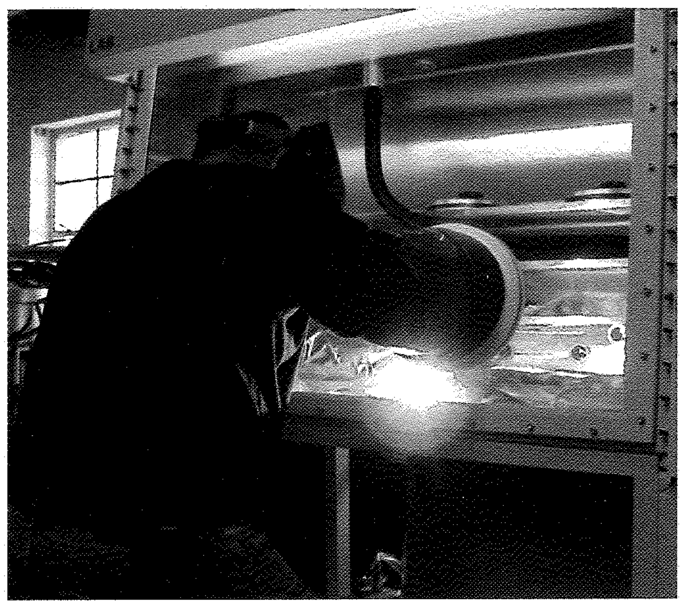
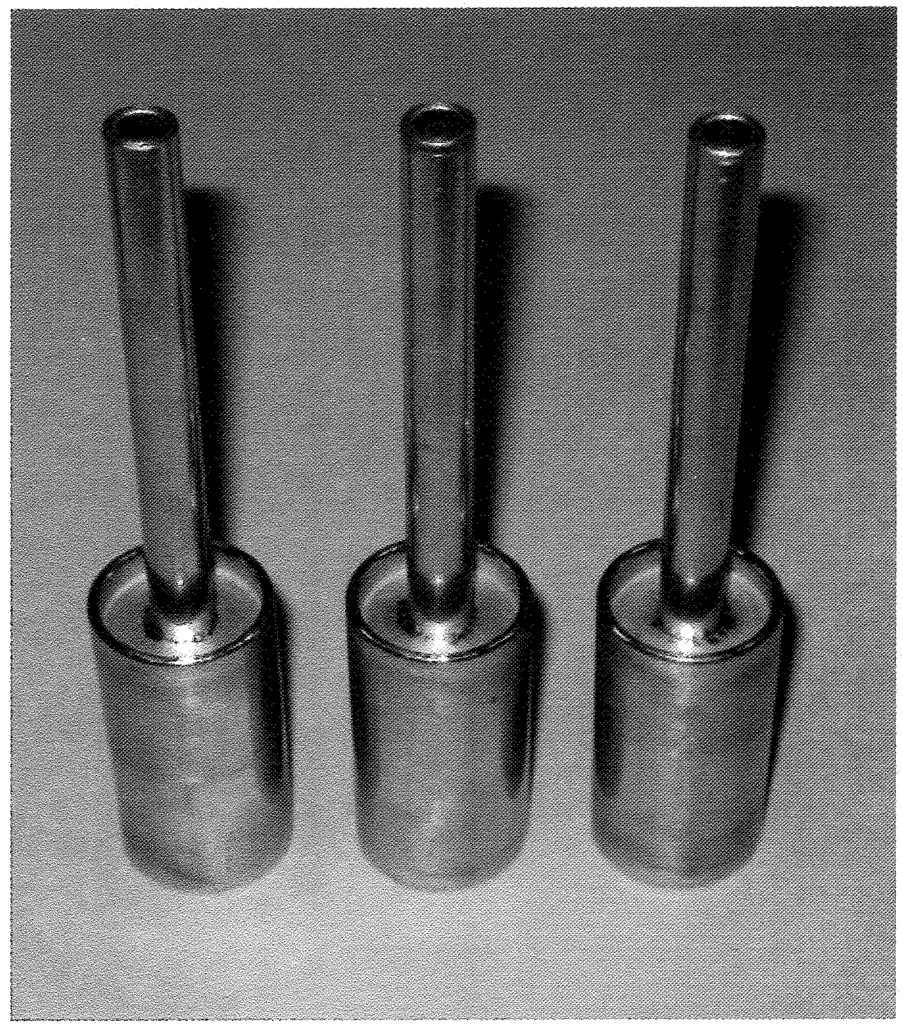
HIP can Assembly







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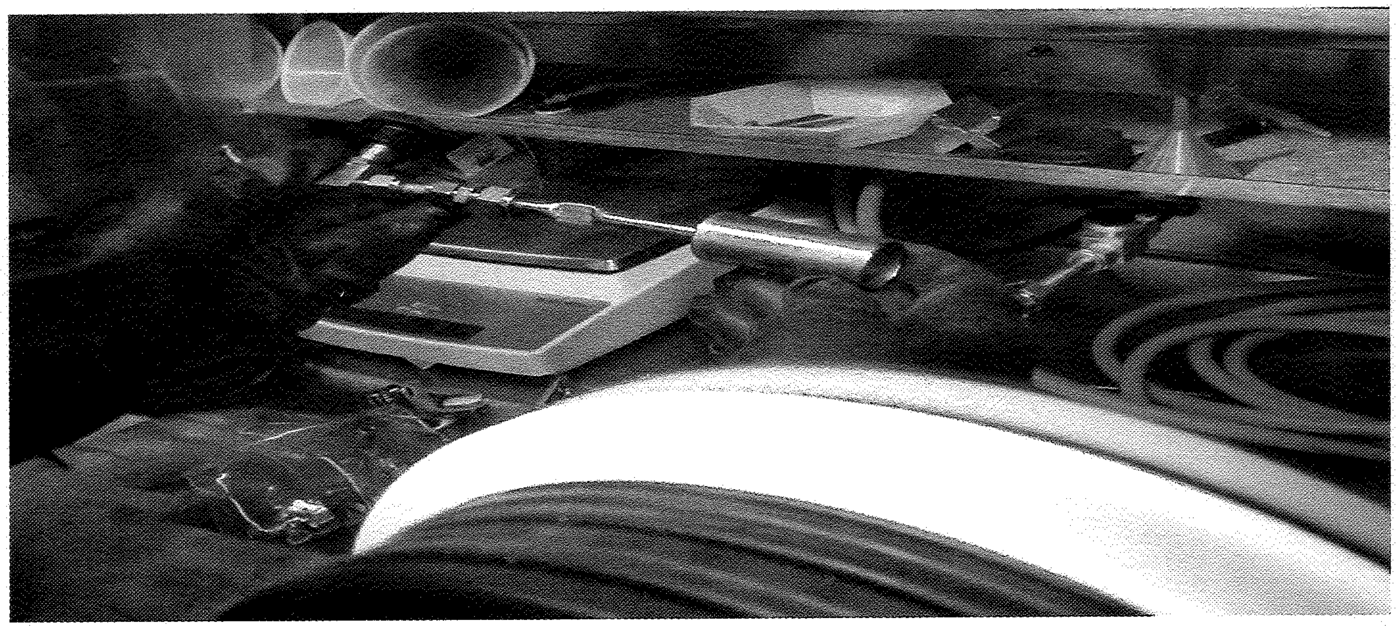
Filling of Cans

- Fill of the HIP can was performed in the glovebox.
- Loading techniques were investigated to a minimum powder packing density of 50% theoretical.
- The packing densities were determined for each powder composition under tapping and vibration conditions to settle the powders.
- Tapping was used during fill due to noticeable segregation of the powders during vibration.
- The feed tube was partially filled to ensure complete fill of the container.

Crimp and Closure

- After filling, the fill tube was partially crimped with a hand operated device to help retain the powder during evacuation.
- Article was attached to vacuum manifold and degassed to a level of ~ 5×10^{-5}, then valved off.



Cutting and Processing

- Valved cans are then pressed to at least 15 tons to fully close fill tube.
- Returned to the glovebox, the can is separated from the valve @ the crimp using TIG and sealed.
- Can is forwarded to commercial HIP processor for thermal cycling.

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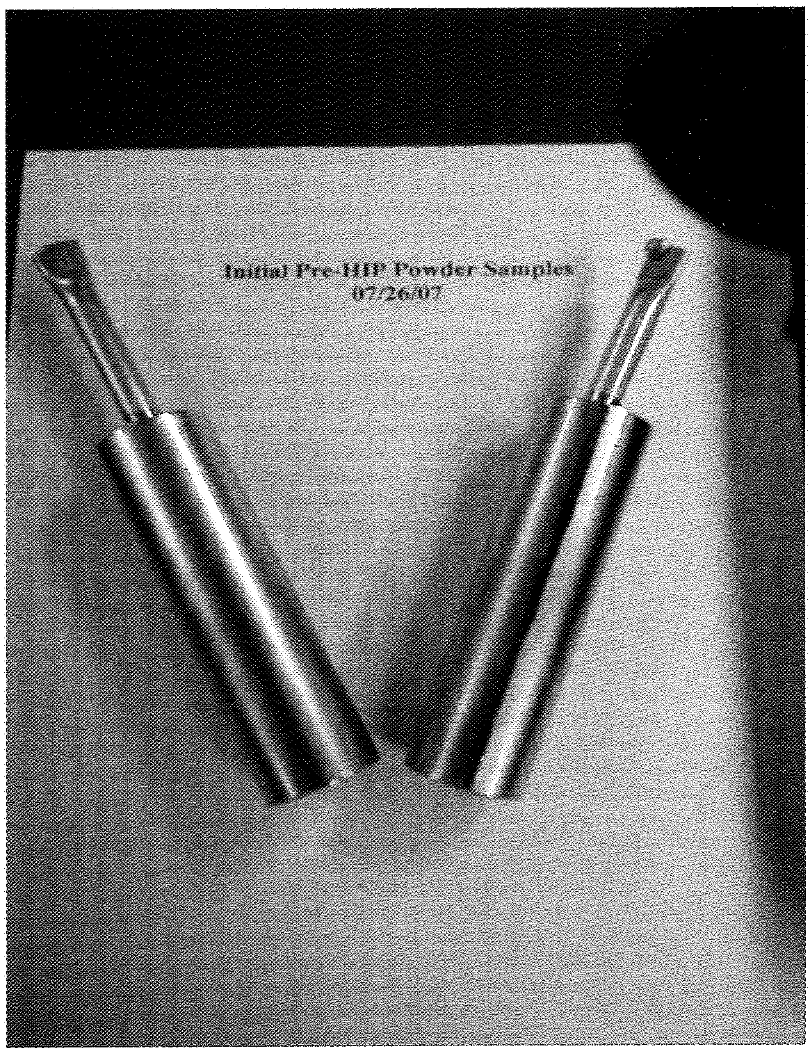
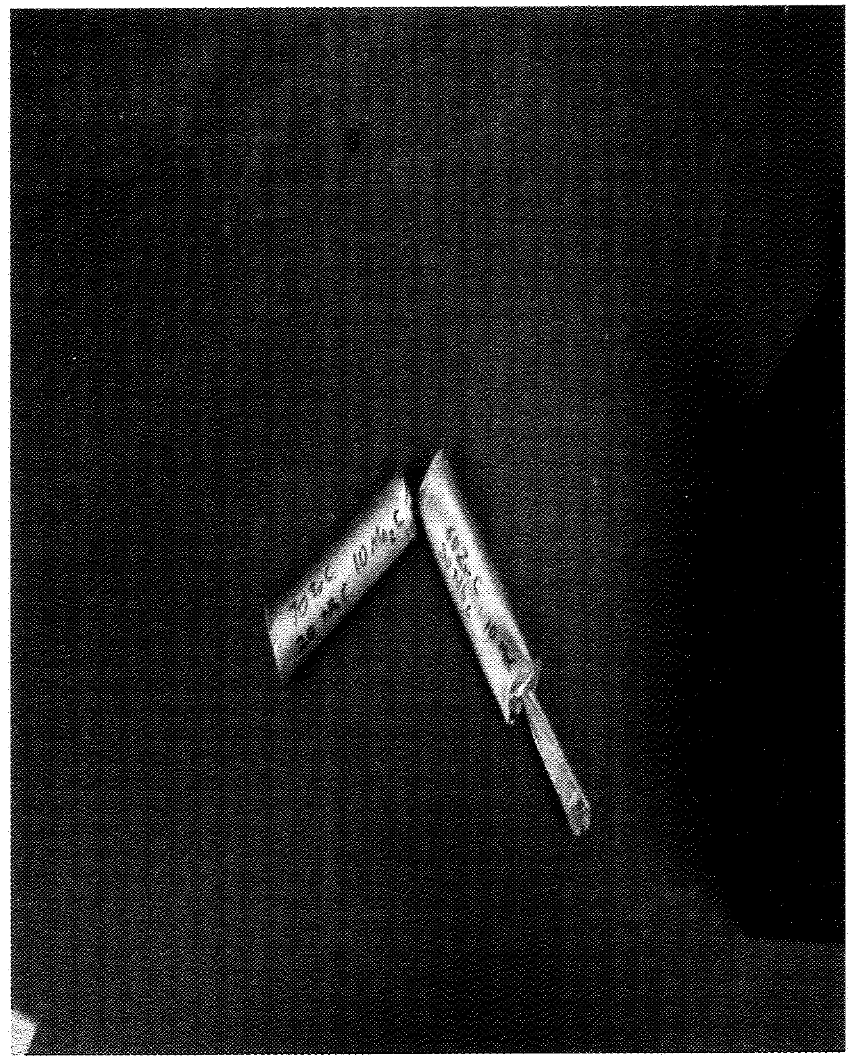
Pre and Post Processed Can





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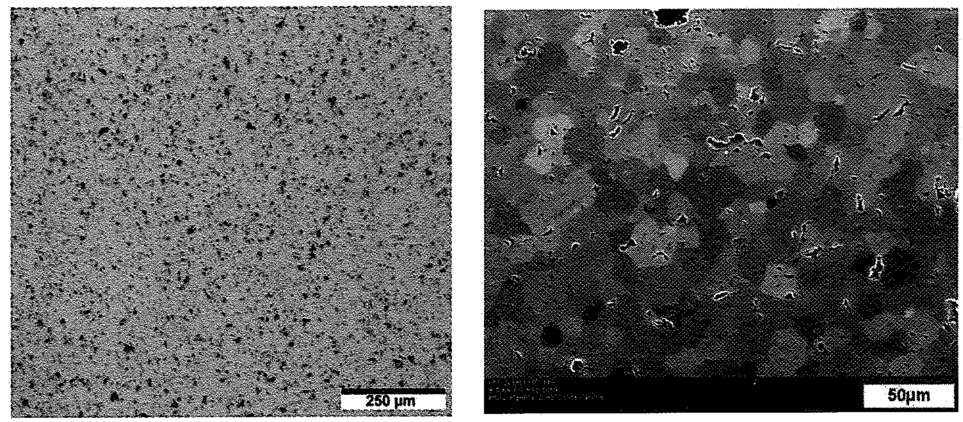
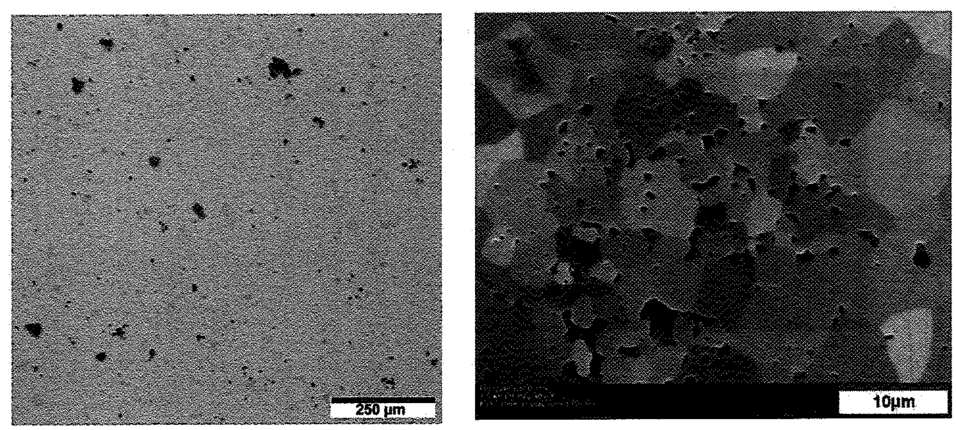
Preliminary Results

- Optical and SEM microscopy carried out along with hardness, chemistry, phases, etc.
- Single carbides showed signs of fine grained and high density.
- Some evidence of free carbon and oxy-carbide phases.



Optical and SEM Micrographs of ZrC



Optical and SEM Micrographs of TaC

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