Impact Testing of Zirconium Diboride

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IMPACT TESTING OF ZIRCONIUM DIBORIDE

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IMPACT TESTING OF ZIRCONIUM DIBORIDE

by

MARK D. FLORES

THESIS

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Department of Mechanical Engineering
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Abstract

Materials research is one of the driving forces that stimulate new ideas and concepts in a wide range of engineering applications. Material research allows engineers and scientists the ability to find new ways to use high performance materials to advance technology in the aerospace industry. In this study, Zirconium Diboride (ZrB2) will be the subject of impact testing at various speeds. The ZrB2 samples will by lying in a crucible and heated up to high temperatures above 1000C waiting for a steel ball bearing to collide into it at high speeds ranging from 115 m/s to 152 m/s. The steel ball bearing is inside of a pressure line and the driving force that propels it will be either Nitrogen or Helium, depending on the desire velocity. Through the experiment, the measurements of the projectile velocity, temperature of the specimen, and the damage length of the specimen are documented for a cold fire test.
# Table of Contents

Abstract ........................................................................................................................................... iv

Table of Contents ............................................................................................................................ v

List of Tables ..................................................................................................................................... vii

List of Figures .................................................................................................................................... viii

Chapter 1: Introduction ..................................................................................................................... 1
  1.1 Project Objectives .................................................................................................................... 1
  1.2 Project Overview ..................................................................................................................... 1
  1.3 Parameters .............................................................................................................................. 2

Chapter 2: Literature Overview ........................................................................................................ 4
  2.1 Ceramics .................................................................................................................................. 4
  2.2 Impact Testing .......................................................................................................................... 4

Chapter 3: Experimental Procedure and Design ............................................................................. 6
  3.1 Materials and Design .............................................................................................................. 6
  3.2 Measurement Methods .......................................................................................................... 10
  3.3 Impact Testing ....................................................................................................................... 18
  3.4 Procedure .............................................................................................................................. 19

Chapter 4: Results and Discussion .................................................................................................. 21
  4.1 Projectile Velocity ................................................................................................................... 21
  4.2 Temperature of the Specimen ............................................................................................... 23
  4.3 Damage Length ..................................................................................................................... 24

Chapter 5: Conclusions and Recommendations .......................................................................... 28
  5.1 Recommendations ................................................................................................................ 28
References........................................................................................................................................29
Appendix A: Damaged Samples........................................................................................................30
Appendix B: Temperatures Profiles ................................................................................................32
Appendix C: Arduino Code ..............................................................................................................33
Vita.....................................................................................................................................................36
List of Tables

Table 2.1: Sample Sintering Process ........................................................................................................9
Table 3.4: Test Matrix with ZrB2 Sample Number ..................................................................................20
Table 4.3-1: Test Matrix of the Experiment ..........................................................................................25
List of Figures

Figure 1.1: Experimental Concept ................................................................. 1
Figure 3.1-1: CAD Model of the Mounting Assembly ..................................... 6
Figure 3.1-2: Mounting Assembly ..................................................................... 7
Figure 3.1-3: CAD Model of the Nozzle and Barrel ......................................... 8
Figure 3.1-4: Steel Ball Bearing and ZrB2 ......................................................... 8
Figure 3.2-1: Hamamatsu Photodiode Arrays ................................................... 10
Figure 3.2-2: Hamamatsu Photodiode Array Assembly ...................................... 11
Figure 3.2-3: Arduino Mega 2560 ..................................................................... 11
Figure 3.2-4: PCI-6132 Data Acquisition Device ............................................. 12
Figure 3.2-6: Experiment Setup with Photodiode Array, Circuit Board, and Microchip ............................................................ 12
Figure 3.2-7: Picture of the Circuit Board ......................................................... 15
Figure 3.2-8: Labview-Analog Data of the Photo-interrupters ........................... 15
Figure 3.2-9: RDO Induction Heating Furnace and a Five Turn Heating Coil ....... 16
Figure 3.2-10: Type K Thermocouple Place Next to Sample ........................... 17
Figure 3.2-11: NI-USB-9263 10V Analog Output Module ............................... 17
Figure 3.2-12: Omega Pyrometer .................................................................... 18
Figure 3.3-1: Test Schematic .......................................................................... 19
Figure 3.3-2: Test Setup ................................................................................... 19
Figure 4.1-1: Velocity Measurements for Helium and Nitrogen ...................... 21
Figure 4.1-2: Experimental Setup of Change in Velocity ................................. 22
Figure 4.1-3: Change in Velocity ..................................................................... 22
Figure 4.2-1: Temperature Measurement of ZrB2 (792), Impact Speed 152 m/s .................................................................................. 23
Figure 4.3-1: a) 792 Sample at 615 C b) 792 Sample at 1000 C ....................... 24
Chapter 1: Introduction

1.1 Project Objectives

The main objective of this experiment was to build an impact apparatus, which can hold ceramics at high temperatures. In order to achieve this objective several goals were set to meet the experiment. The current apparatus was improved to measure the velocities more accurately. A nozzle was created so that a 1/16” steel ball bearing could impact the specimen lying in a crucible. The nozzle fires the projectile using either Nitrogen or Helium at room temperature.

A 1/8” OD stainless steel tube or barrel is connected to an aluminum square nozzle with 1/8” ID hole drilled into it. The nozzle will be held vertically using an assembly mount. The line will be connected to the nozzle and the pressurized tank. The tank is filled with Nitrogen or Helium depending on the desired velocity. Figure 1.1 displays the experimental setup. The projectile will be placed along the line and once the push button valve is pressed the fluid stream will push the projectile toward direct collision of the ZrB2 sample. All the experiments and tests were performed at the Combustion and Propulsion Research Laboratory at the University of Texas at El Paso.

![Experimental Concept](image)

Figure 1.1: Experimental Concept

1.2 Project Overview

The purpose of impact testing is to measure the toughness or energy absorption capability of materials. Understanding how a material fails under certain parameters can help engineers and scientist
improve designs of an application the material is being used for. Impact testing is used in several arenas such as the aerospace industry, automobile industry, and even law enforcement.

Impact testing requires several parameters to understand the energy absorption capability of the material. The specimen parameter is the subject of our experiment. The velocity parameter is used to find the maximum kinematic energy the specimen can absorb through collision without mechanical failure. The temperature parameter is used to understand how ductile the specimen is. The projectile that will be colliding with the specimen will be in the same area respectively.

1.3 Parameters

The impact apparatus will be designed to mount sensors and a nozzle for a cold fire test. The rigid assembly is designed to mount a nozzle and sensors that will allow lateral and translational adjustments to properly aim the barrel towards the specimen. The assembly will hold plates to measure the velocity of the projectile. Fasteners will hold the plates and mounts in their respective place.

1.3.1 Specimen Parameter

ZrB2 is the specimen that will be tested. ZrB2 is a high temperature material that is being explored for the use of aerospace applications. An aerospace applications it’s being considered for is hypersonic jets. More information about the ZrB2 will be supplied in later sections.

1.3.2 Velocity Parameter

The velocity parameter is based on the mass, size, and velocity of the projectile. The mass and size of the projectile will remain constant during the experiment. The projectile’s velocity will be measured prior to the impact testing. Using different types of gases to propel the projectile will yield different velocities. The desired velocities required to cause damage to the specimen will vary 100 m/s to 200 m/s. The particle size and shape are stainless steel ball bearings with a diameter of 1/16 inch (1.5875 mm) and an average mass of 0.0168 grams. The velocity parameter gives us insight on how much energy the specimen can absorb. The kinetic energy is defined below.

\[ K.E. = \frac{1}{2}mv^2 \]
1.3.3 Temperature Parameter

The specimen will be heated up to various temperatures using an electric induction furnace. Operating temperatures play a crucial role in engineering applications from aerospace to nuclear reactors. Increasing the temperature makes certain ceramics and composites more ductile, which result in either higher or lower energy absorption impacts. Temperatures will be varied to understand the properties on the monolithic ZrB2 samples and the composite ZrB2-B4C.

Even though this paper does not study how corrosion affects materials, high temperatures contribute to oxidation, carbide, and other forms of corrosion. Since the samples are being exposed to open air and heating up to high temperatures, several corroding factors may contribute to the material’s performance.
Chapter 2: Literature Overview

2.1 Ceramics

High temperature materials are primarily used in applications whose temperatures exceed 1000°C. The attraction with high temperature ceramics is its relatively low density, high modulus of elasticity, and high melting points. One benefit of advanced ceramics is their capability of maintaining their mechanical properties at high temperatures, which makes them ideal for turbines, propulsion systems, and aircraft. Since advanced ceramics consist of oxides, borides, carbides, and nitride-based systems, they can be developed to be corrosion resistant; depending on the environment they are exposed to.\textsuperscript{11}

Ceramics can be defined as solid compounds that are formed by the application of heat, and sometimes heat and pressure, comprising at least two elements provided one of them is nonmetallic or a nonmetallic element solid. The other element(s) may be metal(s) or another nonmetallic elemental solid(s).\textsuperscript{1} Zirconium, a strong transition metal, will be bonded to a nonmetallic element solid boride, an inert chemical compound with a high melting temperature.

Refractory metal carbide and borides capable of surviving thermal excursions up to 2000°C-2500°C for short times with little material recession, making them a strong candidate (in either monolithic form or as a composite matrix) for the sharp leading edges of hypersonic vehicles.\textsuperscript{12} ZrB\textsubscript{2} mechanical properties such as high melting point, high elastic modulus, high electrical and thermal conductivity, shock and wear resistance make it an essential material candidate for aerospace applications such as hypersonic flight or rocket propulsion systems\textsuperscript{4}. ZrB\textsubscript{2} inherent resistance to high temperature environments and corrosive elements makes this emerging ceramic composite the subject of impact study. One of ZrB\textsubscript{2}’s characteristic traits is that it has a superb thermal shock resistance\textsuperscript{4}.

2.2 Impact Testing

The purpose of impact testing is to measure the toughness or energy absorption capability of materials. The major factors that affect the results of an impact test are specimen, velocity, and temperature. Specimens can have different material properties and it is vital to understand the failure mechanics involved when it comes to cracking or other forms of failure. When a projectile’s velocity increases certain critical values or at hypersonic speeds the impact resistance of the specimen appears to
change respectively. Temperature provides understanding of the strength and ductility of the material. Depending on the temperature that the specimen is exposed to can have an effect of whether or not it will be a high or low energy absorption collision. Understanding how a material fails or acts under certain conditions can help engineers and scientists improve designs of an application the specimen is being used for.

2.2.1 Charpy Test

One of the most common industrial standards for impact testing is the Charpy impact test. The Charpy impact test consists of a rigid specimen holder and swinging pendulum hammer for striking the impact blow to a v-notch specimen. The test is useful in understanding the dynamic response of the materials. The reason for its use is to determine the energy required to initiate crack and also the energy to propagate rather than the total energy for facture. The material is subjected to a high strain rate and triaxial state of stress due to the notch, which promotes failure. This is why it is a preferred industrial tool to measure the material toughness.

2.2.2 Drop Dart Test

The drop dart test is another standard low velocity impact test. The impact behavior of the specimen is determined by the saturation impact energy and the damage degree of the specimen. The machine elevates a fixed mass impact object to a specific height to obtain a low velocity without the use of any energy storage devices. The object is then released to collide with the specimen. Using kinematic analysis of the free falling object, the toughness can be analyzed from an energy viewpoint.

2.2.3 High Velocity Impact

High velocity impacts will be used to determine the toughness of the material using a projectile propelled at high velocities. The high velocity impact test will use the kinetic energy from the projectile to determine the amount of energy to initiate indentation or fracture of the specimen. The temperature will be increased to assess how much energy the specimen can absorb.
3.1 Materials and Design

The impact apparatus utilized a nozzle design for cold fire testing, compatible with Nitrogren or Helium gas as the fluid. Multiple measurements were taken to understand the pressure of the fluid, velocity of the projectile, and temperature of the specimen. The impact apparatus consisted of a nozzle and mounting assembly. The mounting assembly consists of four 1/2” rods threaded, several plates, 1” angles, and several nuts to keep the plates in place. One of the plates was used to anchor the nozzle so that it would remain stationary. The other plates were used to locate the sensors. Figure 3.1-1 and Figure 3.1-2 displays the impact apparatus.

Figure 3.1-1: CAD Model of the Mounting Assembly
3.1.1 Cold Fire Test Nozzle

The nozzle was made of aluminum and is a simple design as represented in Figure 3.1-3. The nozzle was constructed so that it could be connected to the mounting assembly. The particle was put along the pressure line for easier access. The nozzle was a 1” x 1” x 2.5” square block where a 1/8” diameter hole was drilled into it. A 1/18” OD stainless steel tube was put on the opposite end of the pressure line.
3.1.2 Stainless Steel Ball Bearings

A 1/16” (1.588 mm) stainless steel ball bearing was used to impact the specimen. Stainless steel was used for its kinematic properties. The temperature of the stainless steel ball bearings throughout the experiment was room temperature. The average mass of the stainless steel ball bearing was 0.0168 grams. Figure 3.1-4 shows the size of the ball bearing with respect to one of the samples that was tested. The size of the samples was 0.5 cm by 0.5 cm x 0.3 cm.

Figure 3.1-4: Steel Ball Bearing and ZrB2
3.1.3 ZrB2 Specimen

The specimen used in this experiment was ZrB2 as shown in Figure 3.1-4. ZrB2 is well known for its hardness and resistance to thermal loadings. Since ZrB2 is electrically conductive it was heated using induction coils. The size of the specimen varied depending on the ceramic.

In order to produce ZrB2 requires advance methods in the sintering process. An electric arc sintering process assisted in the heating of the pure ZrB2 powder up to 1900 C and depending on the sample was held from 9 to 15 minutes. Sample 796 added 2-wt% B4C to the ZrB2 making it a ceramic composite. The composite ZrB2-B4C is a composite that was also tested to understand the hardness and impact energy. The table below shows the sintering process of the specimens that were tested. Samples 792 and 793 used a 40 mm die during the sintering process. Sample 796 was slightly larger at 55 mm.

Table 2.1: Sample Sintering Process

<table>
<thead>
<tr>
<th>Samples</th>
<th>Composition</th>
<th>Temp. (C)</th>
<th>Time (min)</th>
<th>Diameter (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>792</td>
<td>ZrB2</td>
<td>1990</td>
<td>9</td>
<td>40</td>
</tr>
<tr>
<td>793</td>
<td>ZrB2</td>
<td>1700</td>
<td>9</td>
<td>40</td>
</tr>
<tr>
<td>796</td>
<td>ZrB2-B4C</td>
<td>1900</td>
<td>15</td>
<td>55</td>
</tr>
</tbody>
</table>

Since the specimen was bigger that the crucible, the specimens were cut into smaller samples using a tough SiC wafer blade. The resultant dimensions of the samples varied but were within 3 mm x 4 mm (+- 1 mm). Since some of the specimens (793 and 796) cracked or fractured during the cutting process, only a few samples were left to test. The remaining samples were polished. The specimens went through an ultra sonic bath due to the exposure to oils and water during the cutting and sanding process.

To reduce any human handling of the high temperature ceramics, the ceramics were placed in either an alumina crucible or mullite crucible depending on the nature of the test. For the specimens that were tested under 1000C an alumina crucible was used. For temepratures greater than 1000C, a mullite crucible was used. During one of the experiments the sample burned through the alumina crucible and had to change to a mullite crucible.
3.2 Measurement Methods

3.2.1 Particle Velocity Measurements

Originally a LED and photodiode array assembly was designed to measure the velocity closest to the induction coil. Two 16-element Si photodiode arrays from Hamamatsu (Figure 3.2-1) and 10 LEDs would detect the projectile as in the CAD model Figure 3.2-2. The base that held both the LEDs and sensors were 6” x 6” x 1”. Therefore, the two photodiode arrays were one inch away from each other and the velocity could be determined by the distance between arrays and the time difference between trips. The process of designing a circuit board that would allow one sensor control all sensors proved to be challenging. However, the circuit board was replaced with a more user-friendly Arduino Mega 2560 microchip. Using Arduino software, a program was built such that the each sensor could be monitor simultaneously. Once the low and high operating voltages were determined for each diode, the code was developed such that any sensor trip would signal its respective photodiode array. Once the microchip was programmed, the output data was connected to Labview 2009 Signal Express Viewer (PCI-6132 DAQ) to measure the photodiode trip times.

Figure 3.2-1: Hamamatsu Photodiode Arrays
Figure 3.2-2: Hammamatsu Photodiode Array Assembly

Figure 3.2-3: Arduino Mega 2560
Figure 3.2-4: PCI-6132 Data Acquisition Device

Figure 3.2-5: Sensor Schematic

Figure 3.2-6: Experiment Setup with Photodiode Array, Circuit Board, and Microchip
One of the problems with the original set up was that the projectile still did not completely obscure the diode therefore finding any voltage drop was difficult. Also the light emitted from narrowly spaced LEDs could have impaired the sensors from tripping. To test this was the problem, the distance between the photodiode assembly and the LED assembly was decreased. The microchip was reprogrammed to account for the intensity of the light the LED was producing and photodiode outputting. The voltage drop was insufficient to trip the sensor. The photodiode was able to detect the projectile when the 1/16” (1.588) sphere was replaced with a 3/32” (2.381 mm). Figure 3.2-6 is a graphical display showing what was occurring. The cone of light emitted from the adjacent LEDs could have caused interference with the diodes capability to detect projectiles. During verification, a simple drop test was failed. Therefore this method was abandoned.

Figure 3.2-6: Experiment Setup with Photodiode Array, Circuit Board, and Microchip

The new method would use the photo-interrupters to determine the velocity. Two holes were drilled into the barrel where a photo-interrupter could be placed. The vertical distance between the photo-interrupters was 8 inches. The Arduino Mega 2560 used as a power regulator to produce 5 Volts needed for the photo-interrupter. Using a PCI-6132 data acquisition device for Labview 2009 Signal Express Viewer, voltage drops from the tripped sensors could be detected. One of the initial problems was that the data acquisition device wasn’t fast enough in collecting the data. PCI-6132 had 8 analog/digital simultaneous channels that could collect data at a sampling rate of 2 MHz. Originally a PCI-6220 data acquisition device was used that could only collect data at a sampling rate of 250 kHz.
The Labview code had a trigger for each photo-interrupter. Once one trigger was tripped Labview began recording until the second sensor was tripped. The difference in time was calculated using a playback feature. Figure 3.2-6 shows how the velocities were obtained. Figure 3.2-7 is the circuit board that was used to wire the photo-interrupters. Figure 3.2-8 is a graph of the analog data collected of both photo-interrupters. The velocity is thus the difference in distance divided by the difference in time.

\[ V = \frac{d}{t} \]

Figure 3.2-6: Experimental Setup with Photo-interrupters
3.2.2 Heating of Specimen

An induction heating furnace was used to heat up the ZrB2 specimens. Figure 3.2-9 is a picture of the 3 kW RDO induction heating furnace. The crucible will be placed inside the coil. The specimen will be placed inside the crucible to be heated. Since ZrB2 is electrically conductive, the induction heating furnace can be used to obtain various temperatures. The advantage of using an induction heater is its ability to heat the specimen without coming into contact with it. This would only work if the
specimen were electrically conductive. The electrical conductivity of the ZrB2 specimen allows for wide variety of temperatures.

![Figure 3.2-9: RDO Induction Heating Furnace and a Five Turn Heating Coil](image)

A type-K thermocouple was placed on the side of the ZrB2 specimen. False readings may occur due to the magnetic field produced by the coil of the induction furnace. Therefore the specimen must be oriented in a way that the thermocouple can take adequate measurements such that the specimen continues to heat up. The 1400°C maximum temperature of the thermocouple is another constraint. Some manufacturing companies indicate that the type-K thermocouple can exceed such temperatures if for short duration times. In order to obtain accurate readings an USB-9263 10V Analog Output Module DAQ (Figure 3.2-11) was used.
In order to yield higher temperatures, the specimen must be inside the coil. With the thermocouple, the specimen was placed outside the coil to take temperature measurements. Since the specimen will be place inside the coil, a pyrometer will be used to measure the temperature of the specimen. The constraint with the pyrometer is to ensure the laser is hitting the surface of the specimen. The experiment will be using an Omega Pyrometer as depicted in Figure 3.2-12.
3.3 Impact Testing

3.3.1 Test Setup

Prior to each experiment every piece of equipment will be placed in its respective space in order to perform the impact test. The specimen will be placed in a crucible and situated inside the coil. For lower temperature the type-K thermocouple will be placed on the side of the specimen. For higher temperatures the pyrometer will be used to measure the temperature. Once the temperatures reach steady state with no fluctuations the impact apparatus is moved in. The impact apparatus will be positioned such that the barrel is placed directly above the sample. A push button valve will release the pressurized gas allowing the projectile to collide into the specimen. Figure 3.3-1 is the schematic of the test setup.
3.4 Procedure

The specimen will be heated up to temperatures ranging from 500 C to 1400 C and tested at two different velocities of 115 m/s and 150 m/s. Then the damage length of indentation and the number of cracks that were produced as a result of the collision will be measured. The table below represents the test matrix of how many samples will be tested at certain temperatures and velocities.
Table 3.4: Test Matrix with ZrB2 Sample Number

<table>
<thead>
<tr>
<th>Velocity (m/s)</th>
<th>Temperature (C)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>500</td>
</tr>
<tr>
<td>115</td>
<td>792</td>
</tr>
<tr>
<td></td>
<td>796</td>
</tr>
<tr>
<td>150</td>
<td>792</td>
</tr>
<tr>
<td></td>
<td>796</td>
</tr>
</tbody>
</table>
Chapter 4: Results and Discussion

4.1 Projectile Velocity

Ten projectile velocities were measured at five separate pressures using two different gases as an airstream. Figure 4.1-1 shows the data and the variability. The average velocity was calculated for each pressure. The variability of the data was small and within +/- 5 m/s per average. Helium yielded higher velocities as expected due to its expansion coefficient. The maximum velocity that was obtained for Helium and Nitrogen were 152 m/s and 115 m/s respectively. As shown on the graph below the velocity is directly proportional to the pressure.

![Velocity Measurements for Helium and Nitrogen](image)

Figure 4.1-1: Velocity Measurements for Helium and Nitrogen

Using Reynolds number, the drag coefficient of a sphere, and the principles of drag, the force of the projectile was calculated. Using equations of projectiles the approximate time between the barrel and the specimen was calculated. One of the heights (1”) was when the specimen was above the coil being testing below 1000C. The other height (2”) was when the specimen was inside the coil being testing at temperatures above 1000C. The purpose of these calculations was to determine if drag might
contribute to changes in the velocity. Figure 4.1-2 shows the setup of the experiment. Figure 4.1-3 is a graph of the changes in velocities and the differences in height.

![Experimental Setup of Change in Velocity](image)

Figure 4.1-2: Experimental Setup of Change in Velocity

![Graph of Changes in Velocities and Differences in Height](image)

Figure 4.1-3: Change in Velocity
4.2 Temperature of the Specimen

The specimen was impacted at several different temperatures. For temperatures between 500 C and 1000 C, a type-K thermocouple was used. Since it was increasingly difficult to get temperatures above 1000 when the specimen was outside the coil a different approach was utilized. A Mullite crucible was used to put the specimen inside the coil. Since a thermocouple is inoperable in this condition a pyrometer was used. Temperatures of the specimen were taken at 1200C to 1400C using a pyrometer. Figure 4.2-1 displays the temperature acquired using the thermocouple to achieve 1000C for the 792 sample of ZrB2. This sample was impacted at a velocity of 152 m/s using helium and the graph also shows the drop in temperature from the cold airstream.

![Figure 4.2-1: Temperature Measurement of ZrB2 (792), Impact Speed 152 m/s](image)

For temperatures above 1000 C an Omega pyrometer was used to measure the temperature. One problem measuring the temperature was when the impact apparatus covered the induction coil. The impact apparatus was moved in such a way to take the temperature of the specimen with the pyrometer. After ample time for the specimen to heat up (300 + seconds), the temperature of the sample was taken. Then the impact apparatus was relocated into position and shot.

Several of the samples were cut and therefore had different dimensions. It was difficult to consistently yield a specific temperature using the induction furnace. Several trips within the induction furnace were occurring which resulted in a quick fire. For instance, once the temperature was reached
750°C and a warning was set off, the push button valve was pressed to release the projectile at that moment. Operating the induction furnace via on/off switch to obtain ideal temperatures proved to be impractical and cumbersome.

During the experiment the furnace was not properly configured. To optimized the furnace the distance between each coil was changed so that length apart were the equal, the current was increased to a higher output, and the start up frequency which the furnace uses was reduced from 350 to 250 Hz. Optimizing the furnace dramatically changed the ability to obtain higher temperatures and no limits were tripped.

4.3 Damage Length

The damage length of the sample is defined by the length of indentation created by the impact of the steel ball bearing. To measure the damage length of the samples, a scanning electron microscope (SEM) was used. The SEM software allows the user to measure impacts in millimeters and at least three measurements were taken for each sample. Figure 4.3-1 shows a comparison of 792 samples impacted at 615°C and 1000°C with a velocity of 152 m/s. At lower the temperatures, the sample shows a highly resistant nature to impact. The higher temperature shows multiple things occurring such as corrosion from oxide. There were no fractures that were apparent amongst the specimens that were impacted. The damage length of the remaining samples can be examined in Appendix A.

![Figure 4.3-1: a) 792 Sample at 615 C b) 792 Sample at 1000 C](image)
Table 4.3-1 below shows the damage length for each sample that was tested along side with their respective temperatures and velocities. Figure 4.3-2 is a graph of the data that was obtained during the experiment. The mass of the projectile was 0.0168 kg and from this, the kinetic energy can be determined. The kinetic energy of the projectile for 115 m/s and 152 m/s respectively was 111 J and 194 J.

Table 4.3-1: Test Matrix of the Experiment

<table>
<thead>
<tr>
<th>Sample</th>
<th>Temperature (°C)</th>
<th>Velocity (m/s)</th>
<th>Damage Length (mm)</th>
<th># of Fractures</th>
</tr>
</thead>
<tbody>
<tr>
<td>792</td>
<td>825</td>
<td>115</td>
<td>1.065</td>
<td>0</td>
</tr>
<tr>
<td>792</td>
<td>1020</td>
<td>115</td>
<td>1.112</td>
<td>0</td>
</tr>
<tr>
<td>792</td>
<td>717</td>
<td>152</td>
<td>1.378</td>
<td>0</td>
</tr>
<tr>
<td>792</td>
<td>1000</td>
<td>152</td>
<td>1.358</td>
<td>0</td>
</tr>
<tr>
<td>796</td>
<td>1250</td>
<td>115</td>
<td>1.449</td>
<td>0</td>
</tr>
<tr>
<td>796</td>
<td>1400</td>
<td>115</td>
<td>1.191</td>
<td>0</td>
</tr>
<tr>
<td>796</td>
<td>1200</td>
<td>152</td>
<td>1.430</td>
<td>0</td>
</tr>
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<td>793</td>
<td>1400</td>
<td>152</td>
<td>1.544</td>
<td>0</td>
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</tr>
<tr>
<td>793</td>
<td>1200</td>
<td>115</td>
<td>1.149</td>
<td>0</td>
</tr>
</tbody>
</table>
Examining the tables indicated that the damage length of the specimen increases with temperature and velocity relatively. While temperature certainly affected the results, the mechanisms for failure cannot be readily determined due to the limitation of experimental samples. When the temperature of the samples was increased, the samples became more susceptible to damage due to ductility.

The velocities of 115 m/s it appeared that damage length increased for the 792 samples. For velocities of 152 m/s it appeared that the damage length decreased by a small margin. Since a limited amount of 793 samples were tested, the damage length increases with temperature with respect to the 792 samples. The main reason for this assessment is that the 792 samples are similar to 793 samples in their sintering processes. One unique observation was the outlier sample that was tested at 1600C was that the damage length decreased. It is speculated that there was a reaction with the crucible since vaporization and other forms of corrosion occurred. Furthermore, the geometry could have played a role since every sample was not symmetrical due to the cutting process.
Examining the 796 samples, which were ZrB2-B4C composite, the damage length of the specimen increased with temperature and velocity with respect to the 792 and 793 samples. However, when increasing the temperature from 500 C to 1000 C, it appeared that no damage had been done to the specimens. After heating the specimens up to 1200 C and 1400 C, a crater would form after impact that could be detected using the SEM. In one case where the sample was heated up to 1400 C the damage length was so large that an entire layer had separated.
Chapter 5: Conclusions and Recommendations

Using Helium and Nitrogen as a gas, the maximum projectile velocities that were obtained were 115 m/s and 152 m/s. The method of determining the velocity was precise and was not a factor throughout the experiment. Even though the furnace had a few problems, it was still able to heat up the specimen between 500 and 1400 C. After optimization, the furnace was not a factor in subsequent tests of temperatures over 1000 C. The damage length of the ZrB2 specimens is directly proportionally to temperature and velocity for most cases.

The importance of performing impact tests is to determine the amount of energy a material can absorb. ZrB2 is a high temperature ceramic that has low density, high modulus, and high shock resistance. Through impact testing, the samples were able to withstand between 111 J and an upper limit of 194 J of energy without fracturing. The maximum damage length that ZrB2 endured was 1.544 mm at a temperature of 1400C making it a strong material candidate for supersonic vehicles or any other high temperature application.

5.1 Recommendations

The experiment allowed the ability to perform impact tests, however improvements can be implemented to further advance this method of experimentation.

- Incorporate the use of a pyrometer to measure the temperature of the specimen as was done later in the experiment.
- Manufacture smaller samples that will fit inside the coil.
- Design a coil to fit larger samples if smaller samples are unattainable.
- Experiment with different airstreams such as Hydrogen since it has a higher expansion coefficient than Helium.
- Due to the smoke (ZrO and SiO) created by heating up the ZrB2 to high temperatures, use a ventilator to get rid of the smoke. Ensure masks are worn during testing.
- Use a better crucible to ensure the sample does not stick or sinter to the crucible.
- Measure damage depth since hardness plays a significant role in determines the impact.
References


### Appendix A: Damaged Samples

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Velocity</th>
<th>Thickness</th>
<th>Image 1</th>
<th>Image 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>792, 825 °C</td>
<td>115 m/s</td>
<td>1.065 mm</td>
<td><img src="image1.png" alt="Image 1" /></td>
<td><img src="image2.png" alt="Image 2" /></td>
</tr>
<tr>
<td>792, 1020 °C</td>
<td>115 m/s</td>
<td>1.112 mm</td>
<td><img src="image1.png" alt="Image 1" /></td>
<td><img src="image2.png" alt="Image 2" /></td>
</tr>
<tr>
<td>792, 800 °C</td>
<td>152 m/s</td>
<td>1.358 mm</td>
<td><img src="image1.png" alt="Image 1" /></td>
<td><img src="image2.png" alt="Image 2" /></td>
</tr>
<tr>
<td>792, 1000 °C</td>
<td>152 m/s</td>
<td>1.378 mm</td>
<td><img src="image1.png" alt="Image 1" /></td>
<td><img src="image2.png" alt="Image 2" /></td>
</tr>
<tr>
<td>793, 1200 °C</td>
<td>115 m/s</td>
<td>1.149 mm</td>
<td><img src="image1.png" alt="Image 1" /></td>
<td><img src="image2.png" alt="Image 2" /></td>
</tr>
<tr>
<td>793, 1400 °C</td>
<td>152 m/s</td>
<td>1.544 mm</td>
<td><img src="image1.png" alt="Image 1" /></td>
<td><img src="image2.png" alt="Image 2" /></td>
</tr>
<tr>
<td>793, 1600 °C</td>
<td>152 m/s</td>
<td>1.420 mm</td>
<td><img src="image1.png" alt="Image 1" /></td>
<td><img src="image2.png" alt="Image 2" /></td>
</tr>
<tr>
<td>Temperature</td>
<td>Speed</td>
<td>Thickness</td>
<td></td>
<td></td>
</tr>
<tr>
<td>-------------</td>
<td>-------</td>
<td>-----------</td>
<td></td>
<td></td>
</tr>
<tr>
<td>796, 1200°C</td>
<td>152 m/s</td>
<td>1.430mm</td>
<td></td>
<td></td>
</tr>
<tr>
<td>796, 1250°C</td>
<td></td>
<td>1.449mm</td>
<td></td>
<td></td>
</tr>
<tr>
<td>796, 1400°C</td>
<td>115 m/s</td>
<td>1.191mm</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Appendix B: Temperatures Profiles

792, 815 °C, 115 m/s

792, 1020 °C, 115 m/s **

792, 717 °C, 152 m/s

792, 1000 °C, 152 m/s

**Quick Fire - Instantaneous release upon furnace trip
Appendix C: Arduino Code

/*
Analog Input
Demonstrates analog input by reading an analog sensor on analog pin 0 and turning on and off a light emitting diode(LED) connected to digital pin 13. The amount of time the LED will be on and off depends on the value obtained by analogRead().

The circuit:
* Potentiometer attached to analog input 0
* center pin of the potentiometer to the analog pin
* one side pin (either one) to ground
* the other side pin to +5V
* LED anode (long leg) attached to digital output 13
* LED cathode (short leg) attached to ground

* Note: because most Arduinos have a built-in LED attached to pin 13 on the board, the LED is optional.
Created by David Cuartielles
Modified 4 Sep 2010
By Tom Igoe
This example code is in the public domain.
http://arduino.cc/en/Tutorial/AnalogInput
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Description:
The purpose of this code is to make the circuit go high or low when a projectile passes through a sensor and LED.
*/

int sensorPin = 0;    // select the input pin for the first diode
int sensorPin1 = 1;
int sensorPin2 = 2;
int sensorPin3 = 3;
int sensorPin4 = 4;
int sensorPin5 = 5;
int sensorPin6 = 6;
int sensorPin7 = 7;
int sensorPin8 = 8;
int sensorPin9 = 9;
int sensorPin10 = 10;
int sensorPin11 = 11;
int sensorPin12 = 12;
int sensorPin13 = 13;
int sensorPin14 = 14;
int sensorPin15 = 15;

int DPin = 2;      // select the Output pin

int sensorValue;  // initialize variables to store the value coming from the sensor
int sensorValue1 = 1023;
int sensorValue2 = 1023;
int sensorValue3 = 1023;
int sensorValue4 = 1023;
int sensorValue5 = 1023;
int sensorValue6 = 1023;
int sensorValue7 = 1023;
int sensorValue8 = 1023;
int sensorValue9 = 1023;
int sensorValue10 = 1023;
int sensorValue11 = 1023;
int sensorValue12 = 1023;
int sensorValue13 = 1023;
int sensorValue14 = 1023;
int sensorValue15 = 1023;
int test=0;

//Function to check values of sensors
void check(){
  if( sensorValue < 110 || sensorValue1 < 110 || sensorValue2 < 110 || sensorValue3 < 110 || sensorValue4 < 110 ||
      sensorValue5 < 110 || sensorValue6 < 110 || sensorValue7 < 110 || sensorValue8 < 110 || sensorValue9 < 110 ||
      sensorValue10 < 110 || sensorValue11 < 110 || sensorValue12 < 110 || sensorValue13 < 110 ||
      sensorValue14 < 110 || sensorValue15 < 110){
    digitalWrite(DPin, LOW);
    test = 1;
  }
  if( sensorValue > 110 && sensorValue1 > 110 && sensorValue2 > 110 && sensorValue3 > 110 && sensorValue4 > 110 &&
      sensorValue5 > 110 && sensorValue6 > 110 && sensorValue7 > 110 && sensorValue8 > 110 && sensorValue9 > 110 &&
      sensorValue10 > 110 && sensorValue11 > 110 && sensorValue12 > 110 && sensorValue13 > 110 && sensorValue14 > 110 && sensorValue15 > 110){
    digitalWrite(DPin, HIGH);
    test = 0;
  }
}

void setup() {
  // declare the ledPin as an OUTPUT:
  Serial.begin(9600);
  pinMode(DPin, OUTPUT);
  pinMode(sensorPin, INPUT);
  pinMode(sensorPin1, INPUT);
  pinMode(sensorPin2, INPUT);
  pinMode(sensorPin3, INPUT);
  pinMode(sensorPin4, INPUT);
  pinMode(sensorPin5, INPUT);
  pinMode(sensorPin6, INPUT);
  pinMode(sensorPin7, INPUT);
  pinMode(sensorPin8, INPUT);
  pinMode(sensorPin9, INPUT);
  pinMode(sensorPin10, INPUT);
  pinMode(sensorPin11, INPUT);
  pinMode(sensorPin12, INPUT);
  pinMode(sensorPin13, INPUT);
}
pinMode(sensorPin14, INPUT);
pinMode(sensorPin15, INPUT);
}

void loop() {
  // read the value from the sensor:
sensorValue = analogRead(sensorPin);
sensorValue1 = analogRead(sensorPin1);
sensorValue2 = analogRead(sensorPin2);
sensorValue3 = analogRead(sensorPin3);
sensorValue4 = analogRead(sensorPin4);
sensorValue5 = analogRead(sensorPin5);
sensorValue6 = analogRead(sensorPin6);
sensorValue7 = analogRead(sensorPin7);
sensorValue8 = analogRead(sensorPin8);
sensorValue9 = analogRead(sensorPin9);
sensorValue10 = analogRead(sensorPin10);
sensorValue11 = analogRead(sensorPin11);
sensorValue12 = analogRead(sensorPin12);
sensorValue13 = analogRead(sensorPin13);
sensorValue14 = analogRead(sensorPin14);
sensorValue15 = analogRead(sensorPin15);

  Serial.print(sensorValue);
  Serial.print(sensorValue1);
  Serial.print(sensorValue2);
  Serial.print(sensorValue3);
  Serial.print(sensorValue4);
  Serial.print(sensorValue5);
  Serial.print(sensorValue6);
  Serial.print(sensorValue7);
  Serial.print(sensorValue8);
  Serial.print(sensorValue9);
  Serial.print(sensorValue10);
  Serial.print(sensorValue11);
  Serial.print(sensorValue12);
  Serial.print(sensorValue13);
  Serial.print(sensorValue14);
  Serial.println(sensorValue15);
  Serial.println(test);
  check();
  // turn the ledPin on
  //digitalWrite(ledPin, HIGH);
  // stop the program for <sensorValue> milliseconds:
  // delay(sensorValue);
  // turn the ledPin off:
  //digitalWrite(ledPin, LOW);
  // stop the program for for <sensorValue> milliseconds:
  //delay(sensorValue);
}
Vita

Mark D. Flores was born on August 13, 1982, in El Paso, TX. Mark graduated from J.M. Hanks High School in the year 2000 and soon after began his higher education at the University of Texas at El Paso (UTEP). As an undergraduate student, he participated in a competition with the Texas Space Grant Consortium on designing an unpressurized manned vehicle. In May of 2005 he received his Bachelor of Science degree in Mechanical Engineering. After graduating, he obtained a job position as a Satellites Systems Engineer for Lockheed Martin. Working for three satellite programs he operated, maintained, built test plans, built scripts, built graphical user interfaces (GUIs), and worked with every segment that has to do with satellites such as the satellite subsystems, ground systems, and command and control systems. During his time with Lockheed Martin, Mark continue to learn new exciting things outside of work such as glass blowing, wood working, reiki and beer brewing. He would also volunteer his time for Habitat for Humanity. After working less than 5 years with the company, Mark decided to go back to school to pursue his master’s degree under Dr. Jack Chessa investigating high temperature ceramics. Once finished with his masters he will be pursuing his Ph.D under Dr. L. Roy Xu investigating salt-water corrosion with carbon-fiber composites.

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