# MODULAR HIGH TEMPERATURE SYSTEM FOR MOLDING AND SINTERING

By

CHRISTOPHER JAMES CANTONI and GARRETT JOSEPH COLEMAN

---

A Thesis Submitted to the Honors College

In Partial Fulfillment of the Bachelor's degree
With Honors in

Materials Science and Engineering

THE UNIVERSITY OF ARIZONA

MAY 2013

Approved by:

_______________________________

Dr. Pierre Lucas
Department of Materials Science and Engineering

# The University of Arizona Electronic Theses and Dissertations Reproduction and Distribution Rights Form

The UA Campus Repository supports the dissemination and preservation of scholarship produced by University of Arizona faculty, researchers, and students. The University Library, in collaboration with the Honors College, has established a collection in the UA Campus Repository to share, archive, and preserve undergraduate Honors theses.

Theses that are submitted to the UA Campus Repository are available for public view. Submission of your thesis to the Repository provides an opportunity for you to showcase your work to graduate schools and future employers. It also allows for your work to be accessed by others in your discipline, enabling you to contribute to the knowledge base in your field. Your signature on this consent form will determine whether your thesis is included in the repository.

**Name (Last, First, Middle)**

Cantoni, Christopher, James

**Degree title (eg BA, BS, BSE, BSB, BFA):**

BS: Materials Science + Engineering

**Honors area (eg Molecular and Cellular Biology, English, Studio Art):**

Materials Science + Engineering

**Date thesis submitted to Honors College:**

5/1/13

**Title of Honors thesis:**

Modular High Temperature System for Molding + Sintering

**The University of Arizona Library Release Agreement**

I hereby grant to the University of Arizona Library the nonexclusive worldwide right to reproduce and distribute my dissertation or thesis and abstract (herein, the "licensed materials"), in whole or in part, in any and all media of distribution and in any format in existence now or developed in the future. I represent and warrant to the University of Arizona that the licensed materials are my original work, that I am the sole owner of all rights in and to the licensed materials, and that none of the licensed materials infringe or violate the rights of others. I further represent that I have obtained all necessary rights to permit the University of Arizona Library to reproduce and distribute any nonpublic third party software necessary to access, display, run or print my dissertation or thesis. I acknowledge that University of Arizona Library may elect not to distribute my dissertation or thesis in digital format if, in its reasonable judgment, it believes all such rights have not been secured.

☒ Yes, make my thesis available in the UA Campus Repository!

Student signature: [signature] Date: 4/29/13

Thesis advisor signature: [signature] Date: 4/29/2013

☐ No, do not release my thesis to the UA Campus Repository.

Student signature: ______________ Date: ______________

**Abstract:**

A modular heating system was developed for low cost heat-related processing of materials. This system demonstrated successful molding and sintering of low $T_g$ glass and future work will be expanded to controlling other properties of materials.

**Statement of roles and responsibilities of each group member:**

For this senior thesis, there were no roles or responsibilities that our supervising professor specifically assigned to either partner in our two-person group. In most situations, both members found it advantageous to work together on the project without divvying up any tasks. For the situations that necessitated splitting up the work, however, both team members discussed and then mutually agreed to working on certain aspects of the project. These responsibilities are detailed below.

- **Chris Cantoni**: assumed responsibility for the cost-estimate, parts ordering, and mechanical design aspects of the project. This involved devising a cost estimate of the components used in the project, ordering these components, designing parts in Solidworks that would be machined, working with the campus machine shop to fabricate these parts, performing additional fabrication on mechanical fixtures, and fabricating and assembling the final housing for the project.

- **Garrett Coleman**: assumed responsibility for the glass synthesis, electrical design, and testing aspects of the project. This involved preparing several types of glasses, collaborating with a lab on campus that could ball-mill these glasses, testing the properties of different types of glasses, estimating the optimal processing settings for the project, devising the electrical schematic for the project, and programming the controller used to power the project.

Table of Contents:

## Introduction

### Overview

In materials processing, useful properties can be obtained from simultaneous heating and pressing of finely-ground powders. The most common types of processed powdered materials are metals and ceramics; however, all types of materials, including glasses and polymers, can be ground into powders and experience heating and sintering to achieve useful properties.

Hot press setups allow for the processing of these powdered samples into bulk materials with desired properties. These setups have incredible processing capabilities: powders can be pressed at forces of up to 100 tons at temperatures of $2500^{o}$C in an inert atmosphere of $10^{-6}$ Torr. Although these properties are desirable, the size of these systems is incredible and their cost can vary between $100,000 and $750,000. Ultimately, these systems are major investments, making them impractical for most laboratory environments and budgets.

For materials that do not require significantly high pressures and temperatures for processing, there are relatively few options available for hot-press systems. Additionally, the few viable hot-press setups that exist are limited by their functionality - for systems that process powders inside mechanical dies, these dies must be designed to fit within the heating chamber of the hot-press system, which is not always possible. Often times, the samples produced by these setups are limited geometrically, as hot-press systems have mechanical designs that are only be capable of sintering a material with a certain area or volume.

Given these shortcomings, it is desirable to create a system capable of processing powdered materials at elevated temperatures and pressures that can be practical for laboratory environments and budgets. The samples that are processed by such a setup would not be limited in size or shape, as many different types of mechanical designs could be incorporated into this setup. The system would not possess the same processing power as commercially-available setups; however, the suggested applications for such a system do not necessitate the high temperatures and forces as well as the inert atmosphere that are found in industrial-grade equipment.

Ideally this system would also demonstrate mechanical functionality, meaning that its components could be used for alternative setups as needed. This would enable the use of different mechanical designs within the hot-press system and would allow for thermal control of several different types of setups.

## Project Goal

The goal of this project is to create a system that can heat powdered materials to 300°C while simultaneously applying forces up to 2 long tons. The powders will be molded and sintered at these temperatures and forces within a machined die. After processing, the sintered samples will be removed easily from the die. The system will also be modular so that several types of dies can be used in the hot-press system. The mechanical design will combine machined parts with commercially available products to optimally balance precision with cost-effectiveness. The electrical design will feature a reliable setup for controlling time and temperature settings along with robust heating elements that can be used in several different mechanical dies.

## Design

Mechanical

The mechanical design of the hot-press system consists of all the components of the system that experience heating as well as the components that exert forces on the powdered materials. More specifically, this mechanical design features both a machined die that experiences indirect resistance heating along with a 10-ton commercially-available press used to press the powders.

The machined die consists of three parts: a stainless steel die, a removable support base for the die, and a hardened-steel pin. These components can be seen in Figure 1 and are discussed in more detail below:

- *Stainless steel die*: the main processing component of the hot-press system. This die is made of a cylindrical piece of 304 stainless steel that is 2.75” in height and 2.0” in diameter. The die has a 7mm diameter hole through its center with a mechanically polished surface on its inside, which is where processing of powders takes place. This polished surface is imperative for effective sintering, as a rough surface makes sample extraction nearly impossible. The 7mm diameter was chosen to be the size of the processing chamber and, therefore, the size of processed samples, as certain thermoelectric properties of samples with this specific diameter could be tested using equipment through collaboration between the AME Department and the MSE Department. The 304 stainless steel as well as the precision drilling of the 7mm diameter hole was performed at the University Research Instrumentation Center. The die also features three $3/8$” diameter cylindrical holes drilled at approximately 120$^{o}$ from each other than run parallel with the 7mm diameter center hole. These holes were not drilled at the URIC, since they were did not have critical tolerances and additional machining at URIC would not help to keep costs low. Instead, they were drilled with a $^{3}/_{8}$" diameter Dewalt drill bit and a drill press.

- *Hardened steel pin*: the load-bearing piston that compresses powders within the die. This pin is commercially available from McMaster-Carr and has a 7mm diameter tolerance, which makes it a perfect fit within the machined die. This fit prevents powders from leaving the die during processing and ensures that all powders poured into the die are compressed and sintered into a bulk sample. The sides of the pin are polished, which helps the pin slip easily through the die. The height of the pin was chosen to be $2\,^{5}/_{8}$". This height was chosen because a larger pin height could result in bending under load, whereas a smaller pin height could negatively affect the heating elements of the system.

- *Removable support base*: the key component for sample extraction. This base is made of the same cylindrical piece of 304 stainless steel that was used in fabricating the die. The base is $^{3}/_{4}$" in height with a 7mm diameter piece of hardened steel extruding about $^{1}/_{4}$ " from its center. This extrusion consists of the same hardened steel used for the pin. This extrusion was constructed by inserting the pin through a 7mm hole drilled in the center of the base and then welding the pin and the stainless steel base together on its bottom. This bottom was then polished so as to make a flat surface for supporting the mechanical die. The base supports the die and sustains the large forces that the pin and powders experience during processing.

Except for the pin, each part was devised in Solidworks and sent to the URIC for precise fabrication. The Solidworks drawings of these parts are below:

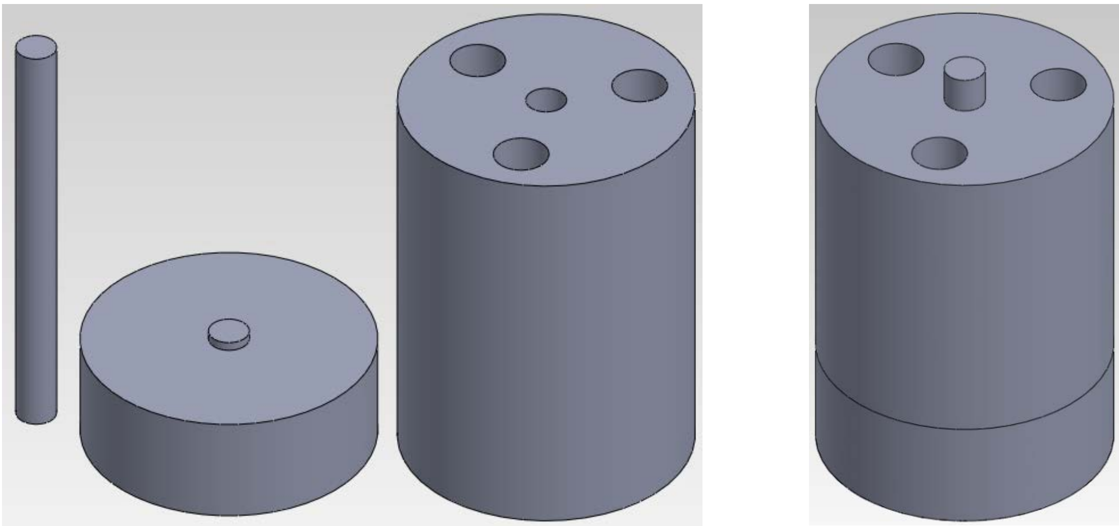


Fig. 1. Solidworks design of the die components (left) and the assembled die (right)

The mechanical design also consists of a 10-ton press used for compressing powders within the die. This commercially-available press (Fig. 2) has a piston which lowers onto the hardened steel pin and applies forces onto the powders. The press also features a force gauge, allowing for continuous monitoring of the applied force on the powdered material. More specifically, this press is the Torin Big Red Hydraulic Shop Press with Gauge Dial, 10-tons (Item #145208).

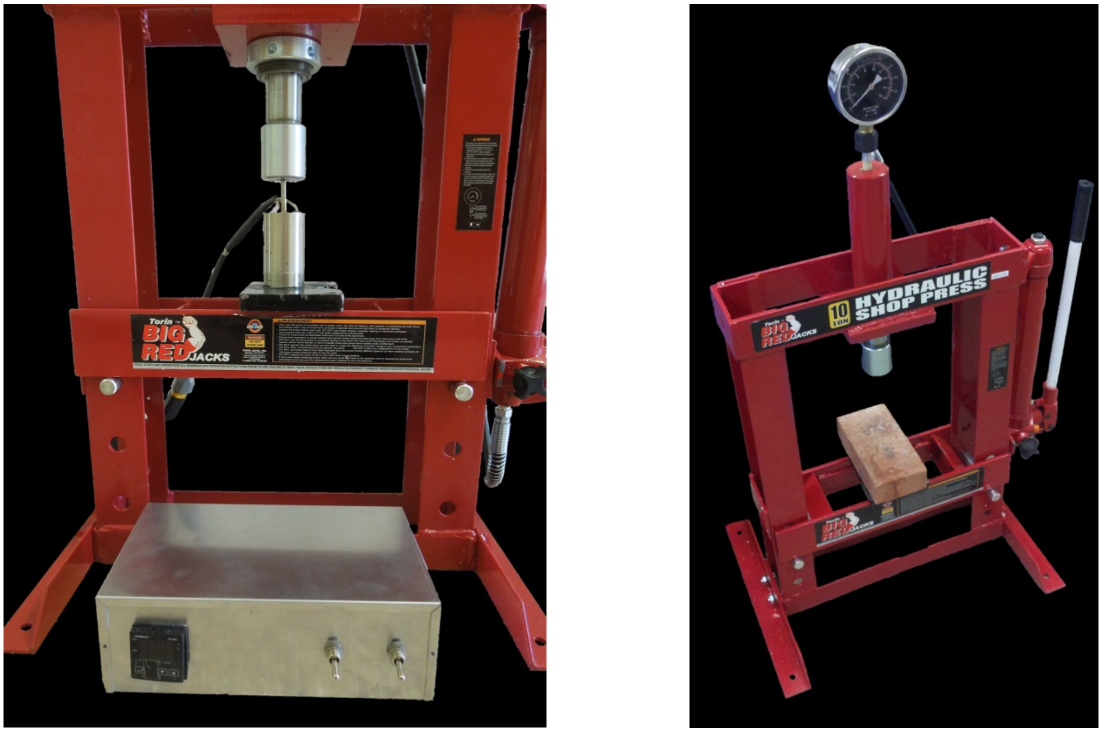



Fig. 2 Full hot-press setup (left) and the press standing alone (right)

<u>Electrical</u>

The electrical design of the hot-press system includes all the electrical components that act as heat sources for the setup as well as the components that control these sources. The main electrical components of the hot-press system are the PID controller, the cartridge heaters, the solid state relay, and the K-type thermocouple. All components are connected with 14 gauge copper wire. A more detailed description of the electrical components is below:

- *PID controller*: the device which controls the heating of the system. This proportional-integral-derivative (PID) controller monitors the temperature of the material and controls the optimal time and temperature for heating using a proportional-integral-derivative algorithm. This controller also has numerous ramp/soak settings that allow for the die to achieve a given temperature over a specified period of time. For qualifying the setup, an OMEGA CN7800 series PID controller was used. This controller demonstrated acceptable functionality, but still left certain aspects to be desired. For the final system design, an OMEGA CNi16 controller with DC pulsed output will be used for controlling the setup. This controller will offer improved temperature precision as well as a more user-friendly interface.

- *Cartridge heaters*: the heating units used to provide heat to the system. These heaters are essentially cylinders that are designed to slip into holes and begin heating an object through indirect resistance heating. For this setup, three OMEGA CIR-2021 120V AC cartridge heaters were used. These cartridges handle 150W and are $^3/_8$" in diameter with a $2\,^5/_8$" height. These three cartridges slip into the three $^3/_8$" diameter holes in the mechanical die and act as the sources of heat for the die. The die is designed so that these cartridges can provide heat to the die while the powders are simultaneously experiencing up to 2 tons of force from the press. These cartridges can be placed into any mechanical setup which features three $^3/_8$" diameter holes, which gives the system optimal functionality. If the system needs to experience higher temperatures, OMEGA offers larger and more powerful heating cartridges that can be used instead of the current 150W cartridges. These cartridges are shown below:

Fig. 3. OMEGA heating cartridges used to heat the hot-press system.

- *Solid state relay*: the switch that passes current to the cartridges. This relay is controlled by the PID controller and allows for current to flow through and heat up the cartridges. The model chosen for this system, G3NA-225B, is a solid-state model that is controlled by a 5-24V DC pulse and allows for a 24-240V and 25A AC output.

- *K-type thermocouple*: reads the temperature of the hot-press system. This thermocouple allows for the PID controller to monitor and control the temperature of the hot-press system. The thermocouple fits securely into a small hole drilled towards the bottom of the die and provides accurate readings of the temperature of the die. The thermocouple does not reach into the very center of the die where processing takes place; however, it can be reasonably assumed that the heat distributed throughout the stainless steel die is uniform and that the reading provided by the thermocouple is the actual temperature that powders are experiencing within the die.

In addition to these parts, there are several miscellaneous electrical components in this setup that deserve mention.

- *Aluminum heat sink*: secures the relay and ensures that heat produced by the relay does not detrimentally affect the electrical system.

- *10-amp fuse*: connected to the main electrical port, prevents the electrical components in the event of a power surge.

- *Switches*: allow for flow of current to both the PID controller as well as the relay.

- *Plugs*: allow for the heating cartridges as well as the power cord to be detached from the electrical system. These plugs feature different configurations for safety so that the plug for the heating cartridges will not fit into the power plug, and vice versa.

These components are connected together in Figure 4 below:

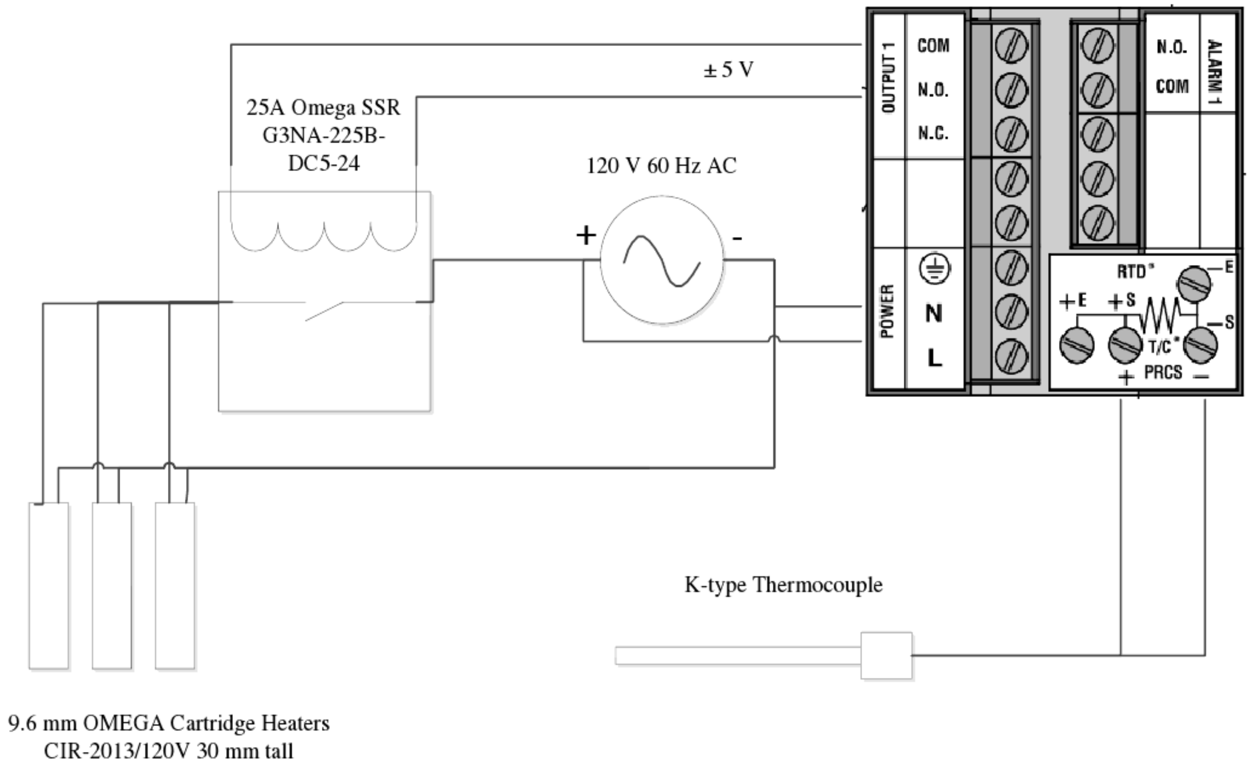



Fig. 4: Electrical Diagram including the PID controller in the upper right, the solid-state relay located at the upper left, and the OMEGA heating cartridges in the bottom left.

## Qualification

In order to demonstrate that the hot-press setup and its mechanical and electrical components are functional, the system must effectively process a powdered sample. This processing has three parts: preparing a powdered sample, sintering the powders together, and characterizing the sintered material. These three components are discussed in more detail below.

Preparation

The material chosen for processing is $GeSe_4$, a low-Tg chalcogenide glass which does not crystallize easily. This glass has a Tg around 170$^{o}$C, which is a temperature that the hot-press setup can achieve. The glass was synthesized at the Arizona Materials Laboratory. The glass was then subjected to high energy ball-milling for 20 hours in order to break the glass into micron-sized constituents which constitute a powdered material. The before-and-after picture of this glass can be seen in Fig. 5:

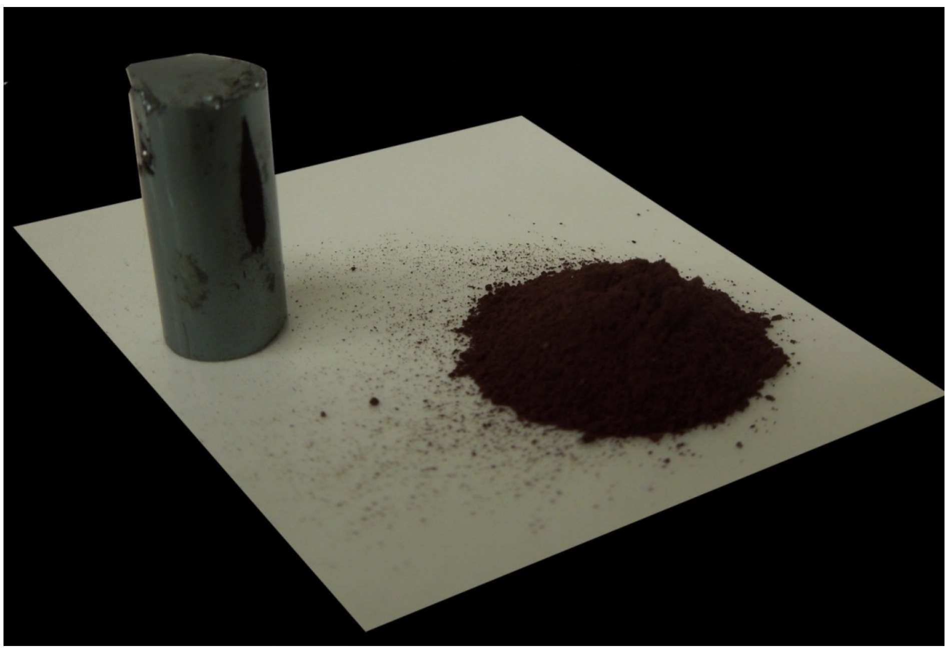


Fig. 5: $GeSe_4$ before ball-mill (left) and after ball-mill (right)

Sintering

The powdered material was prepared and ready to be sintered. However, before the powder can be put inside the machined die, the inside diameter of the die must be cleaned and prepared. The cleaning of the die involved rolling up a Kim-Wipe to a 7mm diameter, coating the wipe with Acetone, inserting the wipe into the center hole of the die, and then moving the wipe back and forth through the die to remove debris. This was done 4-5 times in order to ensure that no residual metal debris or powders from previous samples remained in the die. Once this was complete, another Kim-Wipe was rolled and doused in Isopropyl Alcohol and was then used to clean the inside diameter of the die for a final time.

After the machined die was cleaned, the inside diameter of the die was coated with a Boron-Nitride aerosol release agent. This release agent was sprayed on the inside diameter of the die until all surfaces were coated. The hardened steel pin was then pushed through the die several times to collect residual release agent. This additional residue was then wiped off.

Once the coating was applied, the machined die was placed on its base and the powdered $GeSe_4$ was poured into the die. This powder was compacted into the die with the hardened steel pin. For this qualification, 2 grams of powder were poured into the die to be processed.

The die was placed onto the press and the three heating cartridges were placed in its openings. The press then applied a load of 0.5 tons onto the die. Once this load was applied, the die was heated to 195$^{o}$C, and was held at this temperature for 15 minutes. During heating, the applied force on the powders decreased. In order to counteract this release of force, the press and sample were continuously monitored during processing and the press was adjusted when needed in order to maintain a constant force of 0.5 tons on the powder.

Once the powders were processed for 15 minutes at 195°C under 0.5 tons of force, the heat was switched off and the die was allowed to cool. However, the die could not cool down too much, as doing so would make sample extraction more difficult. Instead, the die was cooled to a temperature of 120°C, at which point the sample was extracted. This extraction was performed by removing the base of the die and pressing the sintered sample out using the force of the press. A metal dish was placed underneath the die in order to catch the sample as it comes out of the die. The final sintered sample is shown below in Fig. 6:

Fig. 6: Sintered $GeSe_4$ sample from the hot-press system.

Characterization

There are several tests that can be performed on a sintered sample in order to evaluate its properties. However, the most important test characteristic for the purposes of this setup is the crystallinity of the sample.

To test for crystallization, the specimen underwent X-Ray Diffraction. The results of this test, shown in Fig. 7 below, indicate that no crystallization of the sample occurred.

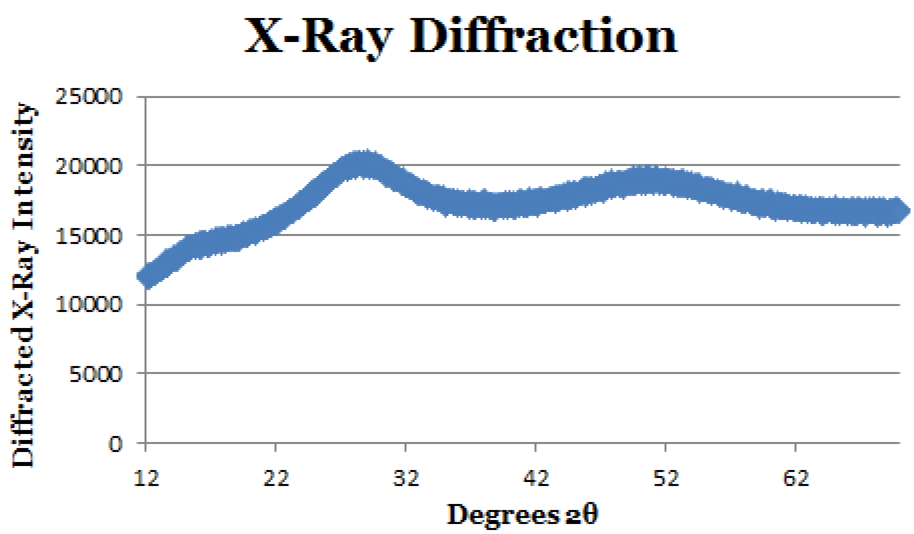



Fig. 7: XRD results of the $GeSe_4$ sintered sample showing no crystalline peaks.

Since the sintered sample did not show any crystalline peaks, it can reasonably be assumed that the sample has an amorphous structure. For the purpose of this setup, it is essential that powdered glasses do not crystallize from sintering. Because this processed sample has an amorphous structure, successful processing of the powders was achieved.

One of the potential applications of this setup is for making improved thermoelectric materials Glasses, which intrinsically exhibit low thermal conductivity values, can potentially possess even lower thermal conductivity values if they can achieve a certain porosity. One way to achieve this porosity is by sintering glass powders together in a setup similar to the one presented in this report.

Increasing the porosity of a material can lead to the development of interfaces between glass and air within the material. These voids disrupt the flow of heat through the material, thereby reducing thermal conductivity.

Additionally, both Germanium and Selenium have large weights that are useful in reducing the flow of heat. As heat flows through a material, the atoms comprising the material absorb thermal energy and oscillate at a frequency corresponding with the amount of energy introduced to the material. Because of their weight, the $GeSe_4$ glass cannot oscillate as much as lighter atoms and the material thereby does not absorb as much thermal energy.

Another attribute of glasses that contributes to their low thermal conductivity values is their absence of long-range molecular order. Instead, glasses have short-range order, which is characteristic of an amorphous microstructure. Because of this short-range order, it is more difficult for atoms to oscillate together. As a result, the glass is unable to conduct heat as well as other materials, which is characterized by its low thermal conductivity.

These characteristics, as well as the processing capabilities of this setup, make the hot-press system a viable option for processing thermoelectric glasses.

**Cost**

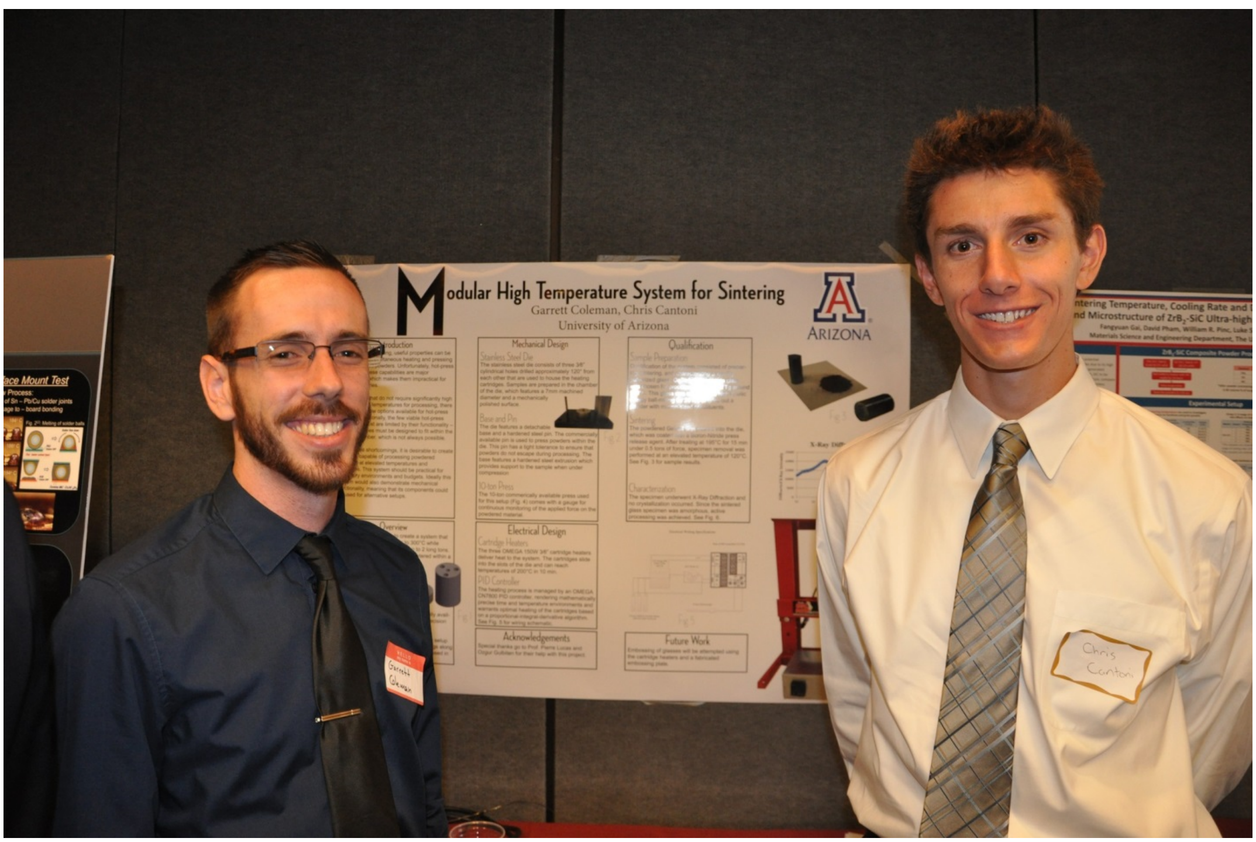
| **Item Description** | **Product ID** | **Price/unit ($)** | **Amount** | **Total ($)** |
|---|---|---|---|---|
| OMEGA SSR 25A | G3NA-225B-DC5-24 | 27.50 | 1 | 32.50 |
| 5cm 150W OMEGA Cartridge Heater | CIR-2021 120VAC | 43.00 | 3 | 134.00 |
| K-Type Thermocouple | Already Have | NA | 1 | NA |
| PID controller | CNi1644-C24 | 295.00 | 1 | 332.50 |
| 10 Ton Press | #145208 | 279.99 | 1 | 371.99 |
| Machined die | Die, base, and pin | 155.00 | 1 | 155.00 |
| *Drill bit* | *3/8" Dewalt* | *10* | *1* | *10* |
| *Heat sink* | *Used at Elliott's* | *3* | *1* | *3* |
| *Aluminum housing* | *New at Elliott's* | *27* | *1* | *27* |
| *Female thermocouple plug* | *New at Elliott's* | *7* | *1* | *7* |
| *Electrical wire* | *14 gauge* | *5* | *1* | *5* |
| *Switches* | *New at Ace* | *5* | *2* | *10* |
| *Plugs* | *Ace and Elliott's* | *5* | *4* | *20* |
| *Heat shrink tubes* | *Ace* | *2* | *1* | *2* |
| Poster for Materials Bowl | Fast Design | 108.00 | 1 | 108.00 |
| Total: $1,217.99 | | | | |

Shipping and tax are included in the "Total ($)" column.

Estimated prices are italicized.

**Poster**

The 10th Annual Materials Bowl took place at the ASU Karsten Golf Course in Tempe, AZ on April 23, 2013. Four University of Arizona (UA) Department of Materials Science and Engineering (MSE) teams squared off against six ASU Ira A. Fulton School of Engineering teams in what is known as The Battle for the Materials Territorial Trophy, a senior project and poster competition created in 2003 by Norm Hubel, President and Founder of Refrac Systems. The event was sponsored by the ASM International Phoenix Chapter and 5 jury members issued from the materials community awarded three top ranked projects. --Maryline Boulay Graduate Program Coordinator, SSES.



Garrett Coleman (left) Christopher Cantoni (right) standing in front of their poster at the Materials Bowl 2013.

**Acknowledgements**

Special thanks goes to Ozgur Gulbiten for his DSC and XRD testing of our samples.

This research was funded and supported by Professor Pierre Lucas.