

**Prototype Development of Remote Operated Hot Uniaxial Press (ROHUP) to Fabricate Advanced Tc-99 Bearing Ceramic Waste Forms – 13381**

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**ABSTRACT**

The objective of this senior student project is to design and build a prototype construction of a machine that simultaneously provides the proper pressure and temperature parameters to sinter ceramic powders in-situ to create pellets of rather high densities of above 90% (theoretical). This ROHUP (Remote Operated Hot Uniaxial Press) device is designed specifically to fabricate advanced ceramic Tc-99 bearing waste forms and therefore radiological barriers have been included in the system. The HUP features electronic control and feedback systems to set and monitor pressure, load, and temperature parameters. This device operates wirelessly via portable computer using Bluetooth® technology. The HUP device is designed to fit in a standard atmosphere controlled glove box to further allow sintering under inert conditions (e.g. under Ar, He, N<sub>2</sub>). This will further allow utilizing this HUP for other potential applications, including radioactive samples, novel ceramic waste forms, advanced oxide fuels, air-sensitive samples, metallic systems, advanced powder metallurgy, diffusion experiments and more.

**INTRODUCTION**

To the date commercial HUP or HIP (hot isostatic pressing) for installation in a standard laboratory size glove box are unavailable due to dimensions and complexity. Therefore the specific mission of this project was to design and build a prototype construction that is capable of providing proper temperature and pressure parameters to produce dense monolithic ceramic waste forms. For now, the Tc-99 oxide structures produced through pressure-less sintering exhibit good chemical homogeneity but high porosity [1] (fig. 1). By applying pressure and heat simultaneously we intend to maintain the chemical homogeneity but increase the density of these samples above 90% theoretical. The synthesis of advanced ceramic Tc-99 waste forms (e.g. pyrochlores, perovskites) is the specific goal for the machine, but ultimately this device should also be able to create new materials at any temperature and pressure parameters between 24 °C to 1150 °C and 0.1 MPa to 600 MPa, respectively. Since the samples to be eventually fabricated inside this machine are radioactive, the press will ultimately be placed in a standard-size glovebox. This one criterion requires the hot press parameters of operation (temperature and pressure) be set remotely from a computer outside of the glovebox. An electrically controlled

hydraulic cylinder will apply up to 98 kN of force to the waste form constituents in a pellet die while a vertical split-tube furnace surrounds the die and heats up the sample to any specified high temperatures up to 1150°C. The real-time application of both temperature and pressure need to be carefully monitored and recorded into a data acquisition system while the machine is operating. This device represents a holistic mechanical, electrical and computer engineering approach to further embrace the future of nuclear waste immobilization.

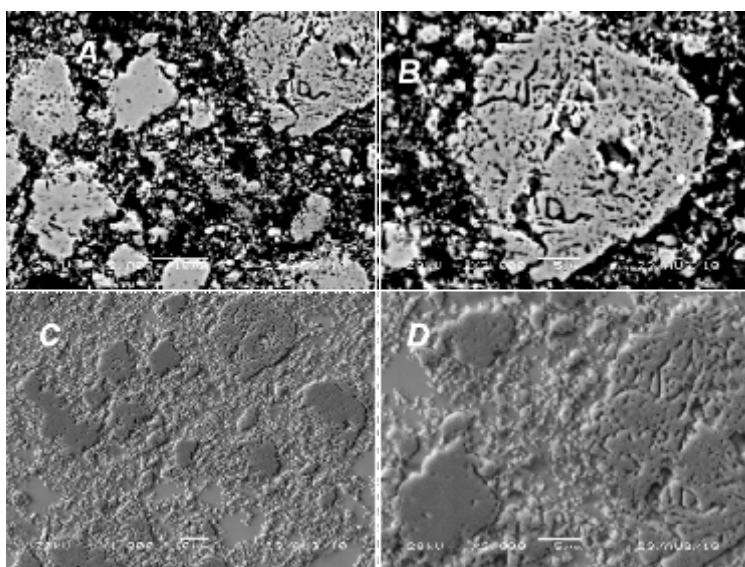


Fig. 1. Microstructures of  $\text{Nd}_2\text{Tc}_2\text{O}_7$  pyrochlore samples [1]. The top images were taken using secondary electron imaging (SEI) and the resulting images show that the microstructure exhibits lots of spongy 'worm-hole' type porosity. The bottom images are backscattered electron imaging (BEI) and show that there is chemical homogeneity of the sample. The mission is that with the HUP, these structures will retain their chemical homogeneity but increase density above 90%.

## METHODS

### *Prototype Design Constraints and Criteria*

The objective of this project is to build a machine that simultaneously heats and compresses radioactive ceramic powders into dense monolithic structures. The dimensions of the pellets should be 2-5 mm in height and range from 3 to 13 mm in diameter. Three major constraints were identified for consideration when developing the final design of this device. The first constraint is that the procedural time that it takes to sinter these particular ceramic powders at high temperature could take anywhere from a few hours to a few days. The exact time of the chemical synthesis will not be known until experiments are executed on the working HUP machine. Using the methodology specified by Hartmann et al. [1, 2] to synthesize  $\text{Ln}_2\text{Tc}_2\text{O}_7$  pyrochlores,  $\text{SrTcO}_3$  perovskites and  $\text{Sr}_2\text{TcO}_4$  layered perovskites the powders must be sintered at 550-1150 °C for 2-10 days. The expectation with this machine is that with the addition of pressure, the total procedural time of the chemical synthesis will be reduced down to a mere few hours. In either case, components in this machine, such as the piston ram and part of the stand

will be exposed to high temperature conditions for significant amounts of time. The second major constraint to consider is the radiological hazards associated with the use radioactive materials. Therefore, engineered radiological barriers need to be present in the design to prevent the contamination of radioactive materials to any surrounding area. The radioactive element, Tc-99, used in the synthesis and these Tc-99 bearing oxides, has the potential to volatilize and disproportionate at 700°C and 1100°C, respectively. For this reason, this machine will reside inside of a glove box. The glove box allocated for this project has total inner dimensions of 76.2 cm x 50.8 cm x 55.9 cm. The machine must be designed compact to fit in this small glovebox but also ergonomic to set up and sensible to use. The total height of the machine should be no larger than 50.8 cm. To reiterate, the three major constraints identified and addressed in our design are: the heat considerations, radiological hazards and control, and the geometric space limitations.

***Design Criteria:***

- Be compact, sensible, and ergonomic to use in a glovebox
- Be remote operated
- Provide pressures of up to 600 MPa over a pellet diameter of 13 mm
- Provide temperatures of up to 1200 °C

***Prototype Design Development - Analysis of Requirements***

This device is designed compact enough to fit in a standard laboratory glovebox. The temperature parameter can be satisfied by means of a resistance furnace. The furnace we designed is a short, 17.8 cm (7 in.), vertical split tube (VST) furnace. The pressure parameter can be satisfied by means of a short, 8.9 cm (3.5 in.), 8.9 cm-diameter hydraulic ram. To apply pressure on the system, *remotely*, the device will require an electrical system to initiate a motor when the signal is triggered by the user, *wirelessly*, via laptop. The device will need a customized signal feedback system to ensure the proper parameters have been reached and are maintained for the duration specified.

**RESULTS and DISCUSSION**

***Mechanical Configuration***

The mechanical force translation system consists of a short hydraulic ram that uniaxially moves through a sealed cylinder providing a maximum force of up to ~109 kN. This amount of force is a factor of 1.2 over the maximum pressure required as specified by the user. This factor is designed for safety. When the cylinder is engaged all of the force translates from the cylinder ram to the electronic load cell. The pellet die is located in between these two components. All of

the parts directly under load must be able to safely withstand the high loads as used. The pressure is first maintained by feeding the hydraulic fluid through a check valve before it is pumped into the cylinder. To release the pressure and retract the ram, the hydraulic line must be relieved through an alternate line that bypasses the check valve. An electric solenoid is placed in the hydraulic line and is used as a dump valve to relieve the pressure in the line after use. Once the pressure has been relieved in the line, the motor must reverse in direction to provide sufficient suction to bring the cylinder back down. When the user sends a signal from the computer, the motor engages the pump which runs hydraulic fluid through the line and through a series of mechanical and electrical devices. The devices include a mechanical pressure relief valve, a check valve, an electrical solenoid (dump) valve, analog pressure gauge and digital pressure gauge. The mechanical pressure relief valve can be regulated and set by an adjustable valve. Presently the pressure setting on the relief valve is  $p \leq 17.2$  MPa (2500 PSI). The stand (fig. 2g) is made of cast iron and all screws used are made steel above grade 8.

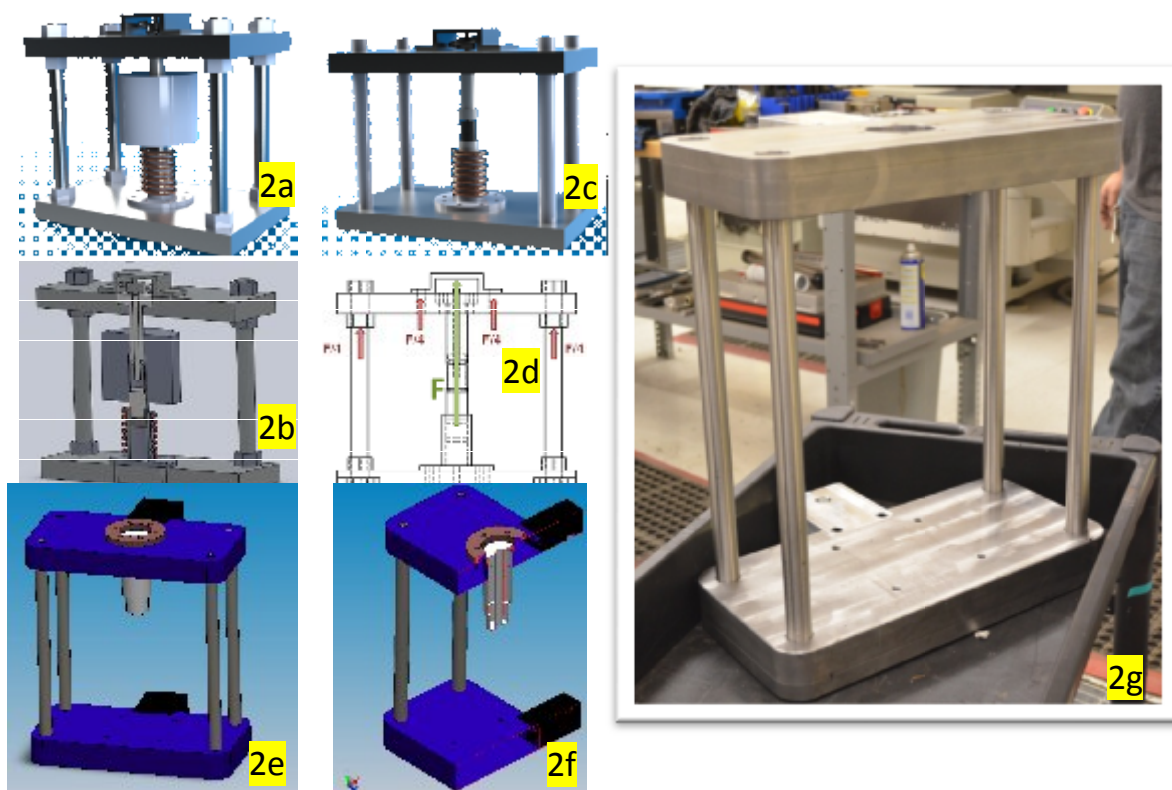


Fig. 2. (a) (Top Left) Conceptual drawing of ROHUP prototype I device featuring the vertical split tube furnace. (b) (Center Left) Cross-sectional conceptual image of the ROHUP prototype I with the vertical split tube furnace installed. (c) (Top Right) Conceptual image of ROHUP prototype I without the vertical split tube furnace. This drawing features the ceramic piston, graphite die and ceramic 'T'. (d) (Center Right) Force body diagram of the ROHUP prototype I. (e) Conceptual drawing of mechanical stand for ROHUP prototype II. (f) Cross-sectional conceptual image of ROHUP prototype II without hydraulic actuator and furnace. (g) Actual mechanical stand of ROHUP prototype II.

### ***Electronic Configuration***

Custom electrical systems had to be designed and fabricated to control the mechanical ram. The motor is the gateway between the mechanical and electrical components and is controlled by a series of electrical safety switches, relays and control boards. The first prototype circuit board (PCB) (fig. 3a,b) on the system features the microcontroller and manages the I/O signals, Bluetooth data capture, and live sensor information from the load cell and the pressure sensor. This customized system allows the user to control the directionality of the motor to engage and retract the piston, wirelessly, as required. The second PCB is a pulsing system designed to control the motor ramping speed. The pulser is desirable when pressure needs to be applied slowly to the specimen. The schematic of this prototype board can be seen in fig. 3c,d. The pulser uses a 555 timer which creates a square electrical signal to turn on and off the motor. The frequency and duty cycle can be changed by modifying a resistor size on the board. Other electrical components on the board, two MCP601 chips, are used to pull all the signals to the board ground and to buffer the output. Both circuit boards have been tested on the machine. As of now, the whole electrical system is controlling the motor and communicating with the wireless signal flawlessly.

### ***Computer Aspects, Code and Graphical User Interface***

The HUP is equipped with a general feedback control system using the ATmega328 controller and a series of sensors. To deliver precise amounts of force to the pellet, the machine is controlled by sending electrical signals processed through computer code to an AC motor that engages the mechanical ram. The device has a wireless remote interface that controls the entire electro-mechanical system remotely on a wireless laptop. The electrical signals are managed by code that considers the user's input, pressure sensor and load cell information. This is displayed by a custom graphic user interface (GUI) (fig. 4) and also provides power to run selected software including Arduino IDE, web browser, and Microsoft Excel 2010 simultaneously. Excel is used to write data acquired from the sensors via code. The HUP is equipped with a Bluetooth – Bluesmif module and uses an 8-bit ATmega328 microcontroller.

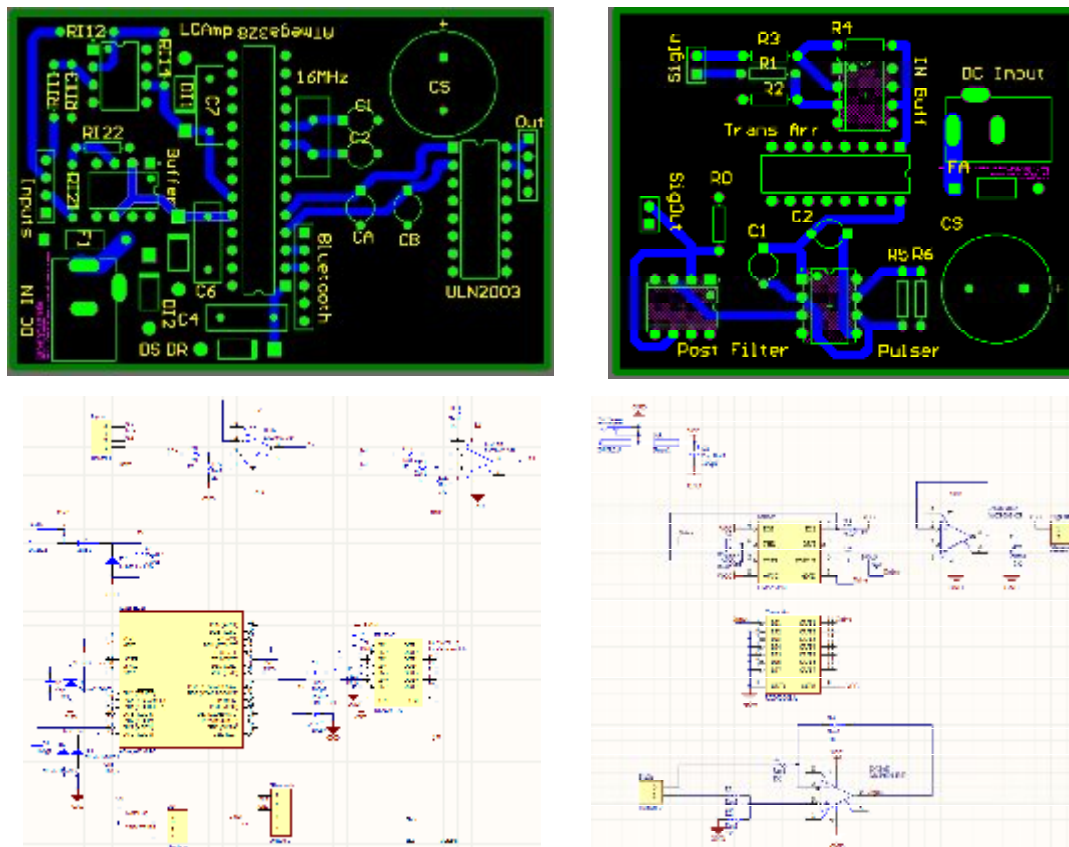


Fig. 3. (a) (Top Left) **Prototype circuit board I (PCB 1)**: manages controller signal, Bluetooth and sensor components; (b) (Bottom Left) Corresponding schematic of PCB 1, (c) (Top Right) **Prototype circuit board II (PCB 2)**: manages pulser, (d) (Bottom Right) Corresponding schematic of PCB 2

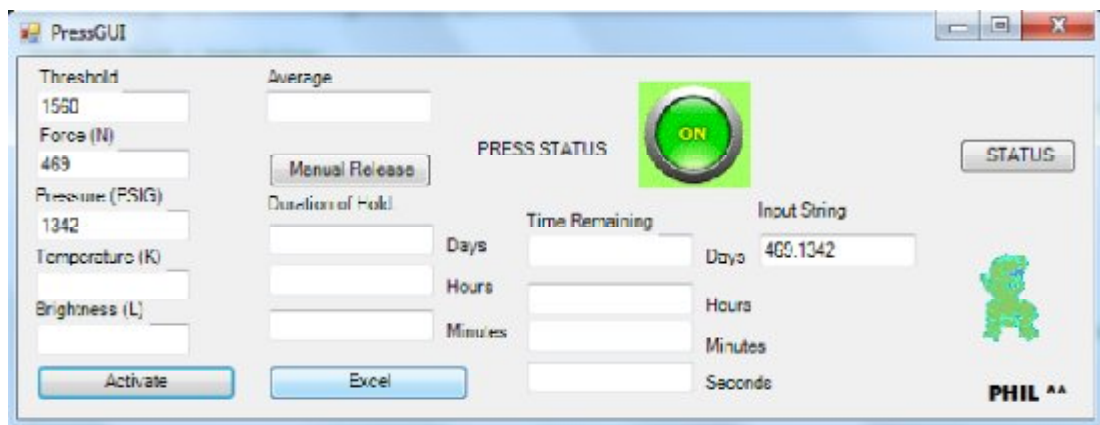


Fig. 4. The display of the graphical user interface (GUI) allows the user to remotely enter their temperature and pressure parameters from a wireless laptop using Bluetooth technology.

## CONCLUSION

In our efforts to design a hot uniaxial press (HUP) to fabricate dense monolithic ceramic pellets under glovebox conditions, we made great progress in completing (1) the mechanical structure of the press, (2) an entire electronic control system and (3) the graphical user interface. Future work includes the fabrication of Tc-99 bearing waste forms [3] as dense monolithic ceramics pellets.



Fig. 5. A 7 x 40 mm-dia. test pellet pressed at 82 MPa controlled by wireless user interface.

## REFERENCES

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