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Combustion and Compaction of Lunar Regolith/ Magnesium Mixtures

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COMBUSTION AND COMPACTION OF LUNAR
REGOLITH/MAGNESIUM MIXTURES

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*A mi Dios,
mis padres
y mis hermanos*

COMBUSTION AND COMPACTION OF LUNAR
REGOLITH/MAGNESIUM MIXTURES

by

ARMANDO DELGADO

THESIS

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ABSTRACT

Feasible techniques that require low energy consumption and reduced costs are of vital importance to enable humans to explore and occupy space. *In-Situ* Resource Utilization (ISRU) is the branch in the Human Exploration Technology that studies the production of consumables and materials using indigenous lunar and planetary resources. ISRU represents an enabling opportunity for establishing long lasting human exploration missions. Construction materials produced *in-situ* are of special interest because of the high payload they represent when launching them from Earth. The *in-situ* production of construction materials that requires low energy input would be advantageous due to the limited energy in exploration missions. Combustible mixtures of lunar regolith with magnesium could be used for the production of construction materials on the Moon and have been demonstrated using JSC-1A lunar regolith simulant; however, the density of the combustion products is not sufficiently high for using them as construction materials. The goals of the present work were to further minimize the content of magnesium and to develop a technology that allowed for the production of dense, and hence strong, structural materials. Both goals were attained by two additional steps to the combustion process of the JSC-1A/Mg system, preheating of initial mixture and compaction of the products after combustion.

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CHAPTER 1

INTRODUCTION

Feasible techniques that require low energy consumption and reduced costs are of vital importance to enable humans to explore and occupy space. A lunar outpost would represent a major accomplishment in space exploration as it would serve as a launching site and as a base that would supply propellants, water, oxygen, and other resources for longer space missions.

Bringing materials, like concrete blocks or steel, from Earth to the Moon to construct a lunar base would be very expensive. Just as an example, in 2012, the cost to put a payload of 6.4 tons in a geosynchronous transfer orbit was 83 million dollars through SpaceX's Falcon Heavy rocket, meaning it costs 13 thousand dollars to put a kilogram into this orbit [1]. Therefore, it is attractive to produce the necessary construction materials in the location where the outpost is desired.

In-Situ Resource Utilization (ISRU) is the branch in the Human Exploration Technology that studies the production of consumables and materials using indigenous lunar and planetary resources. ISRU represents an enabling opportunity for establishing long lasting human exploration missions. Once ISRU technology is applied to exploration missions, risk and cost will decrease and mission feasibility increase. Construction materials produced *in-situ* is of interest because of the high payload they represent when launching them from Earth. The *in-situ* production of construction materials that requires low energy input would be advantageous due to limited energy in exploration missions. The research reported in the present thesis focused on the production of construction materials using combustion synthesis, which provides the advantage of low energy consumption.

1.1 A Lunar Outpost

Space exploration has gained a special interest from the private sector due to the new direction taken by NASA, which aims to enable all public to space. New space exploration companies like Planetary Resources, Shaktel Energy, and Moon Express among others have expressed their commitment on developing the infrastructure needed for sustained space exploration. Also, private sector incentives such as the Google Lunar Xprize [2], worth 30 million of dollars to the first team to send a robot to the Moon by the end of 2015, have caused a positive impact on the public and its interest in the technologies involved in lunar exploration.

Since the beginning of the space race, the Moon has been identified as a primary objective. If a lunar outpost is established, it will represent not only a major accomplishment, but also a first model of what can be done in other planetary locations. For this reason, many have suggested the development of techniques that will process lunar regolith to produce expendable goods. As shown in Table 1, oxygen is an abundant element on lunar regolith. Further, it is known that oxygen is present in the form of silicates, and so reduction processes can be implemented to obtain oxygen, metals, and silicon [3]. Moreover, regolith could be processed to produce structural materials to be used in landing/launching pads, radiation shielding, and even as thermal mass, also known as thermal wadis, that could protect robots/vehicles from the extreme temperature gradients during lunar nights [4].

Table 1. Average composition of lunar regolith [3]

Element	Amount, wt. %
O	42
Si	21
Mg	13
Ca	8
Al	7
Fe	6
Other	3

1.2 Combustion Synthesis

Many alternatives have been proposed to produce structural materials in space, more specifically on the Moon. Some processes proposed go from making lunar regolith concrete to sintering [5]. The construction materials could be produced *in-situ* from regolith by sintering at high temperatures, e.g. using microwave radiation. Heating the regolith to the required high temperatures, however, would require significant energy input. An alternative approach involves combustible mixtures of lunar regolith and energetic materials, i.e. metals. Such process is usually called combustion synthesis or self-propagating high-temperature synthesis (SHS). Upon ignition, exothermic reactions between the metal and regolith cause a combustion front to propagate throughout the green mixture, see Figure 1.1. It has been used for synthesis of numerous ceramics and other compounds. Owing to high temperatures during the process, SHS may produce materials that are sufficiently dense and strong for the construction applications. An important advantage of this method is that a relatively small amount of energy is required for ignition, while the high temperatures during the combustion process are generated by the reaction heat release.

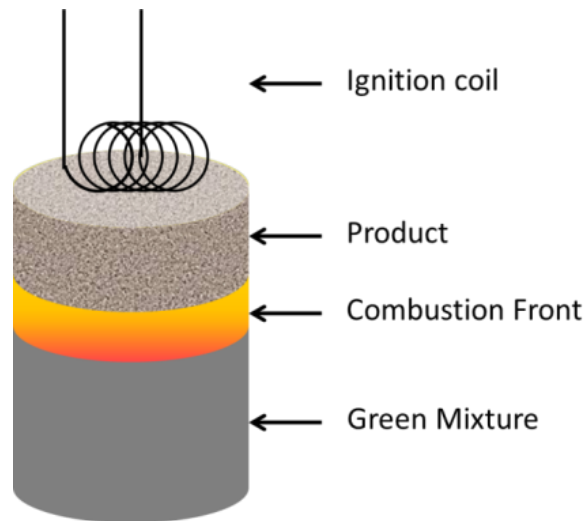


Figure 1.1. SHS Combustion. In the SHS process, energy input is only required for ignition. After ignition, the heat released by the reaction cause self-propagation of the combustion through the remaining green mixture.

1.3 Combustion Synthesis on the Moon

Combustion synthesis using lunar regolith simulant has been experimentally tested. JSC-1A is a commercially available lunar regolith from Orbital Technologies Corporation (ORBITEC). JSC-1A is commonly used in ISRU research. The simulant is obtained after processing mined ashes from a volcanic cinder cone located in the San Francisco volcano field near Flagstaff, Arizona [6]. Table 2 shows the composition of JSC-1A. It is important to note the presence of oxygen and silicates, important for the combustion synthesis process.

Table 2. Mineral composition of JSC-1A lunar regolith simulant [7]

Mineral	Formula	JSC-1A composition, wt. %
Anorthite	$\text{CaAl}_2\text{Si}_2\text{O}_8$	26.48
Albite	$\text{NaAlSi}_3\text{O}_8$	11.35
Orthoclase (K feldspar)	KAlSi_3O_8	0.07
Wollastonite	CaSiO_3	7.77
Enstatite	MgSiO_3	7.38
Ferrosilite	FeSiO_3	4.28
Forsterite	Mg_2SiO_4	9.08
Fayalite	Fe_2SiO_4	3.36
Glass		26.67
MgFeAl Silicate		3.06
Troilite	FeS	0.17
Ilmenite	FeTiO_3	0.11
Calcite	CaCO_3	0.11
Magnetite	Fe_3O_4	0.01
Quartz	SiO_2	0.01
Others		0.07
<i>Total</i>		99.98

Different regolith-based SHS systems have been studied and proven feasible. In 2006, Martirosyan and Luss probed the JSC-1A + Ti + B system [8]. Their system was composed of 40% stoichiometric mixture of titanium and boron and 60 wt% of JSC-1A. In 2010, Virginia Tech studied the JSC-1A + Al system [9]. The system consisted of a mixture of 67 wt% regolith simulant and 33 wt% aluminum ignited through an embedded heating wire in the mixture as seen in Figure 1.2. Lately, a research group from Italy has studied the JSC-1A + FeTiO_3 + Al system where the composition is 30, 45.65 and 24.53 wt%, respectively [10]. Although all of them prove SHS on the Moon to be feasible, a system which is composed of more regolith content is desired.

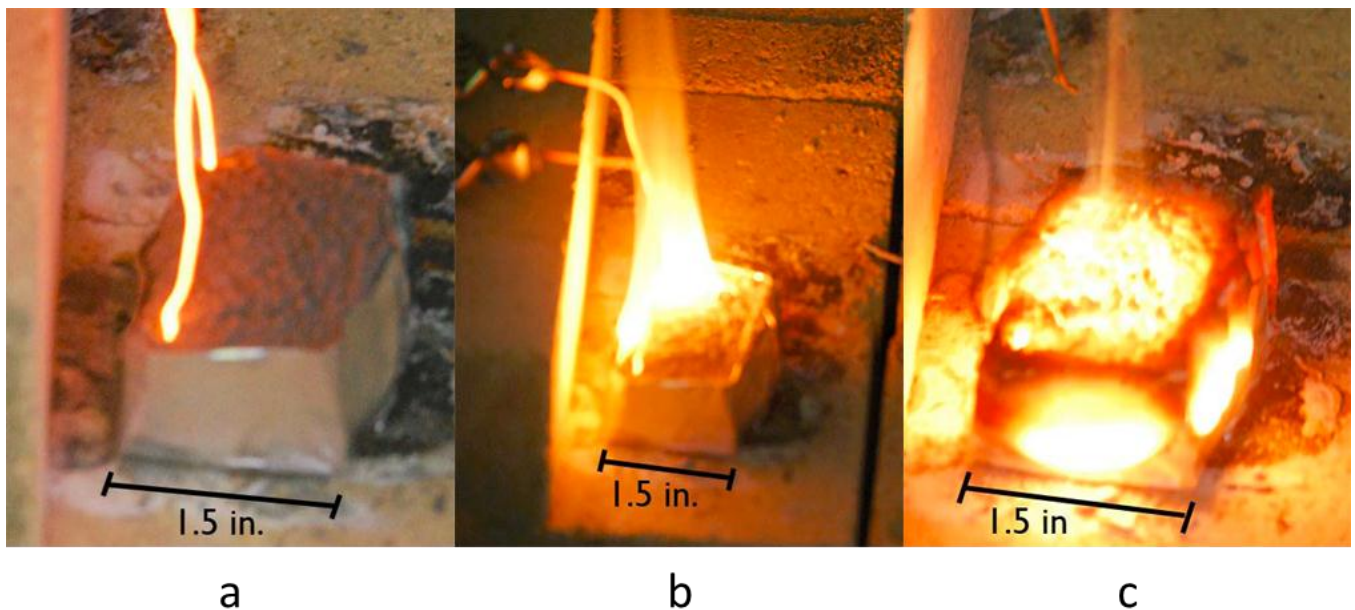
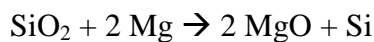


Figure 1.2. JSC-1A + Al system proved by Virginia Tech [9]

Since the creation of the Center for Space Exploration Technology Research in 2009, the University of Texas at El Paso has been conducting research to explore techniques that will enable space exploration to become more feasible. Specifically, the ISRU team under the supervision of Dr. Evgeny Shafirovich investigates the combustion of JSC-1A/Mg mixtures. Previous research conducted by master graduates Christopher White and Francisco Alvarez started the research on SHS of JSC-1A lunar regolith simulant and magnesium. The main reaction driving SHS of the JSC-1A/Mg system is:



Thermodynamic calculations of the adiabatic flame temperatures and combustion products over wide ranges of the mixture ratios showed that magnesium provides higher adiabatic temperatures as compared to aluminum used by Faierson and Corrias et al. Experiments conducted on the combustion of JSC-1A/Mg mixtures, see Figure 1.4, determined the velocity of the combustion front propagation over a cylindrical pellet for different particle sizes of the regolith simulant. The combustion of a thin layer of JSC-1A/Mg mixture (i.e. tile geometry), where the combustion front propagated from the center of the tile, was also demonstrated. For SHS of regolith-based mixtures, aluminum and magnesium could be recycled from used spacecraft, landers, and other structures, as well as from lunar or Martian regolith. Most of the spacecraft debris in orbit is commonly made of alloys composed of aluminum and magnesium [7].

Research was also performed investigating the minimum magnesium content that would be needed for full propagation of the combustion front. SHS findings have shown combustion improvements in combustion temperature and velocity of combustion front. Moore and Feng reported that for many systems particle size and initial temperature play a significant role in SHS [11]. Such improvements in SHS combustion led to minimization of the metal utilized for combustion, i.e. magnesium. This improvement can be understood when comparing high-energy milled powder to roller balled milled powder. In the JSC-1A/Mg system, Álvarez et al. reported spin combustion when using 23 wt% magnesium when using roller-ball-milled JSC-1A, see Figure 1.3. Spin combustion is usually an indicator of combustibility limit [12]. When using a high-energy milled, i.e. small particle size, JSC-1A, the content of magnesium was reduced to 13 wt% [13].

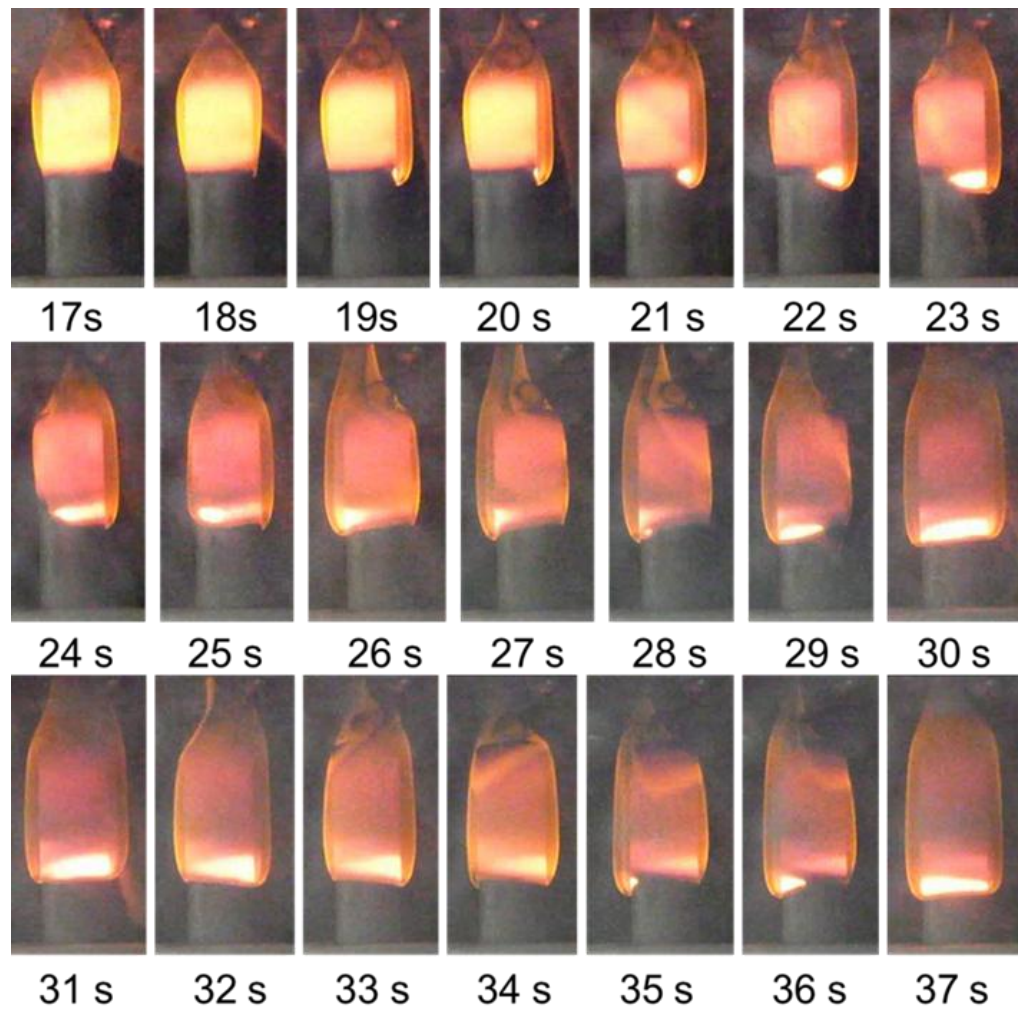


Figure 1.3 Spin combustion observed at 23 wt% magnesium and roller-ball milled JSC-1A (pellet diameter: 12.7 mm) [13]

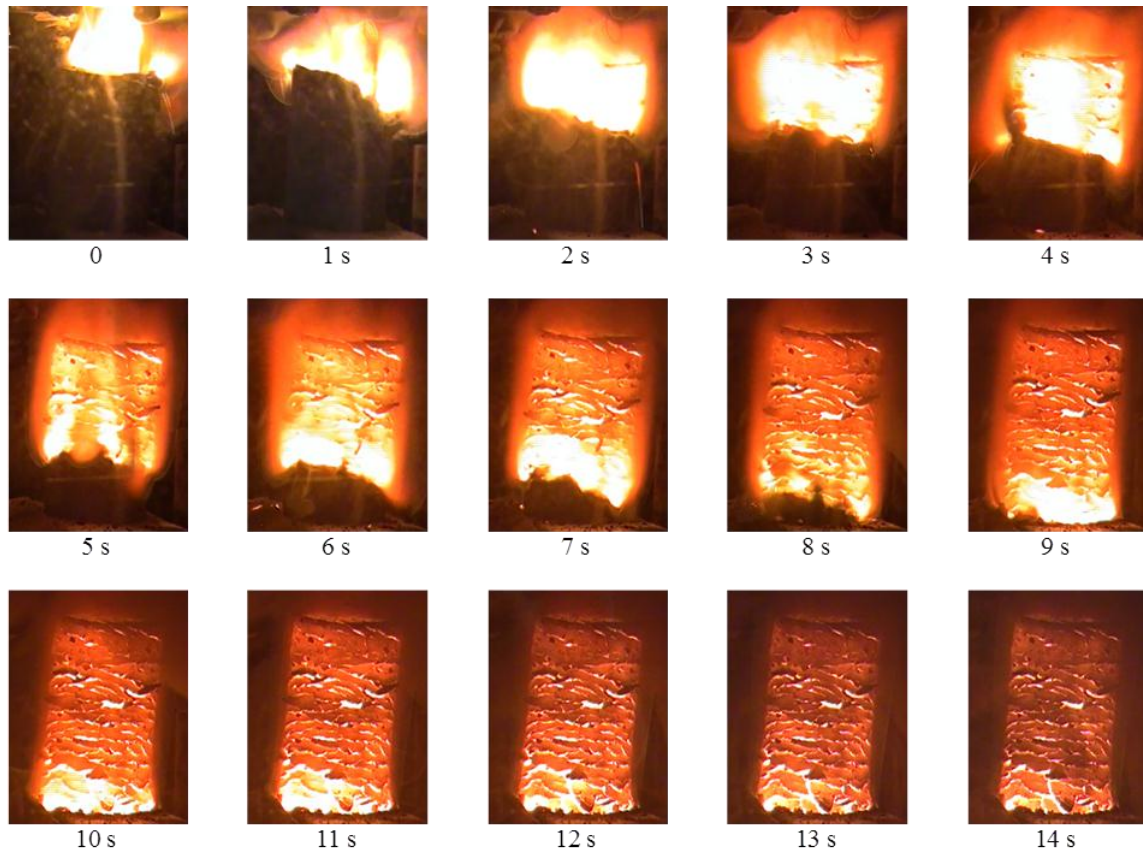


Figure 1.4. Combustion in the mixture of Mg (26 wt%) and JSC-1A milled in the planetary mill (pellet diameter: 25.4 mm) [14].

These experiments revealed, however, that the density of the combustion products is not sufficiently high for using them as construction materials. As noted in Figure 1.5, during combustion, the pellet length increases and numerous cracks are formed as a result of the front propagation pulsations. The products were brittle and vulnerable to break when handling.

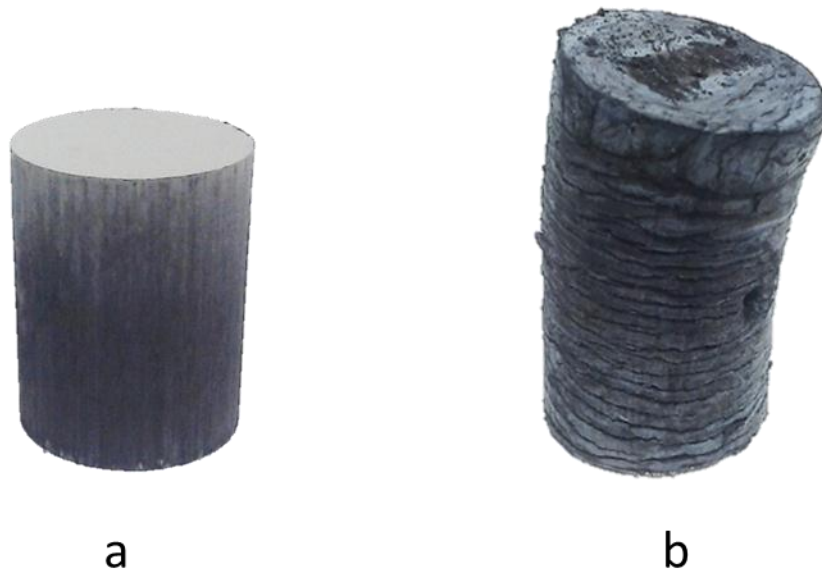


Figure 1.5. SHS green mixture and SHS product. a. Pellet before combustion (green mixture). b. Pellet after combustion (products).

The goals of the present research were to further minimize the content of magnesium and to develop a technology that allows the production of denser and stronger structural materials by combustion of regolith/metal mixtures.

To minimize the magnesium content, a preheating step was added before ignition of the JSC-1A/Mg green mixture. It is common in many SHS systems to raise the initial temperature of the green mixture to increase combustion temperature and combustion front velocity. Different preheated mixtures with low magnesium content were studied.

To produce dense and stronger materials, a technique called SHS compaction was employed. In SHS compaction, the green mixture is located inside some inert powder (e.g., sand) placed under a hydraulic press. The mixture is ignited and after the combustion process, the product mixture is compacted by the press while still at high temperature. This method allows for the production of denser products and items that have complicated shape and can be used with no subsequent treatment. The conducted research involved design and construction of technological equipment for SHS compaction of regolith/magnesium mixtures.

CHAPTER 2

LITERATURE REVIEW

In many SHS systems, it is normal to obtain high porosity products. The JSC-1A/Mg system is no different. Experiments in the laboratory have yielded high porosity products which are also accompanied by low strength. This may be caused by the difference in density of reactants and combustion products, volatile impurities, and also by the solidification of molten products [15]. Thermodynamic calculations of the JSC-1A/Mg system in THERMO software revealed the presence of minor amounts of gas products, seen in Table 3, which contribute to the formation of pores inside the combustion products.

Studies in SHS have demonstrated that many techniques can be implemented in order to reduce porosity of products.

Table 3. Combustion products of JSC-1A/Mg mixture; 26 wt. % Mg; 1 atm. [7]

Formula	Phase	Composition, wt. %
MgO	Solid	43.61
MgAl ₂ O ₄	Solid	17.65
Ca ₂ MgSi ₂ O ₈	Solid	16.04
Si	Solid	10.56
FeSi	Solid	5.82
CaMgSiO ₄	Solid	3.99
Si	Liquid	1.16
Na	Gas	1.03
Mg	Gas	0.10
Na ₂	Gas	0.02
K	Gas	0.01

2.1 SHS Densification Methods

A literature review was conducted to investigate the densification of SHS products. Implementations have been introduced to the SHS process to produce denser combustion products of powder mixtures. It was found, porosity can be controlled by means of either internal or external methods. Internal methods involve control of SHS combustion parameters, e.g. combustion temperature. External methods involve further processing of SHS products, e.g. pressing. Demonstrations show that immediately after reaction of high-temperature SHS, above 1000 K, products can be compacted to obtain non-porous materials and items of complicated shape. It is desired that products are still hot, so that ductility of the material is high. The SHS compaction can be accomplished through different methods such as pressureless densification (sintering), dynamic shockwave, and quasi-isostatic pressing.

2.1.1 Pressureless Densification

High temperatures reached by SHS can cause the formation of a liquid phase in some systems. Researchers have found that by controlling the temperature of SHS systems, i.e. increasing temperature of green mixture, melting of intermetallic compounds can produce liquid phase sintering, and therefore, increase density without the need of applying external pressure. Such process has been confirmed in the Ni + Ti system [16]. Another alternative to pressureless densification is to mill SHS products and then sinter them as a secondary process. Such has been proved in composite powders of Al_2O_3 -TiC and Al_2O_3 -WC [15]. An increase in density can be caused without the need of mechanical work, however high density materials are not obtained from pressureless densification.

2.1.2 Dynamic Shockwave

Dynamic shockwave or explosive compaction involves the use of explosives to improve product properties. Explosive compaction is carried using two main approaches, as seen in Figure 2.1. The first, Figure 2.1.a, consists of a thin-walled cylinder surrounded by explosives, which upon detonation, is reduced causing the compaction of the SHS reaction products. In the second, Figure 2.1.b, compaction of reaction products is achieved through the movement of a massive piston caused by the explosive shockwave. Densities obtained from explosive compaction can reach 97 – 99 % of the theoretical density of the material [17].

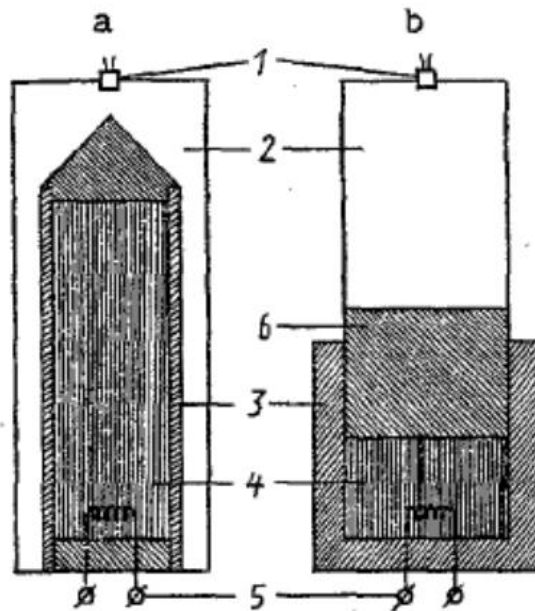


Figure 2.1. SHS dynamic shockwave compaction schemes: 1) electric fuse; 2) explosive; 3) metal container; 4) green mixture; 5) electric ignited; 6) massive piston. [17]

2.1.3 Quasi-Isostatic Pressing

Also known as Ceracon Process, patented process [18], quasi-isostatic pressing is a process that involves uniaxial pressing of SHS products. Pressure is applied to the products, by means of a press, using a pressure-transmitting medium (PTM). This results in closely achieving an isostatic state. In the patented Ceracon process, the PTM was preheated as well as the green mixture, in order to reach an isostatic state and higher combustion temperatures. As seen in Figure 2.2, quasi-isostatic is obtained after PTM is compressed.

The PTM, generally also a heat insulator, is used usually for small-size dense ceramics, while large-size ceramics with complicated shapes can be achieved by using special molds.

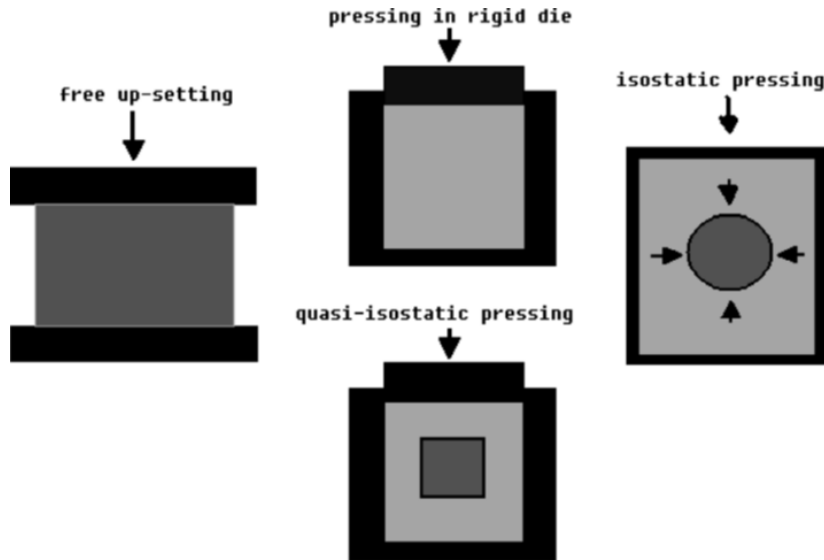
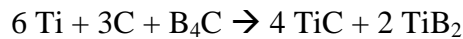
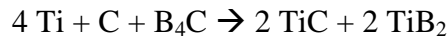
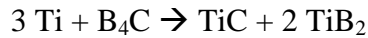


Figure 2.2. Schematics of different densification processes [19].

2.1.3.1 PRODUCTION OF DENSE TiC/TiB₂ WITH HOT QUASI-ISOSTATIC PRESSING

Z. Xinghong et al. used quasi-isostatic pressing to produce dense TiC/TiB₂ ceramics which are attractive for aircraft propulsion systems, space thermal protection, cutting tools, machine tools, etc. Before, complicated processes were used to produce such ceramics which also increased their cost. The SHS process coupled with quasi-isostatic pressing was an alternative that reduced costs and offered a simpler process. In their experiments, they used the following exothermic reactions,



Mixtures of titanium, B₄C, and carbon powders were prepared according to the reactions, and cold pressed to form pellets. The SHS reactants were enclosed in an apparatus filled with PTM which allowed for pressing after combustion, see Figure 2.3. The PTM selected for the quasi-isostatic pressing regime was sand. No details on PTM selection were described. Ignition was carried by a heated coil on one end of the pellet. After combustion, the SHS products were compressed to 160 kPa for 5 seconds.

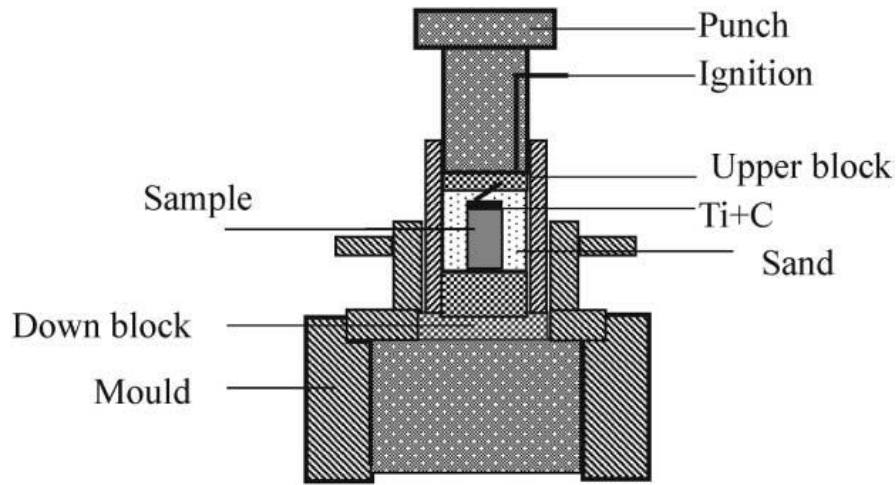


Figure 2.3. Experimental setup of SHS and quasi-isostatic pressing as used by Z. Xinghong et al. [20]

The results showed that relative densities for SHS products were in the range of 90-97%, and when delay time was 5 seconds, the mechanical properties exhibited higher improvement.

2.1.3.2 PRODUCTION OF TiC-BASED FUNCTIONAL GRADED MATERIALS USING QUASI-ISOSTATIC PRESSING

Functional graded materials are of special interest as they offer layered microstructure of two or more phases with different volume fractions. In her thesis, Maria Martinez Pacheco proposed using SHS and quasi-isostatic pressing to develop TiC-NiFe functional graded materials.

Powders of Ti, C, and NiFe were mixed using four different compositions. The composition was varied over the weight percent of NiFe which was 5, 10, 20, and 30. The different mixtures were layered and compacted to form a single pellet with gradient composition.

The layered pellet was placed inside a die submerged in a loosely packed mixture of high purity alumina and graphite powders, which served as the PTM, see Figure 2.4. The pellet was ignited by a booster pellet of stoichiometric mixture of Ti + 2B. Indication of ignition was recognized by the escape of PTM from the die. Pressing delay time was determined according to previous research on the time it took for the combustion to propagate throughout the pellet. The pressure exerted on the pellet was between 150 and 400 MPa, for a period of 5 to 8 seconds.

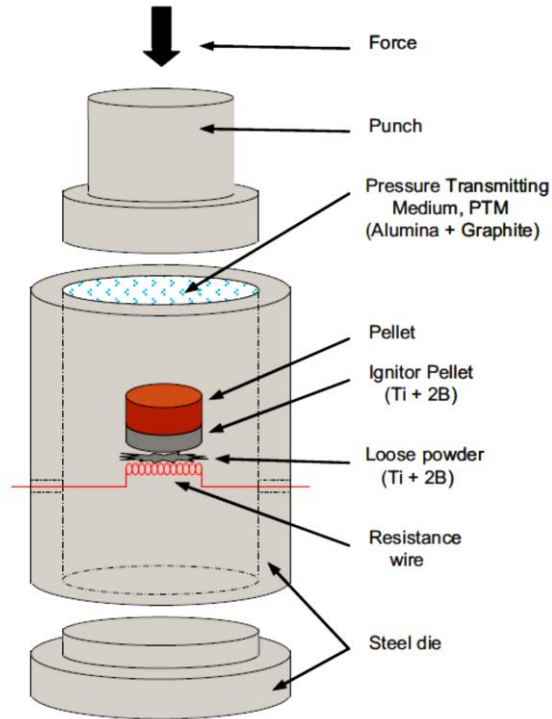


Figure 2.4. Uniaxial pressing SHS compaction schematic used by Maria Martinez Pacheco [21].

Results showed is porosity predominant in layers with higher amount of ceramic content, relative density not greater than 70%. For this reason, pressure distribution in the PTM was investigated using 10 mm by 5 mm pressure sensor films. Overall, examination of the pressure distribution concluded that pressure is exerted more in the horizontal plane rather than isostatic. Also, it was found that there is no strong relation in pressure distribution and the production of TiC-based functional graded materials.

2.2 Summary

Densification techniques for the JSC-1A/Mg systems were considered.

- Pressureless densification may eliminate the need of mechanical work; however the energy input is increased with no fully dense materials as result.
- Although explosive compaction yields greater relative density, the use of explosives in the lab would require much more safety/hazard analysis and special handling, not to mention that in space explosives would need to be transported from Earth.
- Quasi-isostatic pressing is the most viable technique as it can be performed in the lab with simplicity, however it will require further optimization to obtain high density materials.

CHAPTER 3

PREHEATING AND FURTHER MAGNESIUM MINIMIZATION

3.1 Preheating of the JSC-1A/Mg System

Particle size is not the only parameter that can improve the combustibility of SHS systems. Increasing the initial temperature of the reagents can result in an increase of the combustion temperature and front velocity [11]. It is common in SHS systems to preheat the green mixture to a temperature below the ignition temperature, as it eases ignition and results in stability of the combustion. In the JSC-1A/Mg system, preheating of green mixture was studied for different weight percentage compositions.

3.1.1 Experimental Setup

SHS of preheated JSC1-A/Mg mixtures was conducted inside a steel chamber, with a diameter 30 cm and height 40 cm, using a hot plate (Corning, Scholar 170), see Figure 3.1. The hot plate was wired out of the chamber with feedthroughs. A 1/8-inch-thick aluminum plate was placed on top the hot plate to protect from the SHS process. A tungsten coil (Midwest Tungsten Service Inc.) was used for the ignition of the pellet and was connected to a DC power supply (Mastech, HY3050EX). For combustion temperature monitoring, a type C thermocouple (Omega Engineering Inc.) was employed, placed in a hole drilled at half the height of the pellet, perpendicular to the length axis.



Figure 3.1. Hot plate used to preheat pellets.

3.1.2 Sample Preparation

JSC-1A powder, obtained from Orbital Technologies Corporation, was milled in a planetary ball mill (Fritsch Pulverisette 7 premium line). The milling process consisted of pouring 25 grams of JSC-1A in two zirconia-coated grinding bowls with zirconia balls. The milled JSC-1A was mixed with magnesium (-325 mesh, i.e. less than 44 μm , 99.8% pure, AlfaAesar) using a three-dimension inversion kinematics tumbler mixer (Inversina 2L by BioEngineering) for one hour. The obtained JSC-1A/Mg mixtures were compressed into pellets by applying a force of 19.8 kN in a Carver hydraulic press. The mass of the pellets was 25 grams. The relative density was 57% for 8-13 wt% Mg and 59% for 20 and 26 wt% Mg. After compaction, a channel for thermocouple measurements was drilled at mid-length of the pellet to the depth of 10mm.

3.1.3 Experimental Procedure

The sample was placed on the aluminum plate on top the hot plate. Before closing the door of the steel chamber, the hot plate was set on the “high” regime, but was not turned on.

Before ignition was prompted, the chamber was purged three times using a vacuum pump (Fisher Scientific, Maxima C Plus) and argon. The pressure inside the chamber was set close to the 2-psi mark in the pressure gauge (138 mbar) to compensate for the difference of the pressure in the lab (usually 885 mbar) to that of standard ambient pressure. After purging and filling the chamber with argon, the pellet temperature was recorded using a USB DAQ (National Instruments, 9213).

Short after purging, the hot plate was turned on to preheat the sample. Temperature was monitored using the thermocouple inside the sample. In the experiments with preheating, the samples were preheated to a temperature around 100 °C. Once the thermocouples reached such temperature, the hot plate was disconnected and the power supply was turned on for ignition. Experiments with no preheating were conducted for comparison.

3.1.4 Results

Two experiments, with preheating and without preheating, were conducted for the following magnesium content: 7 wt%, 8 wt%, 9 wt%, 10 wt%, 13 wt%, 20 wt% and 26 wt%. As seen in Figure 3.2

and Figure 3.3, temperature of the combustion front is lower for mixtures with less content of magnesium. In both figures, the time at which maximum combustion temperature was observed was selected to be time equal to zero. Generally, combustion temperature of preheated samples is greater than the temperature of non-preheated samples.

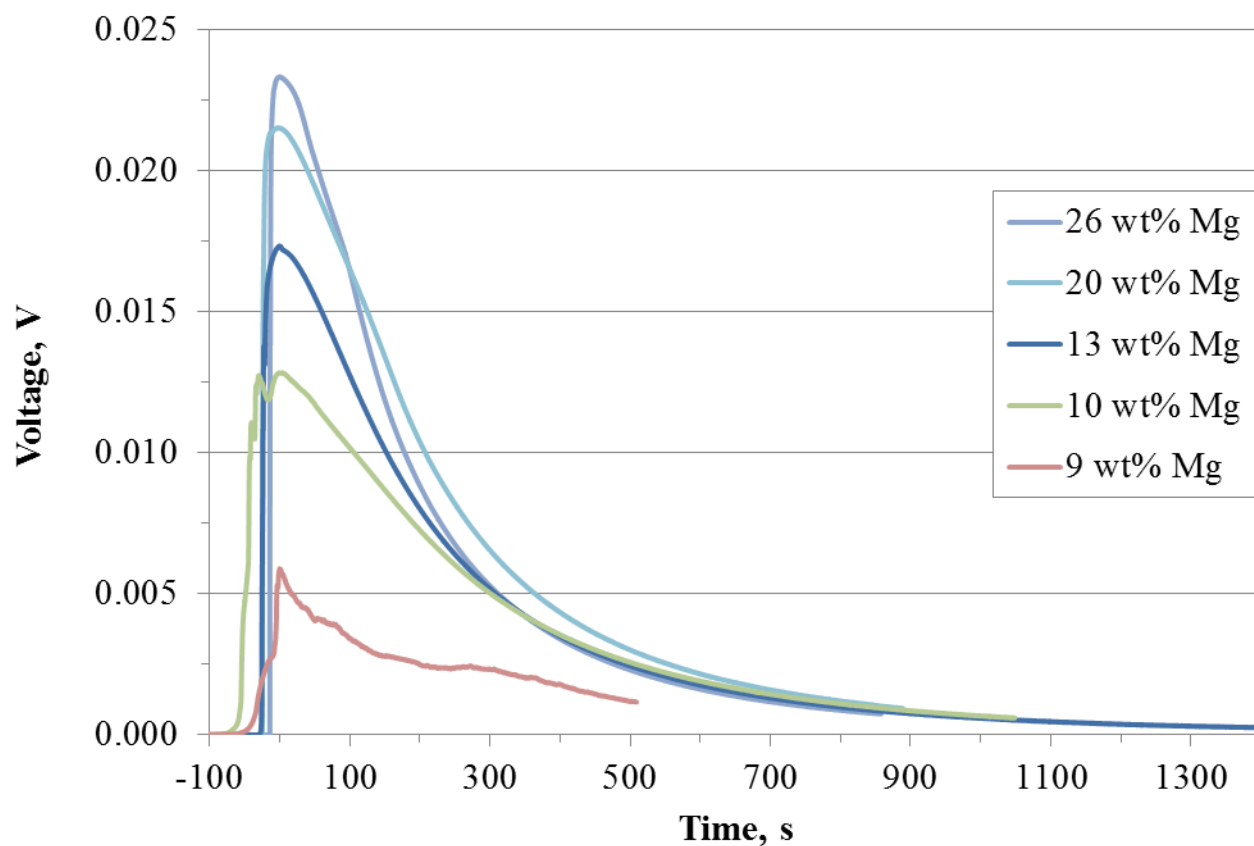


Figure 3.2. Thermocouple signal during combustion with no preheating. Note: 9 wt% front combustion stopped at half the height.

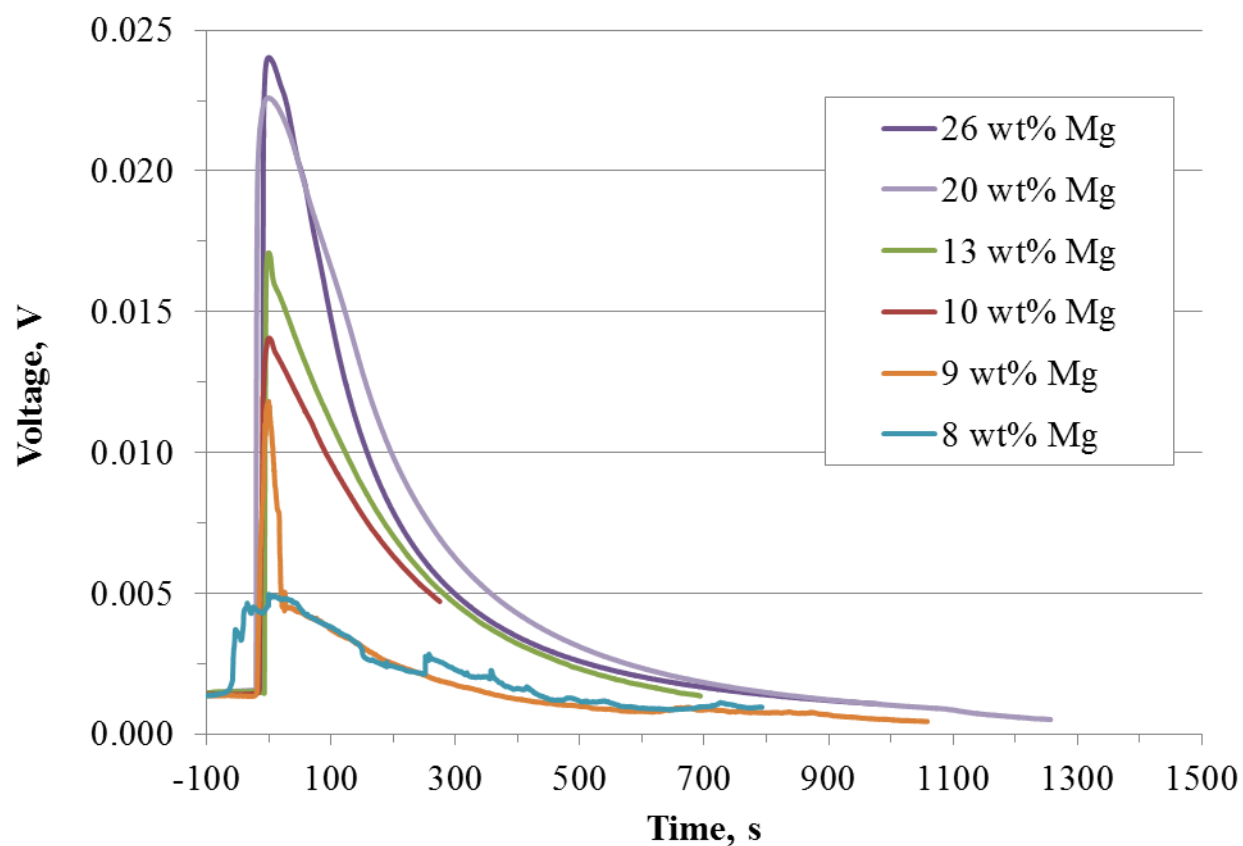


Figure 3.3. Thermocouple signal during combustion of preheated samples (100 deg C).

The minimum content at which full propagation was observed for the preheated case was 8 wt% (Figure 3.4), while for the non-preheated case was 10 wt% (Figure 3.5). As expected, preheating decreased the ignition energy of the JSC-1A/Mg mixtures.

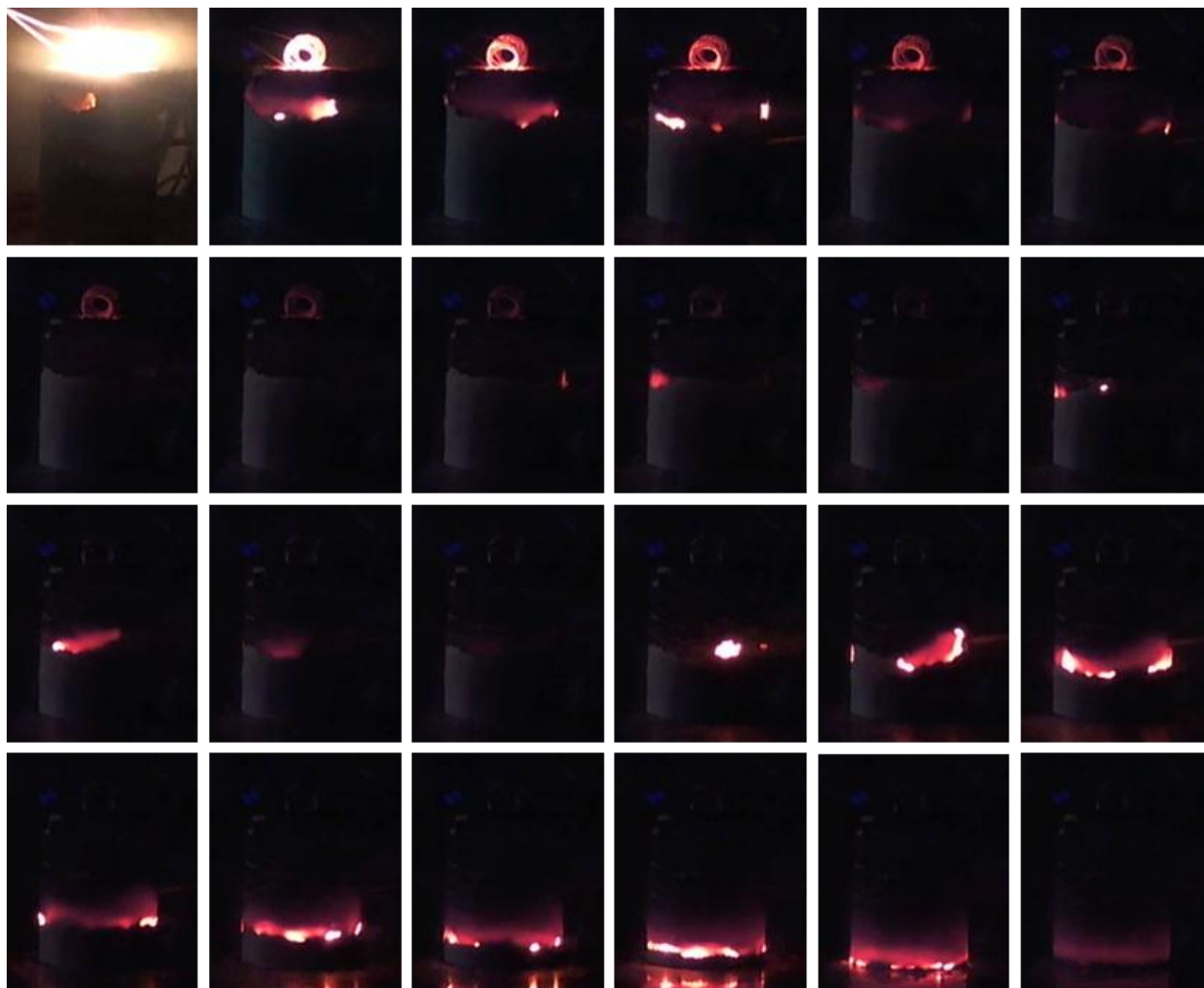


Figure 3.4. Unsteady combustion of JSC-1A/Mg mixture with 8 wt% Mg after preheating to 100 °C. Time interval between frames is 5 seconds.

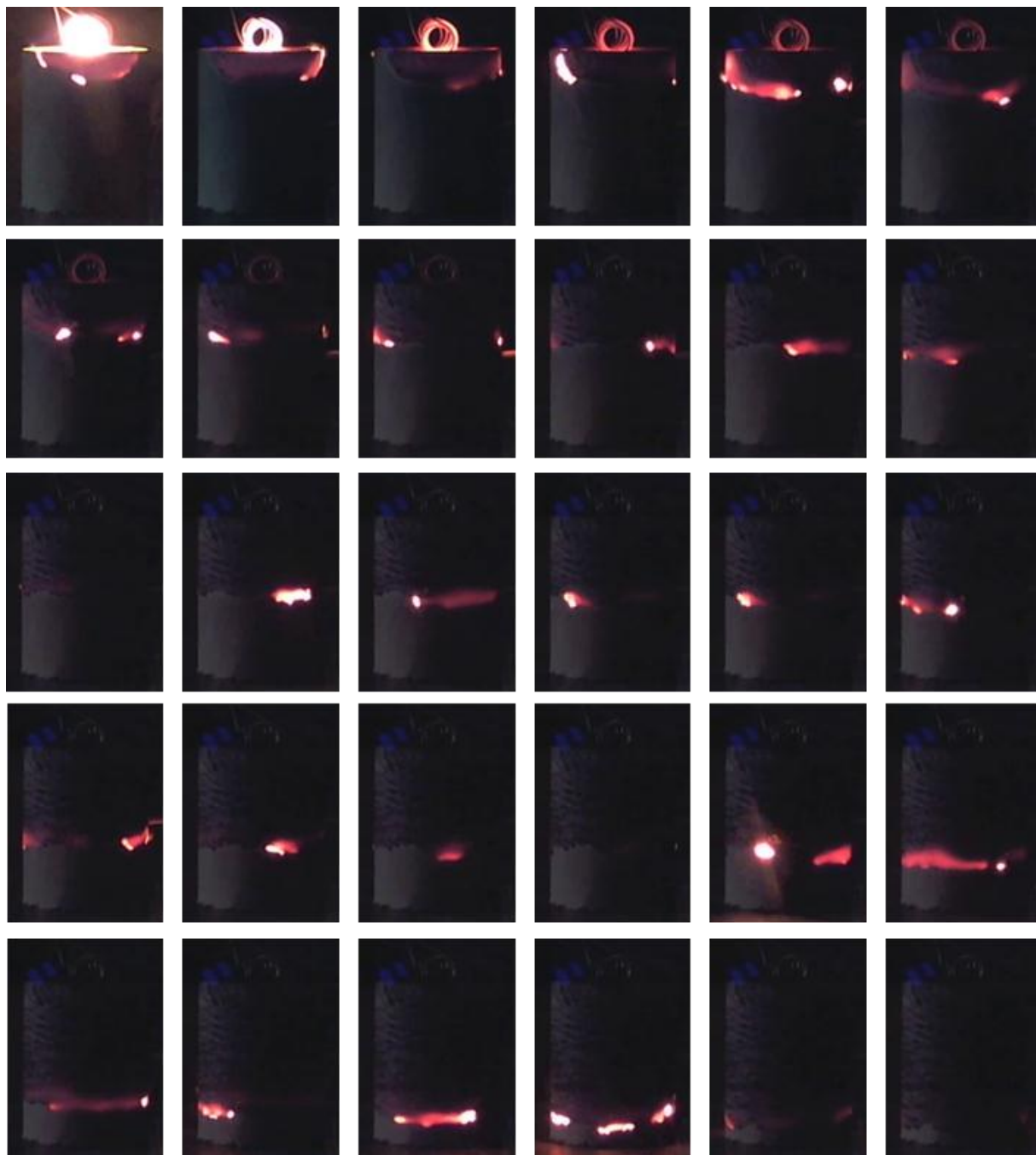


Figure 3.5. Unsteady combustion of JSC-1A/Mg mixture with 10 wt. % Mg without preheating. Time interval between frames is 5 seconds.

Figure 3.6 shows the maximum temperatures obtained for all conducted experiments. It is seen that the maximum temperatures during combustion of mixtures with 13 to 26 wt% Mg was reasonably close to the calculated adiabatic flame temperatures for preheated samples [7].

Unsteady combustion was observed for non-preheated pellets with less than 13 wt% Mg, while for preheated samples it was observed at less than 10 wt% Mg. The front velocity was always higher for preheated samples, see Figure 3.7.

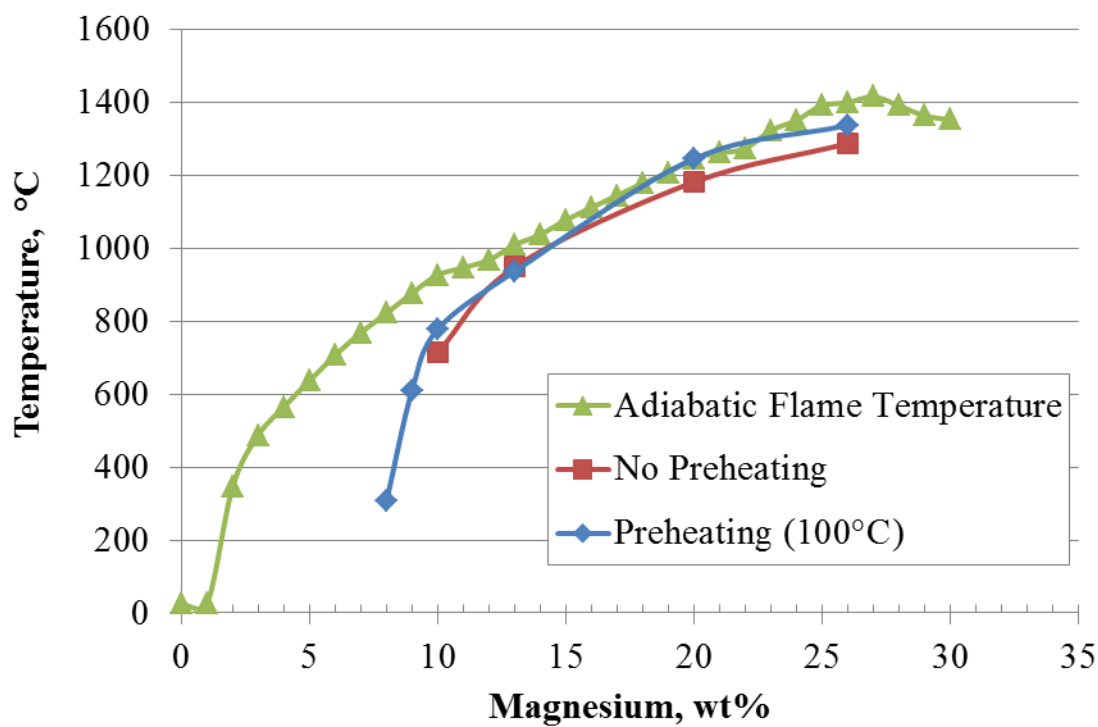


Figure 3.6. Maximum temperatures measured during combustion of JSC-1A/Mg mixtures and adiabatic flame temperatures calculated [7] for these mixtures.

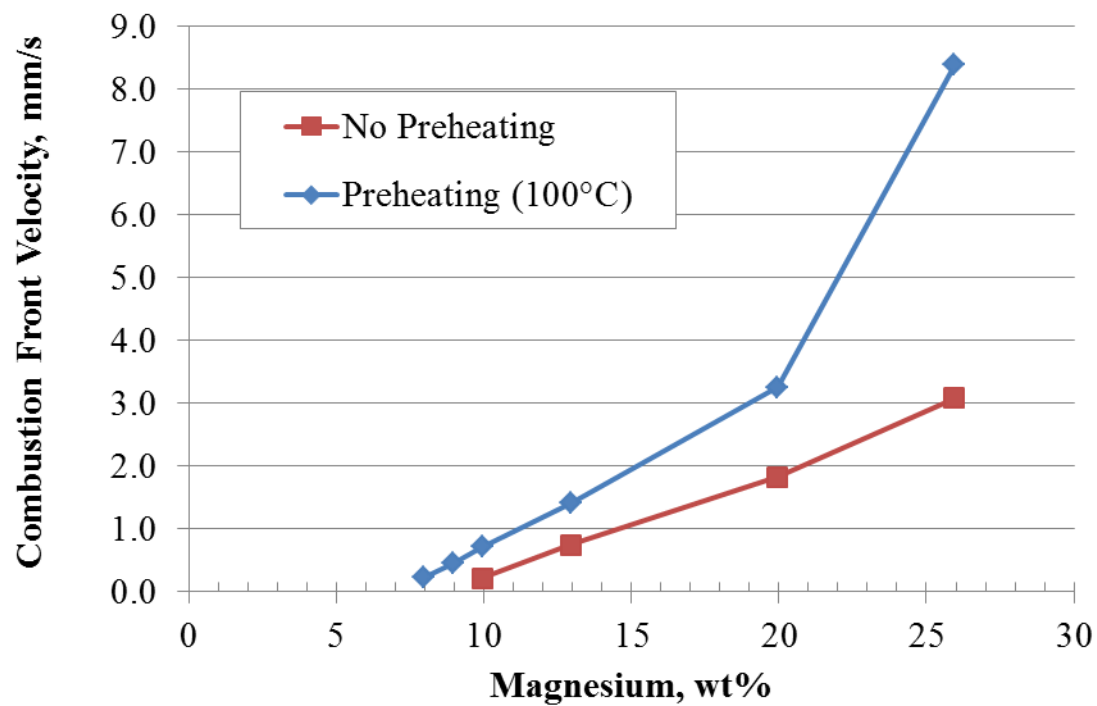


Figure 3.7. Combustion front velocities for JSC-1A/Mg mixtures.

3.2 Preheated JSC-1A/Al system

In the past, research was conducted at UTEP to combust JSC-1A/Al mixtures with SHS [22]. The goal was to conduct experiments to compare to the research performed in Virginia Tech [9]. Even though many experiments were performed, SHS combustion was not achieved. The main factor to prevent the combustion was the low temperature of the green mixture, as compared to preheated temperature of the green mixtures in Virginia Tech [9].

An experiment was performed to check the possibility of igniting and fully combust JSC-1A/Al mixture. The maximum adiabatic flame temperature of the JSC-1A/Al system is attained at 23 wt% Al [7]. The sample preparation and experimental procedure was the same as for Mg-based samples. The preheating temperature was 100 °C.

Ignition was not achieved even when increasing the pellet temperature to 100 °C. The current and voltage were increased to the maximum values, but no ignition occurred.

3.3 Conclusions

The initial temperature of JSC-1A/Mg system was studied to minimize the content of magnesium. Experiments were conducted where a hot plate was used to preheat pellets of different JSC-1A/Mg concentrations to 100°C before ignition. Experiments showed preheating of samples resulted in an increase in combustion temperature and combustion front velocity which provided more stability to the combustion process. Also, it was found that magnesium content can be lowered to 8 wt% when preheated to 100°C.

An experiment of JSC-1A/Al was conducted by preheating the mixture to 100°C however ignition was not obtained.

CHAPTER 4

COMBUSTION EXPERIMENTS IN DIFFERENT ENVIRONMENTS

As discussed in chapter 2, initial temperature of the green mixture plays an important role in the stability of SHS combustion. Moreover, thermal conductivity of the environment/surroundings will affect the combustion process. Low conductivity of the environment will result in smaller heat loss and reliable combustion propagation [21]. Because of this, it was important to determine the effect of the environment in the JSC-1A/Mg system since SHS compaction requires a pressure transmitting medium. The following sections describe the experimental studies of the influence of the environment in the JSC-1A/Mg system during and after combustion.

4.1 Experimental Setup

All experiments were performed inside the aforementioned steel chamber. The chamber was connected to a compressed argon cylinder and a vacuum pump (Fisher Scientific). During the experiment, the pellet was ignited at the top by a 0.43 mm-diameter tungsten wire coil (Midwest Tungsten Service Inc.) connected to a DC power supply (Mastech). Temperature monitoring and recording was accomplished using USB DAQ (National Instruments).

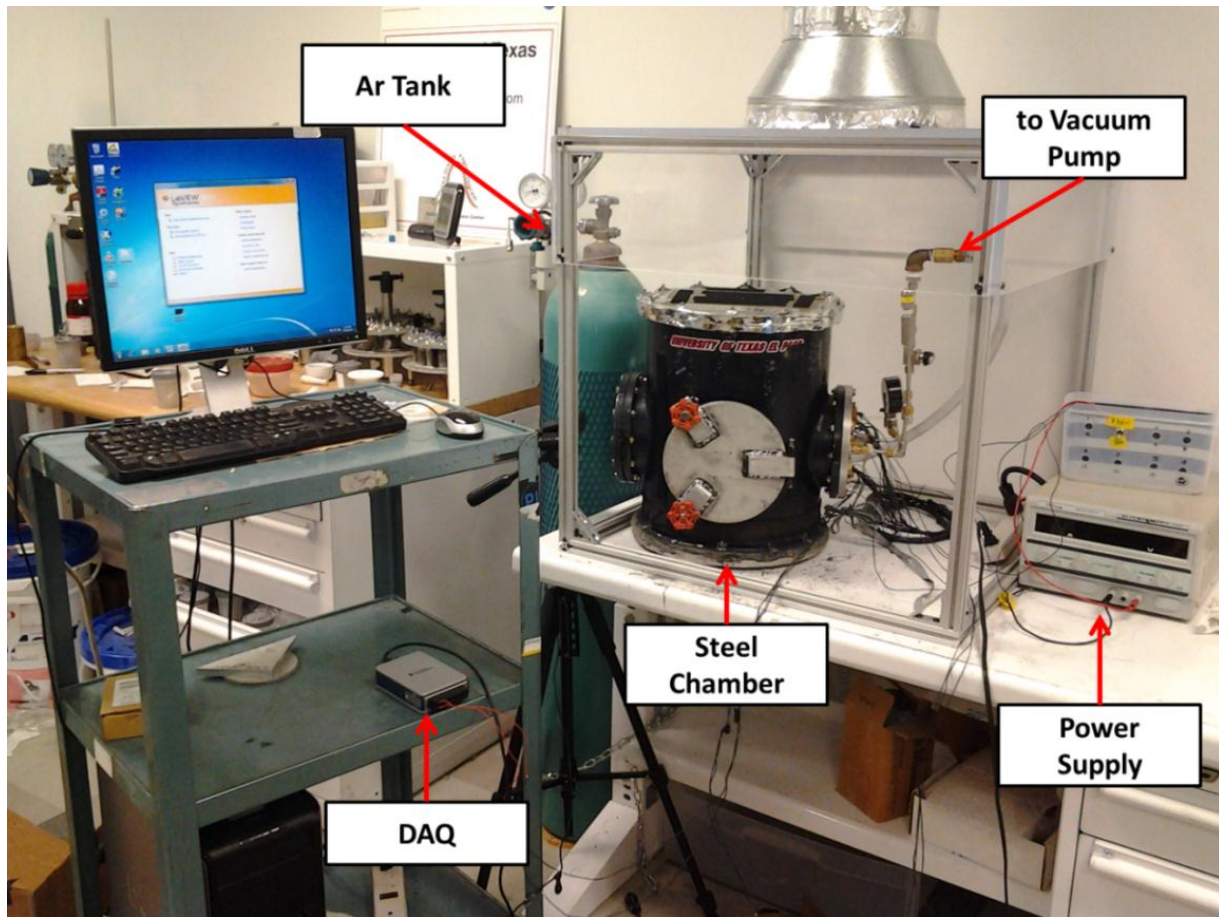


Figure 4.1. Experimental setup used for investigating the effect of environment on the combustion of JSC-1A/Mg mixtures.

4.2 Sample Preparation

To prepare the samples, JSC-1A lunar regolith simulant was treated in the planetary ball mill as described in Section 3.1.2 and mixed with magnesium powder in Inversina mixer for one hour. The composition of the powder mixtures was 74 wt% JSC-1A and 26 wt% Mg. After mixing, the powders were compacted using a 25.4-mm-diameter pellet die. 25 grams were poured in the die to form one pellet. The powders were compressed using a standard manual bench top laboratory press (Carver). The pressing force was 19.6 kN for all pellets. After compaction, pellets had a height of 32-33 mm. Pellets were wrapped with a 3-mm-thick ceramic fiber insulator (Fiberfrax) to minimize heat loss.

4.3 Experimental Procedure

A thermocouple in a two-channel ceramic insulator was inserted into each pellet to measure the temperature during combustion and cooling. Two types of thermocouples were used: W/Re5%-WRe26% (Omega Engineering, type C) and Chromel-Alumel (Omega Engineering, type K). The thermocouples were connected to the DAQ. The pellets were submerged in silica powder, located in an aluminum can, see Figure 4.2.

The chamber was purged three times using the vacuum pump and argon. The purging cycle consisted in pulling the most vacuum usually 846.5 mbar (25 in. Hg mark in pressure gauge) and then filling with argon gas to laboratory pressure (zero in the pressure gauge). After purging and filling the chamber, the pellet temperature was recorded using the DAQ system and LabView software.

During the experiment, the pellet was ignited at the top by a Nichrome wire. The experiments were conducted in argon inside the chamber at 90 kPa.

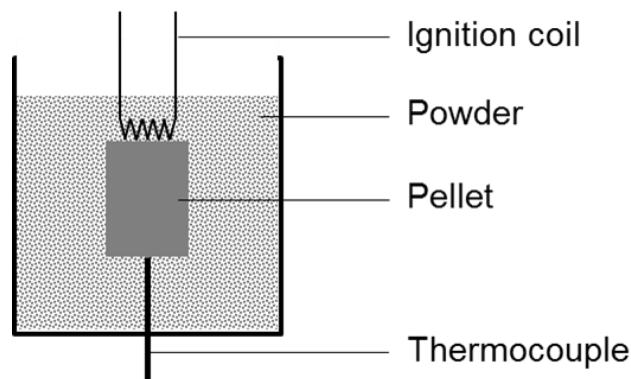


Figure 4.2. Illustration of the pellet setup for experiments analyzing heat loss effect.

4.4 Results

Experiments were successful on the basis that combustion front propagated throughout the entire pellet for all prepared samples. SHS combustion was complete for the different surroundings and thermocouples recorded temperature data.

Unexpectedly, the use of silica as the surrounding medium resulted in the highest strength of the obtained materials. In contrast with the products obtained with no surrounding powder, these materials

could not be easily broken (it was possible to cut them using a saw). Figure 4.3 shows a photograph of the cross-section of the combustion products obtained in silica environment.



Figure 4.3. Cross-section of the product obtained after combustion of JSC-1A/Mg pellet submerged in silica.

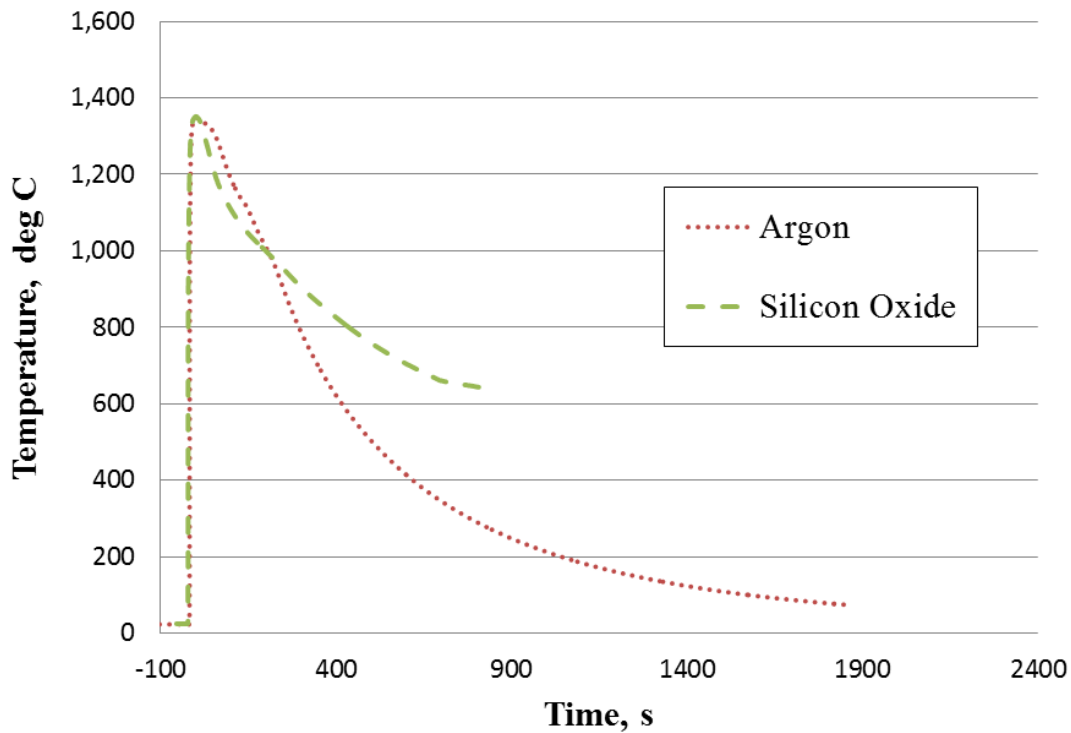


Figure 4.4. Temperature of JSC-1A/Mg combustion in argon and silicon oxide environments.

Figure 4.4 shows typical results of temperature measurements during combustion and subsequent cooling of the mixture pellets. The maximum temperature was 1350-1400°C independently on the environment. It is seen that the use of silica leads to a higher cooling rate immediately after combustion, but the effect becomes the opposite at temperatures lower than 1000°C.

Additional experiments were performed to analyze the velocity of combustion front propagation. To obtain such measurements, two thermocouples were placed 15 mm apart inside the pellet. Velocity was then calculated taking into consideration the time at which each thermocouple reported an increase in temperature due to the combustion front.

Solid combustion products were analyzed using X-ray diffraction analysis (Bruker D8 Discover XRD). Figure 4.5 shows XRD patterns of the products. The highest peaks in both patterns correspond to MgO. The observed differences in other peaks indicate that submerging the sample in silica slightly changes the composition of the combustion products, which include Si and complex oxides of Al, Mg, and Si.

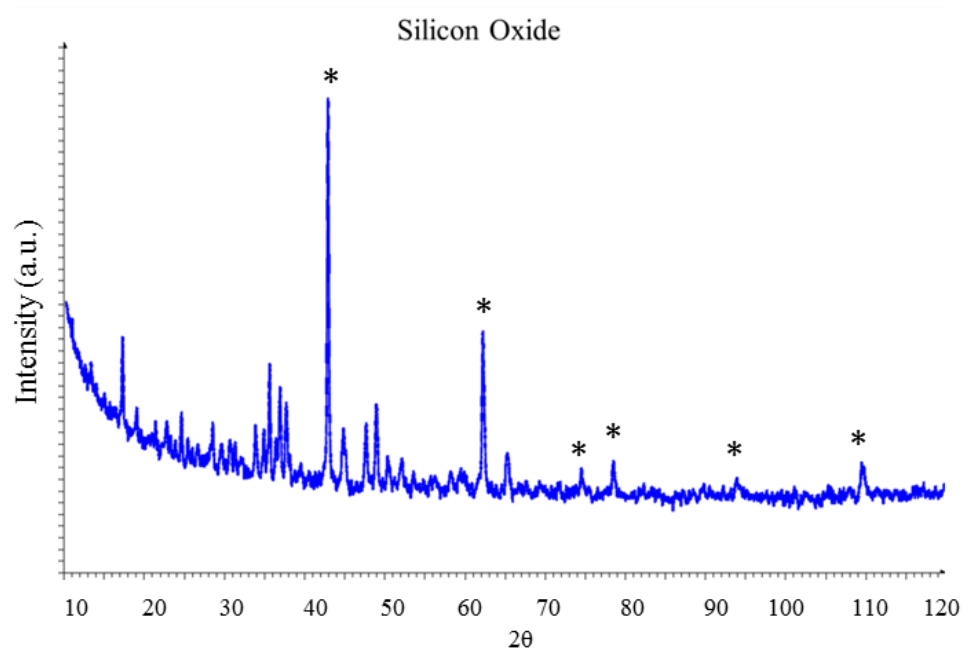
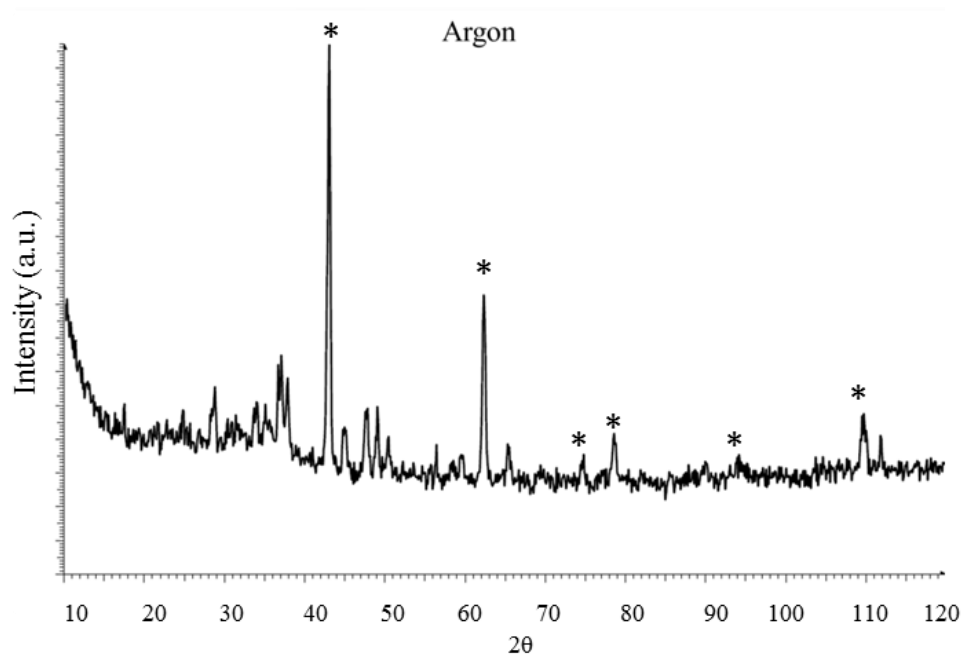


Figure 4.5. XRD patterns of products obtained in argon (top) and silica (bottom) environments. Asterisks indicate MgO peaks.

Table 4. Velocities of combustion front for different environments

Environment	Velocity, mm/s
Argon	4.1
Silicon	4.5

4.5 Compression Test

Compression test was conducted to measure the strength of the obtained products using compression test standards for ceramic materials as reference [23]. The samples were grinded to flatten the surfaces. Samples obtained during combustion in argon, i.e. with no surrounding powder, were not sufficiently strong to withstand the grinding forces. After preparation, the sample obtained by combustion of the pellet submerged in silica was subjected to a compression load using INSTRON 8801 testing machine. The initial length of the sample was 22.8 mm. The machine was setup so that the top fixture displaced at a rate of 1 mm/min. Figure 4.6 shows the load profile over time. The maximum load carried by the sample, 800 N, corresponds to the stress of 1.6 MPa.

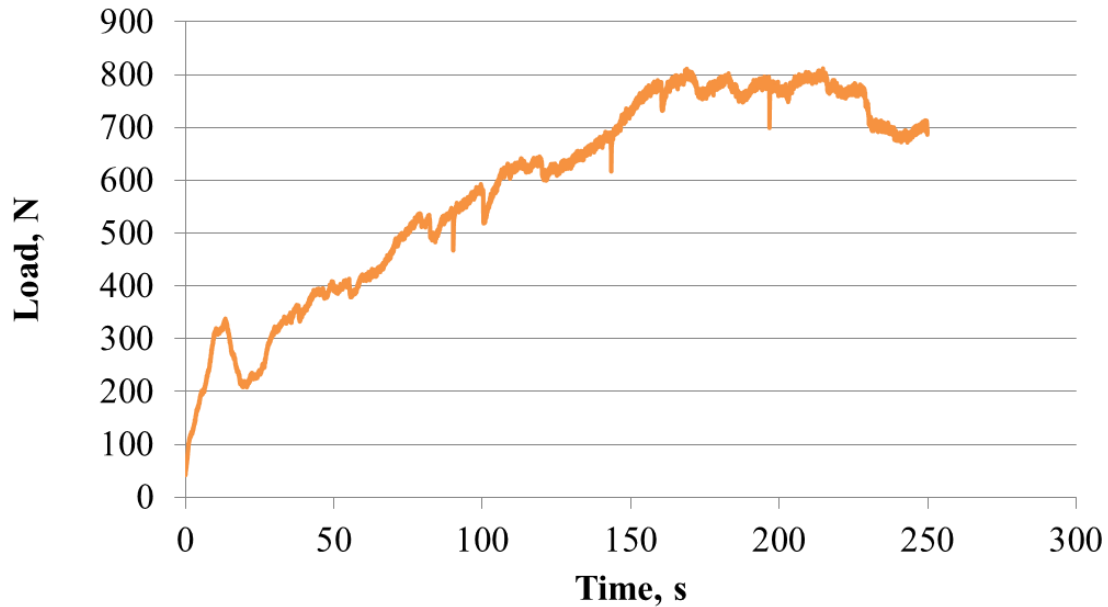


Figure 4.6. Time variation of the compression load.

4.6 Conclusions

Experiments were conducted to investigate the effect of the environment on the combustion of JSC-1A/Mg mixtures. Argon and silica were the environments at which combustion took place. Temperature of the pellet was monitored and recorded during and after combustion.

Submerging the pellets in silica during combustion resulted in a significantly increase in the strength of the products. Cooling rate was the main difference between argon and silica, the former being lower. XRD analysis revealed some changes in product composition. Only products obtained in silica environment were able to sustain compression test preparation; argon samples were not tested. Compression strength obtained for the silica samples was 1.6 MPa. Additional studies are required to explain the observed high strength of the products obtained during combustion of JSC-1A/Mg pellets submerged in silica.

CHAPTER 5

SHS COMPACTION OF JSC-1A/MG MIXTURES

As mention before, SHS compaction allows for the development of dense, nonporous materials. Just after combustion of the green mixture, pressure is applied while products are still hot. Since mechanical equipment was not available in the laboratory for conducting such process, a quasi-isostatic pressing apparatus was developed.

5.1 Design and Manufacture of SHS Compaction Apparatus

The apparatus consist of three pieces: die, punch, and base. The SHS apparatus was design in order to satisfy the following criteria:

Constrains

- 15 cm x 15 cm work area
- Maximum force of the hydraulic press is 12 metric tons.

Requirements

- Pressure exerted on the hot products is a parameter that must be investigated. It was desired to conduct experiments at different loads.
- Withstand up to 10 metric tons with a positive margin of safety.
- Contain 25.4 mm diameter and 32 mm long pellets. Larger diameter pellets may be used in the future, e.g. tile geometry.
- Ignition input
- Thermocouple input

5.1.1 Dimension Constraints and Structural Requirements

The design was constrained to the working area of the available hydraulic press (Carver). The maximum dimensions of the die needed to be 15 cm by 15 cm and 45 cm high. The minimum inner diameter was 2.54 cm which is the sample size of all experiments performed in this research. Also, since future research will require SHS compaction, a larger inner diameter was desired.

Given such constraints and requirements, calculations were made to analyze different size configurations. Initial calculations assumed the die would act as a thick-walled pressure vessel, see Figure 5.1. Equations 1-3 were employed to evaluate the stresses acting on the die [24]. As noticed, the maximum stress is σ_θ at the inside surface. It was assumed that the pressure inside the die, P_i , was caused by the force of the press acting on the die punch. For safety purposes it was set that the design needed to hold a safety factor of 2. Figure 5.2 shows the calculated tangential stress load for different size configurations of the die assuming a constant force of 98 kN (internal pressure is different for every inner radius).

$$\sigma_r = \frac{r_i^2 P_i - r_o^2 P_o}{(r_o^2 - r_i^2)} - \frac{(P_i - P_o) r_i^2 r_o^2}{(r_o^2 - r_i^2) r^2} \quad \text{Eqn. 1}$$

$$\sigma_\theta = \frac{r_i^2 P_i - r_o^2 P_o}{(r_o^2 - r_i^2)} + \frac{(P_i - P_o) r_i^2 r_o^2}{(r_o^2 - r_i^2) r^2} \quad \text{Eqn. 2}$$

$$\sigma_z = 0 \quad \text{Eqn. 3}$$

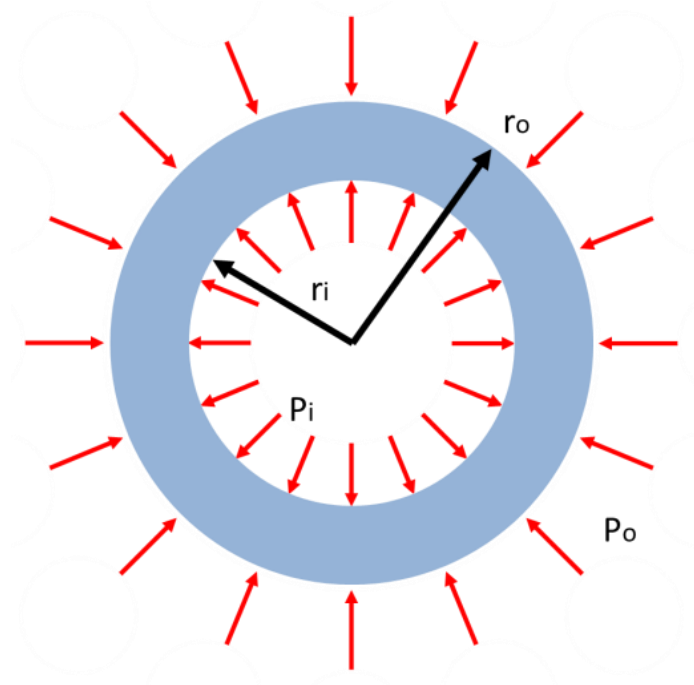


Figure 5.1. Thick-walled cylinder scheme

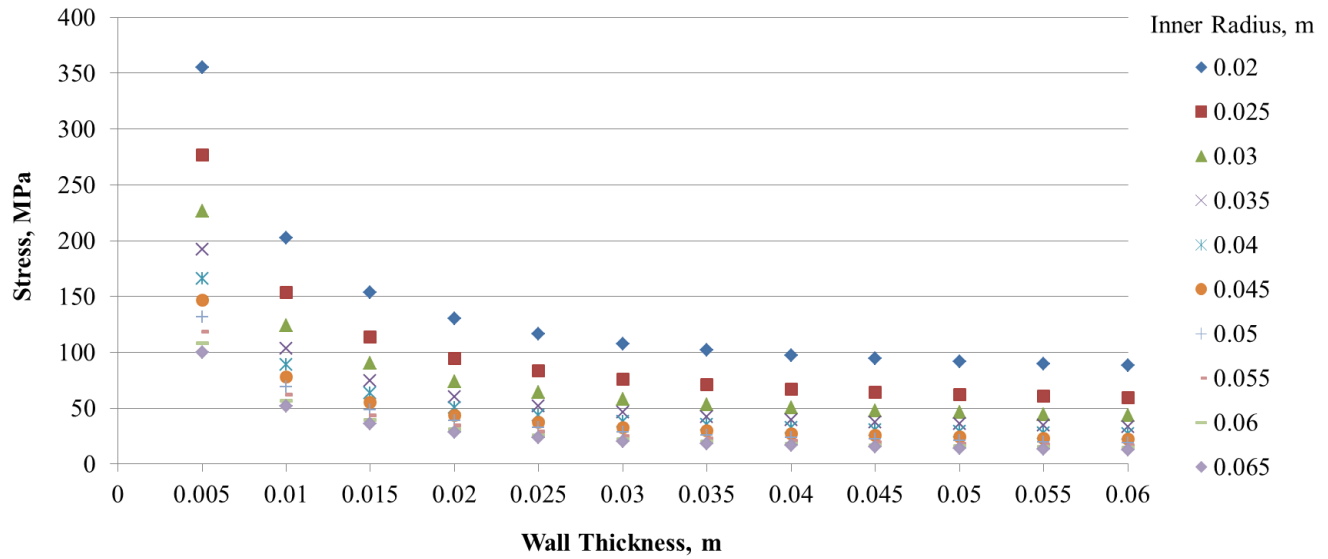


Figure 5.2. Stress calculations for different size configuration of the cylindrical die with a force of 10 metric tons.

The selection was made for the 3.5 cm inner radius and 2.5 cm wall thickness since such configuration allowed testing for larger size of pellet diameters, ensuring low effect of external friction [25] [26]. The selection also took into consideration the accessibility for the user to place ignition wire and thermocouple with no difficulty. The calculated highest stress created by the 10-ton compaction process at such configuration is 37.1 MPa.

Dies are generally made of hardened steel. Reference books were used to design the die. The selection should be based on material's strength, toughness, hardness, machinability and cost [27] [28]. Tool steel A2 was selected as it provides wear resistance, increased toughness, machinability, and was also readily available. Tool steel A2 was heat treated to achieve hardness over 58 HRC.

The margin of safety can be calculated by taking in consideration the stress caused by applying ten tons to the die and taking into consideration material yield strength, see Equation 4 [29]. The safety factor was designated to be 2, as no test was performed to confirm the maximum stress that the die could withstand. The yield strength of 58 HRC hardened A2 tool steel was selected to be 1200 MPa according

to the references [30]. The margin of safety calculated for the design was 16, which is positive and complies with safety design parameters.

$$\text{Margin of Safety} = \frac{\text{Yield Strength}}{(\text{Maximum Stress})(\text{Safety Factor})} - 1 \quad \text{Eqn. 4}$$

5.1.2 Ignition Source and Thermocouple Requirements

Different design configurations were considered, see Figure 5.3. In a first design, it was suggested to put two holes (one through hole, perpendicular to length axis) for the ignition wire and thermocouples.

Of important consideration were the gases produced by impurities in the reaction which cause porosity and may impose a safety hazard for the user and integrity of the lab equipment. It was noted by Martinez Pacheco [21] and Xinghong [20] that SHS compaction dies are designed to allow for the expulsion of such gases. Relief pressure features were suggested, such as drilling of venting holes and making the punch smaller than the orifice of the die. In a SHS compaction in vacuum system, vent holes that are connected to a vacuum pump while SHS combustion and compaction is taking place [31]. Designs were made suggesting that the apparatus base or punch would have a through hole enabling the escape of impurities through them, however it was noted that problems could arise from obstruction of holes caused by hydraulic press bases.

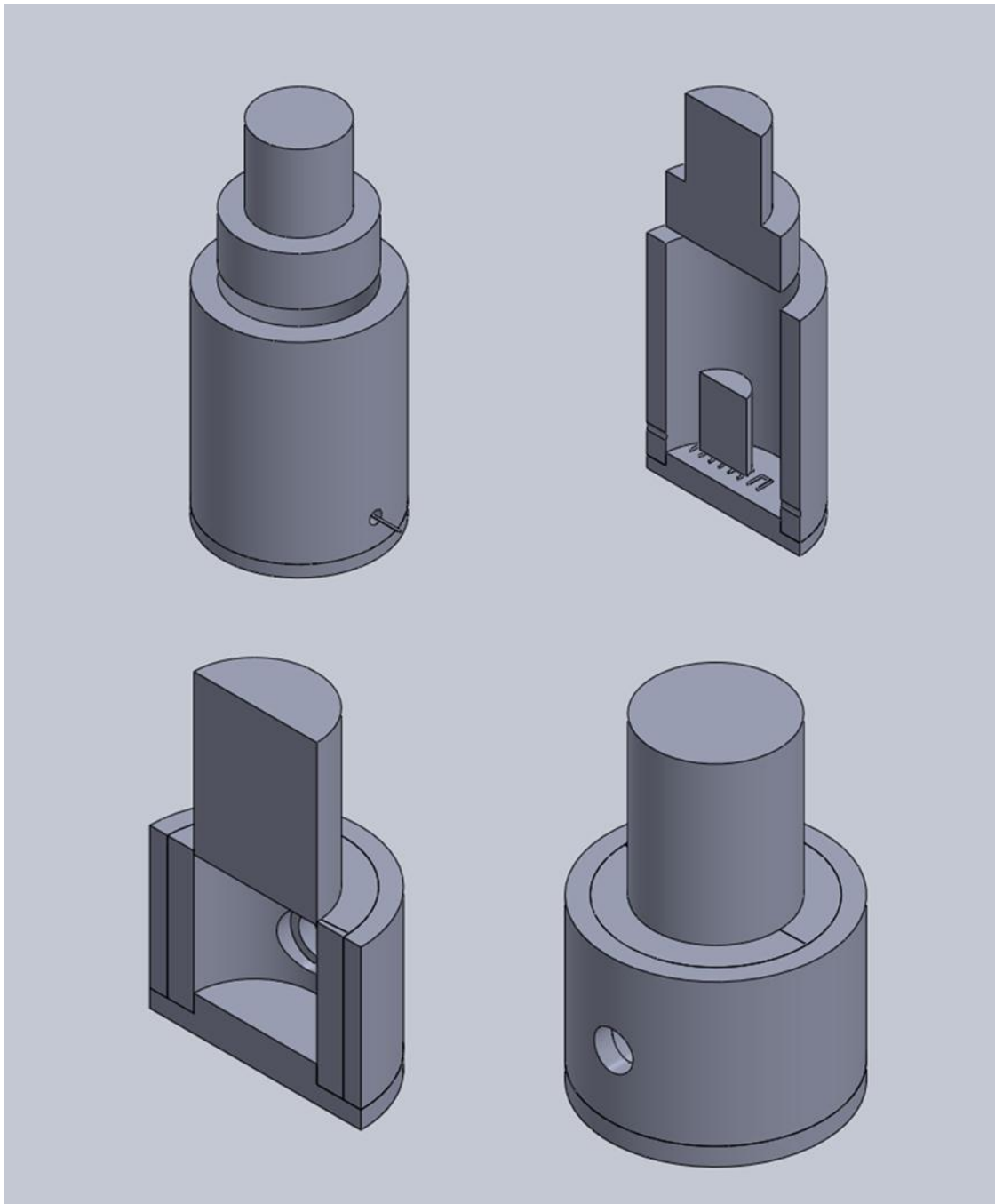


Figure 5.3. Preliminary CAD designs of the SHS compaction apparatus.

5.1.3 Final Design

The final design was the result and iteration of all previous design options. As mentioned, the dimensions of the die were 3.5 cm inner radius and 2.5 cm wall thickness. 6 holes were made into the

die. The bottom holes were envisioned for ignition wire. The remaining four holes were design for either thermocouples or for gas escape, see Figure 5.4.

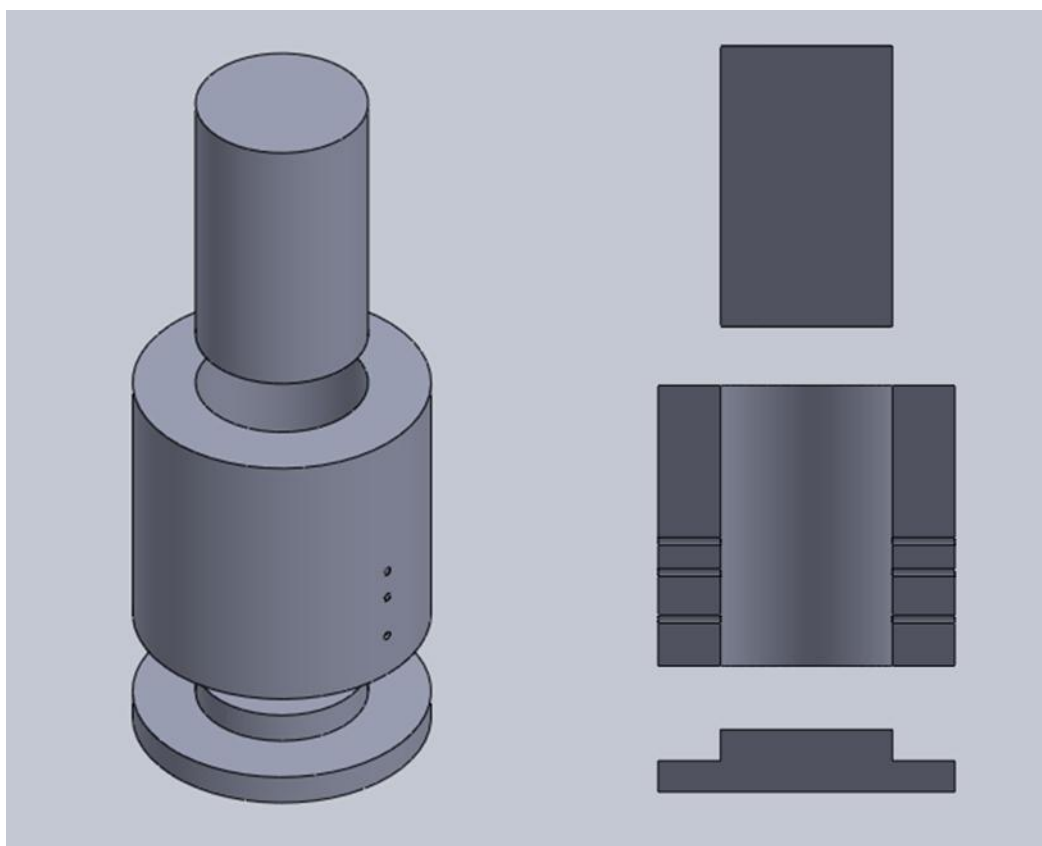


Figure 5.4. Final CAD design

The final design was manufactured. All apparatus components were heat treated to achieve 58 HRC. Figure 5.5 shows the finished apparatus.

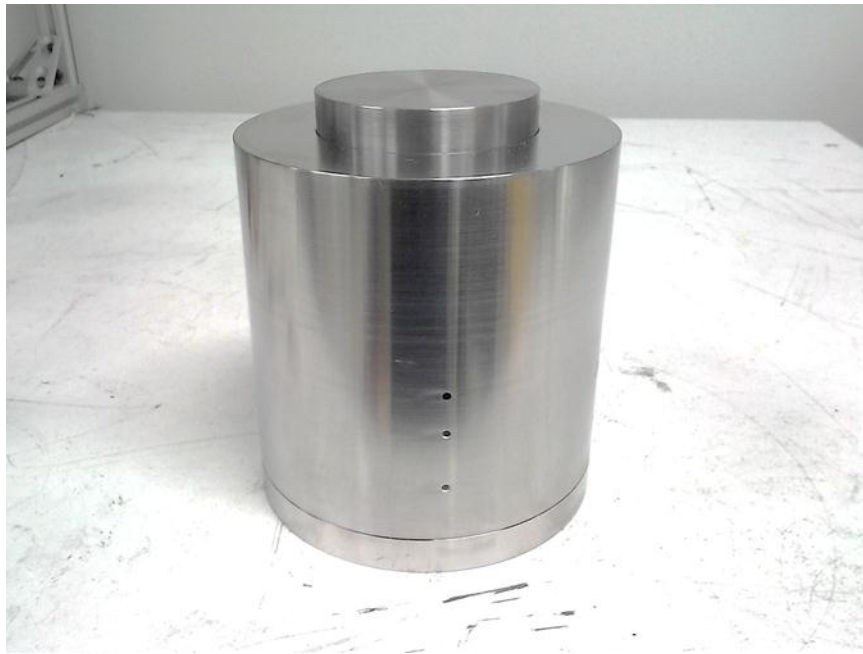


Figure 5.5. Manufactured SHS Compaction apparatus.

5.2 SHS Compaction Experiments

5.2.1 Experimental Setup

The experimental setup of SHS compaction experiments consisted of the SHS compaction apparatus and the manual bench top laboratory press, see Figure 5.6. Ignition was carried using an 18-gage-nichrome wire (Omega Engineering), folded in a waffle pattern. The ignition wire was connected to a 50V power supply (Mastech). Temperature of the front propagation was measured and recorded using type C thermocouples (Omega Engineering). The thermocouples were connected to the computer via USB DAQ (National Instruments).

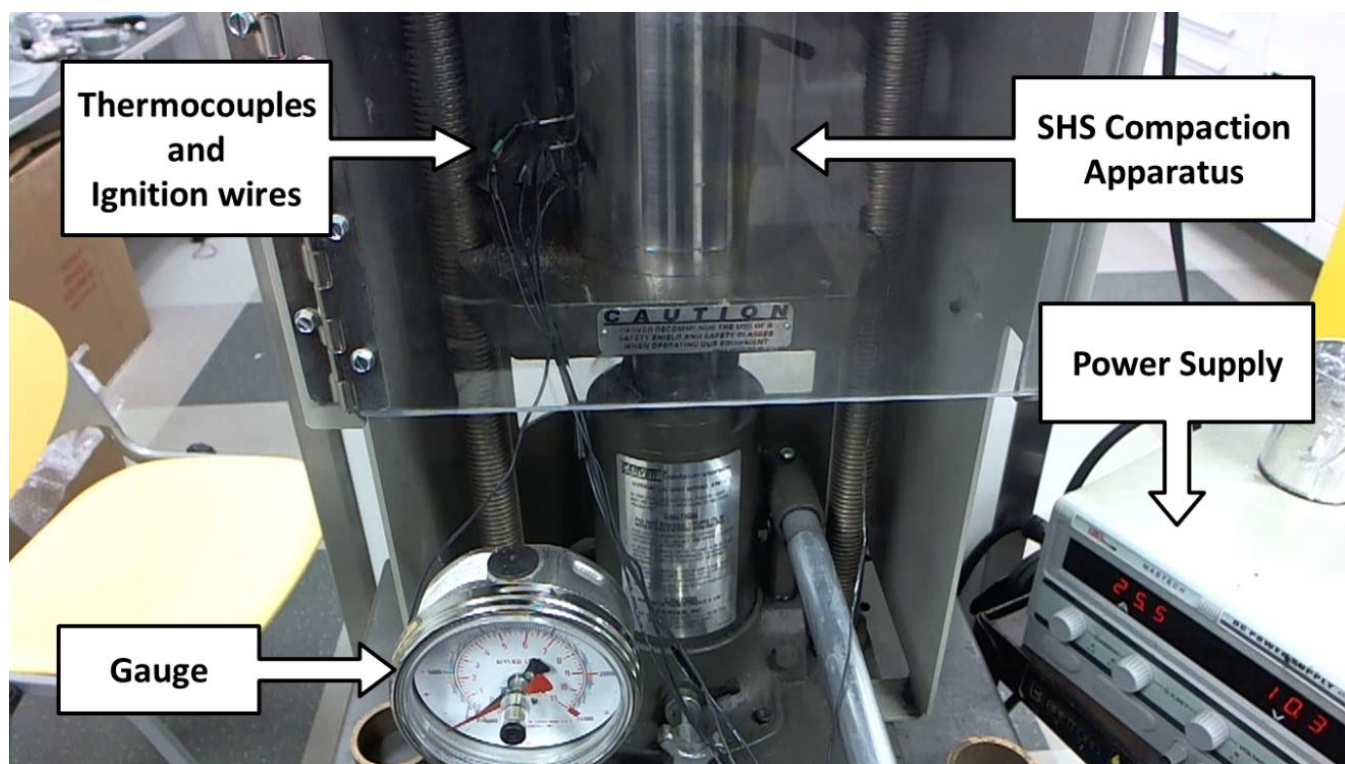


Figure 5.6. Experimental setup for SHS compaction.

5.2.2 Sample Preparation

Samples with a composition of 74 wt% of planetary ball milled JSC-1A and 26 wt% Mg (AlfaAesar) were compacted into pellets. Mixing of powders was conducted in Inversina mixer for one hour. A 10mm-depth hole was drilled in the pellet to monitor the combustion front temperature with the

use of a thermocouple. For experiments analyzing front combustion velocity, two holes 10 mm-depth were drilled 10 mm apart to place a thermocouple in each hole.

5.2.3 Experimental Procedure

Ignition wire and thermocouples were placed inside the apparatus before the pellet was placed in the apparatus. Then, a small amount of silica powder was poured on the apparatus in order to provide support for the pellet. The pellet was placed in the apparatus with carefulness in order to insert the thermocouples. After the pellet was placed, the remaining of the silica powder was loosely packed into the die. In total, the mass of the silica powder was 180 grams. Finally, the punch was slowly placed on the die and the SHS compaction apparatus was placed in the hydraulic press and hooked to the power source and DAQ, see Figure 5.7.



Figure 5.7. SHS compaction apparatus installed in a manual hydraulic press with ignition wires.

Once all cables were connected, the data recording started and power supply was turned on. The current was set on maximum capacity while voltage was slowly incremented. Once ignition was achieved power supply was turned off. Ignition of the sample was monitored via thermocouple readings and also by PTM escaping compaction apparatus.

5.2.4 Results

The experiments performed in the SHS compaction apparatus were conducted successfully. As expected, vent holes streamed jets of silica powder indicating SHS combustion took place.

Pellets were subjected to the SHS compaction pressures immediately after combustion and, as a result, were successfully reduced in volume. Table 5 shows the density values obtained in SHS compaction. Sample no. 1 was obtained in the aforementioned steel chamber in argon environment. Contrary to what was being observed in the past, SHS compaction products exhibit an increase in density as high as 66% when compared to non-compacted products. Figure 5.8 shows a comparison of the JSC-1A/Mg before combustion to the argon, Figure 5.8.a, and the sample as obtained in SHS compaction, Figure 5.8.b.

Table 5. SHS compaction results

Sample No.	Pellet Density, g/cm ³	Compaction Force, metric Ton	Product Density, g/cm ³	Density Change*, %
1	1.46	0	1.14	--
2	1.46	5	1.86	63
3	1.45	6	1.83	60
4	1.42	6	1.90	66

*Relative to non-compacted product.

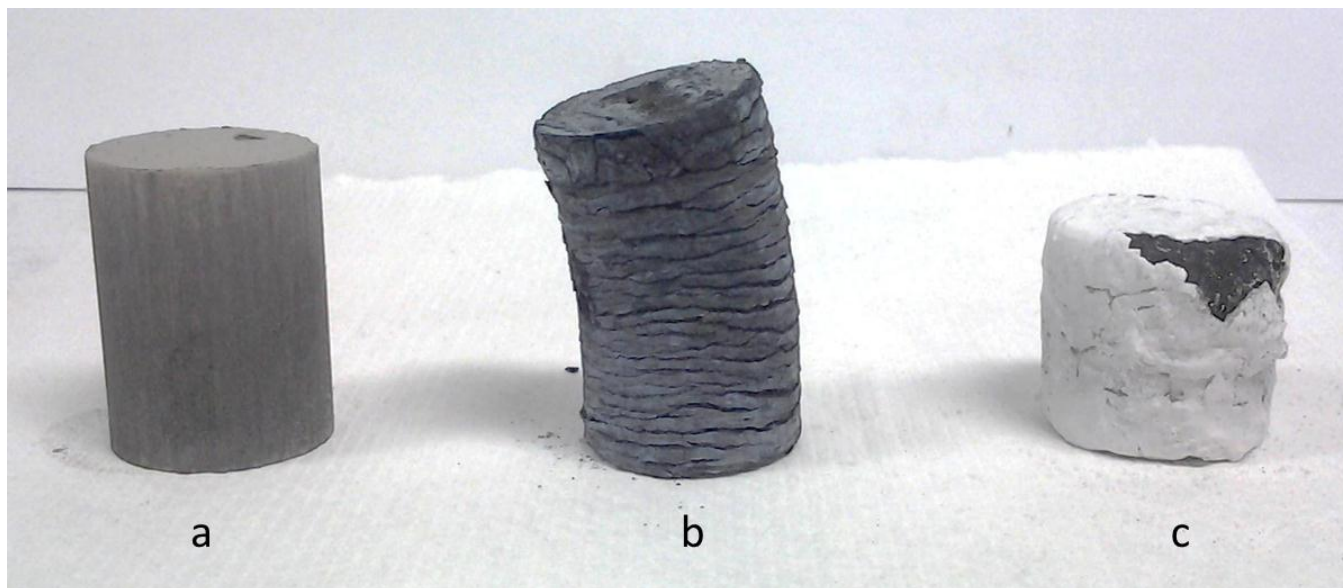


Figure 5.8. a. Original JSC-1A/Mg pellet. b. Product obtained in argon environment (no compaction). c. Product obtained in SHS compaction apparatus.

Temperature was obtained from type-C thermocouples which need mV to degree Celsius conversion, see Figure 5.9. According to the Type C tables [32], the maximum temperature that was recorded by the thermocouple reached 1375°C ; however, it is important to note that most of the time, thermocouple readings were distorted due to the compression of the thermocouples inside the pellet. Maximum recorded temperature before compression was 1316°C . Also, removal of thermocouples after compaction was impossible.

Velocity of front propagation was measured using two thermocouples. The thermocouples were placed 10 mm apart while the time difference was obtained from the thermocouple readings, see Figure 5.10. The velocity of the front propagation was 5.26 mm/s which is higher than in environment effect experiments. The maximum temperature reported by thermocouple 1 was 1301°C while for thermocouple 2 was 1288°C .

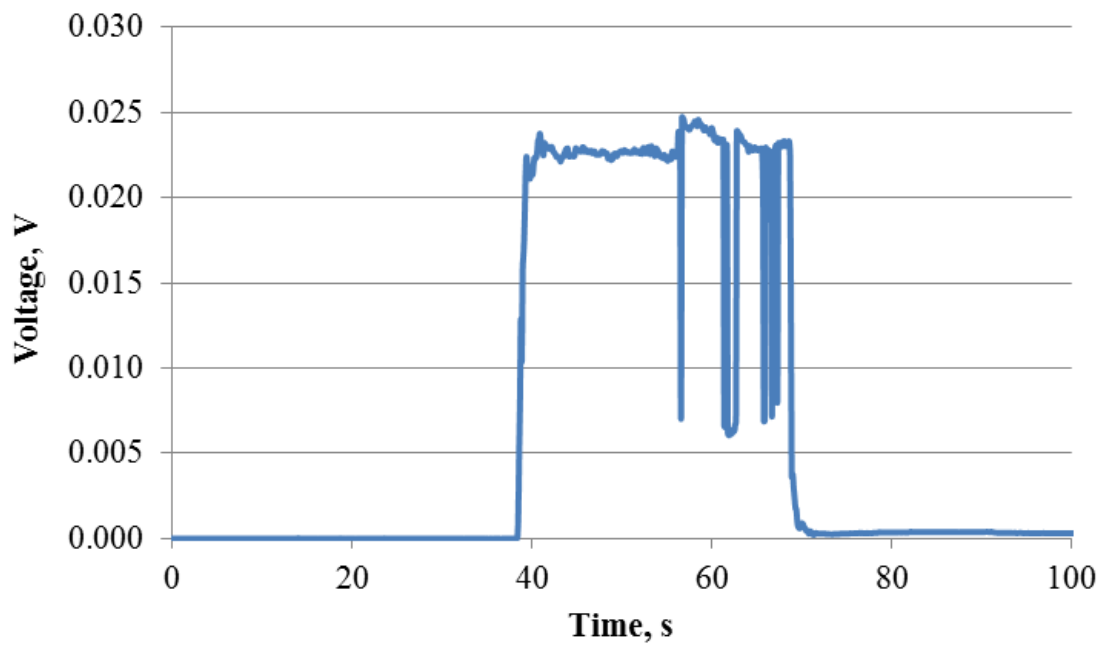


Figure 5.9. Type-C thermocouple reading during SHS compaction for temperature analysis.

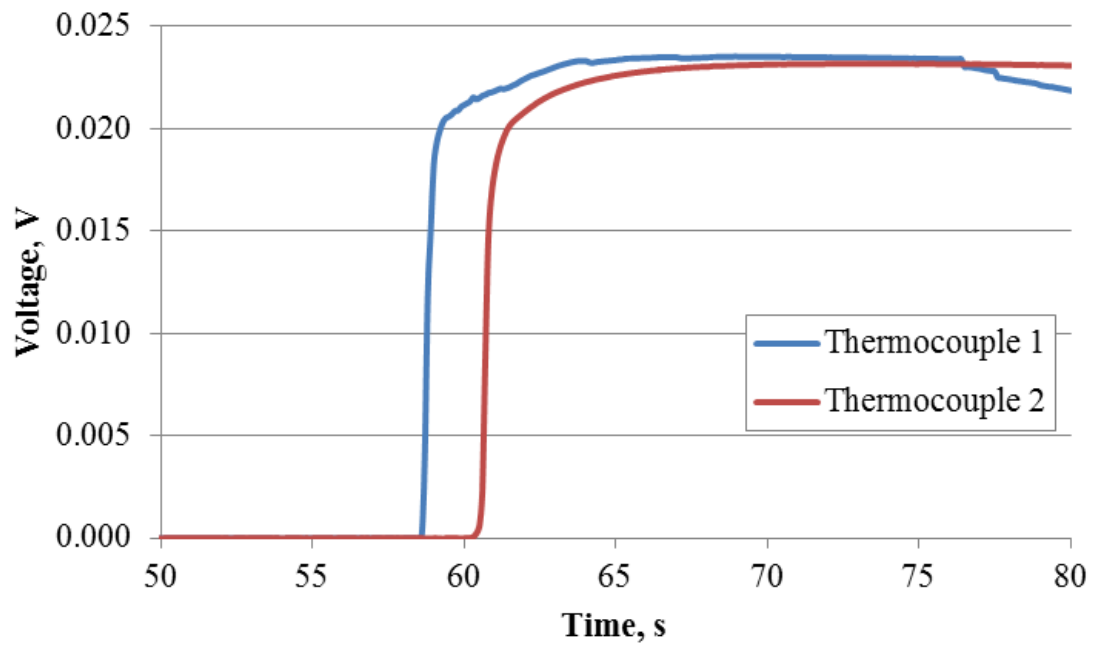


Figure 5.10. Type-C thermocouple readings during SHS compaction for velocity measurement.

5.3 Compression Tests

Compression test were performed in order to determine the compression strength of the SHS compaction products. As in section 4.5, ASTM standard C773 was used as a reference to obtained mechanical properties of the pellets. Test was performed for the 2 metric tons and 6 metric tons compacted products. Before compression test, the silica surrounding the pellets was removed using sand paper. Also, the faces of the pellets were grinded to achieve flat and parallel top and bottom faces.

Compression test was conducted using INSTRON 5866 testing machine with a 10-kN-load cell at the Keck Center. The 2-metric-ton sample had a height of 17.89 mm, while the 6-metric-ton sample height was 25.11 mm. A rate of 5 mm/min was used for the displacement of the top fixture. It was noticed that samples failed along the longitudinal plane. Figure 5.11 shows the testing of the 6-metric-tons compacted sample. Figure 5.11.b shows the failure along the longitudinal plane.

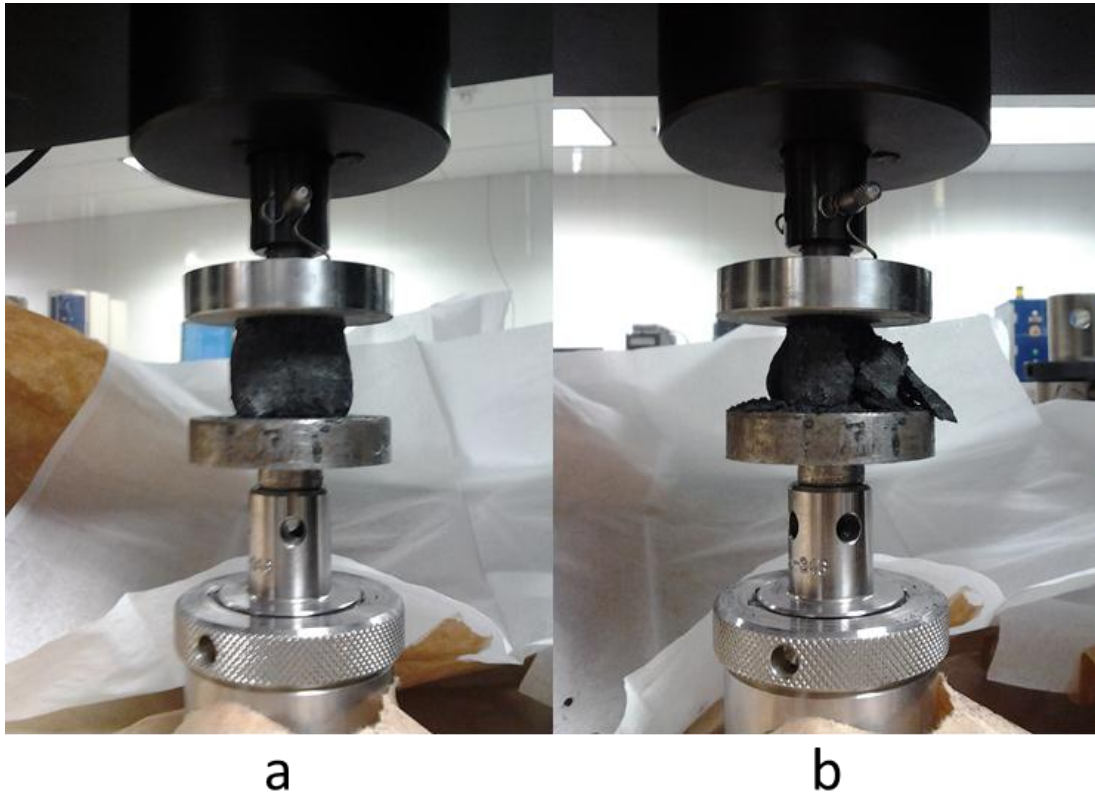


Figure 5.11. Compression testing of 6-metric-ton compacted product. a. Before compression. b. After compression.

Table 6 shows the maximum compression test at which the pellets were subjected. Figure 5.12 shows the stress-strain curve of both tests. The 6-metric-ton sample was subjected to greater compression stress, 11.8 MPa, which is close to the compressive strengths reported by Martirosyan and Corrias et al., 10-18 MPa and 25.8-27.2 MPa respectively.

Table 6. Maximum Compression Stress

Compaction Load, metric ton	Compression Stress, MPa
2	10.2
6	11.8

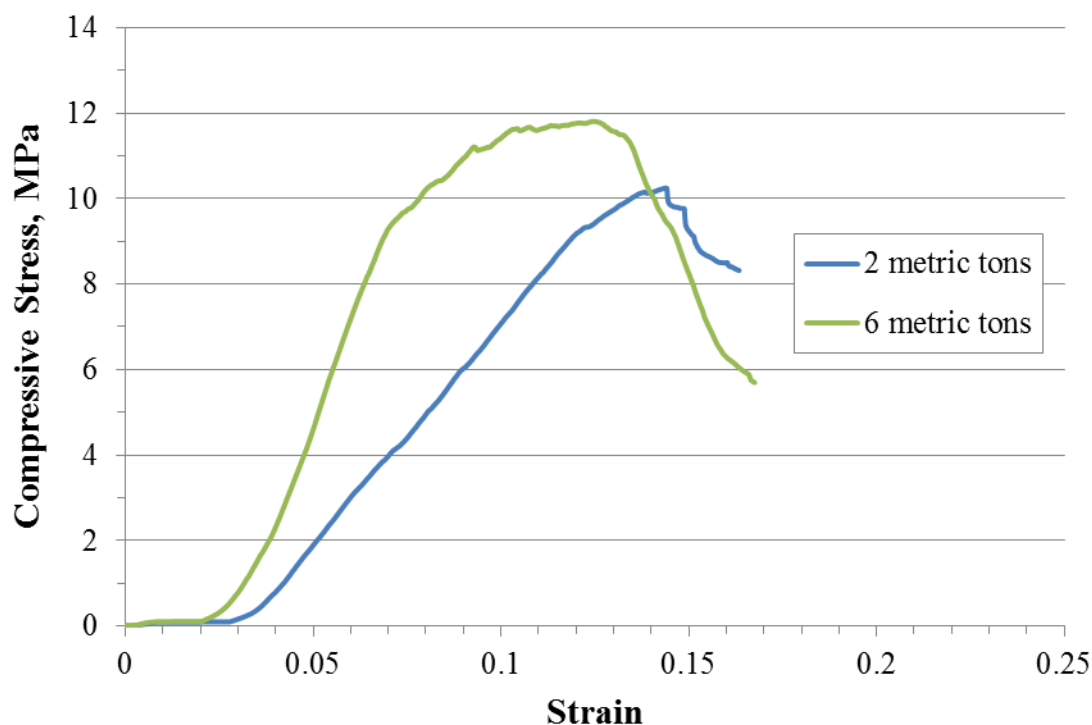


Figure 5.12. Stress-Strain curve of the SHS compaction products.

5.4 Conclusions

SHS compaction apparatus was designed and manufactured after a literature review. The selected method for densification of the JSC-1A/Mg system was quasi-isostatic pressing, as it provides a simpler and safer process.

Various designs were taken into consideration and safety parameters were taken into consideration. The SHS compaction final design consisted of a die with six through holes which allowed for ignition system, temperature monitoring, and release of impurity gases. The final blueprint can be found in Appendix A.

SHS compaction was tested using the designed SHS compaction apparatus, The PTM selected was silica, as used in environment effect experiments (Chapter 4). The SHS compaction products resulted in a 66% increase of density. Temperature and combustion front velocity were monitored.

Compression test were conducted for SHS compaction products. Different Maximum compression strength achieved was 11.8 MPa which is in the range reported by other lunar ISRU groups [21] [10]. For further comparison, the strength of the SHS compaction products is comparable to that of commercial bricks which lowest value can be 9.51MPa [33]

CHAPTER 6

SUMMARY

The presented thesis was the continuation of a research started in 2009 by Christopher White, Francisco Álvarez, and Dr. Evgeny Shafirovich which main objective was the production of construction materials in the Moon utilizing lunar resources. Before the presented research, combustion synthesis of JSC-1A/Mg was demonstrated; however combustion products were porous and fragile. The goal of the present research was to develop a technology that allowed for the production of dense (and hence strong) structural materials by combustion of regolith/metal mixtures.

In direct continuation with the previous work, magnesium content in the JSC-1A/Mg system was minimized through the addition of a preheating step before the combustion process. The initial temperature of JSC-1A/Mg pellets was increased with a hot plate to 100°C. Different JSC-1A/Mg concentrations were preheated and combusted separately: 7 wt%, 8 wt%, 9 wt%, 10 wt%, 13 wt%, 20 wt% and 26 wt%. Experiments showed preheating step increased in combustion stability, combustion temperature, and combustion front velocity. Magnesium content was minimized to 8% when preheated to 100°C. Additionally, JSC-1A/Al system was tested with the preheating step however it could not be ignited.

Different densification techniques for the JSC-1A/Mg systems were considered before to produce strong product: Pressureless densification, explosive compaction, and quasi-isostatic pressing, i.e. SHS compaction. SHS compaction was selected as it provides a simpler and safer process. In this method, the green mixture is located inside some inert powder placed under a hydraulic press. The mixture is ignited and after the combustion process, the products are compacted. Various designs were taken into consideration and safety parameters were taken into consideration. The SHS compaction final design consisted of a die with six through holes which allowed for ignition system, temperature monitoring, and release of impurity gases.

Before SHS compaction, experiments using thermocouples were conducted in argon and silica to evaluate the effect of the environment during and after the combustion of JSC-1A/Mg mixtures.

Submerging the pellets in silica resulted in a significantly increase in the strength of the products (1.6 MPa).

SHS combustion was tested using the designed and manufactured SHS compaction apparatus. The PTM selected was silica, as used in environment effect experiments. Experiments in the SHS compaction apparatus were successful and resulted in a 66% increase in density and compression strength as high as 11.8 MPa.

CHAPTER 7

FUTURE WORK

Optimization of the SHS compaction is needed to develop high density products. This can be done by further analyzing the different times that characterize SHS compaction: delay time and pressing time [34]. Also, increasing pellet size and mass will further increase the temperatures of the combustion and may affect compaction parameters which will result in higher density products.

Additionally, the cSETR has the facilities to make activated aluminum powder from foil which has been demonstrated to readily react with hot water [35]. Such powder could be mixed JSC-1A in order to facilitate ignition of the JSC-1A/Al system.

Furthermore, Martian regolith simulant available and is also rich in silicon oxides [36]. Therefore, an SHS system composed of JSC Mars-1 with a metal additive, e.g. aluminum and magnesium, could be studied for the production of construction materials using Martian resources.

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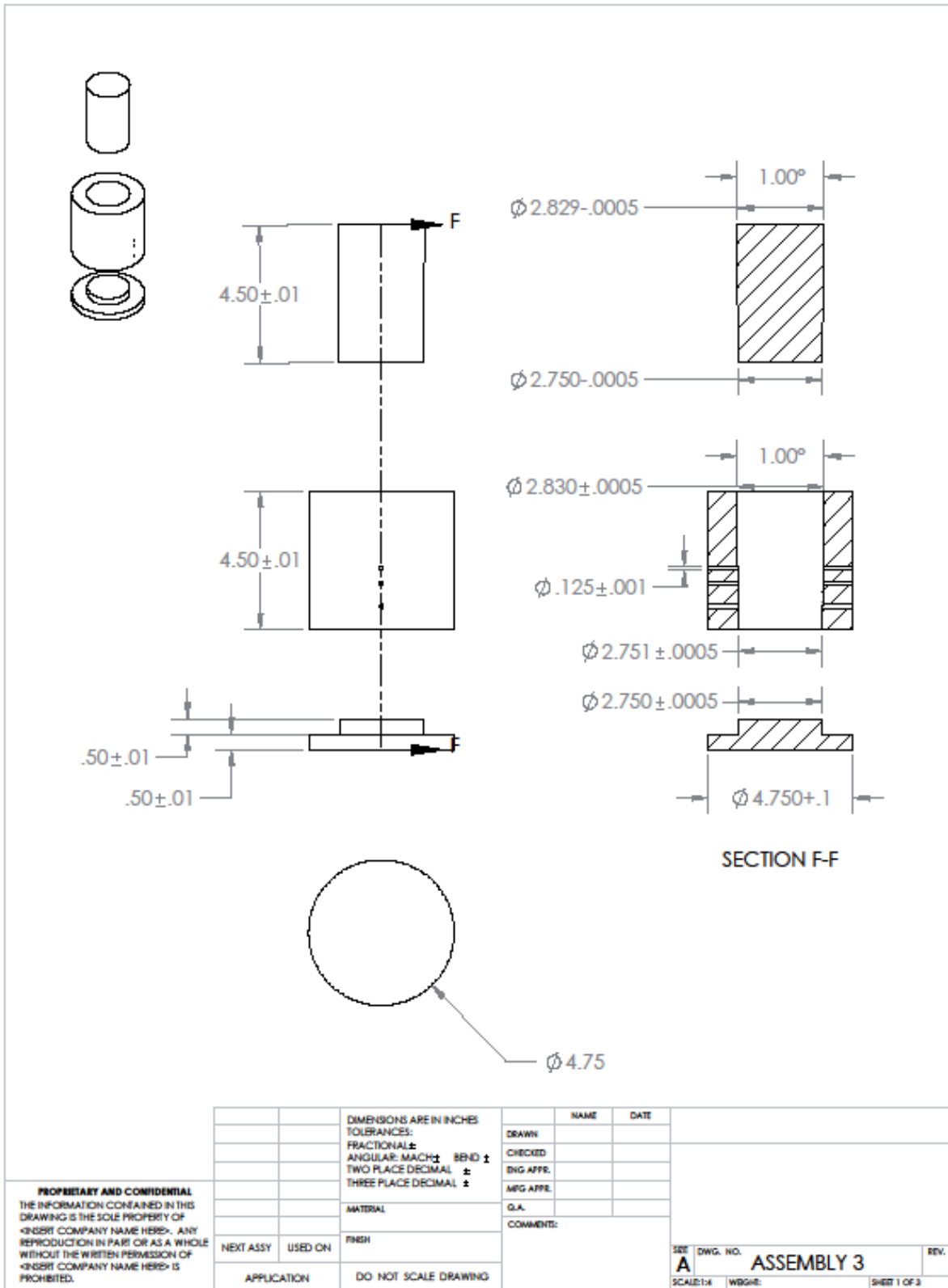
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APPENDIX A



CURRICULUM VITA

Armando Delgado Jr. was born in El Paso, Texas in 1989. He is the son of Armando Delgado and Maria De La Paz Ivione Marentes. He attended J. M. Hanks High School and graduated in December 2006. In January 2007, he began his undergraduate studies at the University of Texas at El Paso, and received a Bachelor of Science in Mechanical Engineering in December 2010. In November 2009, he began working as a research assistant at the Center for Space Exploration Technology Research, working in the In-Situ Resource Utilization team under the supervision of Dr. Evgeny Shafirovich. The research performed at the center led to the publication of “Microgravity Combustion of Thermite Mixtures for Welding in Space and for Production of Structural Materials from Lunar Regolith” by Francisco Alvarez, Armando Delgado, Jorge Frias, Mario Rubio, Christopher White, Ashvin Kumar Narayana Swamy, and Evgeny Shafirovich (AIAA-2012-1119). This also led to the publication of “Combustion of JSC-1A Lunar Regolith Simulant Mixed with Magnesium” by Francisco Alvarez, Christopher White, Armando Delgado, Jorge Frias, Ashvin Kumar Narayana Swamy, and Evgeny Shafirovich (AIAA-2012-4092). He participated in three student teams that conducted microgravity experiments onboard reduced gravity research aircraft at JSC (April 2011 – Flyer, June 2011 – Flyer, and June 2012 – Ground Crew).

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