THE INVESTIGATION AND QUANTIFICATION OF CERAMIC POWDER STICKINESS

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The Investigation and Quantification of Ceramic Powder Stickiness

A Major Qualifying Project submitted to the faculty of **Worcester Polytechnic Institute** in partial fulfillment of the requirements for the Degree of Bachelor of Science in Mechanical Engineering and/or Chemical Engineering

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Abstract

Sticking poses a constant problem to several pressing operations; from glass-forming to pill manufacturing, its potential to ruin the product makes it a hazard that must be managed accordingly. This is especially true in the pressing of green bodies for ceramics manufacturing, where it can potentially ruin entire batches. To account for such an inconvenience and minimize its threat to manufacturing operations, the contributing factors must be determined. Only once the root causes are known can effective measures be taken to alleviate this concern. To do this, potential contributors, including the number of presses and the temperature of operation, are varied in order to determine what influences this issue. The focus of this investigation is on a mixture composed mainly of alumina and zirconia, common in ceramics for a wide range of applications. A model press was employed in this work, with the weight of the sample taken before and after each pressing, with the difference being in the amount that stuck within the press. DSC was also used to determine the glass transition temperature of the binder mixture. It was determined that a higher die temperature correlated to a greater mass loss in the die and made it more difficult to separate the die and continue pressing due to the sticking of the ceramic powder. Further influential factors were the binder package composition, the number of pellets formed before the die was cleaned, and the ambient humidity.

Authorship

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Introduction

Across human history, ceramics have been used for a wide variety of purposes (Ring, 1996). Evidence of ceramics appears around 22,000 BC, with the development of this technology continuing through the bronze age to the modern-day. China, in particular, played a substantial role with its rich ceramic tradition, developing innovations such as the kiln and glazing (Ring, 1996). Today, ceramics are broadly divided into two categories, traditional and advanced. The former refers to inexpensive ceramics used as textiles and other simple purposes, while the latter refers to more expensive ceramics with more refined capabilities for technical purposes (Ewsuk, 1999). The strength, hardness, and robustness of ceramics make them a desirable material for many applications (Ewsuk, 1999; Mohammad & Saheb, 2016).

However, ceramic production still encounters difficulties. The compaction of the ceramic powder into its final shape via the formation of a 'green body' still presents problems. Most ceramic powders do not possess the required strength to maintain the desired shape during the firing process, requiring the inclusion of additives to enhance the green body's robustness (Ring, 1996; Tozzi, n.d.). These additives can take many forms, from binders to add mechanical strength to plasticizers to increase malleability, even dispersants to impart stability to the suspension (Buschow et al., 2001). However, these additions can create problems in addition to solving them; since they constitute an unwanted component in the final product, they must be removed via binder burnout (i.e., thermal degradation). This requirement led to the favoring of binder components that burnout cleanly, such as Polyvinyl Alcohol (Sekisui, 2011; Lewis, 1997; Ring, 1996). Furthermore, during powder compaction in a press, there is often an issue with powder sticking to the pressing dies, resulting in issues for future compactions (Rieser et al., 2008; Tousey, 2003). This powder sticking problem is especially pronounced in modern assembly lines, where some presses can produce hundreds or thousands of green bodies in a batch, with this issue potentially ruining many of them (Ewsuk, 1999).

The issue of sticking and its potential to ruin batches is widespread, impacting the ceramic industry and other industries where similar compaction processes are involved. Similar sticking issues are known to destroy batches of pills in pharmaceutical manufacturing (Tousey, 2003). When forming glass at elevated temperatures, sticking to the press is likewise an issue (Rieser et al., 2008). In the ceramics industry, this issue becomes more relevant at an industrial level due to its batch ruining potential in this area.

Despite the wide-reaching nature of sticking problems, there is no standard for the quantification of stickiness and no standardized unit of sticking. This makes comparing the levels to which various powders or formulas stick very difficult and greatly hinders any efforts to address these issues. Therefore, this work endeavors to establish the groundwork for a standardized method for quantifying stickiness, laying the foundation for future studies and development in the area of stickiness quantification. By determining what factors influence this issue, a more developed understanding of their contribution to the sticking of the powder mixture to the press will aid in mitigating or preventing defects. Ultimately, this will assist in establishing baselines for how

factors such as press temperature and number of presses influence powder sticking to pressing dies.

Background

Ceramic Manufacturing

Ceramics are used in everything from industrial grade cutting tools and abrasives to coffee mugs. Their hard but brittle nature requires a manufacturing method distinct from those used for other materials (Ewsuk, 1999). They must first be shaped into their desired form, which is done with the use of dies and presses, before then being placed in a furnace and fired to achieve their hardened state (Ewsuk, 1999; Ring, 1996; Sun et al., 2009). Afterwards, finishing operations such as coating and glazing are typically employed to finalize the part (Ring, 1996). The state between forming and firing is known within the industry as the green body.

Binder

Green bodies refer to any ceramic that has been shaped into its final form but has yet to undergo firing (Ring, 1996). Pure ceramic powders alone do not typically possess the required strength to maintain the desired shape; as such, supplemental compounds known as 'binders' are used. Binders are numerous compounds used to impart strength to a ceramic powder necessary for green body forming (Tozzi, n.d.). The strength they impart is due to the structure of the binder (typically a long carbon chain as a backbone) and its adhesion to the particles of the powder (typically via side groups), which together serve to increase the cohesion of the green body (Buschow et al., 2001). Binders are typically polymers such as polyvinyl alcohol (PVA), and are mixed thoroughly with the ceramic powder to ensure proper distribution; i.e. particles of ceramic coated with binder (Boursier et al., 2020; Hansen & Park, 1996; Lewis, 1997; Tozzi, n.d.). PVA excels in this role due to its ability to wet particles (maintaining contact with them) and its nature as a strong surfactant (reducing surface tension to ease mixing) (Tozzi, n.d.). It is favored even further due to its ability to burnout in a controlled and clean manner among various atmospheres (Sekisui, 2011).

A plasticizer is added to the binder mixture to impart the necessary malleability to undergo pressing. This is also often a polymer, such as polyethylene glycol (PEG), and serves to impart the necessary flexibility to the powder mixture, complementing the strength provided by the binder (Buschow et al., 2001; Tozzi, n.d.). PEG is especially versatile in this role due to its variable viscosity, with lower molecular weights being more liquidus while higher molecular weights are more solidus (Tozzi, n.d.). It is also water-soluble and readily debinds in water, making for easier removal (Bleyan et al., 2015; Tozzi, n.d.). Often PVA and PEG fulfill their respective roles as binder and plasticizer together, although such systems are subject to variable properties (such as glass transition temperature) corresponding to changes in humidity (Nies & Messing, 1984).

Binder and plasticizer are both necessary for the successful forming of the green body, but their inclusion in the final composition of the ceramic part would undermine the sintering of the ceramic particles to each other and weaken the final product. To prevent this, binders and

plasticizers are typically burned out before sintering via a prolonged period at a lesser temperature sufficient to denature the organic compounds (Ewsuk, 1999; Ring, 1996). Often this is done in the same furnace as firing to prevent a point of risk that would occur when transferring it unnecessarily (Ewsuk, 1999; Ring, 1996).

Binder Sticking

In industry, green body formation is often accomplished with pressing operations, using dies to form the ceramic powder into its final shape (Ewsuk, 1999). Cracks and other defects acquired during pressing or transport to a furnace for firing have the potential to ruin the piece. Ejection is a significant point of risk; it represents the stage when the part is under the greatest forces it will face before firing (Briscoe & Evans, 1991; Chen et al., 2007). The cohesion and adhesion promoted by the binder mixture and required for successful green body formation can cause another problem during ejection; powder sticking to the die during forming operations (Rieser et al., 2008; Tousey, 2003). Powder sticking to the press during the compaction of green bodies has always been an issue in the ceramics industry, but it's especially problematic on modern, industrial scale production lines. If left unchecked due to the widespread integration of automated machinery, it has the potential to ruin entire batches (Ewsuk, 1999; Tousey, 2003). The chemical composition of the green body (namely its additives such as the binder and plasticizer) may greatly influence this issue. Compounds commonly employed in the roles of binder and plasticizer are PVA (poly vinyl alcohol) and PEG (polyethylene glycol), respectively (Tozzi, n.d.). These additives act in conjunction to reinforce the ceramic powder and allow the green body to keep its shape and withstand handling, after which they are removed via burnout before sintering (Buschow et al., 2001). It seems logical that these species contribute substantially to the sticking issue given their role in maintaining the cohesion of the ceramic powder particles and their adhesion to each other (Tousey, 2003). Further investigation will endeavor to confirm or deny this.

Powder sticking to the pressing die during ejection is a prevalent problem across many pressing processes, from pill formation in pharmaceuticals to glass-forming operations (Tousey, 2003; Rieser et al., 2008). Sticking also poses a significant problem for ceramics on industrial-scale production lines (Ewsuk, 1999; Tousey, 2003). There have been efforts to model the process of ceramic powder compaction to aid in alleviating this issue.

Practical solutions to address the issue of powder sticking to the presses have so far been limited to stopgap solutions. Solutions range from polishing or shocking the press and altering its parameters to employing veteran operators to monitor the batch for signs of trouble; such methods are mainly employed in the pharmaceutical industry during pill manufacturing (Tousey, 2003). More elaborate solutions, such as employing lubrication, particularly as a surface coating between the die and the body wall, have been hamstrung by their complexity and the need to slow production lines to reapply lubricant (Tousey, 2003). More successful endeavors include redesigning the press and part geometry and altering the powder composition to reduce sticking; however, these are complex solutions that require significant research and development (Tousey,

2003). Comprehensively understanding the phenomenon of powder stickiness and the parameters that affect it on an operational level would greatly aid efforts to alleviate this issue.

Methodology

To investigate the problem of sticking between the green body and the pressing die, a series of various experiments designed to simulate a scaled down version of pressing multiple pellets was created. This required properly preparing the ceramic powder, utilizing an appropriate die to press pellets, and altering parameters to find a probable cause for sticking. Figure 1 demonstrates a breakdown of the entire process from combining raw materials to pressing pellets.

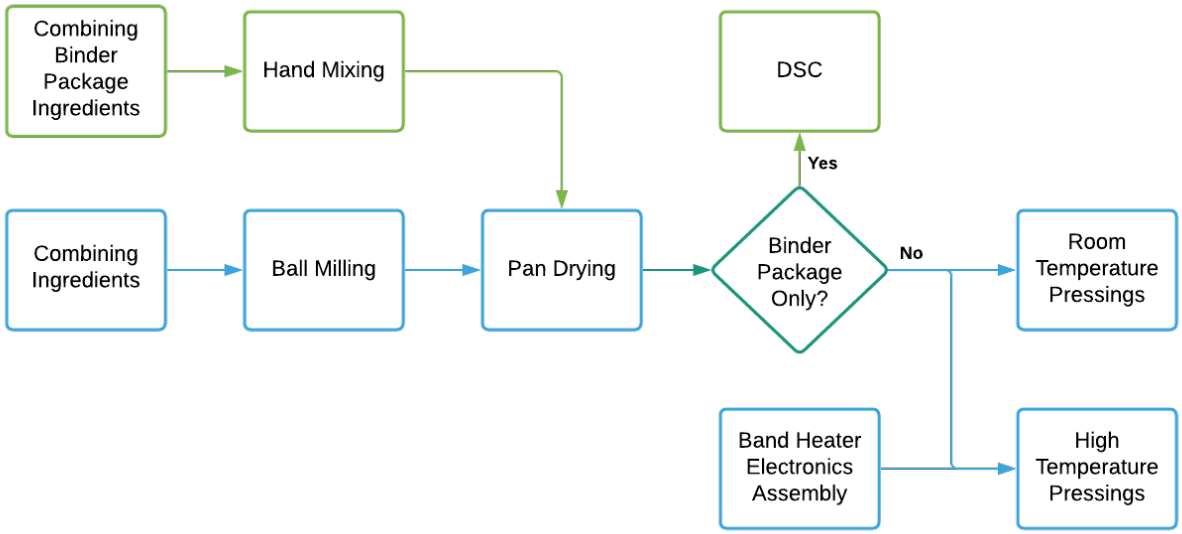

Figure 1: Flowchart of the experimental process

Powder Preparation

In order to create the ready to press (RTP) powder, the ceramic powder and a binder package needed to be mixed in a ball mill (Figure 2), dried in an oven (seen in Figure 3), and finely ground with a mortar and pestle. The raw materials included: alumina powder, zirconia powder, PVA, PEG, an antifoam, and a dispersant. The first attempt at mixing these components was to measure them individually and combine them in a single container to then be ball milled. However, there were two main problems with this method. The first is that measuring very small amounts of the material was difficult and didn't guarantee the proper ratios. The second is that the powder was not homogenous; much of the binder package had only locally mixed with the powder.

Figure 2: Micronano Tools PBM-04 Planetary Ball Mill

Figure 3: RTP powder pan drying with balls from ball milling

Subsequent batches were made using a much more refined process. The process began by mixing the antifoam, dispersant, and PEG, referred to as ADP together, due to their similar viscosities, to produce a large volume for use in creating future batches. Since the viscosity of PVA was comparatively much thicker, and there were concerns that it would separate and not reincorporate easily, the PVA was added separately. In preparing the RTP powder for the ball mill, the different components were measured following the specified ratio of powder to binder package and added to the container in the following order: alumina powder, zirconia powder, ADP, and PVA. The binder package also called for the addition of water to aid in mixing, but a modified amount was used to ensure more homogeneity; the added water equaled approximately double the powder weight. The batch was then placed in a ball mill for several hours. Following ball milling, the mixture was emptied onto a makeshift aluminum foil tray to dry in the oven between 65-70°C. Once the powder dried, further refinement occurred using a mortar and pestle to achieve consistent

particle size. This RTP powder was much cruder than commercially available spray-dried powder but still sufficient for these experimental presses.

Differential Scanning Calorimetry

Since the binder glass transition temperature (T_g) was a critical parameter to investigate, analysis of the binder package using Differential Scanning Calorimetry (DSC) was done (Figure 4). Using products similar to those used for the RTP preparation, a batch of the binder package was prepared separately, mixed by hand, and small droplets were set in an aluminum foil tray to dry in an oven at 65°C. Once they had dried after approximately 1.5 days the solidified droplets were scraped into off the foil and placed in the crucible for the DSC. The DSC slowly burns the sample while raising the temperature at a steady rate. This is done in an isolated chamber where different gases can be used. In this instance dry air, which is the standard composition of air, was used as it accurately reflected the conditions the binder package was used at. There were two temperature ramping rates that were used: 5°C per minute and 10°C per minute. Three different compositions of the binder package were tested, specifically the ratio of PVA to PEG; they were 0.95:1, 0.9:1, and 0.85:1. The ramping rate for the composition testing was 5°C per minute.

Figure 4: TA Instruments SDT 50: external view (left), crucible to hold sample (right)

Pressing

The pressing die is composed of two rods, a sleeve, and a support plate, as shown in Figure 5. To accurately measure the temperature of the die with an infrared thermometer, a piece of tape was wrapped around the outside. Press parameters for the initial pellets were to apply 775 MPa of pressure (10 metric tons) and hold for 5 minutes to allow for densification; however, the pellets displayed several defects, as detailed in the *Results* section. Henceforth, the parameters needed to be adjusted. Since densification is an important result of powder compaction, a series of presses were conducted to compare pressure to density. The force of the press was converted to the corresponding pressure for the specific die, and density was calculated by measuring the height of the pellet and computing the volume using the die inner diameter. From these results, the stable pressing parameters are achieved by applying approximately 200 MPa of pressure (2.5 metric tons)

and holding for 30-60 seconds. The mass of the sample was measured and recorded before pressing and again after compaction. Theoretically, such mass measurements would indicate that powder remained on the die and be a measure of sticking from the pellet.

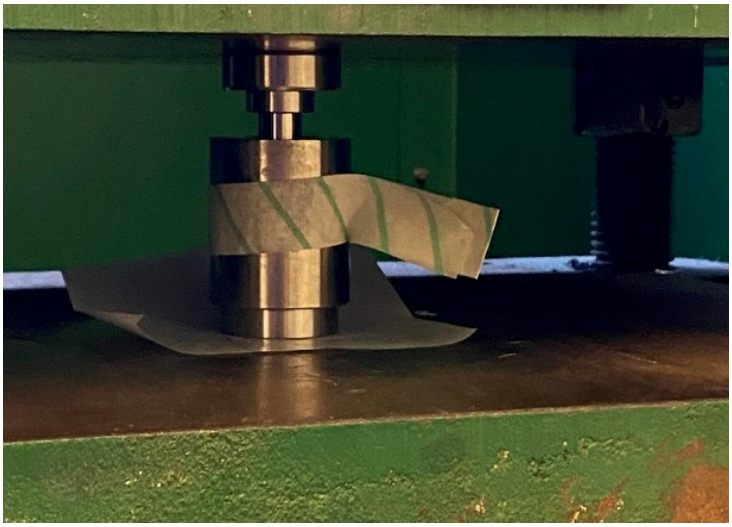

Figure 5: Die under pressure in the press

Elevated Temperature Experiment

A picture of the electronics used to operate the band heater is shown in Figure 6, and a wiring diagram is shown in Figure 7. The band heater is placed around the die and is connected to an Inkbird 25A DA solid-state relay (SSR) to control the current and voltage running through the band heater. The SSR is connected to an Inkbird Digital PID ITC-100VH temperature controller which is further connected to a K-type thermocouple. The PID controller will take the temperature measurement from the thermocouple and determine if the band heater will turn on or stay off based on a threshold value. This threshold value can be controlled by the user for consistent power flow. Since there were difficulties in regulating the temperature, an infrared thermometer was used to measure and verify the outer temperature of the die, and the thermocouple was placed in a beaker of cool tap water to maintain a steady temperature so the PID controller could act as a switch. There were three different times that the temperature was recorded. The first was when the powder was placed in the die, the second was after the powder was compacted, and the third was after the pellet was ejected from the die. The first temperature (powder loading) would be increased in regular increments starting from room temperature (approx. 25°C). For each pellet the initial and final mass were recorded as well.

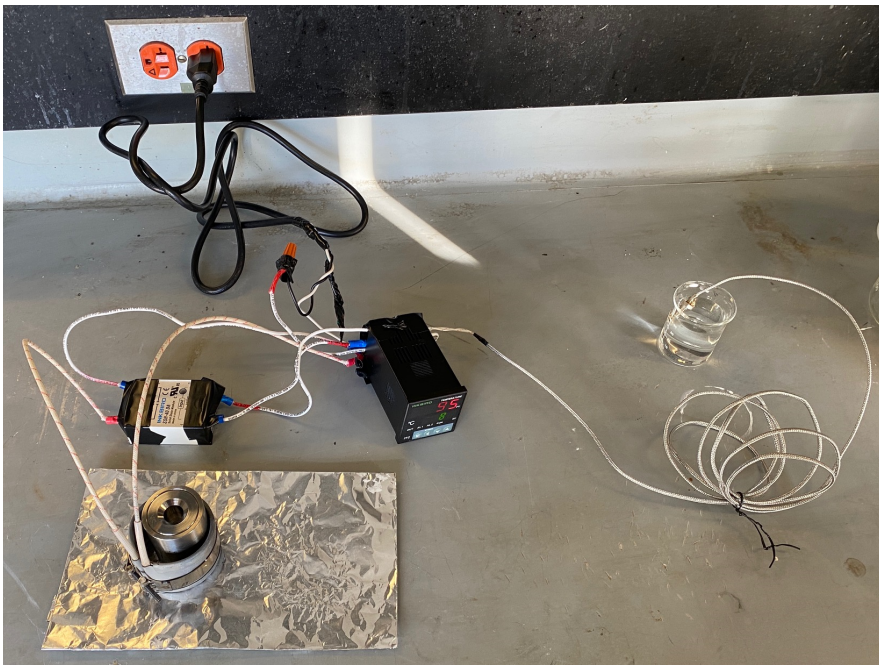

Figure 6: Band heater connected to appropriate controllers

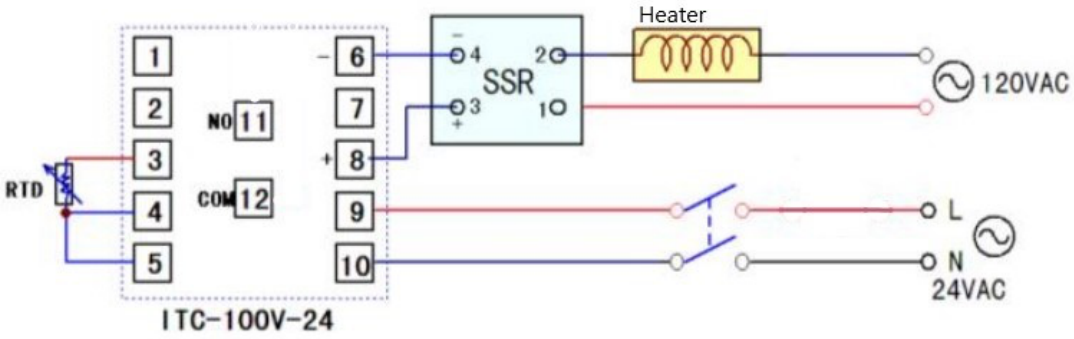

Figure 10:ITC-100VH Wiring diagram

Figure 7: Wiring diagram for the band heater

Results

Experimental data was collected for three key variables: the number of pellets pressed, the compaction behavior of the pellets through densification, and the effect of elevated temperatures on the pellet. Furthermore, DSC measurements on the binder package were used to discern how the glass transition temperature of the binder might impact the pellet stickiness. Stickiness was measured by calculating the difference between the RTP powder mass and the pellet mass. The initial operating theory was that a loss in mass would indicate that the die had retained some amount of powder that would affect later batches, suggesting this behavior as the stickiness observed in an industrial setting.

As previously stated in the *Methodology* section, the initial pressing parameters had to be adjusted to eliminate pellet defects. These changes effectively created a split in the data between the initial high-pressure parameters and the new low-pressure parameters. Consequently, most of the data collected using the initial parameters for the number of pellets pressed simultaneously, with no cleaning between presses, could only be selectively compared to the newer data. Therefore, the primary focus will be on the new low-pressure data to compare both the number of pellets pressed and how elevated temperatures of the powder affect pellet stickiness, and how this relates to the glass transition temperature.

Compaction Curves - Eliminating Defects

As stated in the *Methodology* section, the initial pressing parameters produced several defects among a large portion of the pellets. Some examples of these defects are shown in Figure 8. Since the pressing parameters were the most likely reason for the defects, a comparison of multiple pressures and densities was graphed to inform the best parameters. In Figure 9, a compaction curve can be seen comparing a range of pressures within the operating limits of the die. Until very high pressures, above 500 MPa, are reached, there is no significant difference between densification. Therefore, it is believed that the pellet was overstressed due to high pressure and low powder weight, which ultimately led to breakages after ejection. Pellets tested at lower pressures with larger masses showed no defects upon ejection and had only minimal chips from handling the pellet to weigh them.

Another significant factor contributing to the assumption of overstressing was the hold time. After the pellets were pressed, they were held at that pressure to allow for densification to occur. Decreasing the hold time from five minutes to thirty to sixty seconds also positively affected the pellets. The best results were with a pressure of approximately 200 MPa, as seen in Figure 10.

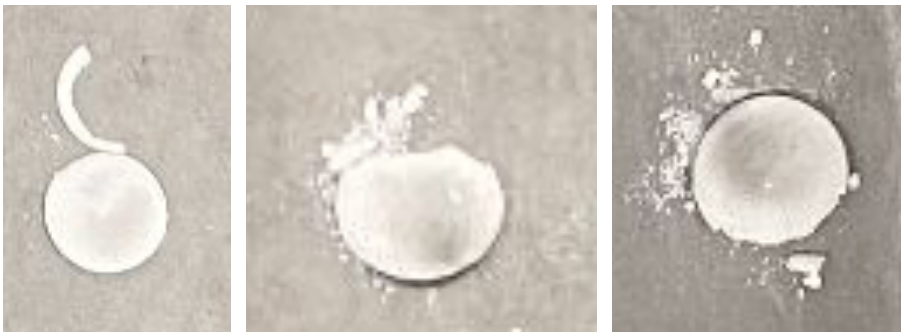

Figure 8: Chipping and uncompressed powder in pellets

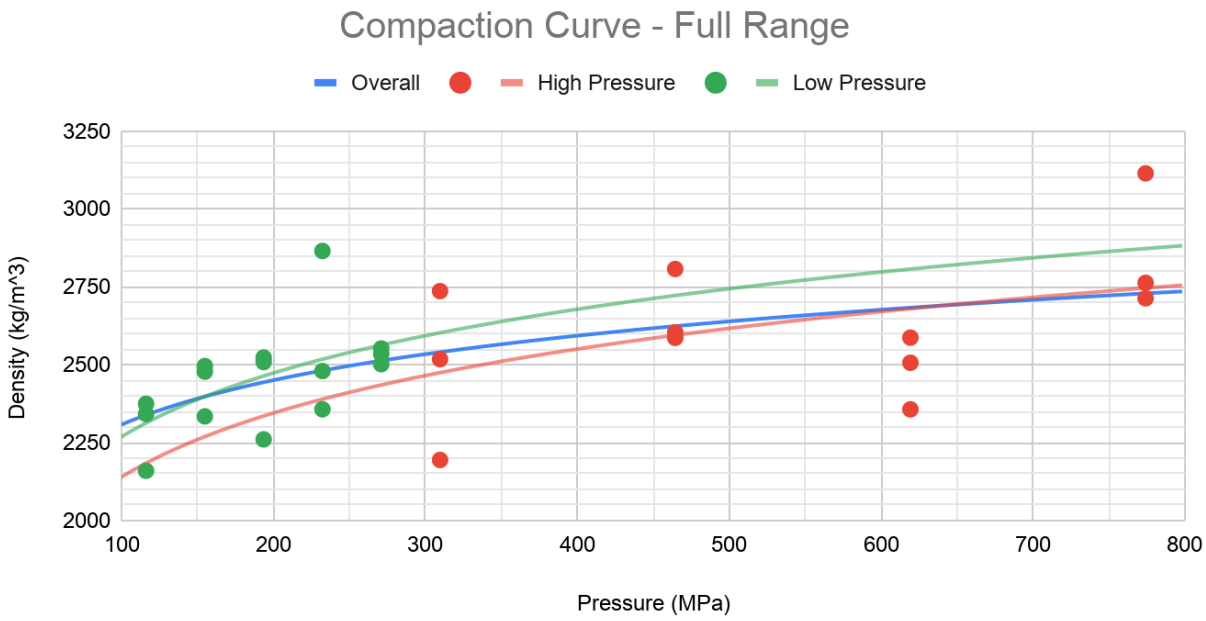

Figure 9: Full Pressure Range vs Density Compaction Curve

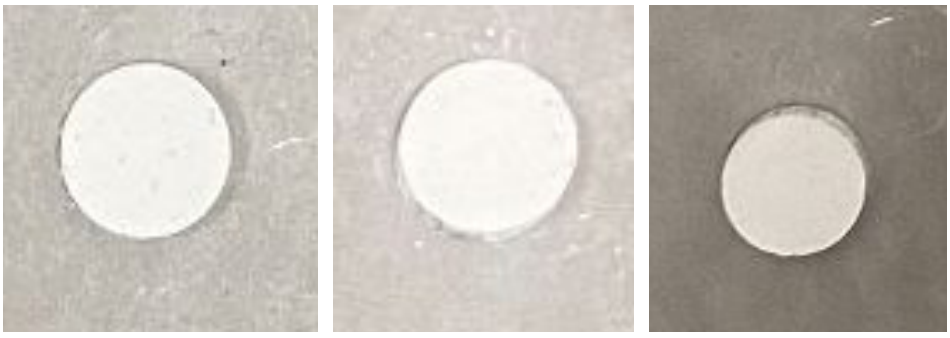

Figure 10: Defect free pellets from lower pressure pressings

DSC Analysis

Most of the experiments involved working with RTP powder, however, the binder package's glass transition temperature is a critical parameter in stickiness so identifying it was crucial. The main reason for determining the glass transition temperature is because the binder will theoretically change viscosity and could result in more residues left on the die to contribute to stickiness. Two different ramping rates were considered, shown in Figures 11 and 12. In Figure 11, there are two "bumps" in the curve at approximately 38°C and 50°C. These are indicators of the transition from a glassy and relatively brittle state to a rubbery and more viscous state. The midpoint between them suggests the glass transition temperature is approximately 44-45°C. In Figure 12 the ramping rate is too fast to clearly show any changes in the curve. This most likely indicates that a larger heat flow results in a faster transition past T_g as is expected. In addition to ramping rates, changes to the ratio between PVA and PEG were explored but did not yield any different results for T_g. Details of the different ratios are in the *Discussion* section.

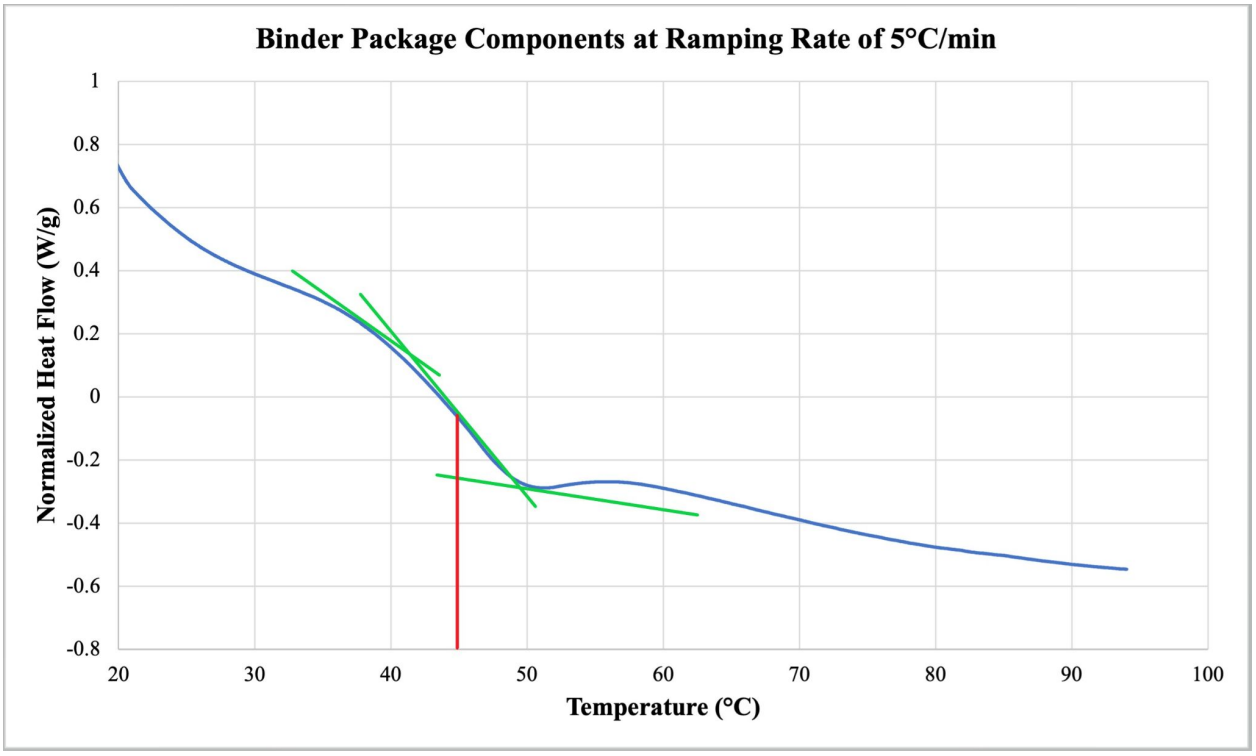

Figure 11: DSC curve at 5°C/min ramping rate with Tg identified

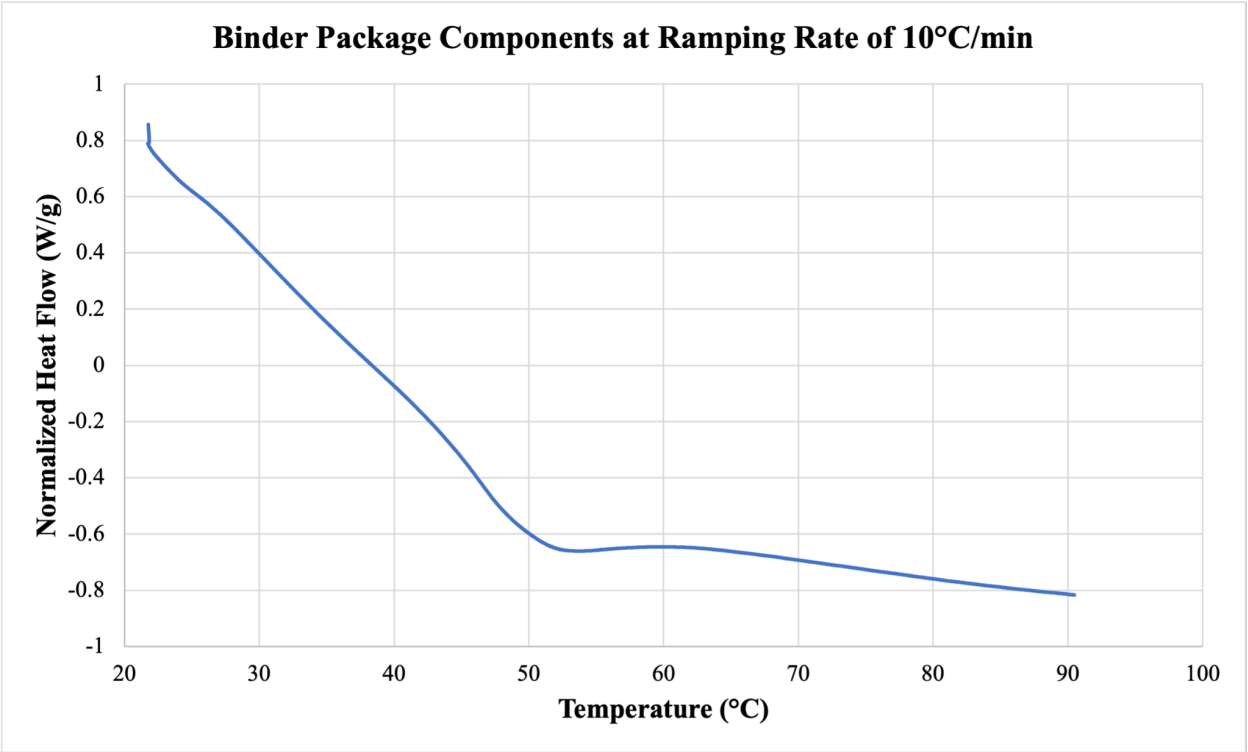

Figure 12: DSC curve at 10°C/min ramping rate

Elevated Temperature Testing

The most significant results were collected during elevated temperature testing. Figure 13 shows the cumulative mass loss over a range of temperatures that were achievable in the lab setting. The general trend of the curves shows that as the temperature increases, the pellet mass loss is greater. Benchmark tests were done at room temperature and were used as a baseline to compare to the other temperatures. The pressing process at room temperature was much more efficient compared to higher temperatures because the die did not need to be heated, which allowed for sixteen pellets to be pressed. The additional time needed to heat the die in other tests limited pressings to ten pellets. Pressing quantity was further reduced at very high temperatures around 100°C, due to difficulties handling the die, thus requiring more time for each press. The next tests were conducted at 35, 45, and 55°C. Powder losses at these temperatures were not significantly higher than room temperature, contrary to the behavior shown at very high temperatures. For 90°C and 100°C, there was much more extreme pellet loss. Interestingly, at 55°C the mass loss was very close to losses observed for the room temperature baseline pressings. This was unexpected, as it was anticipated that the room temperature curve should have the best results. Therefore, a second trial was conducted to verify the initial results, and there was even less pellet mass loss. Details on this behavior are in the *Discussion* section.

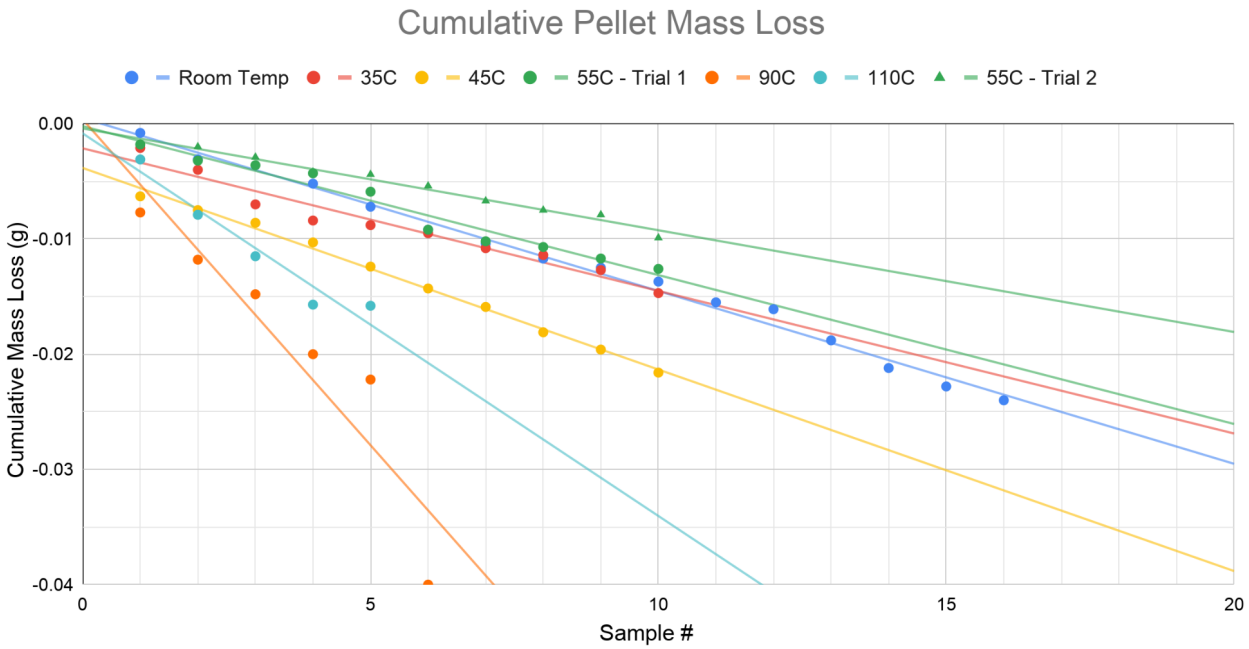

Figure 13: Cumulative pellet mass loss at a range of temperatures

Discussion

As mentioned previously, temperature had the most noticeable effect on the powder. A gradual increase in pellet mass loss was observed as the temperature increased, however, the first trial at 55°C showed similar mass loss behavior to the room temperature experiment. There are several possible reasons.

First, the ambient humidity could have had a significant consequence on the binder package. As mentioned in the *Background* section, humidity can have a significant effect on the T_g of a binder package, particularly with PVA and PEG. A change to the viscosity of the binder package could change how it behaves in the pressing process. To verify this, Table 1 shows the corresponding ambient humidity with each experiment. It can be seen that there is a dramatic difference between the room temperature experiment and the majority of other experiments. If a high humidity was responsible for lower mass loss, then the first trial at 55°C supports this claim compared to room temperature. However, the second trial at 55°C was done at much lower humidity and had even better results. This is a clear contradiction and indicates that humidity cannot be the sole factor contributing to pressing. Further testing at controlled humidity is essential to verify if any correlation exists at all.

Table 1: The Ambient Humidity and External Die Temperatures over Time for Elevated Temperatures Experiments

Die Temperature (°C)	Ambient Humidity (%)[1]	T_1 (°C)	T_2 (°C)	T_3 (°C)
Room Temperature	80	25	25	25
35	35	35.1	34.3	32.4
45	16	44.8	41.6	38.0
55 - Trial 1	65	55.1	50.0	42.7
90	35	93.4	63.5	46.1
110	20	115.8	90.2	63.3
55 - Trial 2	45	55.0	51.6	44.9

[1] Approximate average ambient humidity in the afternoon (2-6pm); data gathered from National Atmospheric and Oceanic Administration

Second, T_g in the RTP powder might be affected by the exact ratio of PVA and PEG. It was suspected that the PVA to PEG ratio in the RTP powders may be different from the nominal ratio, which may have a significant impact on T_g. Additional DSC tests of different binder ratios are shown in Figures 14 and 15. Compared to Figure 11, the curves do not show a significant difference in T_g; i.e., T_g is around 45°C in all these three different conditions.

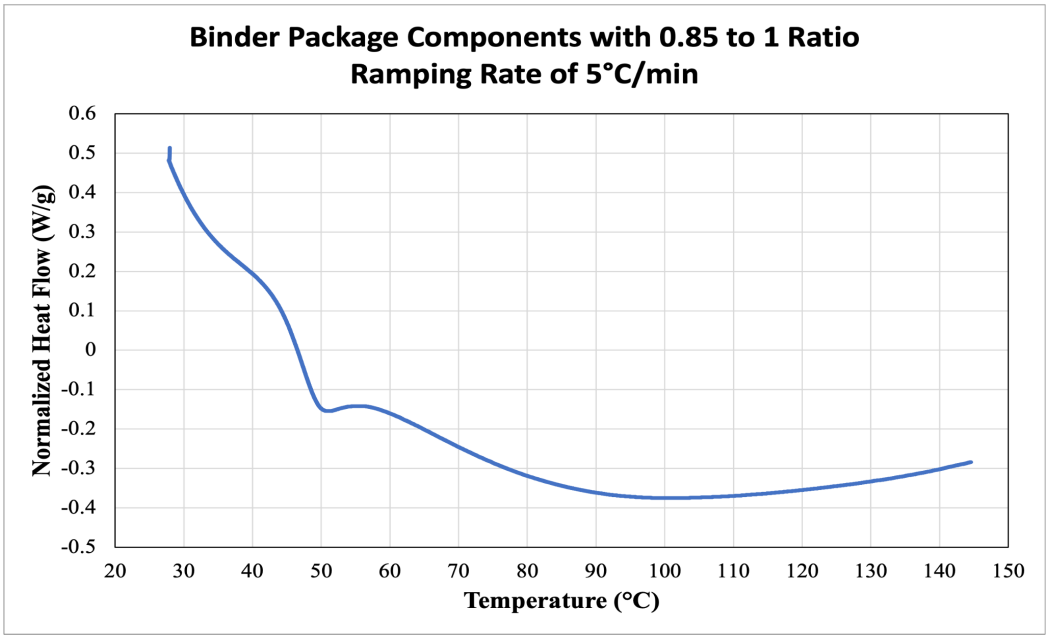

Figure 14: DSC curve at 5°C/min with a PVA to PEG ratio of 0.85:1

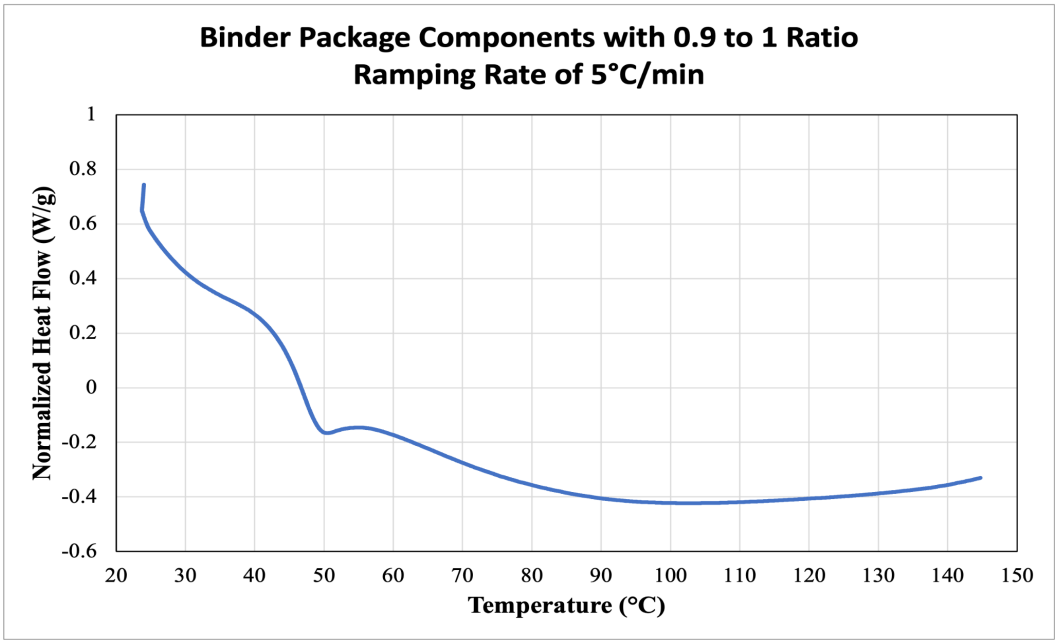

Figure 15: DSC curve at 5°C/min with a PVA to PEG ratio of 0.90:1

Third, the exact temperature of the die throughout the pressing process is different from the nominal temperature. As mentioned previously, only the external surface temperature of the die was measured. However, the internal temperature is the most critical as the interaction between the powder and die will take place there. Since the die was not heat soaked, it is suspected that the interior of the die was lower than the exterior temperature. Throughout the pressing operation, the temperature of the die often dropped by 10°C at lower temperatures and by as much as 30°C at higher temperatures. Table 1 lists these temperatures as averages across the whole experiment. Individually data points are available in Appendices B-G. The nominal temperature, T_1, was measured when the powder was loaded into the die. T_2 was measured after the powder was compacted at the beginning of holding and T_3 was measured after the pellet was ejected from the die. For every single experiment, the temperature differences of these three can be very different. Looking specifically at the two trials of 55°C, there are two main observations. The first is that T_2 is quite close to T_g. In addition to this, it is suspected that the interior of the die was even lower as mentioned above and thus even closer to T_g. Considering both of these factors, it is believed that temperature of the powder and die interior were close to or below T_g, causing the unusual results.

Conclusion and Recommendations

This project aimed to identify and quantify the factors that contribute to ceramic powder sticking during pressing operations. A method to quantify the amount of powder sticking in the press was developed, as was a procedure to operate the carver press both at ambient and elevated temperatures. The glass transition temperature of the binder mixture was also measured via DSC to compare it with the results and how the sticking problem varied with changing temperature, particularly in relation to the glass transition temperature. Extensive testing and subsequent analysis were performed, and the trend of powder behavior at elevated temperature was derived.

The primary parameter investigated concerning the sticking phenomena was temperature. It was found that the mass lost per press at higher temperatures was consistently more significant than at lower temperatures, especially at temperatures much higher than the glass transition temperature. The only exception to this occurred when the temperature was nominally 55°C, at which powder losses were lower than in any other trials, including those at room temperature. It is believed the internal temperature of the die during pressing was probably close to the glass transition temperature of the binder (45°C). This suggests pressing at temperatures comparable but lower than the glass transition temperature would be beneficial. To take advantage of this phenomenon, it is recommended that the glass transition temperature be measured and treated as one additional critical factor during the new binder system design.

The current project explored how to quantify the sticking phenomena during the ceramic powder pressing procedure; it paves the way to investigate and eventually alleviate the sticking issue comprehensively. Here are some recommendations: First, the internal temperature of the die during pressing is critical. A method of monitoring the actual temperature on the inner surface of the die would be greatly needed. Second, besides the temperature factor we explored, several other factors need to be examined to see their impact on the sticking problem. For example, it may be prudent to do experiments with spray-dried powder to see how its sticking behavior differs. Other parameters necessary for further investigation into the sticking phenomenon are the binder package composition and the ambient humidity, die wall friction, ejection force, die lubrication, and part geometry; any could be the subject of future studies (Briscoe & Evans, 1991; Tousey, 2003). In particular, humidity control during the pressing process is recommended to rule out its possible changes in the glass transition temperature and the pressing behavior of the binder mixture. Also, die lubrication also can significantly affect the results by providing a buffer layer or alleviating the jamming of the die that became debilitating at higher temperatures. Overall, future experiments are recommended to confirm and investigate an outlier in the results that diverged from the observed trend, observe the influence of alternative variables, and verify the data.

Broader Impacts

Among the Engineering Code of Ethics tenets, the most crucial is to 'Hold paramount the safety, health, and welfare of the public' (NSPE, 2021). This means prioritizing the well-being of the public over profit and providing quality goods to customers that fulfill their purpose without failure because ceramics are essential and ubiquitous with several commercial purposes and have nearly endless applications. Traditional ceramics are used for their mechanical properties, such as textiles, structural materials, decoration, thermal and electrical insulation, and more. Advanced ceramics were designed and tailored to suit their applications, such as in technical components like spark plugs and catalysts, specialized applications like piston rings and water pump seals, and solid-state electronics as semiconductors, resistors, capacitors, and more (Ewsuk, 1999). This means assuring the quality of ceramic products is necessary to ensure the welfare of the public. Furthermore, the ceramics industry often negatively impacts the communities involved in its harvesting/production of the raw materials and their surrounding environment (Kuasoski et al., 2020). So far, efforts to alleviate these impacts and implement clean, sustainable solutions have been insufficient (da Silva et al., 2017). By eliminating the loss of raw materials associated with powder sticking and its associated costs, this project has the potential to ease the burden of the ceramics industry on consumers, the environment, and society, while simultaneously ensuring the quality of ceramic products produced via pressing operations.

There is no apparent legislation governing powder sticking or pressing parameters; instead, this subject is governed by industry best practices and the practicalities of mass production. Given the prevalence of green bodies in the ceramics manufacturing industry, powder sticking in the press can ruin pieces or even entire batches when defects are introduced during the pressing process. This necessitates a quality control apparatus, which, along with wasted material for ruined products, adds costs that are then passed on to the consumer. Furthermore, if a defective ceramic green body is sintered and subsequently fails or gives a defective product, the additional costs incurred in time and energy will also be passed on to both the consumer and the environment (due to the energy expended to sinter the faulty ceramic). Similar sticking issues have plagued other industries involving pressing operations. When similar powder compaction processes are involved, such as in pill manufacturing for the pharmaceutical industry, or where pressing operations are also conducted at elevated temperature, likewise experiences similar issues and must account for their eventuality (Tousey, 2003; Rieser et al., 2008).

Better quantification of this phenomenon and identifying its contributing factors can significantly aid in mitigating this issue. Once the root causes are identified, meaningful countermeasures can be taken, and progress can be made towards addressing sticking across these industries. If an effective way to alleviate this problem were found, it would prove substantially advantageous. Such a solution could be implemented by altering standard operating procedures, potentially changing the industry standard, ultimately leading to reductions in operating and quality control costs, saving the customer money, the company time and expense, and the environment its associated burden.

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Appendix A: Room Temperature Pressing

Sample #	Initial Weight (g)	Final Weight (g)	Difference (g)	Cumulative Difference (g)	Percent Difference	Notes
1	0.5014	0.5006	-0.0008	-0.0008	-0.16%	
2	0.5	0.4977	-0.0023	-0.0031	-0.46%	
3	0.5062	-	-	-0.0031		Pellet lost during pressing
4	0.5042	0.5021	-0.0021	-0.0052	-0.42%	
5	0.5023	0.5003	-0.002	-0.0072	-0.40%	
6	0.5036	0.5016	-0.002	-0.0092	-0.40%	Started using 3rd batch of powder
7	0.5107	0.5095	-0.0012	-0.0104	-0.23%	
8	0.5037	0.5024	-0.0013	-0.0117	-0.26%	
9	0.5044	0.5036	-0.0008	-0.0125	-0.16%	
10	0.5078	0.5066	-0.0012	-0.0137	-0.24%	
11	0.5016	0.4998	-0.0018	-0.0155	-0.36%	
12	0.5002	0.4996	-0.0006	-0.0161	-0.12%	
13	0.4999	0.4972	-0.0027	-0.0188	-0.54%	
14	0.4993	0.4969	-0.0024	-0.0212	-0.48%	
15	0.5002	0.4986	-0.0016	-0.0228	-0.32%	
16	0.494	0.4928	-0.0012	-0.024	-0.24%	
Total Weights	7.5333	7.5093	-0.024			
	Total Percentage Weight Loss		-0.32%			

Appendix B: 35°C Pressings

Sample #	Initial Weight (g)	Final Weight (g)	Difference (g)	Cumulative Difference (g)	Percent Difference	T_1 (°C)	T_2 (°C)	T_3 (°C)	Notes
1	0.5019	0.4998	-0.0021	-0.0021	-0.42%	34.8	34.5	32.6	
2	0.5066	0.5047	-0.0019	-0.004	-0.38%	34.8	32.9	31.3	
3	0.5088	0.5058	-0.003	-0.007	-0.59%	35.8	34.9	31.8	
4	0.5015	0.5001	-0.0014	-0.0084	-0.28%	34.6	34	32.5	
5	0.5002	0.4998	-0.0004	-0.0088	-0.08%	34.3	33.7	31.1	
6	0.5023	0.5016	-0.0007	-0.0095	-0.14%	35.2	34.3	33.1	
7	0.5002	0.4989	-0.0013	-0.0108	-0.26%	35.3	35.2	32.4	Force needed for die separation
8	0.5051	0.5045	-0.0006	-0.0114	-0.12%	35.2	34.9	32.3	
9	0.5062	0.5049	-0.0013	-0.0127	-0.26%	35.1	35.2	33.6	
10	0.4987	0.4967	-0.002	-0.0147	-0.40%	35.4	33.7	32.9	
Total Weights	5.0315	5.0168	-0.0147		**Average Temperature**	35.05	34.33	32.36	
	Total Percentage Weight Loss		-0.29%						

Appendix C: 45°C Pressings

Sample #	Initial Weight (g)	Final Weight (g)	Difference (g)	Cumulative Difference (g)	Percent Difference	T_1 (°C)	T_2 (°C)	T_3 (°C)
1	0.5022	0.4959	-0.0063	-0.0063	-1.25%	44.1	40.8	35.5
2	0.5011	0.4999	-0.0012	-0.0075	-0.24%	45.1	42.1	38.7
3	0.501	0.4999	-0.0011	-0.0086	-0.22%	45.8	42.7	39.2
4	0.5029	0.5012	-0.0017	-0.0103	-0.34%	45	41.7	38.7
5	0.5001	0.498	-0.0021	-0.0124	-0.42%	44.1	42.8	38.2
6	0.4985	0.4966	-0.0019	-0.0143	-0.38%	44.9	40.1	38.5
7	0.4998	0.4982	-0.0016	-0.0159	-0.32%	44.9	44	37.3
8	0.501	0.4988	-0.0022	-0.0181	-0.44%	45.1	41.5	38.5
9	0.5016	0.5001	-0.0015	-0.0196	-0.30%	45	40.8	37.8
10	0.5008	0.4988	-0.002	-0.0216	-0.40%	43.7	39.8	37.3
Total Weight	5.009	4.9874	-0.0216		**Average Temperature**	44.77	41.63	37.97
	Total Percentage Weight Loss		-0.43%					

Appendix D: 55°C Pressings - Trial 1

Sample #	Initial Weight (g)	Final Weight (g)	Difference (g)	Cumulative Difference (g)	Percent Difference	T_1 (°C)	T_2 (°C)	T_3 (°C)
1	0.5026	0.5008	-0.0018	-0.0018	-0.36%	55.7	52.5	40.4
2	0.495	0.4936	-0.0014	-0.0032	-0.28%	55.7	49	42.2
3	0.5034	0.503	-0.0004	-0.0036	-0.08%	55.3	48.8	40.4
4	0.4998	0.4991	-0.0007	-0.0043	-0.14%	54.5	51.3	43
5	0.5019	0.5003	-0.0016	-0.0059	-0.32%	54.6	49.6	45.4
6	0.5031	0.4998	-0.0033	-0.0092	-0.66%	55.5	49.8	43.4
7	0.4977	0.4967	-0.001	-0.0102	-0.20%	54.8	51.5	44.5
8	0.4971	0.4966	-0.0005	-0.0107	-0.10%	55.6	51.9	44.3
9	0.4986	0.4976	-0.001	-0.0117	-0.20%	55	47.4	43.2
10	0.5032	0.5023	-0.0009	-0.0126	-0.18%	54.2	47.8	39.9
Total Weights	5.0024	4.9898	-0.0126		**Average Temperature**	55.09	49.96	42.67
	Total Percentage Weight Loss		-0.25%					

Appendix E: 55°C Pressings - Trial 2

Sample #	Initial Weight (g)	Final Weight (g)	Difference (g)	Cumulative Difference (g)	Percent Difference	T_1 (°C)	T_2 (°C)	T_3 (°C)
1	0.502	0.5002	-0.0018	-0.0018	-0.36%	55	52	46
2	0.5049	0.5047	-0.0002	-0.002	-0.04%	55.5	54.8	46.7
3	0.4971	0.4962	-0.0009	-0.0029	-0.18%	55.1	52.5	45.5
4	0.5067	0.5054	-0.0013	-0.0042	-0.26%	55.2	48.6	42.4
5	0.4956	0.4954	-0.0002	-0.0044	-0.04%	54.6	51.3	47.7
6	0.5021	0.5011	-0.001	-0.0054	-0.20%	54.6	51.6	41.3
7	0.5045	0.5032	-0.0013	-0.0067	-0.26%	55	50.6	43.1
8	0.5022	0.5014	-0.0008	-0.0075	-0.16%	54.8	52.7	46.2
9	0.5019	0.5015	-0.0004	-0.0079	-0.08%	55	50.6	44.9
10	0.4973	0.4953	-0.002	-0.0099	-0.40%	55.1	51.3	45
Total Weights	5.0143	5.0044	-0.0099		**Average Temperature**	54.99	51.6	44.88
	Total Percentage Mass Loss		-0.20%					

Appendix F: 90°C Pressings

Sample #	Initial Weight (g)	Final Weight (g)	Difference (g)	Cumulative Difference (g)	Percent Difference	T_1 (°C)	T_2 (°C)	T_3 (°C)
1	0.5011	0.4934	-0.0077	-0.0077	-1.54%	94.8	61.2	43.8
2	0.5012	0.4971	-0.0041	-0.0118	-0.82%	96.7	68.7	47
3	0.502	0.499	-0.003	-0.0148	-0.60%	85.5	57	40
4	0.501	0.4958	-0.0052	-0.02	-1.04%	99.8	72.7	56.1
5	0.5064	0.5042	-0.0022	-0.0222	-0.43%	91.5	62.2	47.3
6	0.5008	0.483	-0.0178	-0.04	-3.55%	92.1	59	42.3
Total Weight	3.0125	2.9725	-0.04		**Average Temperature**	93.4	63.47	46.08
	Total Percentage Weight Loss		-1.33%					

Appendix G: 110°C Pressings

Sample #	Initial Weight (g)	Final Weight (g)	Difference (g)	Cumulative Difference (g)	Percent Difference	T_1 (°C)	T_2 (°C)	T_3 (°C)	Notes
1	0.4984	0.4953	-0.0031	-0.0031	-0.62%	106.5	93.6	60.1	
2	0.5006	0.4958	-0.0048	-0.0079	-0.96%	110.6	77.7	52.6	
3	0.4978	0.4942	-0.0036	-0.0115	-0.72%	105.7	85.6	56.2	
4	0.5001	0.4959	-0.0042	-0.0157	-0.84%	140.6	109.2	83.3	Too Hot for repeated presses
5	0.4983	0.4982	-0.0001	-0.0158	-0.02%	115.4	84.7	64.1	
Total Weight	2.4952	2.4794	-0.0158		**Average Temperature**	115.76	90.16	63.26	
	Total Percentage Weight Loss		-0.63%						