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THE EFFECT OF ACCELERATING CURE TIMES VIA ELEVATED TEMPERATURES ON SILICONE GLUE'S ADHESIVE PROPERTIES

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ABSTRACT

The fabrication time of common soft robotics components is severely hampered by the days-long cure times of common silicone glues. Prior research suggests that curing time can be thermally accelerated via curing the glue at elevated temperatures without compromising the glue's mechanical properties [1]. Silicone samples were fabricated using a custom injection molding machine, before being bonded together with two different silicone glues, then allowed to cure at conditions of 21°C for 7 days, 40°C for 24 hours, and 80°C for 3 hours before being torn apart in a peel test employing an Instron. It was found that there is no statistically significant relationship between the either glue's adhesive mechanical properties and the cure temperatures, suggesting that the fabrication time of soft-robotic components can be reduced by a factor of up to 55 times without compromising the material properties of the glue.

Keywords: Silicone Glue, Adhesive Properties, Thermal Acceleration, Silicone Cure Time

1. INTRODUCTION

The field of robotics has grown in both utility and complexity with the unparalleled advancement of technology in recent decades. Robots are currently employed across numerous manufacturing, medical, and logistics industries, and their use will only increase with time. However, many of the design constraints and limitations in robotics today come as a consequence of these machines being primarily comprised of rigid materials such as metals and hard plastics. In an attempt to overcome the drawbacks due to material choice, a significant amount of robotics research over the past two decades has been focused on the subfield of soft robotics [2]. Soft robotics as a field seeks to draw upon inspiration from nature and biology to create adaptive, novel solutions to complex interactions in unpredictable environments. This is achieved by creating robotic systems that primarily utilize soft, compliant, flexible, and/or elastic materials such as silicone in their design. [3].

The typical fabrication process for a soft robotics component involves curing a silicone or other elastomer in a mold for some number of hours-to-days before over-molding the part with another kind of elastomer and/or gluing the component to other parts with silicone-based glues. The cure time of these silicone glues is typically on the order of multiple days, and is by far the largest barrier to rapid part fabrication and thus prototyping. Nevertheless, silicone glues continue to be employed in the field despite their long cure times due to their low cost and chemical compatibility with the silicones which soft robots are most often comprised of. [4]. As such, experimental evidence demonstrating whether silicone glues' cure times can be significantly and safety shortened via thermal acceleration could be extremely fruitful. It has the potential to inform researchers how to most efficiently conduct the fabrication of their soft robots, and to do so without fear of compromising the strength of any adhered joints. Earlier research by Yap et al. into the many possible optimizations of the soft robotics manufacturing process primarily explores the effect of thermal acceleration on the cure times of the component materials themselves–not the bonds between them [1]. The mechanical behavior of silicone glues which have undergone thermal acceleration of their cure times ought to be quantitatively analyzed.

To this end, the adhesive properties of two commonly employed silicone glues, Silpoxy and Red Devil Silicone Sealant, were examined [5, 6]. The glues were used to adhere two injection-molded blocks of Smooth-On's Dragon Skin 30 silicone together [7]. Three groups of these bonded blocks were cured for different periods of time at corresponding elevated temperatures: room temperature (roughly 21°C) for the manufacturer recommended seven days, 40°C for 24 hours, and 80°C for 4 hours. The force, and the distance over which said force was applied in order to completely separate each of the bonded silicone samples, were recorded using an Instron. The area under the resulting force-displacement curves was used to calculate the energy required to pull each sample apart, and served to quantify each sample's adhesive capabilities. These energies were compared to against their curing conditions to determine the governing relationship between the two variables. It was found that there is no statistically significant negative correlation between either glue's adhesive strength and the various curing conditions, suggesting that current soft robotic manufacturing processes can be greatly expedited.

2. BACKGROUND

In the field of soft robotics, manufacturing efficiencies can be greatly improved by leveraging thermal acceleration of curing times for silicone glue used in component assembly. Such leveraging is contingent upon understanding the relationship governing the glue's cure time and temperature, and its resultant adhesive strength, by examining prior works. One method to quantify this relationship is through calculating the adhesive fracture energy, G_a, from force-displacement data obtained during peel tests. We expect the magnitude of this value to differ significantly between Red Devil Silicone Sealant and Silpoxy due to the differing mechanical properties and chemical compositions of each.

2.1 Adhesive Fracture Energy

A very established and well-founded method for quantifying and comparing the adhesive properties of an adhesive is through the calculation of its adhesive fracture energy, G_a [8]. It can be determined experimentally through a peel test, illustrated in Fig. 1. During this test, the laminate is slowly peeled apart while the force being applied to it and the distance over which the peel occurs is recorded. The shape of the resulting force displacement curves most often look similar to those shown in Fig. 2.

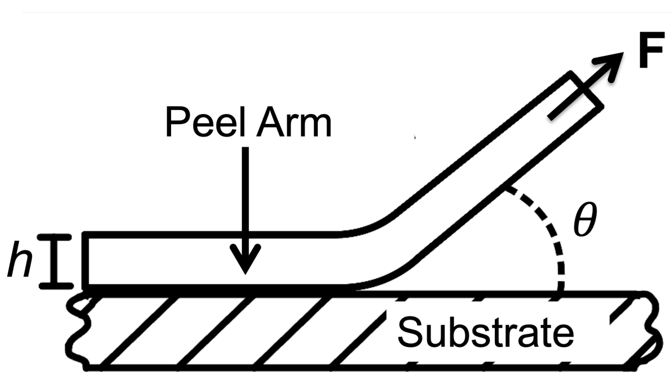

FIGURE 1: Example of a simple peel test, in which a laminate of thickness *h* is separate from a rigidly-secured substrate via peel force *F* at a constant angle *θ*. The average of *F* can be used to calculate the laminate's adhesive fracture energy [8].

Within the context of a peel test, G_a is considered to be a combination of the strain energy stored in the laminate's peeling arm, which is comprised of "the energy dissipated during tensile deformation of the peeling arm, and the energy dissipated due to the bending of the peeling arm" [8]. A specific numerical value

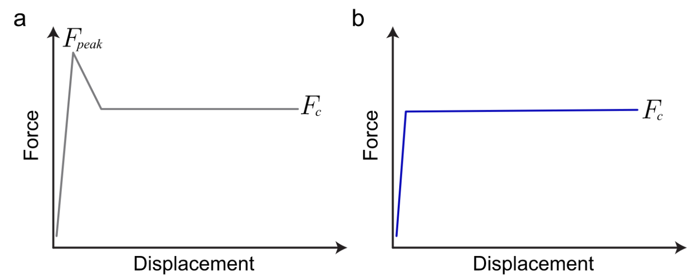

FIGURE 2: (a) Force-displacement curve with a peak force F_{peak} necessary to begin the peel, followed by a steady stated critical force F_c necessary to continue the peel to completion. (b) Force-displacement curve for a peel test in which a steady-state F_c is reached immediately [9].

for G_a for a given sample laminate can be calculated using

$$G_a = \bar{F}(\frac{1 - cos\theta}{w}) + \frac{\bar{F}^2}{2Ew^2h} \quad (1)$$

where $\bar{F}$ is the average of the force applied to the peel arm throughout the peel test, θ is the angle of the peel and is typically held constant during a peel. w is the width of the sample (into the page in Fig. 1), h is the thickness of the sample, and E is the Young's Modulus of the peel arm material [8].

G_a is typically found to be in the order of magnitude of hundreds or thousands of joules, depending heavily on the material the laminate is comprised of (aluminum, silicone, etc.) and what adhesive technique is being used to bond the laminate's components together [10].

2.2 Adhesive Behaviors of Silpoxy and Red Devil

Silpoxy and Red Devil Silicone Sealant are two popular adhesives in the field of soft robotics due to their high flexibility, strong adhesive properties, and compatibility with a wide range of silicone elastomers. It should be noted that their differing chemical compositions mean that we expect the values of the adhesive fracture energy of samples bonded with Red Devil to be on the order of thousands of joules per squared meter, while those bonded with Silpoxy to be on the order of only hundreds. This is due to the fact that when bonded to silicones, Silpoxy is expected to fail adhesively: the glue fails by separating from the materials it is bonded to as illustrated in Fig. 3B. In contrast, Red Devil tends to fail cohesively, meaning that the glue's internal bonds pull apart from one another, while managing to maintain contact and adhesion with each side of the sample. This is shown in Fig. 3A.

2.2.1 Silpoxy. Silpoxy is a silicone adhesive manufactured by Smooth-On, specifically designed for bonding silicone rubbers to each other and to various substrates. It is widely appreciated in soft robotics for its clear finish, flexible bonds, and compatibility with other Smooth-On products like Dragon Skin and Ecoflex. Silpoxy is capable of curing at room temperature and does not require any special treatment [5]. However, the possibility of thermally accelerating its curing process opens up new avenues for speeding up assembly times in soft robotics manufacturing.

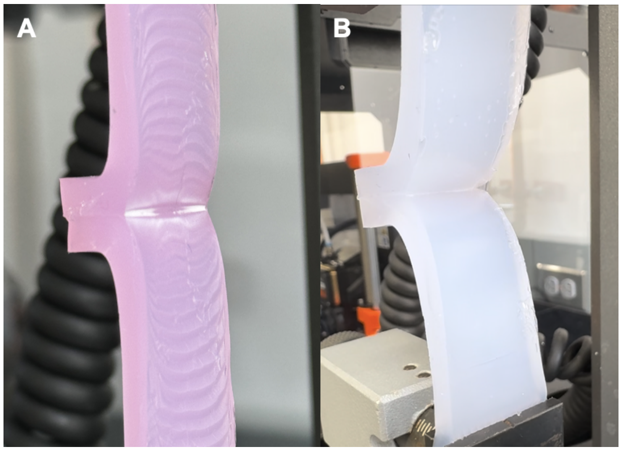

FIGURE 3: (A) Cohesive failure of Red Devil Silicone Sealant, as indicated by the white ripples present in the glue as the sample is ripped apart. (B) Adhesvier failure of Silpoxy, as indicated by a lack of the previoulsy mentioned white ripples. All glue can be seen attached to the top half of the sample.

2.2.2 Red Devil Silicone Sealant. Red Devil Silicone Sealant is a more general-purpose silicone adhesive, known for its durability and resistance to weathering. It is most frequently employed in both indoor and outdoor architectural applications. In the context of soft robotics, Red Devil is favored for its high adhesive strength and tendency to fail cohesively rather than adhesively. Because of this, measured values of G_a for Red Devil are expected to be significantly higher than those of Silpoxy across all curing conditions. But similar to Silpoxy, Red Devil is typically cured at room temperature over a period of several days, and is a primarily silicone based glue [6]. As such, it is reasonable to attempt to thermally accelerate both clue's cure times by assuming they each follow a very similar cure time–temperature relationship.

2.3 Thermally Accelerating Silicone Cure Times

The characteristic shape of any cure time–temperature relationship is specific to that material-type as a result of its particular mechanical and chemical properties. For silicones, the important mechanical factor at play is gelation–the process by which silicone transforms from a liquid state to a gel state, where the fluid becomes thickened and solidified. Recent work by Yap et al. demonstrates that thermal acceleration does not detrimentally affect the mechanical properties of fully cured silicones so long as the time of this gelation, or cure, t, reduces inversely to an increasing cure temperature, T, in a manner which obeys a Arrhenius exponential decay relationship visualized in Fig. 4 and expressed algebraically as:

$$\ln(t) = \frac{E_a}{RT} - \ln(A) \tag{2}$$

where E_a is the material's activation energy, A is a constant that is proportional to the Arrhenius frequency factor, and R is the gas constant.

Unfortunately, Yap et al.'s work was limited to that of silicone elastomers such as Dragon Skin and Ecoflex [1]. While their

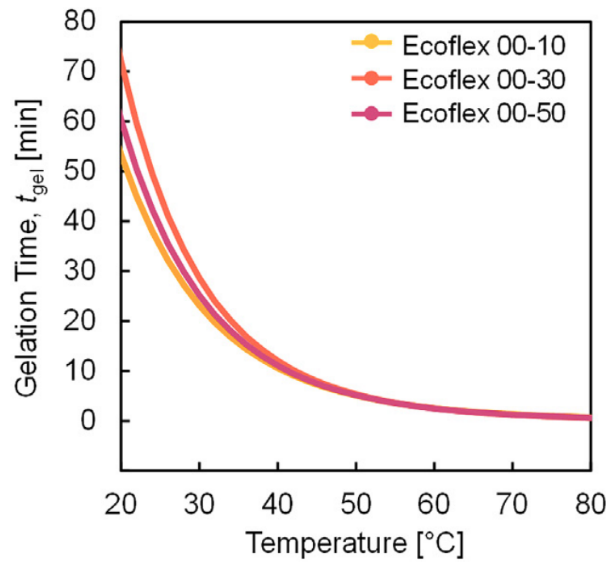

FIGURE 4: Exponential decay relationship between the gelation or cure time t_{gel}, and temperature T of three varieties of EcoFlex silicone elastomer. Each t_{gel} was determined by measuring via mechanical spectroscopy how long it took for the loss tangent between each elastomer sample's storage modulus G' and loss modulus G'' to acheive unity [1]. Examination of these curves allows one to extrapolate the necessary reduction in cure times and elevation in temperatures for the curing of other silicone-based materials.

model can theoretically be extended to Silpoxy and Red Devil by simply inserting appropriate activation energies and proportionality constants for each glue into the equation, at the time of writing, these values are unknown and difficult to determine experimentally. As such, a precise numerical relationship between the glues' cure times and temperatures are currently unavailable. But previous work by Out et al. indicates that all silicone materials share highly similar activation energies, and Yap et al. finds that all silicones have reliably predictable values of A [1, 11].

Taken together, both findings suggest that Silpoxy and Red Devil's curing behaviors are very likely akin to those of more well-understood materials. This allows for a reasonably educated prediction of the necessary cure time for each glue based upon a chosen curing temperature. All of Yap et al's tested silicone elastomers saw roughly a 7-fold decrease in necessary cure time at 40°C and an 55-fold decrease at 80°C as visualized in Fig. 4 [1]. This trend informs this experiment's selection of conservatively estimated 24-hour and 3-hour cure times for each of the respective elevated temperatures in stark contrast with the original manufacturers' recommendations of 7 day cures for both Silpoxy and Red Devil at ambient room temperature (roughly 21°C).

3. EXPERIMENTAL DESIGN

To determine the adhesive fracture energies for each curing condition, it was necessary to custom fabricate samples, then conduct a type of peel test known as a T-Peel test on them using an Instron in order to generate the required force data.

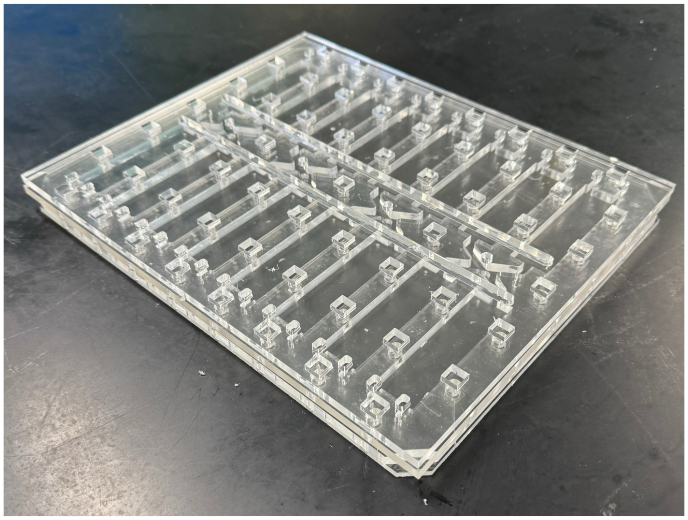

FIGURE 5: Pieces of laser cut acrylic stacked atop one another to form a complete mold shape. This custom mold helps in the manufacturing of 16 blocks of chosen material, which can be bonded together for use in peel test.

3.1 Sample Fabrication

The fabrication process was divided into three distinct steps, consisting of the injection molding of addition-type-silicone and common soft robotic component Dragon Skin 30 (DS30) blocks, the bonding of the blocks using either Silpoxy or Red Devil into testable samples, and the curing of said samples. The specific curing procedure was dependent upon each individual test temperature.

3.1.1 Injection Molding of Silicone Blocks. For ease of handling, design, gluing, and testing, custom molds for the silicone blocks were designed using CAD software. Each mold would allow for the creation 16 separate 75x45x5mm blocks at a time. They were fabricated by cutting multiple pieces of acrylic to form in a laser cutter. These individual pieces were then stacked atop one another to form the desired mold shape, as illustrated in Fig. 5, before being secured together with screws and nuts. Doing so also ensured a sufficient clamping force during the packing phase of the injection molding process.

Once prepared, DS30 was introduced to the molds through the use of a custom, hand-operated injection molding machine as described in Bell et al. and visualized in Fig. 6 [12]. This machine mixes the A and B parts of addition-type-silicones like DS30 just prior to the material's injection into the mold, allowing it to begin curing immediately . Once the molds were completely filled as shown in Fig. 7, they were set on a curing rack for 48 hours to allow the silicone to fully cure, after which the DS30 blocks were removed, and any runners, reliefs, and flash were removed by hand with a razor blade. Finally, the DS30 blocks were left to cure in ambient air for another 36 hours to allow for oxidization of the surface layer of the silicone. Doing so facilitates a stronger bond between the silicone blocks and the glues. After this period, the blocks are considered ready for gluing.

3.1.2 Bonding Silicone Blocks with Glue. The glue-ready DS30 blocks are bonded together following a series of repeated

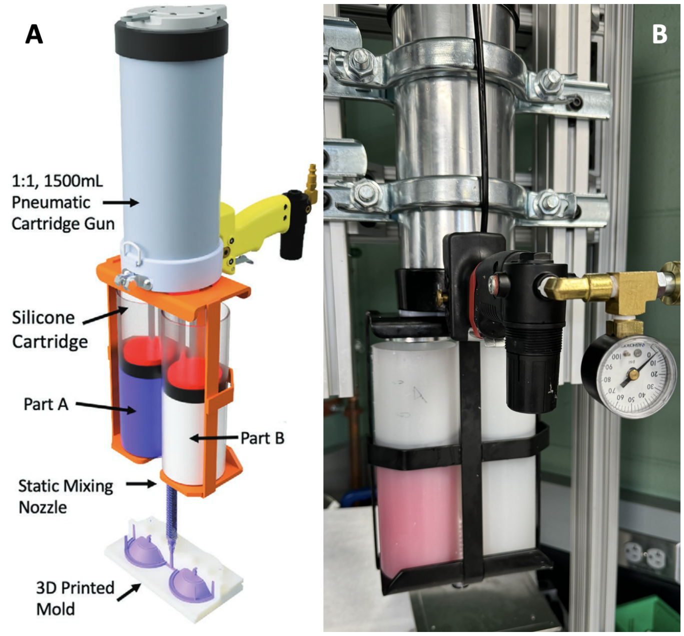

FIGURE 6: (A) Diagram of the custom, hand-operable injection molding machine as presented in Bell et al., and pictured in (B) at the MIT Fabrication-Integrated Design Lab assembled per the paper's specifications. This machine allows for rapid prototyping of parts consisting of addition-type silicones featuring A and B parts which are mixed to form the curable silicone [12].

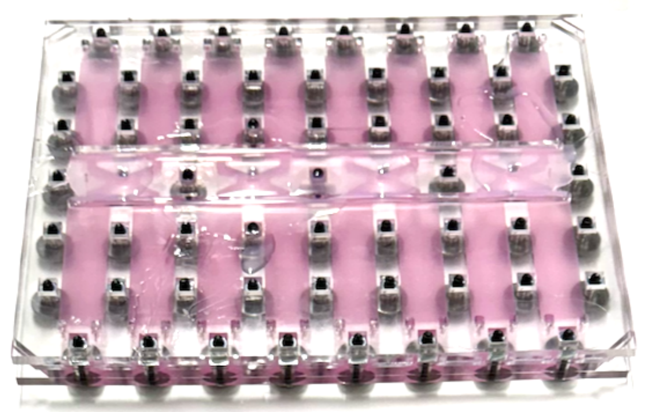

FIGURE 7: The mold shown in Fig. 5 after the pieces have been screwed together, and filled with mixed DS30 using the injection molding machine shown in Fig. 6. The use of this mold allows for the fabrication of many silicone blocks at once.

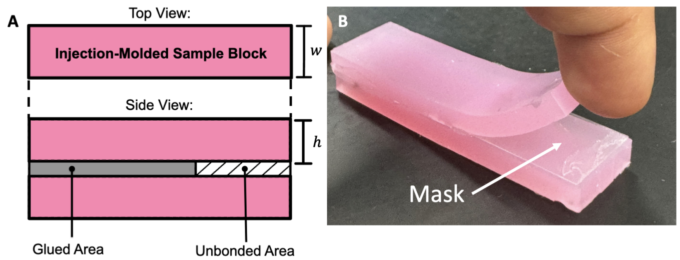

FIGURE 8: (A) Schematic diagram of a bonded DS30 sample, featuring DS30 blocks of width *w* and height *h* partially bonded together by an adhesive of choice. The unbonded area of each sample is achieved through the application of a 0.1mm thick acrylic mask shown in (B). These glued samples are used in peel tests to generate the force data needed to find a sample's Adhesive Fracture Energy [8].

steps. First, the to-be-bonded side of two blocks are cleaned with a 50% Isopropyl Alcohol solution and wiped dry with a paper towel. Then, a 0.1mm thick acrylic mask is placed one of the blocks by lining up a short side of the mask with a short side of the block. This is done to ensure part of each sample remains unbonded, so that it can be easily inserted into the Instron grips for peel testing. Glue is then applied directly from its container or tube onto the exposed area of the block with the mask, before being evenly spread across this area with a popsicle stick. Once glue is sufficiently applied to the DS30 block, the second clean block is placed atop it. Pressure is then applied with one's fingers to the top DS30 block to ensure proper adhesion between the glue and the blocks. The resulting peel test ready sample is illustrated by Fig. 8. This process is then repeated for the amount of samples desired for each curing condition, after which the samples are set to cure.

3.1.3 Curing the Glues. The precise curing procedure for each group of glued samples depends upon the selected curing temperature. For each control condition, 12 bonded samples were placed on the lab's curing rack and left to cure for 7 days at ambient air temperatures per the manufacturer's recommendation for material property testing for both the Red Devil and Silpoxy glue conditions [6]. This ambient air temperature was determined to be 21.0±0.2°C through the recording of the room's temperature over 1 week using a Vernier Temperature Probe whose data-points were collected with a LabQuest2 computer at a 20 sample/hour sampling rate.

For the 40°C and 80°C curing conditions, the elevated temperatures were achieved through the use of a Cascade Tek TFO-1 Forced Air Lab Oven [13]. For both the Silpoxy and Red Devil glues, 12 samples were placed into the oven at 40.0 ± 1.0°C for a period of 24 hours, while 8 samples were cured at 80.0 ± 1.0°C for 3 hours for the Red Devil glue, while 16 samples were cured at 80 ± 1.0°C for the same amount of time for Silpoxy. The uncertainty for the 40°C and 80°C conditions is sourced from the oven's specification sheet. Both cure times were selected per the discussion in section 2.3. Once a set of samples had cured for the full amount of time appropriate to its cure temperature, the samples were considered ready for peel testing in the Instron.

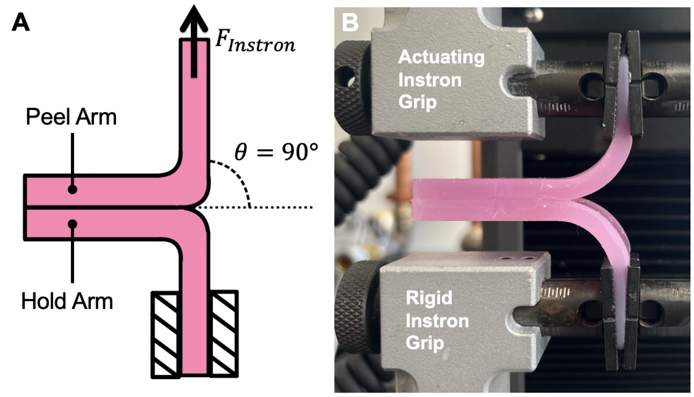

FIGURE 9: (A) Schematic diagram of the T-Peel test used to gather force-displacement data from glued samples. Each block–or arm–of the sample is held in a manual Instron grip and kept at a constant 90angle relative to the normal of the hold arm. The Instron applies a force $F_{Instron}$ continuously until a sudden and sharp drop in the magnitude of $F_{Instron}$ of at least 40% is detected. This drop indicates that the two blocks have completely separated, and that data acquisition for the sample should be ceased. (B) The start of one of these T-Peel test in the lab. The force data from this test can be used to find each sample's Adhesive Fracture Energy.

3.2 T-Peel Testing

As mentioned previously, this experiment employed a specific type of peel test, known as a T-Peel test, to gather force data from each sample. The underlying theory and set-up are the same as that discussed in section 2.1. The only major difference is that the peel angle θ is set a constant 90angle throughout test test [10]. The acrylic mask was removed from each sample, and secured in the 500N manual grips of an Instron 5940 Single Column Table Frame [14]. This Instron was used to peel apart each sample while simultaneously recording force-displacement data for each test as it proceeded. A schematic of the precise experimental set-up is seen in Fig. 9.

4. RESULTS AND DISCUSSION

The findings from the T-Peel tests were analyzed to assess the adhesive properties of Silpoxy and Red Devil silicone glues under the chosen curing conditions, focusing on the force-displacement data from which the adhesive fracture energy G_a for each sample was obtained. These values were compared against the three chosen curing conditions for each glue to reveal that there was no statistically significant negative impact on the adhesive strength of the bonds between DS30 blocks due to thermal acceleration of the glues' cure times.

4.1 Raw T-Peel Test Results

The raw force-displacement curves for each sample were graphed, grouped together by condition for qualitative visual analysis of the relative integrity of the adhesive bonds for each sample across the different cure conditions. They are displayed in Fig. 10. Figure 10 indicates the shape of the force-displacement curves remain consistent for each glue across cure conditions, which suggests that the adhesive strength of the glues is unaffected by thermal acceleration.

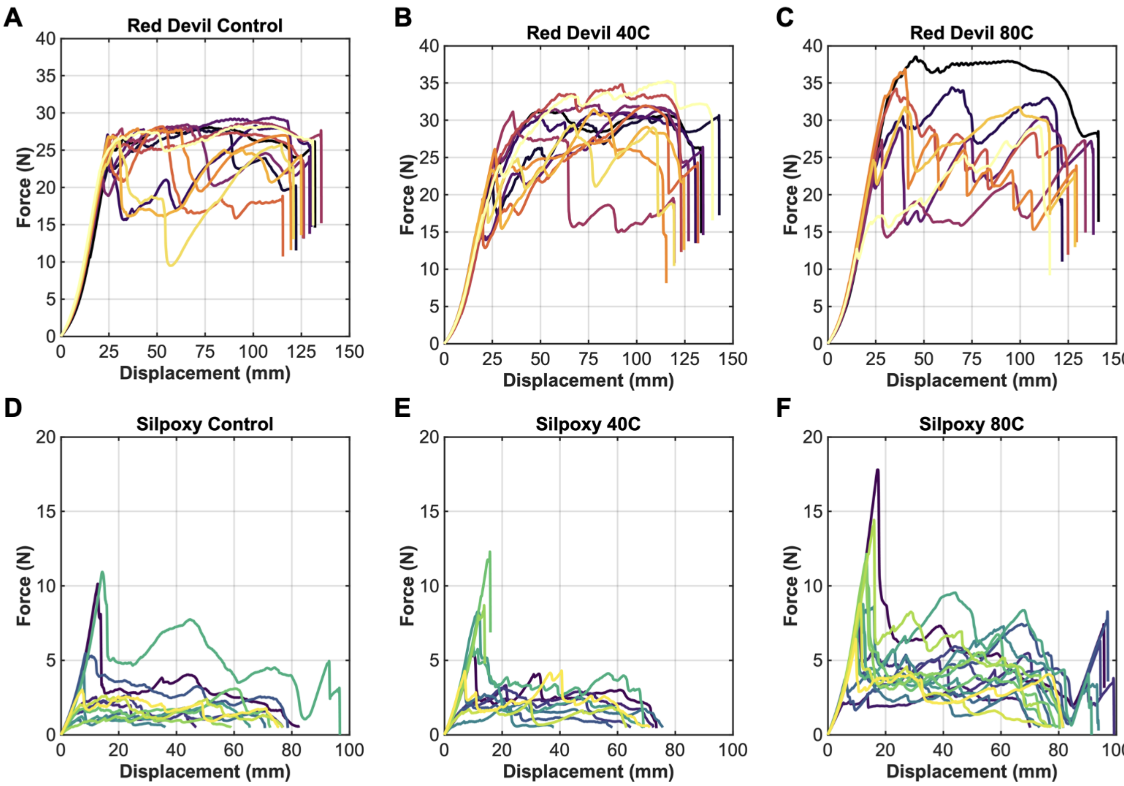

FIGURE 10: Force-Displacement curves taken from Intron T-Peel testing for (A) Red Devil Control (B) Red Devil 40°C (C) Red Devil 80°C (D) Silpoxy Control (E) Silpoxy 40°C (F) Silpoxy 80°C. Analysis of the force values during steady state delamination can be used to calculated the adhesive fracture energy of each sample. [8]

It should be noted that the shape of a typical force-displacement curve for Silpoxy is different than that of Red Devil as a result of the underlying differences in material composition discussed in section 2.2. The fact that Silpoxy tends to fail adhesively while Red Devil fails cohesively directly impacts the expected shape and magnitude of these force-displacement curves [5, 6]. As such, the difference in graph shape between glues is not an indication of differing mechanical properties as a direct result of thermally accelerated curing, emphasized by the difference in graph shapes for both of the control groups in Figs. 10A and 10D.

Additionally, many of these curves show less than the ideal steady state delamination behavior illustrated previously for a peel test in Fig. 2. This is due to the high variance present in the manually gluing process. The glue is often not applied completely evenly [4]. This leads to the formation of air pockets which account for sudden drops in the peel force like that seen in some Red Devil Control specimens in Fig. 9A. There is suddenly much less glue present in one section of the sample to resist the pull of the Instron, which in practice translates to a drop in measured necessary force. Nevertheless, the presence of these air pocket drops across cure conditions further supports that thermal acceleration of glue curing times does little to affect the glues adhesive properties.

4.2 Analysis of Adhesive Fracture Energies

The hypothesis that thermally accelerating silicone glues' cure times has no significant effect on their adhesive properties was scrutinized by first computing the average peel force for each sample. Where the steady state peeling of each sample began and ended was determined by analyzing changes in the slope of the force-displacement graphs, as visualized in Fig. 11. All force data was included between points where a sudden change in slope occurred–the force leveled out near the beginning of each T-Peel test and decreased sharply at the end once complete separation of the sample blocks had occurred.

The value for each average peel force was compared against the shape of its associated curve to ensure the software accurately captured the steady state peel region. Additional checks were in place to automatically account for the presence of air pocket drops in any of the force data. With the average peel forces determined, calculating the adhesive fracture energy for each sample using Equation 1 was straightforward as all other variables in the equation were known constants.

These energy values were subsequently plotted against the temperature conditions their associated samples were cured to find a significant relationship between the glues' adhesive fracture energies and the various cure conditions. Figure 12 illustrates that no statistically significant relationship was found amongst the Red Devil Silicone Sealant samples, which strongly suggests that cure condition of a Red Devil Silicone Sealant sample is uncoupled to

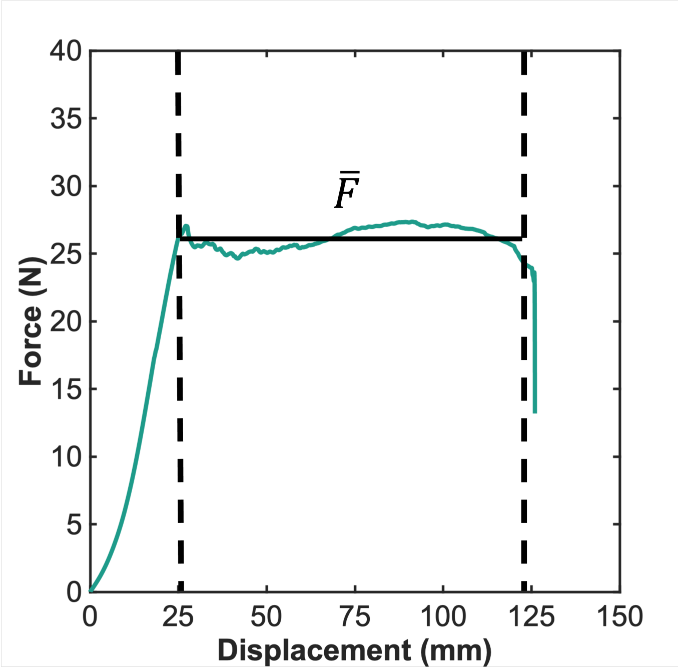

FIGURE 11: Example visualization of how the average peel force $\bar{F}$ for each sample was extracted from its raw force-displacement data for Red Devil Control Sample 7. All force values between the two dashed lines–representing signfigant changes in slope for the curve signifying the beginning and end of the steady state peel period–were averaged to obtain $\bar{F}$. This value can be used to calculate the adhesive fracture energy for each sample per Equation 1 [8].

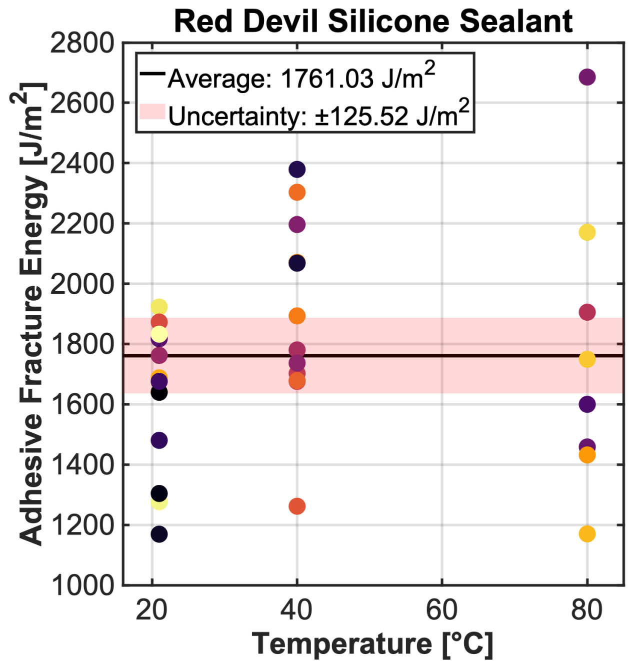

FIGURE 12: Adhesive fracture energy versus curing temperature for samples glued with Red Devil Silicone Sealant. Energies were obtained by plugging the average steady state peel force of each sample taken from raw Instron data into Equation 1. The lack of statistically significant relationship indicates that cure condition and adhesive strength are the glue are independent of one another.

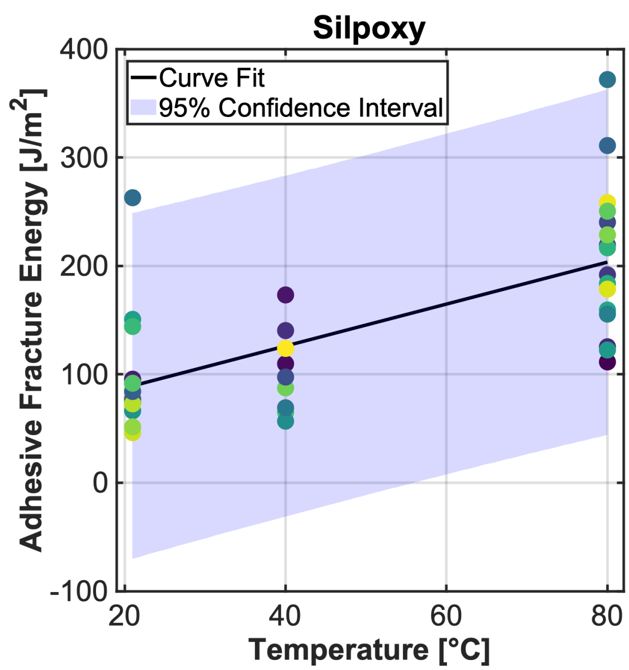

FIGURE 13: Adhesive fracture energy G_a versus curing temperature T for samples glued with Silpoxy. Energies were obtained by plugging the average steady state peel force of each sample taken from raw Instron data into Equation 1. A statistically significant relationship of $G_a = p1 * T + p2$ was found where $p1 = 1.93 \pm 0.78$ and $p2 = 49 \pm 44$. This positive linear correlation indicates a lack of drawbacks in thermally accelerating glue cure times in soft robotic manufacturing.

its adhesive fracture energy value, which indicates that thermal acceleration can be used to decrease the manufacturing time of adhered soft robotic components.

Similarly, no statistically significant negative relationship was found amongst the Silpoxy samples. And while a statistically significant positive relationship was observed as shown in Fig. 13, it is likely due to confounding variables. Notably, the DS30 blocks which comprised the 80°C Silpoxy samples were unintentionally left to air cure for approximately 60 hours–a full day longer than the other conditions. The extended exposure likely allowed for more surface oxidization of the blocks, potentially enhancing the bonding capabilities of the DS30 with the Silpoxy once the glue was eventually applied. Nevertheless, if one were to disregard this possibility, the positive linear relationship found still supports the hypothesis that reducing cure times through thermal acceleration ought to be employed in shortening soft robotic manufacturing times: because by rejecting the possibility of a confounding variable, one must accept that thermal acceleration of glue cure times only *enhances* the component's bond strength. Such a conclusion, combined with the findings for Red Devil, strongly suggests that thermal acceleration of glue cure times can be done with negligible change in mechanical properties, though the positive relationship in Silpoxy does warrant a follow-up study to confirm the findings.

The results of this experiment are exciting as they indicate serious potential for substantially reducing soft robotic manufacturing times without compromising components' adhesive strengths. It should be noted however that this study focused on the effects of thermal acceleration on cure time for specific silicone glues and their adhesive fracture energies only, and did not explore impacts on other mechanical properties like Young's or Shear Modulus, nor on materials other than DS30 or different types of glues. It was also limited in the number of temperature conditions examined and the number of samples per condition. Nonetheless, its findings seriously warrant a more comprehensive follow-up investigation. Such research would definitively determine whether soft robotic manufacturers can accelerate their processes through thermal methods without concern, thereby enhancing production efficiency across various materials and adhesives.

CONCLUSION

The findings strongly indicate that thermally accelerating the cure times of silicone glues does not detrimentally affect their adhesive properties, and may in fact enhance them. As silicone glues are very common soft robotics, this has potential to reduce the manufacturing time of soft robots significantly without compromising their material integrity. Per this study, this could be by up to a factor of 55 times for the glue curing step of the process.

More specifically, the results demonstrated that there was no statistically significant impact on the adhesive strength of Red Devil Silicone Sealant under varied cure conditions, as evidenced by the consistent shape and behavior of the force-displacement curves across all tested temperatures. For Silpoxy, the other glue tested, a statistically significant positive linear relationship between cure temperature and adhesive fracture energy was observed, but the potential of confounding factors such as extended air cure times at higher temperatures indicate that these results should be interpreted with caution.

Future studies should aim to confirm these findings for a broader range of substrate materials, glues and under more varied temperature conditions, as well as investigate other mechanical properties of the adhesives that could be affected by thermal acceleration. This would provide a more comprehensive understanding of the implications of this manufacturing technique, ensuring that the field of soft robotics can continue to advance with both speed and confidence.

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