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SCHOOL OF ENGINEERING

INVESTIGATING THE EFFECTS OF MODIFIERS ON THE MECHANICAL PROPERTIES
OF INJECTION MOLDED LIQUID SILICONE RUBBERS

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Abstract

The affects on physical properties of blending different levels by weight of talc, silica, and glass fibers into liquid silicone rubber (LSR) systems was investigated. Specifically, tensile strength, ultimate elongation, hardness, compression set, and specific gravity were measured on unmodified resins as well as successive levels of modified resins. Filler dispersion was also examined in an effort to correlate mixing, molding, and testing procedures with the results that were obtained. Two generations of materials were produced in the course of the experiment, with the second being a direct improvement upon the first in the areas of mixing, molding, and testing. It was shown that all the tested modifiers had sometimes drastic affects on the physical properties of the LSRs, while displaying both reinforcing and extending and cost saving effects. Better filler dispersion was obtained in the second generation of materials, as well as more consistent mechanical property data. Overall, it can be said that the chosen modifiers had definite effects on every physical property of the LSR systems, and the results obtained have interesting cost vs. property implications in the expansive market of LSRs.

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GENERATION 1

Introduction

The extensive use of additives, fillers, and modifiers in plastics and rubbers has become one of the foundations of success for these industries. These modifiers have the capability to turn one material into a multifaceted system that is suitable for a range of applications much wider than is even possible for their base resins. They are also able to make more expensive materials much more affordable. Applying this same methodology to liquid silicone rubber systems could yield the same benefits. Important considerations for success in this area are based around the ideals of cost effectiveness, overall property modification, and ease of use and manufacturing. Specifically for the modifiers themselves, important considerations include the dispersion of the modifier within the system as well as the compatibility between base resin and modifier. After taking all of these factors into account, the overall effect of a modifier on a resin system can be determined. This experiment was an effort to characterize modified LSR in the same manner that a traditional thermoplastic or rubber would be characterized, with the hopes of yielding useful information on how LSR resin systems perform and react when modified in traditional ways.

Statement of Theory and Definitions

Liquid Silicone Rubber

Liquid Silicone Rubber (LSR) is part of a family of thermosetting synthetic elastomers used in a wide range of engineering applications. An LSR exhibits useful properties over a wide range of temperatures, such as low viscosity for complex molding, chemical resistance, and no toxicity or contaminants. Prominent applications for these resins include electrical connectors, seals and sealing membranes, and food/medical products. [1] The unique structure of this polymer, as shown in Figure 1, is the main factor in determining these properties. These injection moldable LSRs are a combination of injection molding and thermosetting elastomer resins.
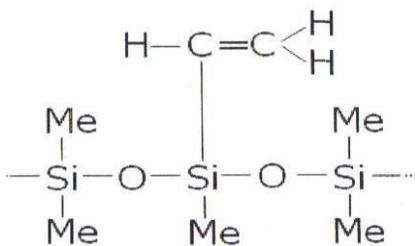
Figure 1 – Dimethyl Siloxane Monomer The foundation for the production of most LSR resins. The repeating Silicone-Oxygen backbone chain gives flexibility to the polymer, while the vinyl group gives a site for curing reactions. Courtesy of Polymer Engineering Center, U of Wisconsin-Madison.

While there is a range of variations on the LSR's structures, the most common LSR is comprised of a dimethyl siloxane polymer chain. As shown in Figure 1, the Si backbone atoms have methyl and vinyl groups attached along the chain. [2] The resin is crosslinked through a platinum catalyst curing operation, as illustrated in Figure 2.

Figure 2 - Platinum Cure Reaction The platinum catalyst reacts with the double bond in the vinyl group, creating an active site for polymer chain propagation. Courtesy of Polymer Engineering Center, U of Wisconsin-Madison.

LSR resins can also be cured using a peroxide catalyst in a similar free-radical chemistry reaction. Typically, processing these resins involves pumping and mixing the two components (A + B) that contain the ingredients for curing through a specialized injection molding machine. Once the material is pumped into the mold, it is heated to 150+ °C to start the curing process. The ejection process usually involves sweepers or robots to remove the parts from the mold. [1] Some resins require a post cure after molding to remove the remainder of the catalyst within the part. Part of an LSR's versatility stems from the different curing processes that can be used to make them. [2]

The properties of an LSR are first determined by the structure of the polymer. [2] Desired properties from LSR resins usually include extreme elongation without yield, excellent thermal stability, resistance to extreme heat and cold, and low surface energy which results in a smooth surface. [1] These properties can be changed by modifying the chemistry and number of the side groups (such as the Me groups in Figure 1). This is the most direct and widely used method to achieve specific properties. [2] Their mechanical properties, such as tensile strength, are low and therefore the uses of LSRs are usually restricted to parts that do not require high mechanical properties.

Traditional vs. Silicone Rubbers

Fillers and additives can produce a wide range of properties in LSRs as well as traditional rubbers. [3] There are a wide range of reinforcing fillers that are currently used in the rubber industry to increase the spectrum of applications of rubbers, including carbon black, silica, calcium carbonate, and mineral clay. [3] Using these additives is a simple way to change the rubber's properties. The properties that are most

affected by using these reinforcers are tensile strength, hardness, and elongation. [4] Another important factor in determining the effects of these additives is the use of coupling agents, which increase the interaction between the rubber and the filler. [4]

Since the use of fillers is so successful in the rubber industry, the same effects could possibly be seen when applying similar methods to LSRs. Instead of applying different polymerization techniques, readily available fillers could be blended in using proven techniques for rubbers. Overall, this could be a possible avenue for expanding an LSR's application range.

Processing of LSRs

Processing LSRs involves a special injection molding system that meters the two parts of the resin through a compression-free screw to provide a homogenous mix. [6] When blending additives into the system, the best control can be obtained through mixing the coupling agent and additives into the resin before it is pushed through the injection molding system. If the additives are properly dispersed within the resin before it is mixed, the combination of the two-part resin through the molding machine will be an effective blending method in terms of dispersion and interaction. Care should be taken, however, to observe the effects on the viscosity that the additives have on the resin. [6] The polymerization mechanism could also be affected by using additives. This could create problems when the resin is injected. If the additive-mixed resin can be successfully used in the molding machine, the end product will be a blended LSR.

Description of Equipment and Processes

The base LSR resins were the KE-1950-40 and KEG-2000-40 provided by Shinetsu Materials. The additives mixed into the silicones were as follows:
1. Cab-O-Sil 17D Fumed Silica, provided by Cabot Corp (referred to as SIL)
2. *Spheron 5000A Carbon Black, provided by Cabot Corp (referred to as CBL)
3. Gojo J-Glass, provided by Plastics Services Network (referred to as GLF)
4. Techfil Talc provided by Luzenac (referred to as TLC)
5. *American Wood Fibers Maple 10010 Wood Flour provided by Plastics Services Network (referred to as WFL)

Z-6094 Silane coupling agent was provided by Dow Corning.

*(denotes that these blends were never tested)

Equipment necessary for the completion of small scale testing included a Kitchen Aid kitchen mixer (model K45SSWH serial WM1412587), a Blue M Stabil-Therm Gravity Oven (model OV-8A serial 16499), disposable aluminum pans, and an OHAUS analytical balance (model GT400 serial 1951).

A single cartridge feeding system was used to store the batches of LSR after they were mixed. This included 20 oz Catridges and WH Wipers provided by Nordson EFD. These cartridges were sealed with duct tape and zip ties and stored in a kitchen freezer until molded.

The injection mold used was the Hyperelastic Mold, designed as a part of a senior level research project conducted at Penn State Behrend. This tool molded a 10.2cm x 15.2cm x .20cm plaque along with a 2.54cm diameter by 1.27cm thick disk. This tool was used in an Arburg ALLROUNDER LSR injection molding machine (model 370A 500-70 serial 212841). A single cartridge feeding system provided by Nordson EFD was used in place of the pumping system of the injection unit, and interfaced with it via brass fittings. Components of this feed system included the 7012440 20 oz retainer body and the 2532 20 oz retainer cap (both used for all runs), and a quantity of the 7012416 cartridges and 7012421 cartridge pistons (for storage of individual mixtures).

To provide test samples, a non-standard tensile sample die cutter was used to make a 1.9cm gauge length bar (shown in Figure 3). These were stamped using a Phase II arbor press (serial 260-101). The tensile tester used was an Instron 4400R that employed a 1kN load cell (model 2525-806 serial 285). Compression set tests used flat plates in the same Instron. Hardness testing used a Shore Instruments Shore A Hardness Tester (serial 49838-A).

Figure 3 – Die Cutter and Samples Shows the die cutter used to make samples from plaques, as well as cut samples.

Application of Equipment and Processes

Small scale testing involved blending of the base LSR with each additive in order to determine the maximum filler content by weight that was viable for each additive, as well as viability of using the silane coupling agent in the blends. All of the small scale testing involved mixing A and B components in 1:1 ratios and combining it with the desired amount of additive and/or silane in the kitchen blender. These blends were then placed in the oven at 260°C and monitored in two minute intervals for progression of cure. Extreme differences in viscosity of the blend and curability were noted, as well as the LSR's ability to wet the additive (or blend completely into the material without excess). The maximum desired amount of any additive to be added was 40%, which was treated as the "worst case" for every blend. Therefore, every additive was attempted at 40% by weight first and then decreased in proceeding experiments if not viable.

The A and B components of the LSR were combined in 10g parts to form a 50/50 blend. This blend was then mixed by hand and cured in the gravity oven to obtain a control sample. To test the silane, 1% by weight silane (0.2g) was added to a 50/50 blend of A and B (9.9g each) and cured in the same manner. The testing was then scaled up to 50g batches using the kitchen mixer to simulate the blending method that would be used for the injection molding of the batches. These were mixed at medium speed until all the filler had been visually mixed into the LSR. These batches were used to determine the maximum filler level that was attainable for each respective modifier.

After the additive levels were chosen, each of levels was mixed into single cartridges of material weighing 800g (based on the maximum capacity of the cartridges). To achieve the percentages by weight of the additives, equal amounts of A and B component were mixed into the calculated amount of additive in the kitchen mixer. For example, for a 10% blend, the cartridge was composed of 45% A, 45% B, and 10% additive, to achieve 100% and the desired weight. The blends were mixed at medium to high speed until all additive had been visually wet, and were then transferred by hand into the cartridges (as shown in Figure 4). They were then sealed before placed in temporary storage in a freezer to prevent any curing prior to molding.

Unmodified KE-1950-40 and KEG-2000-40 were mixed 50/50 and stored in the same manner. Two unmodified resins were used due to insufficient amounts of KE-1950-40 (KEG has very similar properties). The additives mixed into the KE resin were the talc, glass, and carbon black. The additives mixed into the KEG were the silica and wood flour. Specific gravity tests were conducted in an attempt to confirm the filler content of the blends.


Figure 4 – Cartridges After Loading Shows Carbon Black charges loaded into feed system cartridges. Cartridges were placed in the retainer body and fed through the injection molding machine.

These cartridges were molded using the Nordson EFD feeding system with shop air being applied to pump into the barrel. The pressure used was varied by how far the valve was opened (higher viscosity blends required more air pressure to feed). All of the blends that were molded were run until the material in the cartridge was depleted, which usually resulted in approximately 15-20 parts. The process used was set up to make full parts with minimal flash and varied slightly with each blend. An optimized process was not set up because the effects of the molding process were not the focus of this research. The blends that were ultimately run were talc, glass, and silica. Between each additive run, the barrel was cleaned by running unmodified resin through it.

Shore A hardness testing was conducted near the gate of the plaque parts, followed by the tensile testing of seven samples using the non-standard tensile bar at a rate of 5.08cm/min. The maximum load and extension were collected from this test. Finally, compression set testing of two samples at 25% compression for 22 hours was conducted (some samples had greater than 25% compression due to differences in height between the samples). These sometimes extreme height differences were due to the blow out of disks that were not completely cured in the mold, as illustrated in Figure 5. The real compression set percentages are shown in Table 1, using the abbreviations stated previously:

Sample	Comp. %	Sample	Comp. %
KE1	25.45%	2 SIL 1	25.45%
KE2	24.40%	2 SIL 2	26.18%
KEG1	26.47%	4 SIL 1	38.83%
KEG2	26.61%	4 SIL 2	31.19%
2.5 TLC 1	26.33%	6 SIL 1	24.70%
2.5 TLC 2	28.16%	6 SIL 2	23.47%
5 TLC 1	26.90%	5 GLF 1	41.59%
5 TLC 2	26.90%	5 GLF 2	23.78%
10 TLC 1	28.44%	10 GLF 1	40.48%
10 TLC 2	26.47%	10 GLF 2	41.59%
20 TLC 1	24.70%	20 GLF 1	41.22%
20 TLC 2	24.40%	20 GLF 2	33.86%
30 TLC 1	23.63%	30 GLF 1	42.75%
30 TLC 2	23.94%	30 GLF 2	42.57%
		40 GLF 1	23.47%
		40 GLF 2	24.40%

Table 1 - Real Compression % for Testing Shows the variation in compression percentage while testing, due to differences in height between samples.
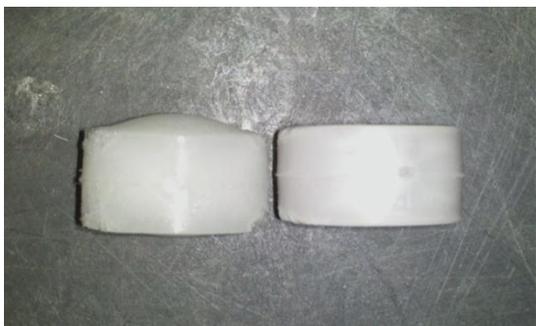
Figure 5 – Height Blow Outs of Compression Disks The left shows a blown out disk due to removal from mold before complete curing, while the right shows a disk of proper height.

Presentation of Data and Results

Small Scale Testing

The first test conducted was determining the viability of using a silane coupling agent to improve the filler loading in the LSR system. All of the samples tested using silane were unable to cure under the test conditions. These blends were also left to cure at room temperature over the course of several weeks and were still unable to completely solidify.

The next test conducted was determining the maximum percentages by weight of fillers that could be used in the LSR system. After the maximum had been determined, four lower loading levels were chosen. The results of this testing are shown as follows in Table 2. Levels one through four were

chosen to provide the most consistent range of difference between the blending levels and different additives, and were based off of the determined maximum.

Filler	Lvl. 1	Lvl. 2	Lvl. 3	Lvl. 4	Lvl. 5
Talc	2.5%	5%	10%	20%	30%
Wood Flour	2.5%	5%	10%	20%	30%
Carbon Black	2.5%	5%	10%	20%	30%
Silica	2%	4%	6%	8%	10%
Glass Fiber	5%	10%	20%	30%	40%

Table 2 - Blend Levels from Small Scale Testing Shows the determined blend levels to be used based off of the maximum allowable percentage by weight.

The glass fiber filler was the only system that was able to be added in 40% batches and not have problems with wetting of all the filler. Talc and carbon black only presented minor problems with wetting, as did the wood flour. The wood flour, however, began to burn during curing due to high temperatures used. Silica was only wettable up to 10% by weight.

Molding and Testing Processes

The cartridge feeding system proved to be an effective method of testing many different formulations of LSR. The only limitation that was encountered was that the shop air used to feed the material into the barrel was insufficient to push higher viscosity blends and therefore rendered them unmoldable. The molding process itself was not limited with the blends that were molded. Fill pressures for unmodified resins were around 50 MPa and raised only 3.5 to 4 MPa for successive levels of filler loading.

All levels of the talc blends molded with no issues, and the final parts appeared to have good dispersion after visual inspection. The glass levels molded with no issues until the 40% blend, which could not be fed using the cartridge system. The dispersion of the glass within the LSR was also very poor, with large aggregates of glass following the predominant flow pattern into the part, and more finely dispersed fibers in the perimeter. The silica levels molded well and had good dispersion up until 8%, where feeding issues again arose. For this same reason, the 10% silica blend was not molded either.

Again, due to limitations on the amount of material available, two different base resins were used. Testing showed that the materials demonstrated similar properties in most categories; but even so, comparative studies using additives were only

conducted against the base resin that they were blended into, and not against other blend types.
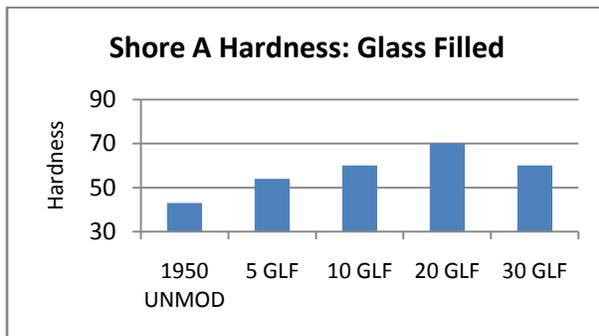
Hardness

Shore A Hardness: Talc Filled
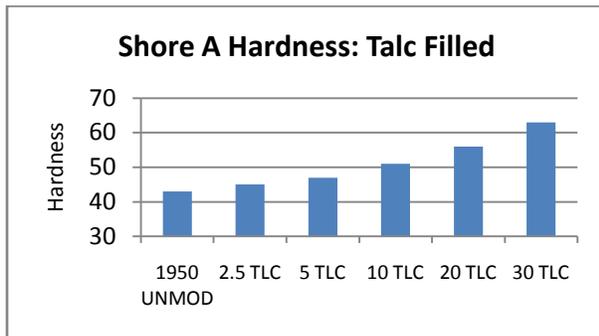
Figure 6 - Hardness of Talc Filled Shows the hardness values for every level of talc percentages.

Shore A Hardness: Silica Filled
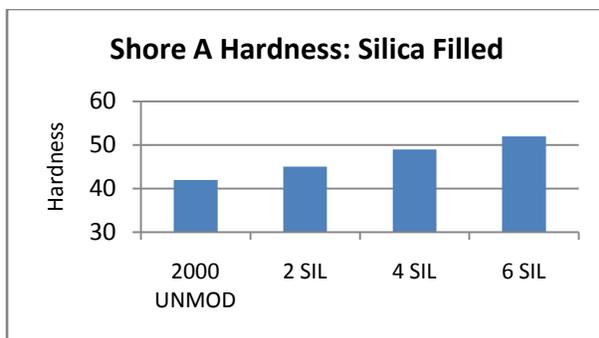
Figure 7 - Hardness of Silica Filled Shows the hardness values for every level of silica percentages.

Shore A Hardness: Glass Filled

Figure 8 - Hardness of Glass Filled Shows the hardness values for every level of glass percentages.

The hardness data for all tested filler systems is shown in Figures 6, 7, and 8. All show a steady increasing trend over the unmodified resins, except for the highest percentage of glass.

Tensile Properties

Tensile Strength: Talc Filled
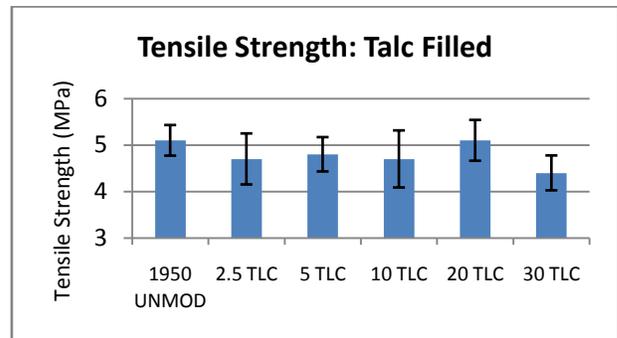
Figure 9 - Tensile Strength of Talc Filled Shows the tensile strength values for every level of talc percentages.

Figure 9 shows that the tensile strength data for the talc filled LSR is inconclusive due to the size and overlap of the standard deviation error bars. The experiment either showed that there were no detectable differences in data, or that the testing procedures were not conclusive enough to tell.

Tensile Strength: Silica Filled
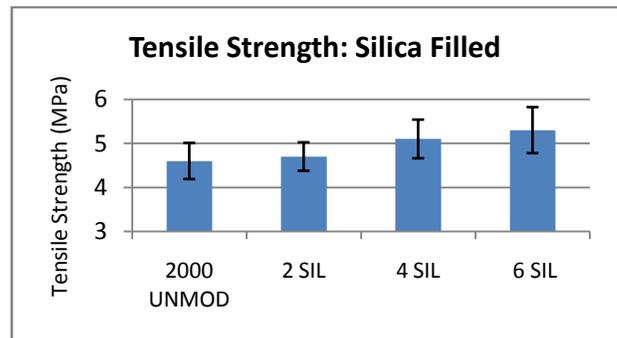
Figure 10 - Tensile Strength of Silica Filled Shows the tensile strength values for every level of silica percentages.

The tensile strength of silica filled LSR, as shown in Figure 10, shows a slight increasing trend as the filler loading increases. However, due to the size and overlap of the error bars, these conclusions should be critiqued carefully.

Tensile Strength: Glass Filled
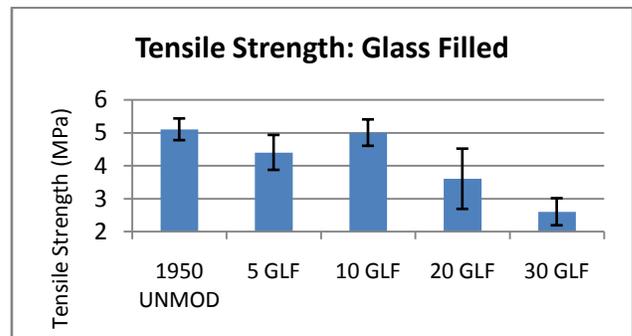
Figure 11 - Tensile Strength of Glass Filled Shows the tensile strength values for every level of glass percentages.

The tensile strength values of glass filled LSR showed large variations and some error, as shown in Figure 11. The 30% blend showed the smallest strength values, even considering error and overlap, while lower levels showed similar strengths to that of the unmodified.
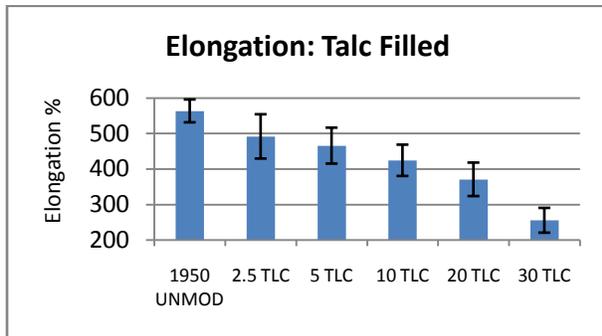
Elongation: Talc Filled

Figure 12 - % Elongation of Talc Filled Shows the percent elongation values for every level of talc percentages.

Figure 12 shows a slight overlap in error for these tests, but overall, the data for elongation of talc filled LSR showed a decreasing trend as the filler loading increased.
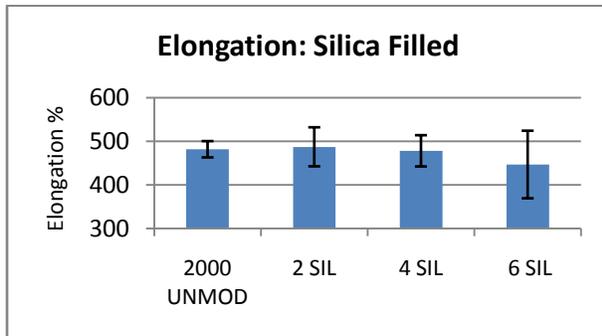
Elongation: Silica Filled

Figure 13 - % Elongation of Silica Filled Shows the percent elongation values for every level of silica percentages.

The percent elongation of silica filled LSR in Figure 13 shows that it is not affected much by the filler, even without considering the size and overlap of the error bars.
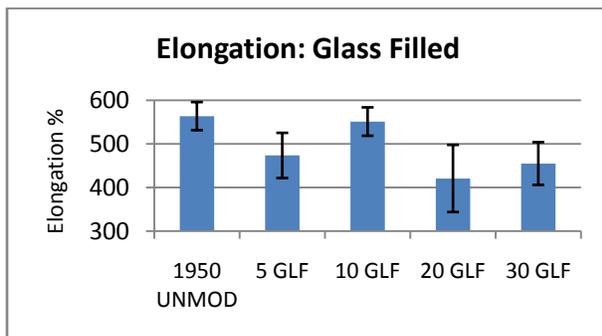
Elongation: Glass Filled

Figure 14 - % Elongation of Glass Filled Shows the percent elongation values for every level of glass percentages.

The percent elongation values shown in Figure 14 show differences in the 5%, 20%, and 30% as compared to the unmodified, while the 10% shows very similar values. However, most of the filled levels could be considered similar to each other due to the large error and overlap.

Compression Set

Compression Set: Talc Filled
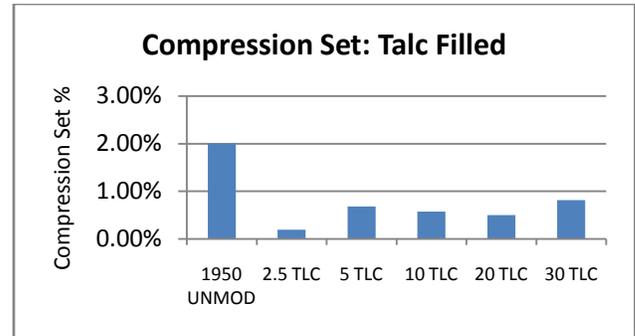
Figure 15 - Compression Set % of Talc Filled Shows the compression set % values for every level of talc percentages.

Figure 15 shows that all samples of talc filled LSR showed a great decrease in compression set. The 2.5% blend showed the least compression set, while 30% showed the most (but still much less than the unmodified).

Compression Set: Silica Filled
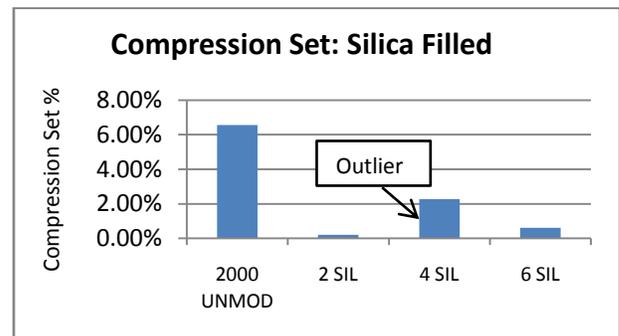
Figure 16 - Compression Set % of Silica Filled Shows the compression set % values for every level of silica percentages.

Figure 16 shows that there is a great decrease in the amount of compression set experienced by the filled material. The 4% blend should be considered an anomaly due to the large compression percentage that those samples experienced. Still, all level showed that the silica decreased the amount of compression set, and it can be predicted that 4% samples under similar conditions to the other two levels would produce similar results.

Compression Set: Glass Filled
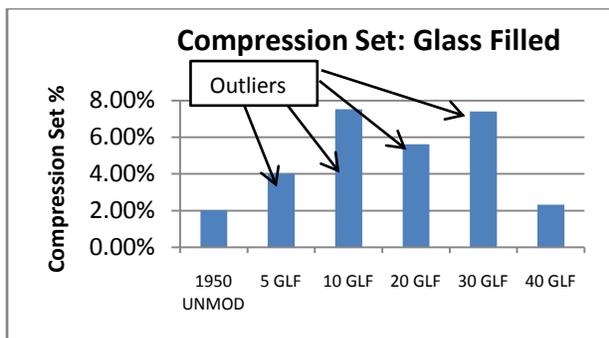
Figure 17 - Compression Set % of Glass Filled Shows the compression set % values for every level of glass percentages.

All but the 40% compression set data in Figure 16 should be considered outlying again due to the large compression that was used during testing. The 40% actually showed more compression percentage than the unmodified, and no comparative conclusions can be drawn for the others.

Interpretation of Data

Small Scale Testing

The use of a silane coupling agent was not viable for improvement of filler loading because of its inhibiting of the normal curing process of the LSR. The tackiness and lack of cohesiveness in the blends that contained it signified either an inhibiting effect on the kinetics of the cure or a decomposition of polymer structure that prevented cure. Since the blends with silane were unable to cure at room temperature, the reaction kinetics were not the issue, and it was determined that the silane actually decomposes the LSR's structure. This was further supported by the blends with silane beginning to brown in the curing oven when the neat LSR had not. Because of this, the silane was not used for any future blends.

The glass fibers were viable up to 40% because there were no extreme effects on curing and wetting of the fibers was not an issue. This could have been due the glass being in fiber form rather than powder. Every other additive was powder and showed wetting concerns, with the worst being the fumed silica. The density of this silica was so low that the volume of it needed to reach 40% by weight was much greater than the volume of the LSR. It was therefore impossible to blend in more than 10% of it into any amount of LSR. These density concerns carried over to the other additives, only in much lower degrees.

Molding and Testing Processes

Overall, the molding processes used were very robust and capable of molding many different additives and levels. Since the injection molding machine was not pressure limited, in theory much higher levels of additives could be run with these systems without issue, if they could be fed through the barrel. Small scale testing could not have predicted that the barrel would not take the higher viscosity blends due to the lack of pressure from the shop air line, but to run these higher loadings a new system would have to be implemented.

Dispersion of the additives, however, proved to be an issue. Without proper dispersion, the reinforcing effects of these fillers will not show. While the powder additives (talc and silica) seemed to be visually dispersed, no means of quantifying this was employed. The glass had terrible dispersion through the part, mostly due to jetting of material within the cavity. Opening the small gate could have alleviated some of these dispersion problems. Also, more vigorous mixing or shear during processing or compounding could have assisted this dispersion. The issue with using these additives with the LSR system is that they cannot be compounded in using heat and high shear, and are therefore limited in how much dispersion can be obtained before molding. This is the major limitation in this research.

Hardness

Overall, the trend experienced by every blend was an increasing hardness as filler loading percentages increased. This follows the logic that the hardness and stiffness will increase as more inert material of a higher hardness and stiffness is added to the system. LSR grades of the same family usually differ by their Shore A hardness, and this testing shows that it is possible to modify a system using an inexpensive additive instead of purchasing a different grade of material. The glass filled LSR showed the greatest increase in hardness (even while the highest loading level tested showed the lowest comparative hardness, due to some incomplete curing in the samples), but the silica showed respectable increases at a much lower percentages. This shows that even at low loading levels, the properties of these LSRs can be modified in significant ways.

Tensile Properties

Tensile properties are where the traditional "reinforcement" would take place when considering thermoplastics. However, this apparently is not the case when considering LSRs, as most of the data collected showed no detectable patterns in affects on tensile strength or elongation. Most of this could be due to the large amount of variation and error in the data.

The talc filled samples showed no significant trend or improvement over the unmodified resin, but

the elongation showed a steady downward trend. Since the elongation was obviously affected by the increasing filler loading but no strength improvements were gathered, it can be concluded that the talc served no purpose as a reinforcement filler in the traditional thermoplastic sense. Overall, the talc served as more of a modifier and not a reinforcement filler in the traditional sense. From a material standpoint, this could be due to poor dispersion of the filler within the LSR or lack of interaction between the filler and LSR.

From a statistical standpoint, the tensile strength data showed a large amount of variation and overlap in error. It is possible that not much confidence can be placed in the conclusions made from it. Reasons for this variation could inconsistencies in the testing methods (such as area of the plaque where the part was taken, stress concentrations due to dull cutters, etc.) or too small of a sample size. However, given that the elongation data proved to be reliable, it can be said that most of the effects observed were due to the filler itself instead of statistical error.

The silica filled LSR showed the only trend of reinforcement, with an increasing tensile strength following an increasing filler load. Also, there were no differences in ultimate elongation that could be considered significant for these samples. Again, the inconclusiveness of the elongation data could be statistical, but given that some of the data is reliable, it is more likely that the material itself is the cause. From these tests, the silica showed to have a comparatively higher tensile strength with no detectable difference in percent elongation, and can be concluded to be an effective reinforcing filler. While the dispersion of the silica is still in question, it most likely had better interaction with the LSR than other systems due to the volume of it used and its powdered form.

None of the glass filled data showed any reinforcement or reliability, and was likely a direct result of the very uneven dispersion of the fibers within the final parts. Care was taken to select samples that visually had a good dispersion (no clumped aggregates), but even these samples showed no reinforcement properties. The highest glass percentages actually showed a much lower tensile strength than the unmodified and similar elongations. Another major contributing factor to this is the interface between the dispersed glass and the LSR. There was most likely very little interaction and therefore the glass acted more like a placeholder instead of a reinforcer.

Compression Set

Overall, the comparative conclusion that can be drawn from the compression set data is that talc and silica filled systems showed much lower compression set percentages (better elastic recovery, no permanent deformation) than the unmodified resins, while glass showed no conclusive results. This is intuitive when considering that the addition of a harder filler in place of the elastic LSR would decrease its tendency to set. The fillers themselves do not compress in the way that the resin does, and for this reason the filled samples retained their original dimensions better than the unmodified resins.

As previously stated, the talc and silica showed the best improvements in elastic recovery over the unmodified resins. Compared to the tensile properties, the fillers had a much more concrete effect on these properties. This could mostly be due to the dispersion of the additive not being as important in the compression properties as in the tensile properties.

The glass compression set data was unreliable due to the blown out samples being tested for the 5-30% samples. The 40% samples were properly cured and shaped, and can be considered the only reliable glass filled sample. This actually showed a slight increase in compression set. This could have been due to the poor dispersion of the glass or the lack of interaction between the filler and LSR, reducing the reinforcing effectiveness.

Extending vs. Reinforcing

While the initial scope of this research was looking at reinforcing effects of additives and fillers, the possible extending and modifying effects should not be ignored. The data collected does not show much reinforcing taking place, in the traditional thermoplastic sense. However, since LSR systems are very unique, their behavior should not be compared to traditional rubbers and thermoplastics. Using these fillers as extenders that are more for cost saving instead of reinforcing is viable given that not much change in properties has been observed, and the changes in properties that have been observed are mostly favorable.

Sources of Error

Potential sources of error have been previously highlighted, and include most prominently the blending method employed to make batches of material with good dispersion of filler. This error was minimized by carefully and vigorously mixing small amounts of additive into the systems at a time

without overloading and clumping the additives. Another source of error in the testing data was the die cutting system used to make tensile samples. The cutters were sharpened between each set of plaques being cut to reduce stress concentrations, and care was taken to take the samples out of similar areas on the plaques for every part. Testing only one part for the compression set data possibly gave an inaccurate picture of the real compression behavior of the materials.

Conclusion

Expanding the application range through mechanical reinforcement of LSR resins could possibly open up avenues that were previously unexplored by this family of materials. At the very least, an investigation into the modifying and blendability of these additives is beneficial because it gives a picture of future viability of blending different filler systems into LSRs and provides a baseline look at their effects and problems that will be encountered.

Achieving a good dispersion with additives using these systems is a major hurdle since they cannot be heat compounded into a homogenous mixture. Within mechanical tests that require good dispersion (tensile properties), this proved to be an issue since the uneven dispersion possibly created local weak areas within parts that were tested.

Talc filled LSR showed a dramatic decrease in ultimate elongations but no detectable improvement in strength, due to the presumed poor dispersion of filler. In compression set and hardness, however, improvements were present due to just the presence of the filler itself. Those properties were seemingly unaffected by dispersion or interface of filler and LSR.

Silica filled LSR showed a slight increasing trend in tensile strength while showing no reliable differences in elongation. Silica can therefore be concluded to have a slight reinforcing effect, and at the very least does not affect the properties (except for hardness and elastic recovery) much. The hardness increased even at low filler levels and the elastic recovery was concluded to be greatly improved over the unmodified resin, due to the mere presence of the filler in any dispersion.

Glass filled LSR showed almost no reliable results for any testing, mostly due to the lack of proper dispersion of the filler. The low tensile strengths at high levels of filler could be due to the lack of true interaction between the glass and the LSR, and the compression set data cannot have any conclusion drawn from it due to the error in the samples.

Future Work

The first area for future development is to find a blending system that achieves a guaranteed good dispersion within the LSR system, since that is the single most important factor in mechanical reinforcement. This experiment should also be expanded upon by using a more reliable testing system based on the conclusions that were drawn, such as using samples of more similar height in the compression set testing. More additives should also be explored after establishing a better mixing system, and more samples of each of the present blends should be tested using these improved methods as well. Afterwards, established non-linear test methods including Hyperelastic Analysis could be employed to get a broader picture of the behavior of these resins.

GENERATION 2

Introduction

After completion of the first series of testing and data collection, there was a significant realization of improvements that could be introduced to the project in order to better develop the project results and validity. The research and work that had already been completed was greatly expanded upon, and as a result, the new and improved data set will be discussed as a continuation of the future work and previous conclusions that were prepared. The main objectives of the second iteration of this project were to enhance the dispersion of modifiers in the molded parts in order to provide more data consistency, develop an optimized process for the molded parts in order to ensure proper curing of the LSR resins, and to summarize all of our new findings into a more accurate and valid data set.

Description of Equipment

Enhancements

Unlike Generation 1 of the liquid silicone molding, Generation 2 employed a uniform base resin for the entirety of the part collection. Wacker Elastosil LR 3003-40 was used as the control material for the mechanical property testing through the completion of Generation 2 molding, whereas the base resins utilized in Generation 1 included both Shinetsu KE-1950-40 and KEG-2000-40. A Kenrich Petrochemicals titanate coupling agent was also experimented with as another attempt to enhance curing and material properties. Being able to provide a comparative analysis between all modifier types and their separate effects on the standard LSR resin was an important aspect of this project, and unlike Generation 1, the improvements that were made (using 1 base material) allowed for a more comprehensive data set.

Generation 2 materials were mixed using a paint mixing attachment for a power drill. This was arguably the most significant improvement to the project as it allowed every subsequent step in the research to utilize more uniform and accurate test samples.

Figure 18 – Kitchen Mixer from Generation 1 Mixing Methods Shows the exact mixer that was used in all of the mixing of the Generation 1 material blends.

Figure 19 – Paint Mixing Attachment for Power Drill Used in Generation 2 Mixing Methods Shows the paint mixing attachment that was used to improve the mixing stage of the research project.

Generation 2 materials were cut using an ASTM D412 standard cutter that made a 3.3cm gauge length bar. This was a change and improvement to the previous collection of data because this tensile bar shape was relevant to an ASTM standard test, whereas the last samples were cut using a non-standard tensile shape. The tensile tester used for both generations of testing was an Instron 4400R that employed a 1kN load cell (model 2525-806 serial 285). Compression set tests for both generations used flat plates in the same Instron. Hardness testing for both generations used a Shore Instruments Shore A Hardness Tester (serial 49838-A).

Figure 20 – Non-Standard Tensile Bar Cutter Was used in Generation 1 material testing.

Figure 21 – ASTM D412 Standard Tensile Bar Cutter Was used in Generation 2 material testing in order to better produce tangible data.

Additions

DSC testing was added as a part of the data set in order to capture exactly what was happening to the small scale samples during the curing phase, as to hopefully provide an explanation for the coupling agents' failures.

A scale was used with a large glass beaker and distilled water to conduct specific gravity testing according to ASTM D792. The goal of the specific gravity testing was to provide some sort of validity to the amount of modifier in the test samples that were mixed. Using the base LSR material's specific gravity as well as the subsequent blend's specific gravity, the change in specific gravity values could be recorded and charted to display the increasing effect of the specific gravity as additional modifier was added to the system.

A Celestron LDM, model 44340 microscope was used to look at filler dispersion in all molded samples. The microscope was utilized as an attempt to compare Generation 1 molded plaques to Generation 2 molded plaques. Based on the enhancements made for Generation 2 parts, the objective of looking at parts in a microscope was to microscopically prove the progress that was made, rather than to visually inspect the parts.

Application of Equipment and Processes

After analysis of the Gen. 1 materials was complete, another round of experiments were commenced that attempted to resolve some of the glaring issues that reduced the reliability of the data, as previously mentioned. The overall procedure remained similar for these Gen. 2 materials, with improvements being made in the specifics of how mixing, molding, and testing were conducted. Also, a different base resin (LR 3003-40) was used in the Gen. 2 materials in order to keep the testing within the generation consistent.

Small scale testing was conducted in the same manner as before while replacing the kitchen mixer with the paint mixer. Also, a titanate coupling agent was tested in addition to the silane. Differential scanning calorimetry tests were conducted on unmodified resin and coupling agents employing a temperature ramp from 30-220°C at a rate of 10°C/min. Blend percentages were based more off of what had worked previously rather than the new round of small scale testing.

When mixing the material for molding, the proper amount of LSR and additive were combined in a bucket and mixed by hand with the paint mixer. Batches were mixed at the molding machine and immediately placed into the feeding system instead of being stored for later use. The goal of changing the mixing method was to improve the dispersion of the fillers by adding more shear when mixing to the system before molding.

During molding, a more consistent process was set up for each additive. Injection velocities were kept consistent except in cases that jetting or non-fills occurred. The cure time was also raised to a point where the compression disks were completely cured and showed no blow-outs.

Hardness and compression set testing followed the same procedures as in Gen. 1. Tensile testing followed more closely to the ASTM standard for rubber tensile testing. By using the standard bar cutter, die punching all samples from the plaques in the flow direction rather than randomly, and increasing the testing speed to 50.8 cm/min the results for tensile testing would undoubtedly produced immense error reduction. This was all done in an effort to obtain more consistent and reliable data when compared to Gen. 1.

To complete the data set, samples were cut out of molded plaques with a utility knife from the end of fill and the gate regions of all unmodified resins and blends, and were then tested for specific gravity in an effort to characterize the filler content of the blends. These samples were also put under the microscope to look at filler dispersion.

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# Presentation of Data and Results

Small Scale Testing

The first test conducted for both Gen. 1 and Gen. 2 materials was determining the viability of using coupling agents to improve the filler loading in the LSR system. Neither silane nor titanate showed an effective use in these blends as all of the samples that were tested using them were unable to cure under the test conditions. These blends were also left to cure at room temperature over the course of several weeks and were still unable to completely solidify. DSC graphs showing the cure of A+B resin, resin with silane, and resin with titanate are depicted in Figures 22-24. These DSC results are exclusive results only yielded from the second generation of the project. Notice should be taken to the spikes that occur on the down slope of graphed lines. The A+B system shows an apparent cure at roughly 120°C, whereas the titanate system shows only a hint of a cure. The silane system demonstrated absolutely no signs of any curing.
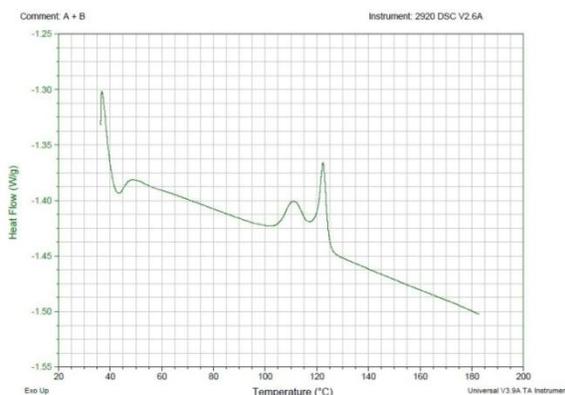
Figure 22 – LR 3003-40 A+B Silicone DSC Curve This DSC graph depicts the curing that takes place during the heating ramp-up of just the base silicone resins.
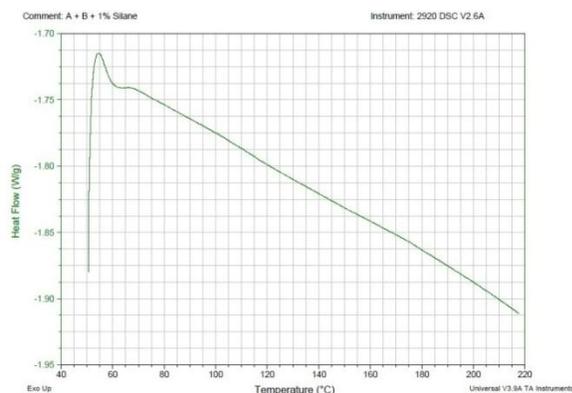
Figure 23 – LR 3003-40 A+B+Silane DSC Curve This DSC graph depicts the inability of the silane coupling agent and silicone materials to cure during the temperature ramp-up.
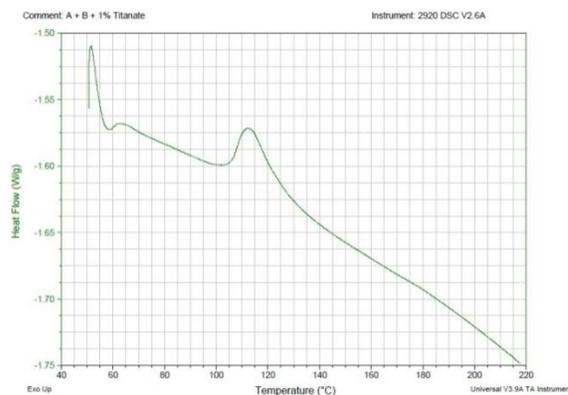
Figure 24– LR 3003-40 A+B+Titanate DSC Curve This DSC graph depicts the attempt of the titanate coupling agent and silicone materials to cure during the temperature ramp-up.

Generation 2 blend levels were chosen based on the results of the Generation 1 testing. Glass fiber was eliminated from the blending and the levels of talc were changed to reflect the numbers in the following table. The silica levels used were the levels that proved to be successful in the previous generation of testing.

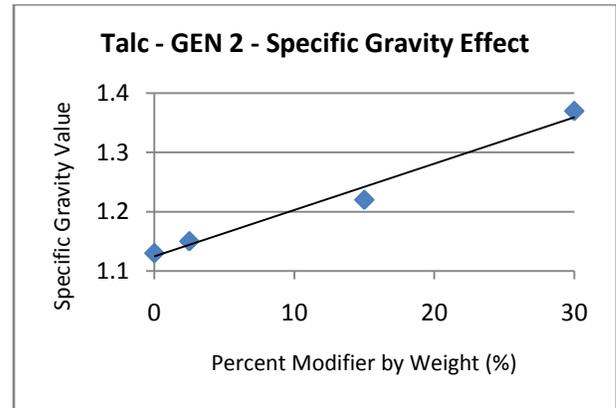| Filler	Lvl. 1	Lvl. 2	Lvl. 3
Talc	2.5%	15%	30%
Silica	2%	4%	6%

Table 3 – Blend Levels from Gen. 2 Mixing Methods The table represents the new levels for modifier mixing as per the Generation 2 material mixing methods.
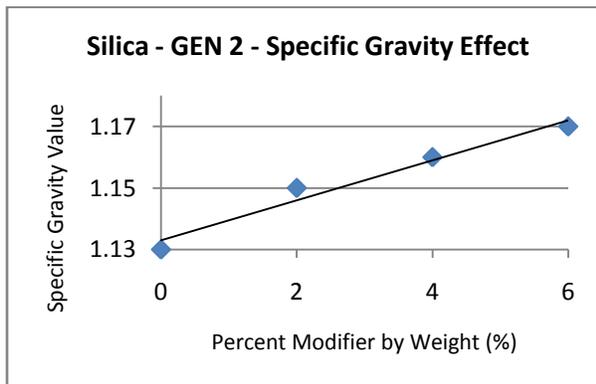
Filler Content

The filler content data is a newly presented section that was directly focused on improving the validity of the entire data set. The goal of the filler content experiments was to prove that a consistent increase in the actual loading percentage of modifier existed in each respective blend. The results were achieved by collecting specific gravity figures for unmodified resins as well as each subsequent blend of filler. The line graphs in Figures 25-28 explicitly depict the phenomenon of the blended resins specific gravity value increasing in a linear fashion toward the specific gravity of the respective filler that the blend was loaded with. If there was a significant loss of modifier throughout blending, feeding, molding, or part extraction, then the charts would have suggested that the specific gravity of the blends was not increasing as per the loading percentages. The actual specific gravity values were not of particular interest; instead, the upward trending of the SG toward the SG of the modifier was the objective of this experiment.
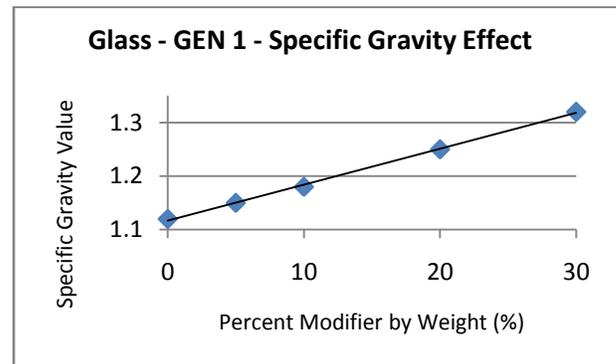
Silica - GEN 1 - Specific Gravity Effect
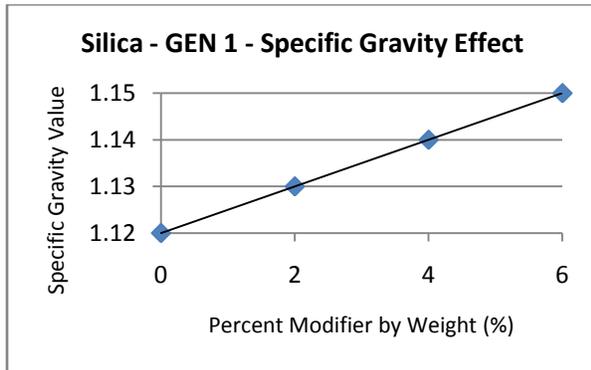
Figure 25 – **Specific Gravity Effect for Silica Filled** Shows the specific gravity values for every level of silica percentages from Gen 1 testing.

Silica - GEN 2 - Specific Gravity Effect

Figure 26 – **Specific Gravity Effect for Silica Filled** Shows the specific gravity values for every level of silica percentages from Gen 2 testing.
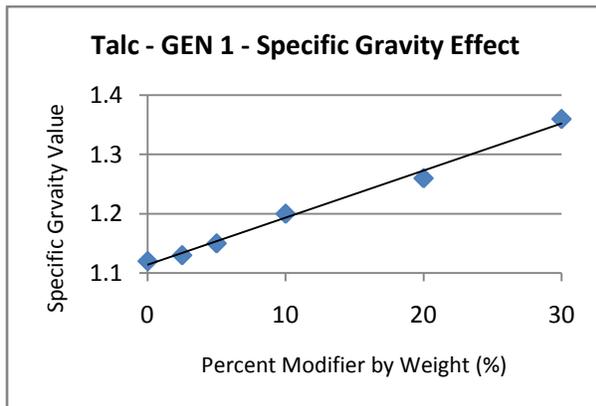
Talc - GEN 1 - Specific Gravity Effect

Figure 27 – **Specific Gravity Effect for Talc Filled** Shows the specific gravity values for every level of talc percentages from Gen 1 testing.

Talc - GEN 2 - Specific Gravity Effect

Figure 28 – **Specific Gravity Effect for Talc Filled** Shows the specific gravity values for every level of talc percentages from Gen 2 testing.

Glass - GEN 1 - Specific Gravity Effect

Figure 29 – **Specific Gravity Effect for Glass Fiber Filled** Shows the specific gravity values for every level of glass fiber percentages from Gen 1 testing.
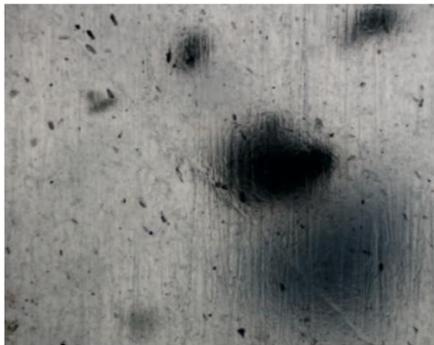
Filler Dispersion

The improvement of filler dispersion was a clear and well defined goal of the second iteration of material mixing. By applying the new methodologies of mixing, drastic measures were taken in the development of better dispersed fillers throughout any given part's cross section as well as lateral flow dispersion. The following images were taken from the microscope, in order to better characterize the differences seen for filler dispersion from Gen. 1 to Gen. 2. The large black specs in Figure 30 represent an agglomerate of filler that was not broken up during mixing, whereas much better blending can be seen in Figure 31. These images were taken from exactly the same type of modifier as well as modifier percentage, the only difference being which iteration the part represents. A 2.5% talc filled blend was chosen since it was a blend that was collected from both generations of the project. This blend was also of particular interest because of it being translucent to the point where it was easy to recognize the dispersion differences. The microscope was utilizing a 4X optical zoom at the time the pictures were taken. Since the same mixing methods that were used for Figure 31 were employed throughout the entire Gen.

2 mixing, it was determined that all the successive blends taken form Gen. 2 would also represent this improved dispersion, and that no further microscope testing was needed. This should be the case for both talc and silica blends.


Figure 30 – Filler Dispersion from Gen. 1 Mixing Methods Shows the filler dispersion as seen in Generation 1 mixing.


Figure 31 – Filler Dispersion from Gen. 2 Mixing Methods Shows the filler dispersion as seen in Generation 2 mixing.
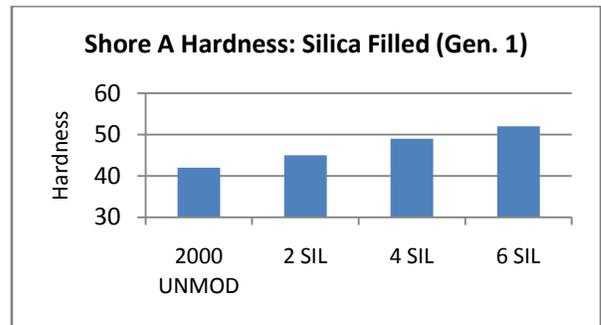
Hardness

The hardness data for all tested filler systems is shown in Figures 32-35. All show a steady increasing trend over the unmodified resins. Comparisons between Generations 1 and 2 show the same trends for both the talc and silica.
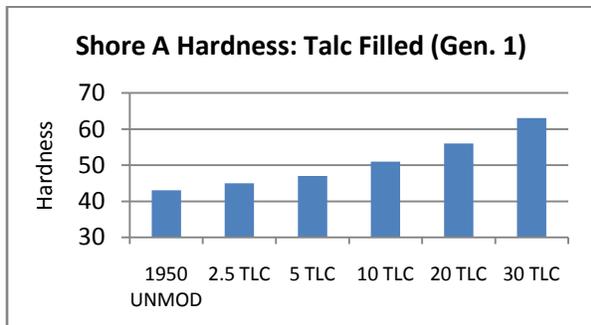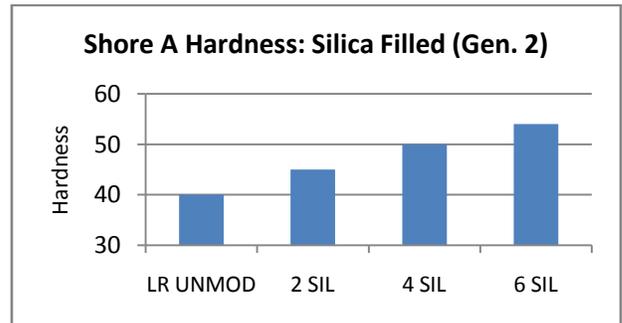

Figure 32 - Hardness of Talc Filled Shows the hardness values for every level of talc percentages from Gen. 1 testing.
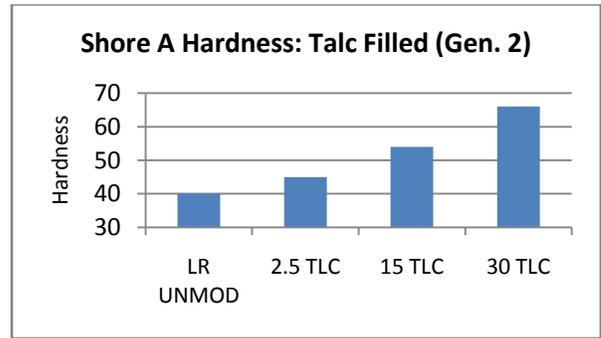
Figure 33 - Hardness of Talc Filled Shows the hardness values for every level of talc percentages from Gen. 2 testing.
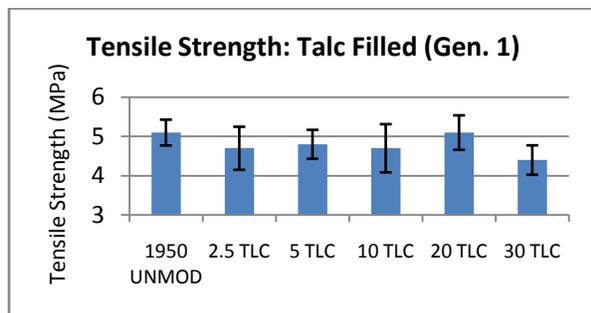

Figure 34 - Hardness of Silica Filled Shows the hardness values for every level of silica percentages from Gen. 1 testing.
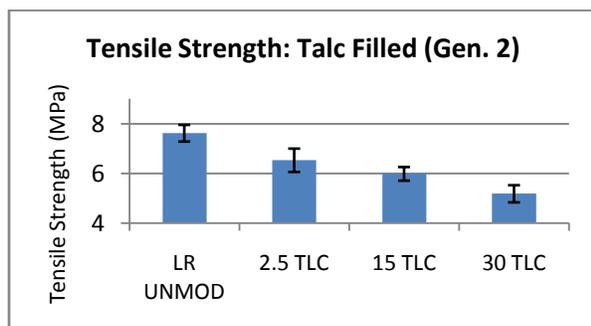

Figure 35 - Hardness of Silica Filled Shows the hardness values for every level of silica percentages from Gen. 2 testing.
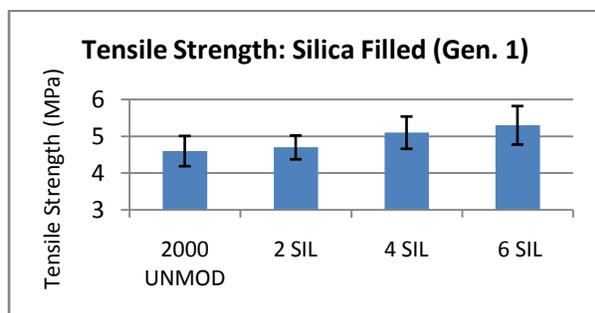
Tensile Properties

Figure 36 - Tensile Strength of Talc Filled Shows the tensile strength values for every level of talc percentages from Gen. 1 testing.
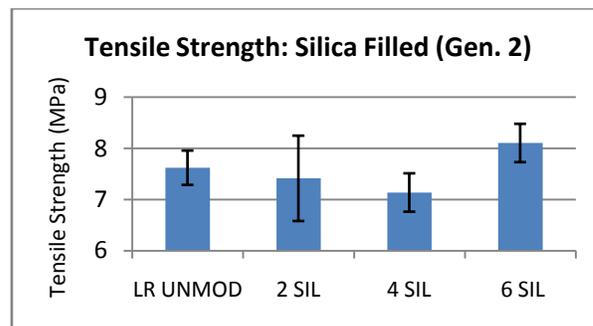

Figure 37 - Tensile Strength of Talc Filled Shows the tensile strength values for every level of talc percentages from Gen. 2 testing.

Figures 36 and 37 show that the tensile strength data for the talc filled LSR blends from Gen. 1 is inconclusive due to the size and overlap of the standard deviation error bars. The Gen. 1 experiment either showed that there were no detectable differences in data, or that the testing procedures were not conclusive enough to tell. However, Gen. 2 data yielded some significant improvements to the data trends. It is very apparent from the new tensile strength data that a general decrease in properties existed as modifier percentage was increased.


Figure 38 - Tensile Strength of Silica Filled Shows the tensile strength values for every level of silica percentages from Gen. 1 testing.


Figure 39 - Tensile Strength of Silica Filled Shows the tensile strength values for every level of silica percentages from Gen. 2 testing.

The Gen. 1 tensile strengths of silica filled LSR samples show a slight increasing trend as the filler loading increases. However, due to the size and overlap of the error bars, the graphical representations seen in both Gen. 1 and Gen. 2 provide no statistical significance that can be attributed to the silica loading of LSR molded parts.
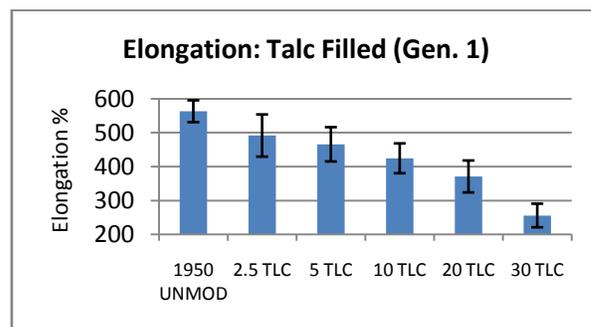
Figure 40 - % Elongation of Talc Filled Shows the percent elongation values for every level of talc percentages from Gen. 1 testing.
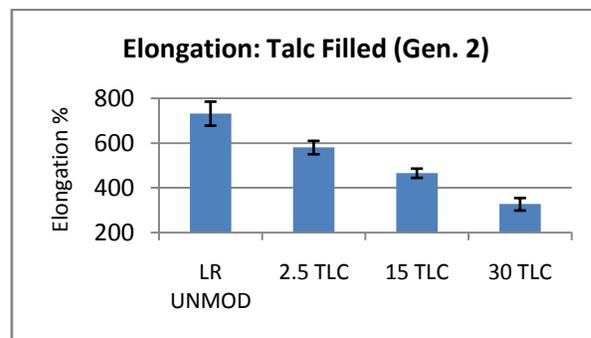
Figure 41 - % Elongation of Talc Filled Shows the percent elongation values for every level of talc percentages from Gen. 2 testing.

The elongation figures of talc filled LSR show a slight overlap in error for these tests completed in Gen.1, but overall, the data for elongation of talc filled LSR showed a decreasing trend as the filler loading increased. This was especially apparent in the second generation of part and data collection since the error bars were drastically minimized in height.
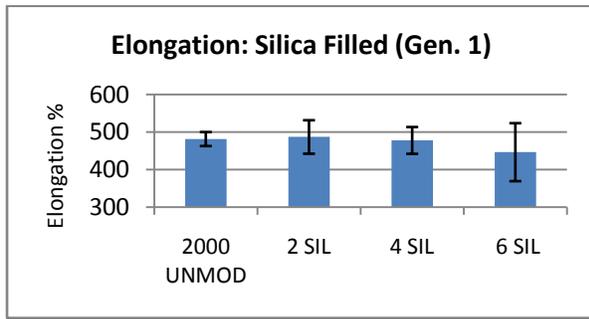
17

Figure 42 - % Elongation of Silica Filled Shows the percent elongation values for every level of silica percentages from Gen. 1 testing.
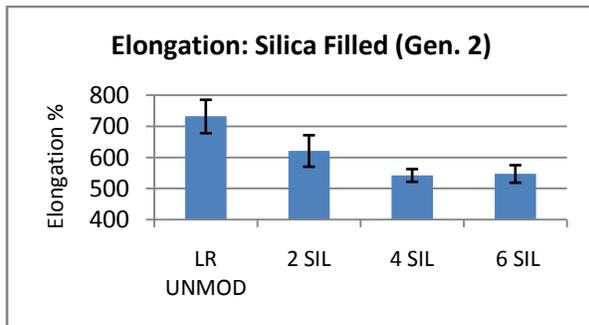
Figure 43 - % Elongation of Silica Filled Shows the percent elongation values for every level of silica percentages from Gen. 2 testing.

The percent elongation of silica filled LSR shows that Gen. 1 results were not affected much by the filler, even without considering the size and overlap of the error bars. However, Gen. 2 data suggests that there is a decreasing trend in the elongation of parts molded with silica modifier as the percentage of silica is increased.

Compression Set

The compression set data is a representation of exactly how the molded pucks were affected by a uniform loading over a prolonged period of time. These tests are run over the course of 22 hours and are a measurement of the permanent deformation of the pucks as a result of the compressive forces acted upon them. A higher percentage means that the material is more apt to lose is elastic recovery properties, whereas a lower percentage suggests the resin has increased elastic recovery characteristics.
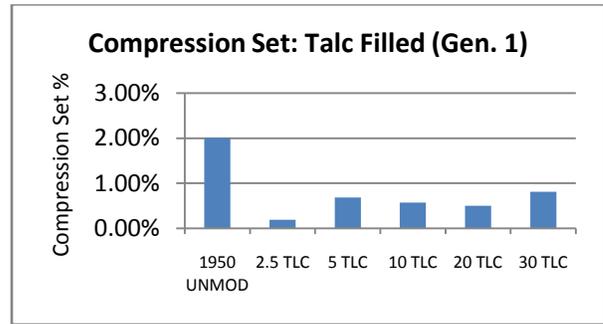
Figure 44 - Compression Set % of Talc Filled Shows the compression set % values for every level of talc percentages from Gen. 1 testing.
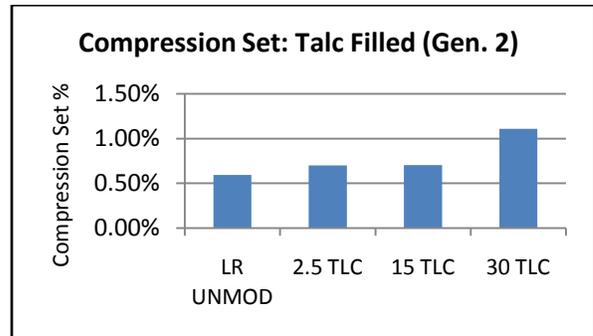
Figure 45 - Compression Set % of Talc Filled Shows the compression set % values for every level of talc percentages from Gen. 2 testing.

Figure 44 shows that all samples of talc filled LSR in Gen. 1 proved a significant decrease in compression set. This data suggests that there is an increase in the elastic recovery of the LSR resin if talc is introduced at these tested percentages. Despite the increase in elastic recovery of the test samples, no trend is apparent for an increasing modifier percentage. However, Gen. 2 compression set data seemed to suggest just the opposite. The results of the Gen. 1 compression set are likely skewed since the modified resin pucks experienced the swelling effect and were compressed much more than the unmodified versions.
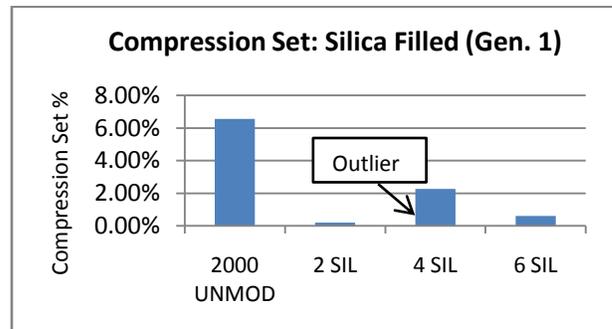
Figure 46 - Compression Set % of Silica Filled Shows the compression set % values for every level of silica percentages from Gen. 1 testing.
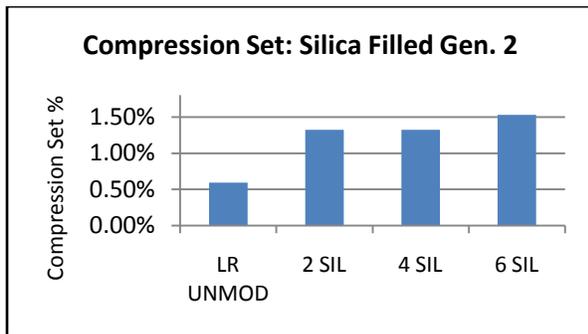
Figure 47 - Compression Set % of Silica Filled Shows the compression set % values for every level of silica percentages from Gen. 2 testing.

Figure 46 shows that all samples of silica filled LSR in Gen. 1 showed a significant decrease in compression set. This data suggests that there is an increase in the elastic recovery of the LSR resin if silica is introduced at these tested percentages. Despite the increase in elastic recovery of the test samples, no trend is apparent for an increasing modifier percentage. However, Gen. 2 compression set data seemed to suggest just the opposite. The results of the Gen. 1 compression set are likely skewed since the modified resin pucks experienced the swelling effect and were compressed much more than the unmodified versions. It should also be noted that both talc and silica compression set data experienced the exact same reversing effect from Gen. 1 data to Gen. 2 data.

Interpretation of Data

Small Scale Testing

The Gen. 2 small scale testing results reinforced the results that were collected in the first iteration. The small scale testing that was completed in Gen. 2 was primarily done in order to test the feasibility of yet another coupling agent in that of the titanate. The DSC results again just solidified the first generations results that coupling agents inhibit the curing of the LSR systems. The coupling agents were to be added in at very small percentages, and despite careful measurements, the minimal amount was likely still an overload for the LSR materials. It is very likely that during the addition of the coupling agents, the agent was inhibiting the overall curing of the material because it very quickly cured several short chains rather than curing the entire charge of LSR. This would produce the "non-cure" effect that was seen in the experiment. The coupling agents, as a result, acted to flood the system and undoubtedly inhibit the overall progress of curing, even when left to cure for several weeks.

The levels that were determined for the second iteration of mixing were taken directly from the predetermined levels from Gen. 1 as well as the results of prior molding experiments. Glass fiber mixing was completely eliminated from the mixing procedures due to its poor results throughout the entirety of Gen. 1. The silica and talc ratios again reflected the range of Gen. 1 mixing, however, lesser iterative levels were chosen so that larger differences in data from one to the next could be apparent if any existed.

Filler Content

The results of the filler content data (specific gravity) suggests that as the percentage of modifier is increased, so too is the specific gravity of the respective blend. The linear slope of the line means that the specific gravity is uniformly being increased at the rate of the increase in filler loading. The summary of this data is that there effectively no loss of modifier in the system. It makes sense that the specific gravity should increase toward the respective filler's specific gravity value, and progress away from the LSR's specific gravity value as the blend becomes made up of more filler than it is LSR. Hence, a 100% LSR system would have the specific gravity of the LSR, and 100% modifier system would have the specific gravity of the modifier, and anything in between should be proportional to the percentage of each. The only reason that the values may not match up perfectly (as they should with matching respective percentages of each) is because there are likely micro-voids in the system that are introduced as the modifier is mixed in. This could also be a result of minimal or no bonding adhesion between the base LSR polymer and the modifiers. Obviously the voids or air pockets would have no specific gravity, and would pull the values down some from where they should technically be. Thus, the linear increasing trend in the data is the primary take away from the data.

Filler Dispersion

The filler dispersion results were undoubtedly a direct result of the enhanced mixing methods. The paint mixing attachment for the power drill was able to produced increased velocities, and in turn increased the shearing effects on the LSR system. The increased shear on the LSR and modifier being added was able to better the dispersion and breaking up of the agglomerates so that a more uniform blend could be utilized. As previously mentioned, this was undoubtedly the most significant change from Gen. 1 to Gen. 2 because it allowed every subsequent step to be improved. The microscope test verified that Gen. 2 mixing methods were indeed quite an improvement from Gen. 1 mixing methods.

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Hardness

The hardness data was some of the best data that was collected in the entire project. The main reason for such consistent data is because the hardness test is a very quick and easy test to perform, and during this test, the tester is able to have a high sample size with very good repeatability. The data did not very much from Gen. 1 to Gen. 2, and is another reason why there is much confidence in the data. Not only is the hardness data very consistent, it is also likely the most important data in the entire project. LSR materials are almost always characterized based solely on the level of hardness, i.e. the base LSR's in this research were all 40 Shore A. However, with the addition of the modifiers, each respective blend demonstrates a different hardness. This could be of vital importance to an application where costs savings, material extending, or different properties are of importance. To be able to mirror the properties of perhaps, a more expensive, higher hardness material, with a lower hardness and filled material could be the best take away from the research.

Tensile Properties

The tensile properties of interest were the tensile strength as well as elongation. Based on the variability of Gen. 1 data and tensile testing, Gen. 2 tensile data is of particular interest.

The talc data from Gen. 2 suggests that as filler percentage is increased, the tensile strength as well as elongation values are decreased. When talc is added to the base LSR, it inherently stiffens the material as was seen in the increased hardness data. The decrease in elongation was definitely expected for this very reason. A stiffer material should inherently produce a smaller elongation percentage. The surprise in the data came from the tensile strength results. The obvious thought would be that if a material had a smaller elongation that it would also have a higher strength, and this was simply not the case. The decreased tensile strength properties can likely be attributed to the talc actually interfering with the LSR's polymer matrix, and as a result tends to somewhat reduce strength.

The silica data from Gen. 2 suggests that as filler percentage is increased, the elongation values are decreased. When silica is added to the base LSR, it inherently stiffens the material as was seen in the increased hardness data. The decrease in elongation was definitely expected for this very reason. A stiffer material should inherently produce a smaller elongation percentage. The tensile strength data for silica molding samples did not produce any significant results. A conclusion can be drawn that silica may actually have no significant effect on changing the tensile strength properties of the base LSR. It is likely that because silica is so similar to the chemistry of silicone, that this no change phenomenon is actually the resultant effect.

Compression Set

The talc filled LSR in Gen. 1 showed a significant decrease in compression set. This data suggests that there is an increase in the elastic recovery of the LSR resin if talc is introduced at these tested percentages. Despite the increase in elastic recovery of the test samples, no trend is apparent for an increasing modifier percentage. However, Gen. 2 compression set data seemed to suggest just the opposite. The results of the Gen. 1 compression set are likely skewed since the modified resin pucks experienced the swelling effect and were compressed much more than the unmodified versions. Therefore, the focus of the compression set data should be on the Gen. 2 data. With the addition of a modifier, the results show that the blend has a decreasing effect of elastic recovery from a non-modified material. The addition of the modifier will act to inhibit the LSR material's inherent abilities to spring back and therefore can be deemed as more permanently compressible. It may also be the case that it just takes the filled materials a longer period of time to recover its shape than the test allows for.

Gen. 2 compression set data for silica molded samples seemed to suggest just the same as the talc samples. A decrease in elastic recovery is apparent in the data and can be attributed to the more compressible nature of the modified materials. Silica also seemed to inhibit the material's inherent abilities to spring back and therefore can be deemed as having a higher compression set value as well.

Sources of Error

The sources of error seen in Gen. 1 mixing, molding, and testing processes were the very reason of the need for Gen. 2 mixing, molding, and testing. It is very clear from the Gen. 2 presentation of data that a number of these sources of error were either eliminated or significantly minimized. However, some error is still likely to have occurred in Gen. 2 of the project. Some errors that may have existed include variation across the plaque in properties and inaccurate designation of modifier percentages. There is also the potential for error in all of the compression set data since the results were based on a single test sample size.

Throughout many of the mechanical tests, samples were cut out of a section of the molded plaques. One potential result of the project that was out of the scope of the project was the chance that variability may exist depending on where the section of the plaque was taken. There were extensive efforts to try and minimize this error effect as many of the sections were purposely taken from identical spots of the plaque, i.e. the gate region, end of fill region, in flow direction, and transverse to flow direction.

The exact percentages that were labeled in all of the data may have deviated from actual molded in percentages of modifiers. However, this was not seen as a major avenue of error since it would have affected all the samples the same. The validity in the data exists because, despite the potential of the blends not being the exact percentage of filler that was thought to have been tested, all the blend levels were successive and therefore accurately captured the desired trends in the data.

Final considerations for general research error in this project include the potential for minimal or no bonding/ adhesion of the modifiers to the respective base LSR polymer matrix. If this were the case, extensive efforts could be made to try and improve the viability of the wetting of the modifiers by trying new coupling agents, or experimenting more with the titanate that showed some potential. Lastly, a larger sample size should be collected for tests like compression set and hardness testing in order to drive out error from the experiments.

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