Influence of Processing Pressure Differential and Mixing Module Configuration on Volumetric Ratio and Physical Properties of a Spray Polyurea Elastomer.

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ABSTRACT

Physical properties for an experimental fast-set spray polyurea were measured under various processing pressure and temperature conditions. Statistical analysis was applied to the physical property results, over a range of pressures and temperatures, to identify the relative influence of each on the physical properties. Often times the gauges used for measuring the process pressure for each component are not equal, due to various equipment configurations and physical characteristics of the spray system. Low and high-pressure differential conditions were created to study how the volumetric ratio and physical properties are influenced under these conditions. Further investigation and experimental data reveal the impact of mixing module nozzle diameter, nozzle orientation, and spray tip assembly, on the pressure differential and physical properties.

INTRODUCTION

Polyurea technology is currently being used in a wide variety of commercial applications from concrete coatings to control joint fillers.[1,2] In this study, we focus on how the final properties of an experimental spray polyurea system with a very short gel time (fast-set), are directly connected to the process parameters.[3] The connection rests in the ability of the process parameters, such as temperature and pressure, to influence how well the streams of chemicals mix in an impingement process. In this paper, we studied how processing temperature, processing pressure, and spray gun configuration influence the impingement mixing efficiency, and ultimately the quality of this spray polyurea system. In the literature there are several methods reported that provide accurate means for observing mixing efficiency, and in our study we chose tensile strength and elongation as our measure of mixing quality.[4, 5, 6]

One unique characteristic of fast-set spray polyurea systems is their ability to cure rapidly, offering end-users excellent mechanical properties in a very short period of time. Mechanical properties are not reached immediately after application, but rather they increase over time, and the rate at which these mechanical properties increase, is dependent upon mixing efficiency. In this study we have investigated how favorable, and unfavorable processing conditions, as well as different spray gun configurations, influence the cure rate and final mechanical properties.

Impingement mixing is reported as the most efficient means of mixing fast reacting polymers.[7] Several references and publications specify the use of impingement mixing for applying spray polyurea systems, but little is said as to how the impingement process produces a quality spray polyurea product. The impingement mixing process, and how it is capable of producing homogenous mixtures, has been extensively studied for RIM applications.[8] The fundamental principles of impingement mixing discussed in these applications, also apply to the impingement spray gun equipment commonly used in the industry today. In this study, we employ a model to help describe the mixing process, and we identify the critical steps in the impingement mixing process.

BACKGROUND

Mixing Process

In the mixing chamber, the kinetic energy of both reactant streams is transferred into various forms of mixing—macromixing and micromixing. During high velocity impingement, intense turbulent motion, shearing, and stretching of the reactants reduces the individual stream sizes to a very fine scale. Efficient macromixing is critical in the mixing chamber to create uniform concentration regions of each reactant. Furthermore, the smaller scale or shorter distance between each reactant stream, the greater the efficiency of subsequent forms of mixing. These regions, or layers, are typically on the order of 100um. Further reduction in stream thickness occurs with micromixing, or interfacial activity and diffusion, between the two reactants.[8] The thickness and distribution of these layers due to macromixing and micromixing, significantly affect the diffusion rate at which the reactants come in molecular contact to complete the reaction.

The efficiency and time for each of these steps in the impingement process has been considered in detail.[7] Essentially, if sufficient energy is achieved in the mixing chamber, an isotropic state is reached and each form of mixing can be considered independent of each other. Nguyen and Suh (1986) compared the approximate efficiency of each mixing step and found the efficiency to decrease in the following order; impingement mixing, stretching, shearing, and diffusion. Even though all the mixing steps occur simultaneously, and at different levels of efficiency, the residence time is too short to complete the final mixing step—diffusion. So, diffusion mainly takes place outside of the mixing chamber, and it is responsible for smoothing out the concentration gradients between the reactants. Since the majority of diffusion takes place outside of the mixing chamber, its rate is subject to ambient temperature conditions and can be adequately described by an Arrhenius equation:[7]

$$D = Do\ e\ –Q/RT$$

in which D is the diffusivity of the material, Do is known as the frequency factor, Q is the activation energy, and R is the gas constant. Process parameters are known to influence the efficiency of mixing, and calculating the Reynolds number is one method of measuring this influence. The Reynolds number (Re) is an expression used to quantitatively describe the individual contribution of each reactant stream in terms of kinetic and viscous forces.[8]

$$Re = 4\ \rho\ Q\ /\ \pi\ \mu\ d$$

Re is simply the ratio of density (ρ) and flow rate (Q), to viscosity (μ) and nozzle diameter (d). Given this relationship between viscosity and flow rate, it is obvious how process parameters, such as temperature, pressure, and nozzle diameter strongly influence the Reynolds number. Typical Re values range from 100 to 1500, and the minimum Re to provide adequate mixing is usually between 300-500.[8]

Residence Time

In order to compare the influence of mixing chamber geometry on mixing efficiency, it is important to understand how long the material resides within the mixing chamber. This information can then be utilized to estimate the time for each step in the mixing process. Previous kinetic studies confirmed that aliphatic and aromatic amine resins, which are commonly used in formulating spray polyurea systems, react almost instantaneously with isocyanates.[9] Even though these chemical reactions are thermodynamically and kinetically favored, in reality, the time required for micromixing and diffusion is sufficiently slow to extend the time of reaction, therefore allowing more time for macromixing to occur.

Previous visualization studies have demonstrated that turbulent motion during the residence time, decays into laminar flow within 2-3 chamber diameters of the impingement point.[8] Furthermore, the characteristically high reaction viscosity, which occurs when polyurea is formed, suggests that turbulent motions will be dampened soon after the point of impingement. Since the chaotic motion of turbulence rapidly decays into laminar flow, this would suggest that the final polymer network in the sprayed elastomer is a good representation of the degree of macromixing or turbulent mixing.[7] With this assumption, we can use the physical properties of a spray polyurea elastomer to study how the mixing chamber geometry will affect macromixing.

Macromixing time (M_t) is typically on the order of 0.2 – 3.0 milliseconds and it can be calculated from the relationship between the mixing chamber length (L) and the stream velocity (u): [7]

$$M_t = L / 2u$$

Even though diffusion occurs simultaneously with macromixing, the rate of diffusion is controlled by the concentration gradient between reactants. The diffusion time (D_t), typically on the order of seconds, can be approximated using known constants for typical polymer solutions and the Kolmogorov microscale value (n):

$$D_t \sim n^2 / D \qquad D = K\,M^{-d} \sim 10^{-6} \qquad n = (v^3 L / u^3)^{1/4}$$

in which D is the molecular diffusivity, K & d are constants, M is the molecular weight, L is the chamber length, and u is the average stream velocity.[7] The actual diffusion time, which works to smooth out the concentration gradients between reactants, is strongly dependent upon the macromixing efficiency and the post-mixing temperature.

EXPERIMENTAL

Spray Polyurea Elastomer

The experimental spray polyurea elastomer used in this study meets the Polyurea Development Association's definition of a polyurea.[10] The resin side (B-side) was a specially formulated blend of multi-functional, high molecular weight amine-terminated resins, and low molecular weight amine chain extenders. The isocyanate side (A-side) is a quasi-prepolymer, specially formulated to impart flexibility and fast-reactivity to the spray polyurea elastomer. The fast-set nature of this experimental spray polyurea elastomer is achieved without the need of catalysts or hydroxy-terminated resin, since isocyanates and amines resins react rapidly to form high molecular weight polymers within seconds. The effective gel time and specific gravity for each component of this spray polyurea elastomer is listed in Table 1.

Table 1: Characteristics of the experimental spray polyurea elastomer.

Description	Specific Gravity, g/cm3	Effective Gel Time, seconds	Hardness, Shore A
MDI quasi-prepolymer (A-side)	1.12	4 seconds	93
Polyamine resins (B-side)	1.01		

Procedures

Each spray polyurea elastomer sample was approximately 0.080 inches in thickness and the flow data were recorded during the preparation of each sample. Each spray polyurea sample was allowed to cure at 70ºF and 45% relative humidity. Tensile strength and elongation measurements were recorded with an Instron 5565 according to ASTM D412. Specific gravity measures were calculated from the average thickness and weight of a 3 in^2 sample. A Brookfield CAP 2000, cone and plate viscometer was utilized to measure the viscosity of each component between 120ºF to 160ºF.

Application Equipment

A hydraulically operated, plural-component, proportioning unit was used to process the experimental spray polyurea elastomer. The proportioning unit was a Gusmer® H-20/35-35, capable of dispensing 24 lbs./min. with a maximum static pressure of 3500 psi (Figure 1). The unit was specially equipped with a flow monitoring system to provide output data every 2 seconds during operation. During our study we set the hydraulic pressure at 420, 640, 940, and 1,120 psi to generate a processing pressure of 1000, 2000, 2500, and 3000, respectively. The unit is equipped with two 6000-watt primary heaters, which are capable of raising the temperature of each component by 70ºF at the maximum output. The digitally controlled primary heaters and hose heater were each set at the same temperature, and the processing temperatures for this study were 120ºF, 140ºF, and 160ºF.

Three Gusmer® spray guns were chosen for this study to evaluate how different mixing module geometries influence the physical properties of the spray polyurea elastomer (Table 2). Figure 2 (A) is the GX-7 direct impingement spray gun, which is a mechanically purged gun with the mixing module nozzle orientation set at 180º (B). The GAP spray gun, Figure 3 (C), is a Gusmer® air purge spray gun, which has a smaller mixing chamber diameter than the GX-7 series, and its nozzle orientation is set at 180º. The GX-7 400 series spray gun (Figure 4) is mechanically purged, and its nozzle orientation is 180º, but offset so the A-side nozzle is closer to the front of the mixing module than the B-side nozzle.

Table 2: Mixing module and spray gun details.

Spray Gun	Reference Number	Mixing Chamber Diameter, Inches	Mixing Chamber Length, Inches	Number of Nozzles A / B	Pattern Control Disc
GX-7 453	17190-453	0.125	0.43	2 / 2	212
GX-7 452	17190-452	0.125	0.43	1 / 1	212
GX-7 DI	22190-2-125	0.125	0.43	1 / 1	212
GAP	35160-1-F-00	0.046	1.72	1 / 1	212
GAP	35160-1-F-00	0.046	1.72	1 / 1	None

Figure 1: Gusmer® H-20/35 proportioning unit.

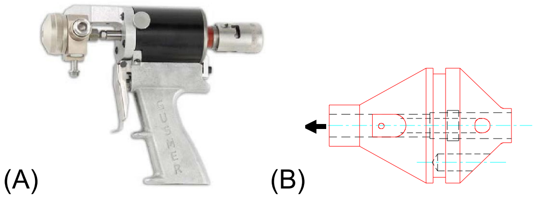

Figure 2: GX-7 Direct Impingement spray gun (A) and mixing module (B) (courtesy of Gusmer®).

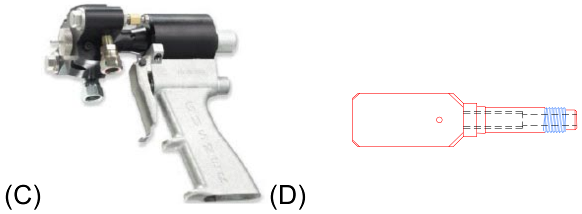

Figure 3: GAP spray gun (C) and mixing module, 00/01 (D) (courtesy of Gusmer®).

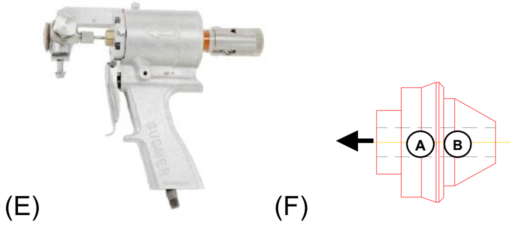

Figure 4: GX-7 400 series spray gun (E) and 452 / 453 mixing modules (F) (courtesy of Gusmer®).

RESULTS AND DISCUSSION

Process Variables

Table 3 contains the tensile strength of the spray polyurea elastomer processed at different temperatures and pressures. These samples were created with a GX-7 453 spray gun and cured for seven days at 20ºC. The average tensile strength and standard deviation was calculated at each temperature over a range of pressures, and also, at each pressure over a range of temperatures. The standard deviation was between 223-283 psi when the temperature was held constant and the pressure was increased from 1000 psi to 3000 psi, which is significantly higher than 81-102 psi when pressure was held constant and the temperature was increased from 120-160ºF. These results suggest a greater dependence of mixing efficiency on processing pressure rather than processing temperature. This is not to say that processing temperature is unimportant, but these results only speak to the relative importance between pressure and temperature.

Table 3: Tensile/Elongation vs. Range of T & P

	Spray Processing Pressure (Hydraulic pressure)				
Spray Processing Temperature (Primary and Hose)	**1000 psi** (420 psi)	**2000 psi** (640 psi)	**3000 psi** (1,120 psi)	**Average Tensile Strength (1000-3000 psi)**	**Std. Dev. (1000-3000 psi)**
120°F	1704	1922	2150	1925	223
140°F	1582	2032	2103	1906	283
160°F	1736	2126	2274	2046	278
Average Tensile Strength (120-160°F)	1674	2027	2176		
Std. Dev. (120-160°F)	81	102	88		

The volumetric flow rate is one critical variable in calculating the Reynolds number. Figure 5 shows how the pressure and temperature influence the output. As the processing pressure increased from 1000 psi to 3000 psi, the output increased by 79%, when the temperature was held constant at 160ºF. At a constant temperature of 120ºF, the output is increased by 75% over the same pressure range. So, the output is significantly dependent upon processing pressure within this temperature range.

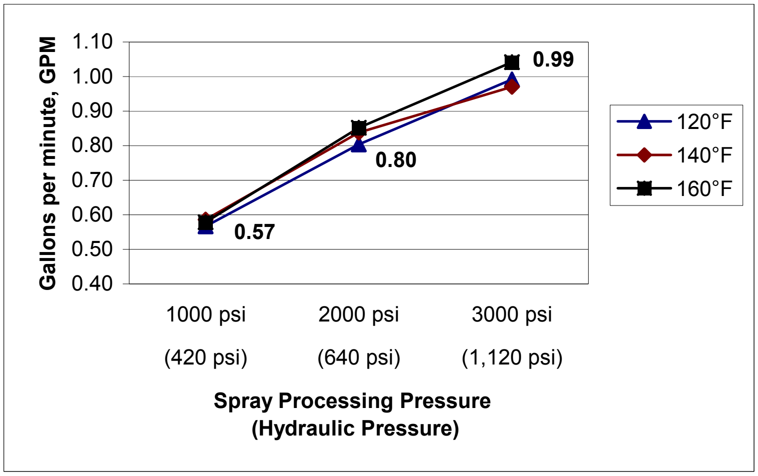

Figure 5: Effect of pressure and temperature on output.

Even though the calculated Reynolds number is influenced by these process parameters (Figure 6), the physical properties do not reflect an equal contribution to mixing efficiency by each of these parameters. In other words, if the processing temperature is 160ºF while the processing pressure is 1000 psi, this would result in a Reynolds number of 428 for the isocyanate stream. If the parameters are changed to 120ºF and 3000 psi, the Reynolds number for these parameters is 347. Theoretically, the mixing efficiency should be lower for these parameters and will result in a lower tensile strength. In reality, the opposite result is seen when reviewing the results in Table 3. Given this observation, we can conclude that the critical Reynolds number is reached at these lower process parameters, and that the *reaction* viscosity, is dominating the mixing efficiency. Increasing the processing pressure delivers more kinetic energy to the impingement zone to help overcome the dampening effect of a high reaction viscosity on turbulent motions.

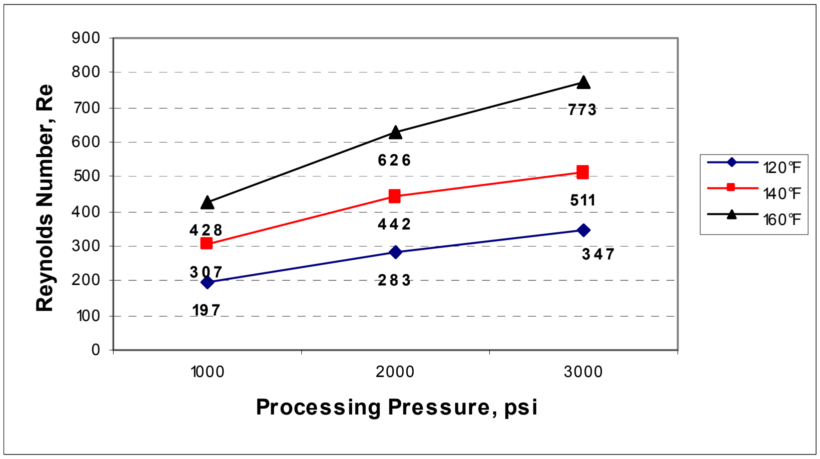

Figure 6: Effect of pressure and temperature on Reynolds number (453 module, Part A nozzle).

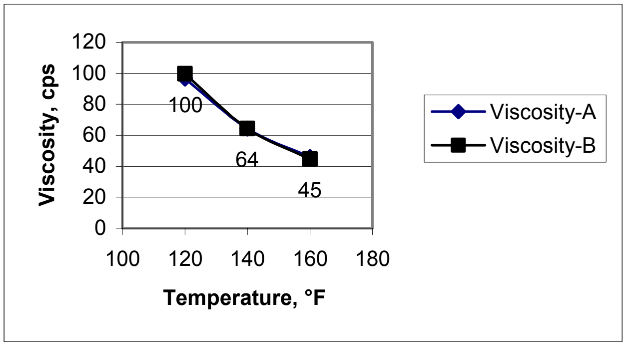

Figure 7: Effect of temperature on viscosity.

Spray Gun Configuration

Choosing the proper spray gun configuration for a spray polyurea system is important for producing consistent and reliable mechanical properties. A manufacturer of spray polyurea systems is responsible for providing this important gun configuration to the end-user, so proper application and optimal performance of the spray polyurea elastomer is realized in the field. Table 4 lists the various spray gun configurations we used to study how pressure differential, nozzle diameter, and nozzle orientation influence volumetric ratio and mixing efficiency of this experimental spray polyurea elastomer.

To create different gauge pressures for the isocyanate and resin components, we adjusted the nozzle diameter of a 452 and DI mixing module, and processed the spray polyurea elastomer at 140ºF and 2500 psi. When we changed the nozzle diameter the output changed proportionately, and the impact of this change on the Reynolds Number can be seen in Table 4. We were able to produce pressure differentials from 0 to 1100 psi with these different nozzle diameters, and there was a negligible influence on the volumetric ratio under high pressure differential conditions. This is to say that the spray polyurea elastomer was “on-ratio” even when the processing pressure difference between A-side and B-side was 1100 psi. This result was measured with a hydraulically driven unit with horizontally opposed proportioning pump (Figure 1), and proportioning units with different designs (e.g., vertical proportioning pumps, air-driven units, etc.) might produce different volumetric ratio results.

Applying plural component systems when the processing pressure differential is greater than 200 psi is generally not recommended, and the acceptable difference must be defined for each individual system.[11] One objective of this study was to evaluate how the volumetric ratio, mixing efficiency, and physical properties change with different pressure differentials. Generally, a pressure differential greater than 200 psi is a symptom of either a restriction in the nozzle, or a lack of material being supplied to the proportioning unit. In such cases, the application of a spray polyurea elastomer should be stopped immediately to investigate the source of the large pressure differential.

Table 4: Effect of spray gun configurations on processing pressure and Reynolds Number.

Mixing Module	Gallons per Minute	Nozzle A In.	Nozzle B In.	Δ P	Vol. Ratio A / B	Hydraulic Pressure Psi	Process Pressure A Psi	Process Pressure B psi	Reynolds Number A-side	Reynolds Number B-side
453-25-23	0.88	0.025	0.023	200	1.00	940	2200	2400	502	545
452-25-20	0.66	0.025	0.020	0	1.00	940	2300	2300	744	930
DI-25-20	0.62	0.025	0.020	100	1.01	940	2400	2300	699	874
DI-28-28	0.82	0.028	0.028	0	1.01	940	2300	2300	826	826
DI-23-23	0.63	0.023	0.023	300	1.01	940	2100	2400	780	780
GAP00-212	0.79	0.029	0.029	0	1.01	940	2400	2400	778	778
GAP00R	1.10	0.029	0.029	0	1.01	940	2300	2300	1,069	1,069
DI-32-20	0.78	0.032	0.020	1100	1.02	940	1700	2800	718	1,148
452-32-20	0.73	0.032	0.020	1100	1.03	940	1700	2800	643	1,029

Pressure Differential

During the course of this investigation, we discovered a relatively large difference in physical properties after 24 hours of curing at 20ºC (Table 5). We were surprised to learn that samples prepared with a large pressure differential performed better than samples prepared with balanced pressures. Multiple samples were created for each spray gun configuration, so we compared the test results within each set and again found the same results. We verified our initial findings by repeating the study, paying close attention to create samples of equal thickness (0.08 +/- 0.01), as well as storing the samples under the same conditions (20ºC). We found our initial results were repeatable, so we continued to monitor the change in tensile strength over time.

Table 6 lists the tensile strength after 7 days, and when we compared these results to the 24-hour tensile strength results, we found that the cure rate changed for different spray gun configurations. Overall, most of the samples that displayed lower tensile strengths at 24 hours did achieve tensile strengths close to, if not greater than, the other samples after 7 days. Even though some of these results might seem surprising, we applied our understanding of the mixing process to study which variables in the mixing chamber were influencing the final polymer network of the spray polyurea elastomer.

Table 5: Effect of spray gun configurations on physical properties after 24 hours.

Mixing Module	Tensile Strength, psi	Standard Deviation Psi	Elongation, %	Standard Deviation %
453-25-23	1389	126	317	19
452-25-20	1358	44	309	12
DI-25-20	1533	50	340	6
DI-28-28	892	36	161	15
DI-23-23	906	117	198	26
GAP00-212	979	87	226	19
GAP00R	962	36	160	13
DI-32-20	1574	50	350	10
452-32-20	1530	33	341	5

Table 6: Effect of spray gun configurations on physical properties after 7 days.

Mixing Module	Tensile Strength, psi	Standard Deviation psi	Elongation, %	Standard Deviation %
453-25-23	1870	96	407	10
452-25-20	1586	105	346	14
DI-25-20	2010	61	368	15
DI-28-28	1836	36	405	10
DI-23-23	1736	65	379	9
GAP00-212	1922	89	383	14
GAP00R	2299	79	375	11
DI-32-20	2179	88	415	13
452-32-20	1720	122	358	17

Nozzle Diameter

In Figure 8, we compare how the nozzle diameter for each stream influences the tensile strength after 24 hours. In one mixing module, DI-25-20, the isocyanate stream (A-side) diameter was 0.025 inches and the amine resin (B-side) stream diameter was 0.020 inches. Two other modules with equal nozzle diameter for each stream, 0.028 (A & B) and 0.023 (A & B), were used for comparison. When the nozzle diameters were equal, we found the tensile strength was approximately 40% lower than the sample prepared with a module with unequal nozzle diameters. After seven days, this difference between was only 11%.

The large initial difference, followed by a smaller difference after 7 days, indicates a slower diffusion rate in the samples prepared with equal nozzle diameters. A slower diffusion rate implies a larger concentration gradient exists between the amine and isocyanate regions, or in other words, lower macromixing efficiency. The lower macromixing efficiency might be explained by recognizing that 180 degree impingement points are not always stable. Previous studies have found that 180-degree impingement points rarely realign if disturbed, and they don't always occur in the middle of the mixing chamber.[12]

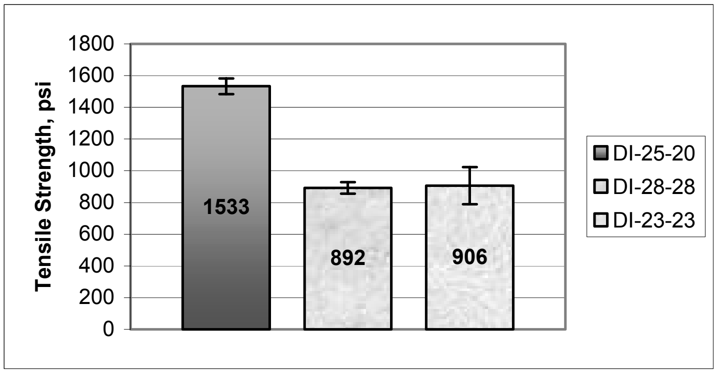

Figure 8: Effect of nozzle diameter with direct impingement on tensile strength at 24 hours.

Nozzle Orientation

Figure 9 shows how the nozzle orientation influences the tensile strength at 24 hours and 7 days. The 452 module has one nozzle for each component, and the isocyanate nozzle diameter was 0.025 inches, while the resin nozzle diameter was 0.020 inches. These nozzles are offset from one another by 0.100 inches, so the two streams do not directly impinge. The nozzle diameters for the DI-25-20 module were identical to the 452-25-20, but aligned for direct impingement at 180 degrees.

After 24 hours, the tensile strength for the modules with different orientation was within 11% of each other, indicating similar macromixing efficiency. The difference in tensile strength between the DI-25-20 and 452-25-20 after 7 days increased to 21%. The macromixing efficiency may appear to be similar in both modules of different orientation, but limited growth in tensile strength of the 452-25-20 sample suggests nonuniformity in flow in the mixing chamber. The lack of uniformity in flow will give rise to a large distribution of concentration gradients in the amine and isocyanate layers, leading to lower performance properties in the experimental spray polyurea elastomer.

Directly impinging the isocyanate and resin streams readily disperse the two reactants in the mixing chamber and allows them to form layers, which are thin and uniform in concentration. The combination of thin, uniform regions of isocyanate and amine resin, allows the individual molecules to diffuse rapidly and form a polymer network with less irregularities.

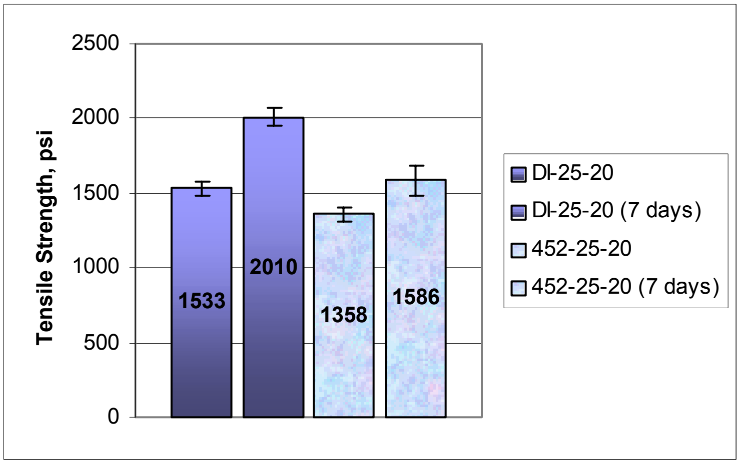

Figure 9: Effect of nozzle orientation on tensile strength at 24 hours.

Mixing Chamber Geometry

Mixing chamber geometry is known to play a role in the quality of impingement mixing.[8] The difference in mixing chamber geometry between the DI-28-28 and GAP00R is quite large, which accounts for a significant difference in residence time and turbulent mixing volume (Table 7). Figures 10 & 11 show the difference in tensile strength and elongation in the spray polyurea elastomer after 24 hours and 7 days.

One notable difference between the two mixing chambers is that the GAP00R does not use a pattern control disc to form a spray pattern. The lack of a pattern control disc increases the specific gravity and slightly darkens the sample color prepared by the GAP00R (Figure 12). Even with greater specific gravity, or less air voids that lighten the polyurea elastomer color, the tensile strength was approximately equal to the DI-28-28 after 24 hours.

The approximately equal tensile strengths after 24 hours for both mixing chambers suggests that geometry of these two mixing chambers do not significantly influence the diffusion rate, or mixing quality. The difference in tensile strength at 7 days between the two geometries, DI-28-28 vs. GAP00R, is attributed to the greater specific gravity in the sample prepared without a pattern control disc. When we fitted the GAP00R with a pattern control disc (GAP00-212), and measured the specific gravity, output, and tensile strength, the values decreased from 1.04 to 0.92, 1.10 to 0.79, and 2299 to 1922 psi, respectively.

Table 7: Effect of mixing chamber dimensions on mixing time.

	Gallons per minute, GPM	**Mixing Chamber Volume (mm^3)**	**Mixing Chamber Diameter, (mm)**	**Residence Time, ms**	**Turbulent Mixing Volume (mm^3)**
DI-28-28	**0.82**	**86.4**	**3.17**	**1.79**	**50.2**
GAP00R	**1.10**	**47.8**	**1.18**	**0.74**	**2.6**

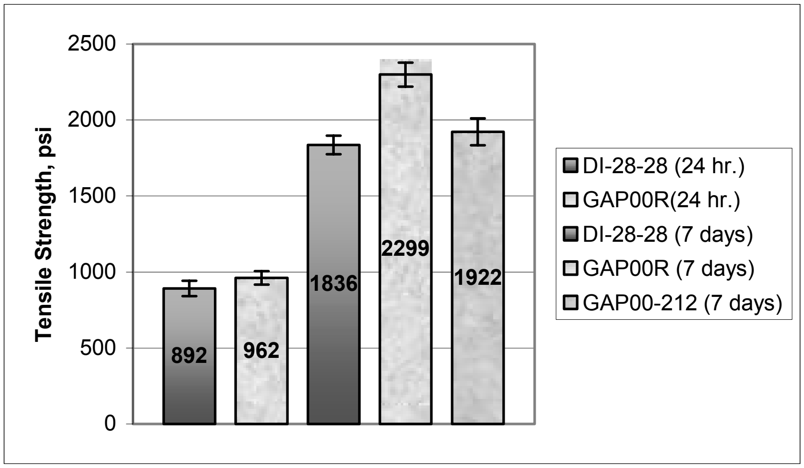

Figure 10: Effect of mixing chamber dimensions on tensile strength (24 hrs. & 7 days).

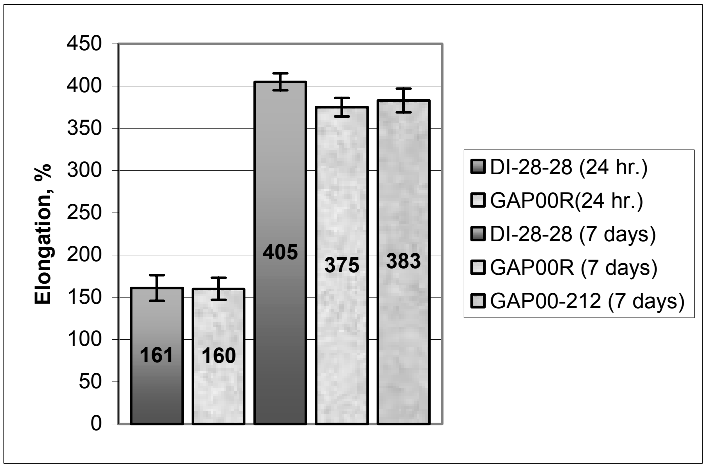

Figure 11: Effect of mixing chamber dimensions on elongation (24 hrs. & 7 days).

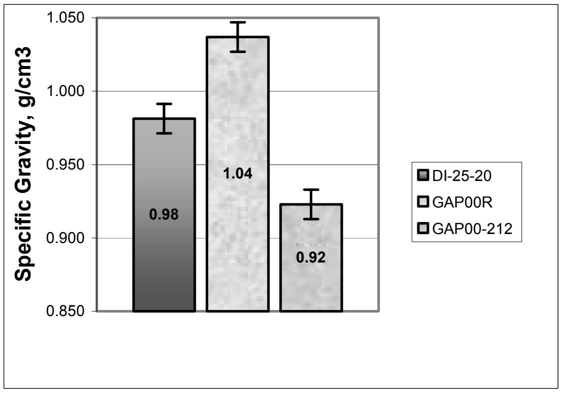

Figure 12: Effect of spray tip assembly on specific gravity.

CONCLUSION

The fast, consistent reactivity of spray polyurea elastomers produces excellent physical properties in a very short period time. For fast-set spray polyurea elastomers with gel times of only a few seconds, impingement mixing is the most efficient means of mixing both the isocyanate and amine reactants. Impingement mixing is a mechanical-free form of mixing which uses the velocity of each reactant to create turbulent motions within the mixing chamber, reducing the thickness of each reactant stream to a very fine scale. Efficient mixing is associated with creating very thin, alternate layers of each reactant, which allows for diffusion between each layer to take place in a short period of time. Faster diffusion results in greater physical properties in the spray polyurea elastomer within just a few hours, whereas less efficient mixing will require more time for diffusion, resulting in a slower increase in physical properties.

Processing pressure plays a prominent role in maximizing the mixing efficiency of a spray polyurea elastomer by overcoming the intrinsically high reaction viscosity seen in the mixing chamber. Processing temperature aids in reducing the fluid viscosities, and contributes to reaching the critical Reynolds Number for efficient mixing. The effect of large processing pressure differential on volumetric ratio was explored and found to be negligible, and the spray polyurea elastomer application under these extreme conditions was determined to be “on-ratio.” Applying a spray polyurea elastomer in the field with a large processing pressure differential is not recommended for various reasons.[11] We found the final physical properties of the spray polyurea elastomer, or mixing efficiency, were influenced in the following order of importance; nozzle diameter, nozzle orientation, and spray tip assembly.

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BIOGRAPHY

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