Novel CO₂-based Polycarbonate Polyols for Cast Urethanes

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Abstract

Novomer is an emerging sustainable chemistry company which has pioneered a new-to-the-world polycarbonate polyol for urethane applications. Using a proprietary catalyst system which enables CO₂ to react precisely and efficiently with epoxides, Novomer produces highly precise polyols with a unique, “high density” polycarbonate backbone, perfect –OH functionality, and very narrow polydispersity. In urethane applications, these polyols deliver extremely high tensile & flexural strength, excellent chemical & hydrolytic stability, high UV resistance, and very good transparency. In addition to exceptional performance, these new polyols are up to 50% CO₂ by weight and have a 3-9x advantage in carbon footprint vs. existing petroleum-based polyols used in cast urethane systems.

Introduction

Significant effort has been expended in recent years in the pursuit of more renewable and sustainable materials for polyurethanes. A large sub-segment of this work has been focused on biomass-based materials, such as “natural oil polyols” from sources such as soy and castor oils. Novomer was founded to commercialize an entirely different approach to sustainable polyols in which waste carbon dioxide is used as a raw material and sequestered in a useful polyol product.

The core of Novomer’s technology platform is a highly active and selective catalyst which delivers a perfectly alternating copolymer of epoxides with CO₂. Building upon the pioneering work of Coates et al. in the field of cobalt salen complexes [1], Novomer has advanced the catalyst technology and developed a family of proprietary catalysts with significantly improved activity and selectivity [2]. These catalysts maximize CO₂ incorporation and provide polycarbonates with perfectly alternating CO₂-epoxide backbones and low polydispersity indices. In addition to improving the performance of the resulting polymers, these catalysts allow Novomer to conduct polymerizations using parts per million levels of catalyst. Recently, Novomer has developed and has been granted intellectual property [3] on the use of chain transfer technology to produce CO₂-based polyols with low molecular weights (500 to 10,000 g/mol). Novomer has proven the ability to design and produce tailored poly(propylene carbonate) (PPC) and poly(ethylene carbonate) (PEC) polyols with very specific molecular weights, degrees of branching, and hydroxyl endgroup functionalities. This highly flexible polyl technology
platform can thus be applied to a wide range of thermoset applications including coatings, foams, adhesives, elastomers, and TPU.

Polycarbonate polyl-based polyurethanes are known for their excellent strength properties, hydrolytic stability, oxidative resistance as well as heat resistance properties [4-6]. However, the commercial application of these petroleum-derived polycarbonate polyls is still limited, primarily due to their high cost relative to traditional polyether and polyester polyls.

The polycarbonate polyls produced by Novomer are a first-in-the-world technology and exhibit unique performance properties versus existing polycarbonate, polyether, and polyester polyls. The studies described in this paper had two objectives: first understand the fundamental structure/property relationship of PPC in solid urethane systems, and then to understand how PPC behaves in formulated PU systems which also contain traditional polyester polyl.

**Experimental – Part 1 (Structure / Property Relationship)**

**Chemicals:**
Raw materials used in this study are shown in Tables 1 and 2. Prior to preparation of NCO-prepolymers, polyls and 1,4-BD were dried for 24 hours at 75°-80°C under vacuum of 1-3 mm Hg and continuous mixing by magnetic stirrer. The water content after drying was checked by Karl Fisher Titrator.

Diphenylmethane 4,4’-diisocyanate (MDI) was used as received from the supplier and its isocyanate content was checked by di-n-butylamine titration method (ASTM D-5155).

**Preparation of NCO-prepolymers:**
The NCO-prepolymers were synthesized utilizing a standard laboratory procedure for prepolymer preparation as follows: MDI melted at 60°C was placed in the heated reaction kettle, which was equipped with a stirrer, thermometer, and continuous flow of nitrogen. Preheated polyl was added slowly to isocyanate at 60°-65°C and reaction was continued at 70-80°C for about 105 minutes. The NCO% of the prepolymer was checked periodically during the synthesis. Afterwards, the prepolymer was degassed under vacuum, transferred into glass jars and sealed under dry nitrogen. The NCO% of the resulting prepolymer was checked after 24 hours according to ASTM D5155.

**Preparation of polyurethane elastomers:**
Cast polyurethanes were prepared by reacting NCO-prepolymers with a chain extender at an isocyanate index of 1.02.

Urethane sheets and round bottom samples were prepared to test their physico-mechanical properties. The urethane sheets were prepared using a laboratory compression molding method (Carver press). NCO-prepolymer was preheated at 80°C, weighed into a Speed Mixer cup and heated at 120°C for 15
minutes in an air circulation oven. A chain extender (conditioned at 80°C) was added to the prepolymer and all components were mixed via Speed Mixer (FlackTek Inc.) for 20 seconds at 2200 rpm and transferred into an aluminum mold covered with a Teflon sheet that was preheated at 120°C. At the gel time, the mold was closed and cured for 2 hours at 120°C. Afterwards, the samples were post-cured for 20 hours at 110°C.

Urethanes with 50% hard segment concentration based on Fomrez 44-160 were prepared by adding straight MDI to the prepolymer prior to adding the chain extender. Urethanes based on UH-50 at 50% hard segment concentration were prepared by adding calculated amount of UH-50 polyol to the chain extender.

Cylindrical “button samples” (6.5cm² x 1.3 cm) for testing of hardness and resilience were prepared by casting of degassed polyurethane system into a Teflon coated mold with multiple cavities which was preheated at 120°C. The mold was then covered with Teflon coated aluminum plate, transferred into an oven at 120°C, cured for 2 hours and then post-cured for 20 hours at 110°C.

The urethane were kept in the desiccators and aged for seven days at RT prior to testing.

**Testing:**

**POLYOLS:**
The following measurements were carried out on polyols (Table 2):
- Hydroxyl number, ASTM D 4274-05
- Acid number, ASTM D4662-08
- Viscosity, 50° and 70°C, ASTM D 4878-08
- Water, Karl Fisher method, ASTM D 4672-08
- Glass transition temperature, DSC (DSC Q 10, TA Instruments at heating rate of 10°C per minute)
- Mn and Mw, GPC analysis

**NCO-PREPOLYMERS:**
NCO% was measured according to ASTM D 5155 and viscosity at 70°C via Rheometrics.

**ELASTOMERS:**
The following properties were measured on the finished urethanes:
- Hardness, ASTM D-2240, Shore D
- Tensile properties (Tensile strength at Yield and Break and Young’s modulus), ASTM D 412
- Toughness at yield (tensile strength x elongation% at yield)
- Tear Strength, Die C, ASTM D 6240
- Abrasion Resistance, ASTM D 1044 (H22 wheels, weight load 500g, 2000 cycles)
- Flexural strength and modulus (ASM D 790)
- Resilience, % (Bashore rebound), ASTM D2632
- Dynamic mechanical analysis, DMA, in bending mode (DMA 2980, TA instruments)
- Thermo-mechanical analysis, TMA (TMA Q 400, TA Instruments)
• Differential scanning calorimetry, DSC (DSC Q 10, TA Instruments at heating rate of 10°C per minute)
• Heat resistance of elastomers: tensile properties at 50° and 70°C were measured by using heat chamber attached to Instron tester.

Solvent resistance to various polar and non-polar solvents, including oil and water, was measured as weight change and retention of properties of the urethanes upon their immersion in the solvents at RT for seven days.

Moisture resistance of the urethanes was measured after their exposure to 50°C and 100% relative humidity for seven days; the moisture uptake and retention of properties was measured.

Oxidative resistance was measured upon immersion of the urethanes in 30% hydrogen peroxide solution for two weeks at 37°C.

Experimental – Part 2 (PPC in formulations with other Polyols)

Raw Materials
The formulations evaluated in this report are all based on the following key raw materials:
• Novomer polypropylene carbonate (PPC) diols, with molecular weights ranging from ~800 to ~3000 grams/mol
  o Polydispersity index on all polyols was <1.2
  o All diols were 2.0 functional with no unsaturation or other functionality
  o Hydroxyls are a mixture of ~85% secondary and 15% primary
• Linear aliphatic ethylene / butylene polyester diol with molecular weight of 2000 grams/mol (EBD)
• Modified diphenylmethane-4,4’-diisocyanate (MDI) with functionality of 2.2 (MM103)
• Ester-based isocyanate-terminated two functional prepolymer
• Traditional tin-type polyurethane catalysts
• Adhesion promoters and other additives

Procedures
• Polyols were heated and degassed under 2 mm vacuum to remove residual solvent, water and any other volatile components.
• In some cases, prepolymers were created. When the experiment called for a prepolymer, it was made by reacting the specified polyol with MM103. These prepolymer ingredients were mixed thoroughly, and then degassed, again under 2mm of vacuum, until bubbling was minimal, and then blanketed with dry nitrogen at the specified temperature and time to complete the reaction.
• For prepolymers, the remaining unreacted isocyanate groups were measured using the standard dibutylamine titration method.
When necessary, the effective hydroxyl number of the polyol was then "back calculated" from the final NCO measurement, to yield a practical basis for further experiments.

For some formulations the final polymer was made by the "one shot" approach, bypassing the prepolymer step.

Once characterized for the unreacted isocyanate, the prepolymer was then reacted into the given experimental formulations, using a variety of co-reactants at various stoichiometric ratios. Typically, a final degassing also occurred after all ingredients were thoroughly mixed in a lab mixer. The mixture was then applied to bonding substrates as specified, in some cases after curing, or in some cases before substantial curing occurred, thus creating a hot melt system, or a 2K reactive system.

A subjective screening examination of bond strength was later used to determine which experimental materials exhibited sufficient bonding for the creation of ASTM bond samples for further analysis and testing.

Also, as specific formulations showed promise as hot melt or reactive systems, sheet samples were made for testing ASTM D412 properties, including ultimate tensile strength, ultimate elongation, and stress measurements at 100%, 300%, and 500% elongation.

Tear strength measurements were made in accordance with ASTM D624 Die C, and ASTM D1938.

Results and Discussion

Polyols:
Results of GPC analysis carried out on poly(1,2-propylene carbonate) diol (Novomer polycarbonate) and two reference diols, poly(1,6-hexametlene carbonate) diol (Eternacoll UH-50) and poly(1,4-butylene adipate) diol (Fomrez 44-160) are shown in Table 2. The Polydispersity Index (Mw/Mn) of Novomer polycarbonate polyol was low at 1.37 (Table 2 and Figure 1), indicating a narrow molecular weight distribution (Figure 1). It is well known that molecular weight distribution has an impact on properties of urethanes. Polydispersity Index of Eternacoll UH-50 polyol was 7.7, which is relatively high. The Polydispersity Index of Fomrez 44-160 polyol was 2.75, which is typical for commercial aliphatic polyester polyols.

All three tested polyols are solid at room temperature. Their viscosities at 50° and 70°C are reported in Table 2. The viscosity of the Novomer polyol was lower than that of Eternacoll UH-50. However, Tg of Eternacoll UH-50 polyol was lower than that of Novomer polyol (Table 2), which can be ascribed to the longer hexamethylene chain in the polyol backbone of Eternacoll UH-50 polyol.

Polyurethane elastomers containing only PPC:
Polyurethanes plaques based on all three types of polyols were prepared by the prepolymer method at a 1.02 isocyanate index and hard segment concentration of 50%. Their properties are shown in Table 3.
At 50% hard segment concentration, the PU based on Novomer polyol was harder at RT (Shore D 78) than PUs based on Eternacoll UH-50 polyol (Shore D 67) and polyester polyol (Shore D 62). The hardness of PUs based on Novomer polyol changed slightly when heated to 50° and 70°C, while hardness of PUs based on Eternacoll UH-50 changed significantly (Table 3).

As expected, the resilience measured by Bashore rebound was relatively low in the case of all three PUs. The resilience of PU based on Eternacoll UH was somewhat higher (30%) than that of Novomer type PU (22%), which can be ascribed to the longer sequence of CH2-chains between carbonate groups in the case Eternacoll UH-50 polyol (Table 3).

The stress-strain test results indicate that all three types of elastomers exhibited yield at relatively low strains. PUs based on Novomer polyol exhibited significantly higher tensile strength at yield (11,712 psi at 6% strain) in comparison to other two types of urethanes (Table 3).

The toughness at yield of PUs based on Novomer polyol was similar to urethanes based on Eternacoll UH-50 polyol and 50% higher than the toughness of PUs based on polyester polyol (Table 3).

Stress-strain measurements indicate that PUs based on Novomer polyol are very hard, high strength plastic materials.

PUs based on Novomer polyol exhibited higher flexural strength and modulus in comparison to PUs based on commercial polycarbonate and polyester polyols (Table 3).

The abrasion resistance of all three types of PUs was about 1% (Table 3).

The glass transition temperature of PUs based on the Novomer polyol, as measured by DSC, was slightly higher as compared to PUs based on Eternacoll UH-50 polyol and significantly higher than that of PUs based on polyester polyol. Similar relationship was obtained when the glass transition was measured with DMA (Figures 2 and 3) and TMA method.

Coefficient of thermal expansion measured by TMA below respective Tg’s was lower and above respective Tg’s higher for PU based on the Novomer polyol than those based on reference polycarbonate and polyester glycols (Table 3).

Heat resistance of elastomers was measured as retention of tensile properties at 50° and 70°C relative to those at room temperature (Table 3, Figure 4). The retention of tensile strength at yield of PUs based on the Novomer 076 polyol at 50°C was excellent; it retains the strength measured at room temperature. The tensile strength at yield of PUs based on Eternacoll UH-50 polycarbonate polyol decreased significantly at 50°C. The tensile strength of PUs based on Novomer 58-076 at 70°C was significantly lower than that at 50°C; still tensile strength at yield of PUs based on Novomer 58-076 polyol was higher than that of other two types of PUs.
The oil and water resistance of all three PUs, as measured by weight gain upon immersion in solvents at RT, was excellent and very similar (Figure 5). However, PUs based on Novomer polyol exhibited better resistance in non-polar solvent (toluene and xylene).

The retention of tensile strength of PUs based on the Novomer polycarbonate polyol upon exposure to different media for 7 days at room temperature was good (Figure 6). The retention of properties was good in non-polar solvents such as oil and xylene as well as in hydrochloric acid and sodium hydroxide solution.

The oxidative resistance of PUs based on Novomer polyol was excellent. The retention of tensile strength at yield after immersion in 30% hydrogen peroxide oxidative solution for two weeks at 37°C was over 80% (Figure 6). It is well known that polycarbonate polyols impart excellent oxidative resistant properties to polyurethanes [4].

The water stability (moisture resistance test) was measured by exposing PUs based on the Novomer polycarbonate polyol to 100% relative humidity at 50°C for 1 week. The retention of the tensile strength was 74% which is good (Figure 6).

**Polyurethane elastomers made with blends of PPC and Polyester Polyol:**

The Novomer polyols reviewed can create a very broad range of urethanes when blended with existing polyols.

This initial evaluation looked at the Novomer polyols in relatively simple formulations that did not have any types of additives or fillers. In addition to the very high tensile strength, very high tear properties can be achieved with these formulations, in which the Novomer polyols represent from one third to one half of the weight percent of the material. A common ethylene butylene adipate based 2000 molecular weight diol works well in this regard.

Very soft and very hard materials can be formulated to address specific urethane needs. For example, soft formulations can deliver elongations over 1000% providing shock resistance and energy absorption. A continuous range toward hard urethane is straightforward.

**Example Formulation #1:**

- A prepolymer with MM103 was made from 74-083 to a measured NCO% of 7.79. A final system was made with a blocked diamine and MDI. The system was heated to 300F for 30 minutes, and then the temperature was reduced to 275F for 16 hours.
- Result: The hardness at 275F was 75A—much harder at this temperature than any other system in which the PPC diol was the only large molecular ingredient. Like early attempts with just the PPC polyol, at RT the specimen hardened to 76D and was brittle.

**Example Formulation 2:**
• A polyester-based prepolymer was combined with a PPC polyol-based prepolymer (NCO 8.1%) and cured with traditional tertiary amine catalyst system and BDO. The formulation was 30% PPC polyol by weight. Gel time was ~2 minutes. Multiple test plaques were created (demold time ~1 hour) and tested.

• ASTM D412 Test Results (See Figure 7 below)
  - Tensile: 7,160 psi / 7,460 psi / 6,200 psi
  - Elongation 500% / 520%
  - Moduli: 100% 1,215 / 1215 psi; 300% 2,670 / 2,760 psi; 500% 7,160 / 6,840 psi
  - Tensile Set: 25% after 500% + elongation
  - Durometer: 85A at room temperature

**Example Formulation 3**

• The prepolymer used in experiment 5 (PPC polyol to a measured NCO% of 8.1) was reacted with EBD, and after that reaction was complete, it was further reacted with a blocked diamine. In this formulation the PPC comprises 28% of the total polymer weight. The mix was put into the mold and oven at 300F. After 1/2 hour the material was solidly gelled. After further curing the material at 280F for 4 hours the hardness was 80A at 275F.

• Result: At RT the hardness increased to 88A and was tough and elastic. Additional material was cured at 275F overnight. After 22 hours at 275F the material measured 81A, and at RT its hardness increased to 85A. A single tensile sample was cut from the sheet and measured 3860, only 2 hours after it had been cooled to RT and the 100% mod was only 720 psi., indicating thermoplasticity.

**Example Formulation 4**

• A formulation was created with an increased amount of PPC relative to EBD (approximately 2:1).

• Result: The physical properties were measured. The tensile graph showed very high elongation of over 900% with continual gradual yielding of the polymer; the D624, Die C tear also showed some yield but no sudden failure as is normally expected. Additionally, the D1938 split tear showed a remarkable gradual increase in tear resistance indicating toughness. (See Figures 2, 3, 4 below).

**Conclusion**

Novomer PPC polyols exhibit a unique set of performance properties when compared to existing specialty polycarbonate polyols and commodity polyester polyols in representative PU formulations. The PPC-based PUs are high performance products delivering exceptional hardness, very good tensile and flexural strength, good hydrolytic stability and good chemical & oxidative resistance.

The addition of PPC polyol to a standard polyester-based two component polyurethane delivers results similar to what one would expect when blending compatible polyols in a urethane system. The more
flexible ester polyol increased flexibility and elongation of the finished urethane vs. a PPC only system, while retaining some of the unique strength offered by PPC. This indicates that the poly(propylene carbonate) backbone does appear to impart improved high strength properties in formulated urethanes, opening up a wide range of application opportunities. It is fully compatible with existing polyester polyols and reacts well with common urethane catalysts and other additives. Thus, this report only begins to explore where the PPC backbone can be of use to improve the performance of urethane applications – many more opportunities almost certainly exist.

**Acknowledgment**

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**References**

# Tables and Figures

## Table 1. Materials

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<thead>
<tr>
<th>Designation</th>
<th>Identification</th>
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<tr>
<td>Eternacoll UH-50</td>
<td>Poly(1,6-hexylene carbonate) diol</td>
<td>UBE Chemical</td>
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<td>Fomrez 44-160</td>
<td>Poly(1,4-butylene adipate) glycol</td>
<td>Chemtura</td>
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<td>Novomer 58-076</td>
<td>Poly(1,2-propylene carbonate) diol</td>
<td>Novomer</td>
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<td>1,4-Butane Diol</td>
<td>1,4-Butane diol</td>
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<td>Mondur M</td>
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<td>Rubinate 44</td>
<td>Diphenylmethane 4,4'-diisocyanate</td>
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## Table 2. Polyol Properties

<table>
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<tr>
<th>Polyol Designation</th>
<th>Eternacoll UH-50</th>
<th>Novomer polyol</th>
<th>Fomrez 44-160</th>
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<tr>
<td>Acid Value, mg KOH/g</td>
<td>ASTM D 4662-08</td>
<td>0.39</td>
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<td>Hydroxyl Value, mg KOH/g</td>
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<td>182</td>
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<td>Moisture, %</td>
<td>ASTM D 4672-00</td>
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<td>Viscosity at 50°C, cPs</td>
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<td>3600</td>
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<td>Mn</td>
<td>GPC</td>
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<td>Mw/Mn</td>
<td>GPC</td>
<td>7.70</td>
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## Table 3. Properties of PUs

<table>
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<tr>
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<th>UH-50</th>
<th>Fomrez 44-160</th>
<th>Novomer polyol</th>
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<td><strong>Properties</strong></td>
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<td>Hard Segment, %</td>
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<td>Hardness, Shore D</td>
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<td>@ RT</td>
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<td>@ 50°C</td>
<td>53 ± 5</td>
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<td>@ 70°C</td>
<td>30 ± 3</td>
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<td>Resilience, %</td>
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<tr>
<td>@ Yield</td>
<td>8639 ± 218</td>
<td>5037 ± 428</td>
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<td>@ Break</td>
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<td>Elongation, (Room Temp.), %</td>
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<td>@ Yield</td>
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<td>19 ± 4</td>
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<tr>
<td>@ Break</td>
<td>60 ± 29</td>
<td>351 ± 26</td>
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<td>Toughness at yield, psi*</td>
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<td>Tear Strength, lbf/in</td>
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<td>Flexural strength, psi</td>
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<td>17990 ± 2404</td>
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<td>Flexural modulus, psi</td>
<td>316657 ± 11621</td>
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<td>• Via DSC</td>
<td>34°C</td>
<td>4°C</td>
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<td>52°C</td>
<td>3°C</td>
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<td>• Via TMA</td>
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<td>Coefficient of thermal expansion via TMA, µm/m°C</td>
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<td>• Below Tg</td>
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*Toughness at yield: calculated as tensile strength times elongation.
** Abrasion, 2000 rotations, 500 g, 60% vac.
Figure 1. GPC graph of Novomer polyol (Mw = 624; Mn = 456)

Figure 2. DMA Graph for Novomer elastomer with 50% hard segment concentration
Figure 3. Loss Modulus Overlay for three elastomers with 50% hard segment concentration (Green: Novomer elastomer; Blue: UH-50 elastomer; and Black: Fomrez 44-160 elastomer)

Figure 4. The effect of temperature on tensile strength at yield (psi) of elastomers with 50% hard segment concentration.
Figure 5. Resistance of PUs with 50% hard segment concentration to various solvents measured as a weight change after immersion.

Figure 6. Retention of tensile strength at yield of PUs based on Novomer polyol after immersion in different solvents (Retention of properties in water was measured by exposing PU samples to 100% RH at 50°C; In all other solvents samples were immersed at RT for one week, except for hydrogen peroxide in which samples were immersed for 2 weeks at 37°C)
Figure 7: ASTM D412 Tensile Test for Example Formulation #2

Figure 8: ASTM D412 Tensile Test for Example Formulation #5
Figure 9: ASTM D624-Die C Tear Test for Example Formulation #5

Figure 10: ASTM D1938 Tear Test for Example Formulation #5