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Experimental Investigation of Polyurethane Camouflage Coating Using Dynamic Mechanical Analysis

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Request recipient make the following pen-and-ink changes to subject report:

Page 9, Figure 4: The last line of the key for this figure, which reads "24 days," should be changed to read "SOL."

Page 11: The first word on this page, which reads "for," should be changed to read "from."

Army Research Laboratory

Aberdeen Proving Ground, MD 210055069

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Experimental Investigation of Polyurethane Camouflage Coating Using Dynamic Mechanical Analysis

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Abstract

Three polyurethane coatings were evaluated using dynamic mechanical analysis (DMA) to investigate the relationship between dynamic mechanical properties and durability properties of coated test panels. The current polyurethane solvent-based (SOL) formulation, used as a chemical-agent-resistant camouflage top coat on all military tactical vehicles, was investigated, along with newly developed water-reducible (WR) polyurethane coatings. The WR coatings offer significantly reduced volatile organic compounds (VOC), compared to the SOL system, and thus represent environmentally compliant coatings. DMA investigations revealed that the two classes of polyurethane coatings exhibit different dynamic mechanical properties, which are attributed to different **cross-linking** mechanisms involved in **film** formation. The more uniformly cross-linked SOL coating provides the best chemical-agent resistance (CAR) but the poorest mechanical properties. Properties measured using DMA were sensitive to the degree of isocyanate to hydroxyl indexing in the WR formulations, as well as the drying time of coatings prior to evaluation. DMA investigations indicated that longer cure times at ambient temperature (6 or more months) may adversely affect the mechanical properties of the SOL system and potentially enhance CAR of the WR coating. Further studies involving aged coatings are planned.

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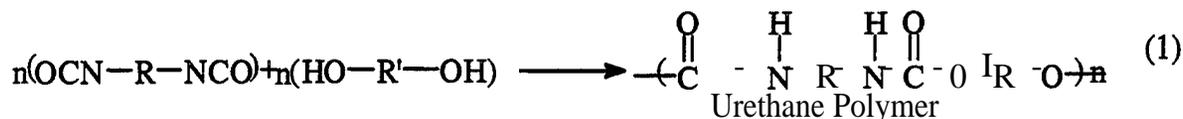
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1. Introduction

The U.S. Army utilizes polyurethane coatings as camouflage “topcoats” on all Army tactical vehicles and aircraft. These coatings not only serve to camouflage vehicles but also provide protection against chemical warfare (CW) agents. The coatings must retain their physical properties over a broad temperature range in widely varying climatic environments. Presently, camouflage topcoats used on Army vehicles are comprised of a two-component solvent-based (SOL) polyurethane. However, in an effort to meet current and anticipated Environmental Protection Agency (EPA) regulations, as well as military requirements, the U.S. Army Research Laboratory (ARL), Weapons and Materials Research Directorate (WMRD) at Aberdeen Proving Ground (APG), MD, has developed and patented a water-reducible (WR) two-component polyurethane coating for military vehicles that exhibits a 50% reduction in volatile organic compounds (VOC) compared to the SOL system. The WR polyurethane maintains required chemical-agent resistance (CAR) and exhibits superior properties compared to the SOL formulation.

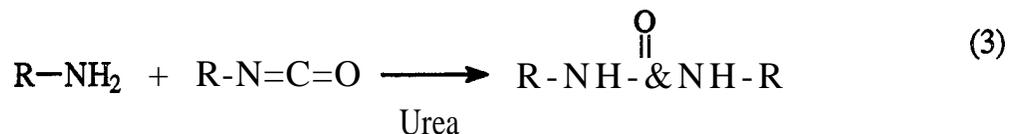
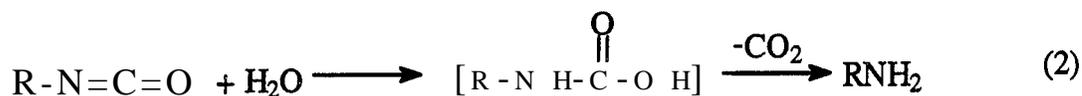
2. Background

The urethane polymer is formed by the reaction of a hydroxyl-terminated polyol with a diisocyanate, as shown in reaction 1.



SOL systems are formulated with a slight excess of isocyanate (NCO). NCO-to-OH ratios approximately equal to 1.1: 1.0 are typical for SOL polyurethane coating formulations. The excess NCO ensures complete reaction of the polyol and provides optimal film properties. Great care is taken to ensure that excess moisture is not present in nonaqueous two-component polyurethane formulations due to its undesirable reaction with isocyanate [1, 2]. The reactions of

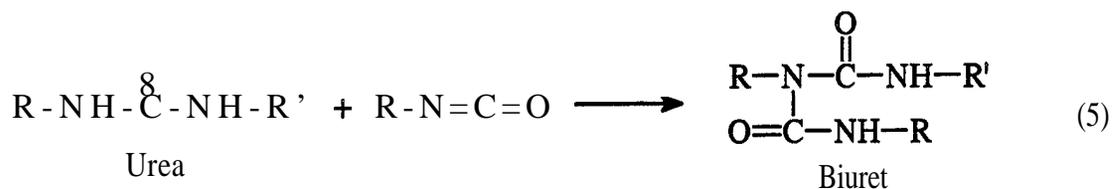
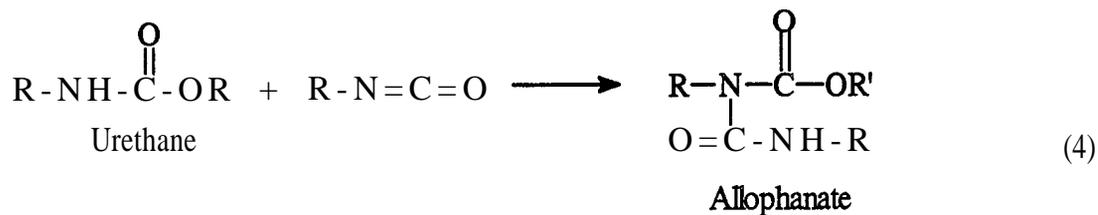
isocyanate with water are shown in reactions 2 and 3. An unstable carbamic acid is formed, resulting in generation of CO_2 and amine. CO_2 causes foaming and porosity in the film, and the amine can further react with isocyanate, resulting in the formation of urea. In short, the resulting film exhibits lower molecular weight and poor properties.



Recent developments in water-borne polyurethane technology have enabled high-performance coatings to be formulated using water-dispersible polyisocyanates and hydroxyl-functional polyurethane dispersions [3]. Hegedus et al. recently proposed a mechanism for film formation of two-component water-borne polyurethane systems, suggesting that the reaction between the isocyanate and water is sluggish compared to the reaction between isocyanate and the hydroxyl terminated polyol, therefore enabling urethane formation to take place [4, 5]. In water-borne formulations, greater excess of **NCO** is required to account for the competing reaction between isocyanate and water. Typically, water-borne formulations are indexed using excess **NCO** ranging from 1.5 to 3.5. Early efforts within our laboratory focused on formulations with **NCO** to **OH** ratios of 2.0: 1.0 and 3.5: 1.0. While these films exhibited enhanced properties compared to the **SOL** coating, they did not have the necessary **CAR** to pass the Army's requirement for nerve and blistering agents. For this reason, further investigation led to the most recent formulations with **NCO** to **OH** ratios of 5.0: 1.0. This level of indexing provided **CAR** without a significant change in coating properties.

It is important to note that, although urethane formation occurs in the water-dispersible formulations, other reactions producing amine and ureas are also prevalent (reactions 2 and 3). Additionally, because of the excess isocyanate present in these systems, additional cross-linking

reactions resulting in the formation of allophanate and biuret are also likely. Cross-linking reactions of urethanes and ureas with isocyanate are shown schematically in reactions 4 and 5.



It is a likely assumption that the cumulative effect of the side reactions occurring during film formation of the water-dispersible coatings results in films with less uniform or more heterogeneous cross-links compared to the SOL films. Dynamic mechanical analysis (DMA) was utilized to investigate the mechanical properties of the different systems in an effort to further our understanding of the structure/property relationships of these complex coatings.

3. Experimental

DMA and was performed on nonsupported coating films. These films were prepared by spraying the coating onto release paper. The films were dried for varying lengths of time at ambient temperature ($25 \pm 2^\circ \text{C}$) before separating them from the release paper. Coating film thickness varied between $160 \mu\text{m}$ – $280 \mu\text{m}$. Data were normalized according to individual sample dimensions.

DMA of the nonsupported films was performed using an Imass Inc. autovibron (automated Rheo-200 rheovibron, Toyo Instruments). The samples were evaluated between -100°C to $+150^\circ \text{C}$ at a heating rate of $2^\circ \text{C}/\text{min}$. Data were collected at 1.1 Hz.

Formulations reported in this paper were pigmented conforming to color no. 34094 (green 383) as stated in MIL-C-46168D [6], the U.S. Army specification for two-component polyurethane coatings. Tests on coated panels were conducted according to MIL-C-46168D [6].

4. Results and Discussion

A complete evaluation of the coating properties and test methodology according to MIL-C-46168D has been previously reported [6, 7]. WR 3.5 and WR 5.0 both pass **all** of the specification requirements, with the exception that WR 3.5 does not meet the necessary requirement for CAR. Both **WR** formulations exhibit vastly improved VOC compared to the conventional SOL system. Additionally, the WR formulations exhibit significantly improved impact resistance and low temperature flexibility. These properties, determined in accordance with MIL-C-46168D [6], are shown in Table 1.

Table 1. Selected Properties of Coated Panels

Sample	VOC ^a (g/liter)	CAR ^{b, c} (μ g)	Forward Impact Resistance (in-lb)	Low-Temperature Flexibility ^{c, d}
SOL	-420	-20	52, ^e 48, ^f 28 ^g	F
WR 3.5	-180	-510	160, ^e 148, ^f 148 ^g	B
WR 5.0	-180	-98	148, ^e 84, ^f 100 ^g	P

^a ML-C-46168 requirement = \leq 420 g/liter.

^b ML-C-46 168 requirement = \leq 180 μ g.

^c Data reported after **7-day** dry time.

^d **P** = pass, **B** = borderline/pass, and **F** = fail; failure is indicated by visible cracking in the coating after bending the coated panel around a cylindrical mandrel at 0° C.

^e Data reported after **10-day** dry time.

^f Data reported after **17-day** dry time.

^g Data reported after **24-day** dry time.

Figure 1 shows the storage modulus, E' , vs. temperature for the three polyurethane coatings. **WR 3.5** exhibits the highest values for E' in the rubbery plateau region of the curve. E' is a

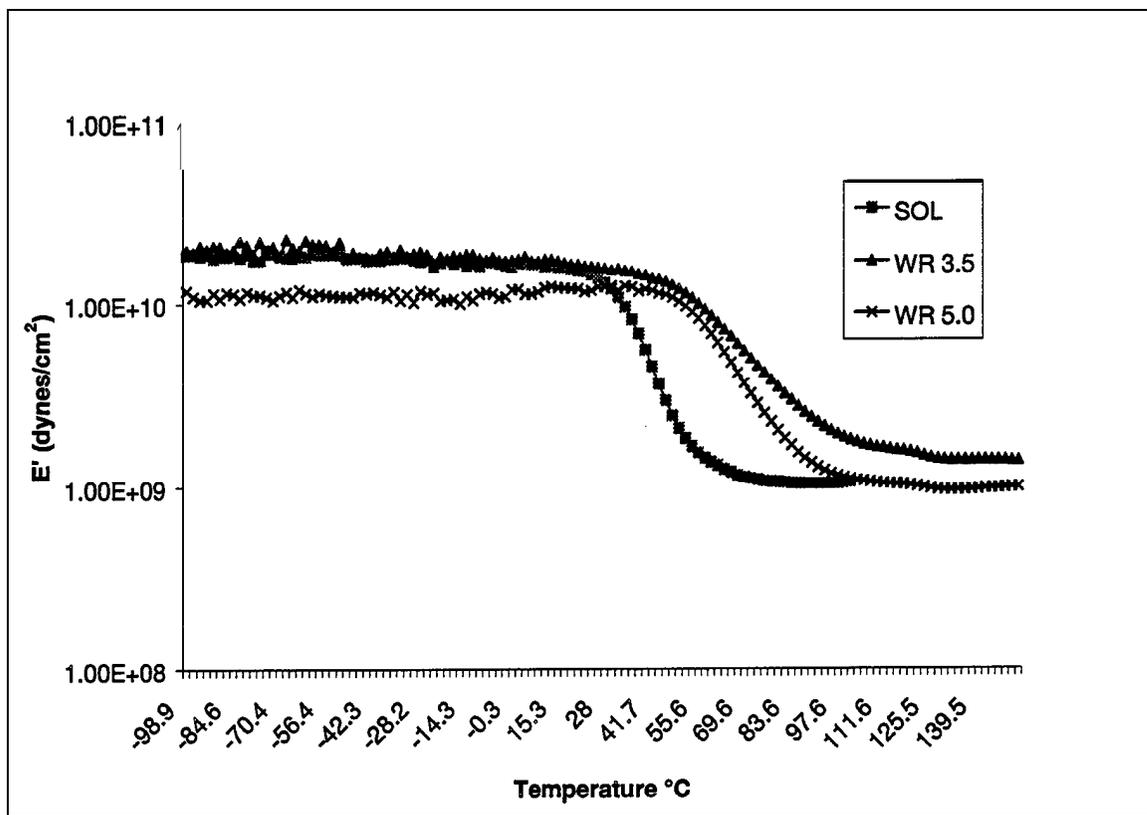


Figure 1. E' at 1.1 Hz After a 10-Day Dry Time.

measure of material stiffness and can be used to provide information regarding polymer molecular weight, degree of cure, and cross-link density [S-lo]. The difference between the storage modulus ($\Delta E'$) in the plateau regions before and after the glass transition is related to the degree of cross-link density. A smaller $\Delta E'$ is associated with greater cross-link density [8,9]. Measurements of $\Delta E'$ for the three polyurethane coatings indicate that WR 3.5 and WR 5.0 have slightly higher cross-link densities (smaller $\Delta E'$) compared to the SOL system. These experiments were performed on coatings allowed to dry for 10 days prior to evaluation.

Tan δ of the polyurethane coatings is shown in Figure 2. The SOL coating exhibits a much larger and sharper tan δ peak compared to the WR coatings. The temperature associated with the peak magnitude of tan δ is defined as the glass transition temperature (T_g). The SOL system exhibits a significantly lower T_g (43°C) compared to the WR coatings (73°C) following a

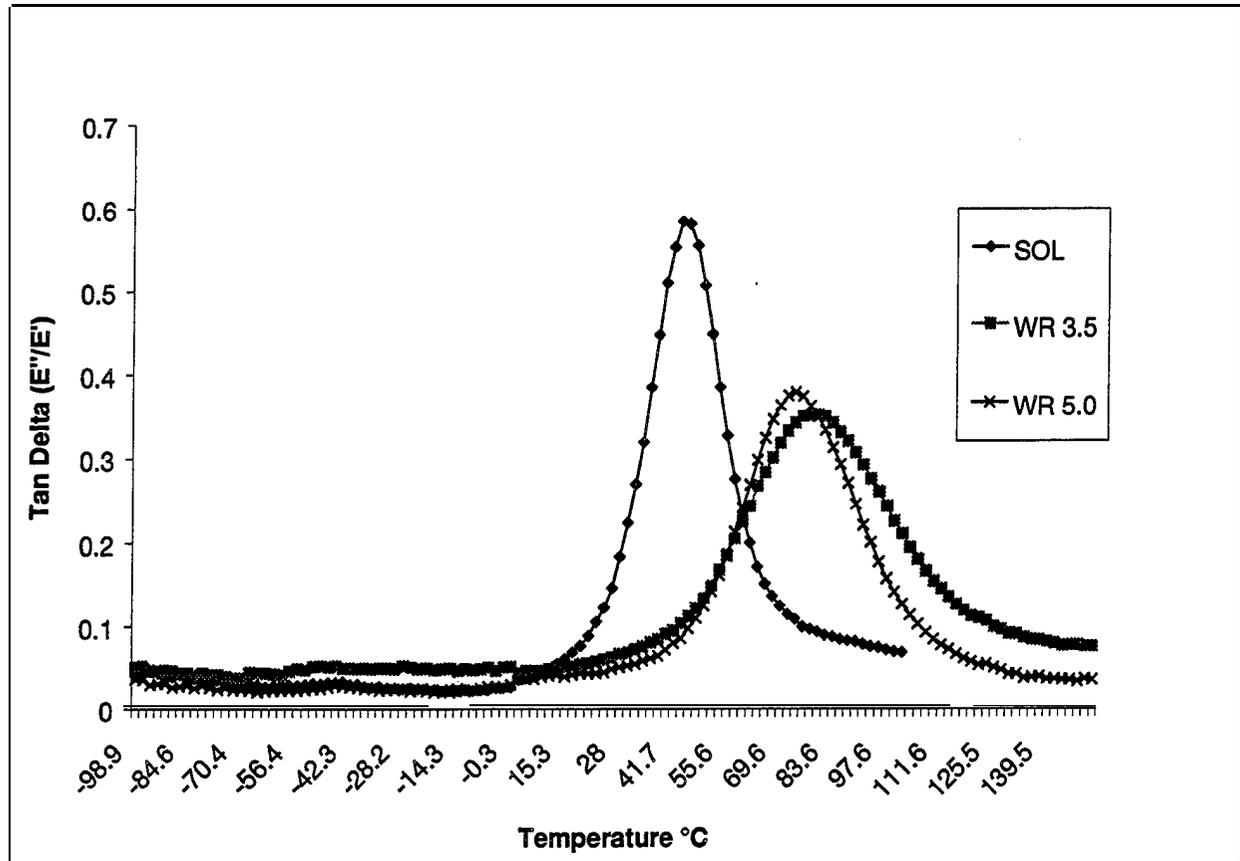


Figure 2. Tan δ at 1.1 Hz After a 10-Day Dry Time.

10-day dry time. The sharper tan δ transition observed for the SOL coating suggests more uniform cross-links compared to the WR coatings. WR 3.5 exhibits the broadest tan δ transition of the three coatings, suggesting the greatest degree of heterogeneity of cross-links. These results were also observed in the loss modulus (E'') vs. temperature curves as shown in Figure 3.

WR 3.5 exhibits a very broad E'' transition that is notably high over a broad temperature range (-98°C to $+80^{\circ}\text{C}$). E'' of WR 5.0 is also broad compared to that of the SOL coatings. High values of E'' suggest greater mobility of the polymer chains associated with dissipation of energy when the polymer is subjected to deformation [11]. Thus, coatings exhibiting a high and broad E'' transition have the ability to absorb energy associated with impact. Therefore, the DMA data helps to explain the superior impact resistance of WR 3.5 and WR 5.0 compared to

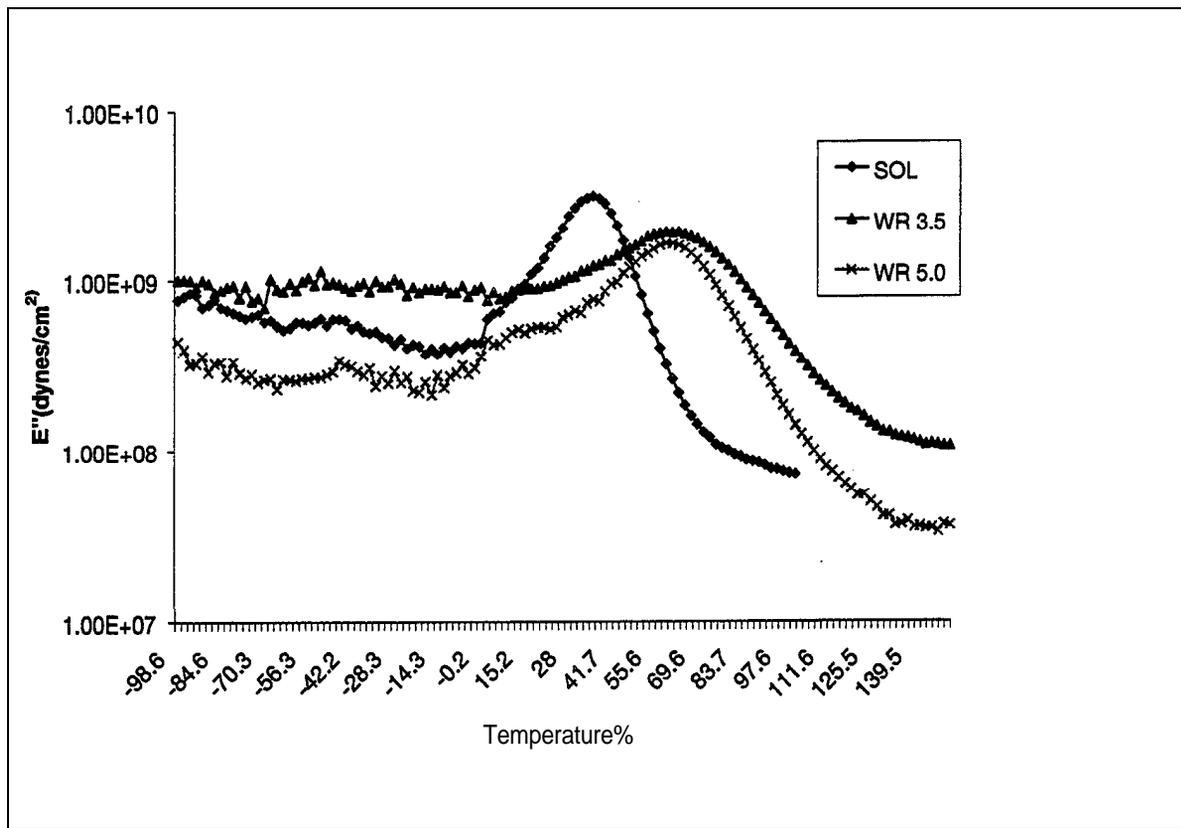


Figure 3. E'' at 1.1 Hz After a 10-Day Dry Time.

the **SOL** system. Although impact resistance was measured at ambient temperature ($25^{\circ}\text{C} \pm 2^{\circ}\text{C}$), the DMA data suggest that WR 3.5, in particular, may also exhibit superior low-temperature impact resistance. The DMA data suggest that, while broad loss transitions indicate superior mechanical properties such as impact resistance, sharper loss transitions, such as those exhibited by SOL coatings, appear to be related to superior CAR. The nonuniformity of cross-links, indicated by the broad E'' and $\tan \delta$ transitions, may introduce voids in the polymer matrix that allow greater amount of chemical agent to penetrate and become trapped in the matrix. Therefore, DMA provides some insight into the fact that differences in cross-linking as a result of film formation in the SOL and WR coatings result in optimization of different properties.

While WR 5.0 currently provides the best balance of chemical and mechanical properties, WR 3.5 exhibits the most enhanced mechanical properties. Additionally, WR 3.5 requires less isocyanate for formation of the coating, which is an important cost consideration for full-scale production. It is interesting to note that, while SOL coatings exhibit a significantly lower T_g than WR coatings, they also exhibit poorer low temperature (0°C) flexibility. At 25°C , all three polyurethane coatings exhibit acceptable flexibility as measured by bending the coated panel over a cylindrical mandrel (specified in ASTM D 522 Method B [IO]). The enhanced low-temperature flexibility of WR 3.5 and WR 5.0 is believed to be the result of the broad loss transitions shown in Figures 2 and 3.

DMA was also used to study the effect of coating dry time on mechanical properties. The dynamic mechanical properties of SOL coatings were found to be much more sensitive to coating dry time compared to the WR coatings. After all dry times, E' (in the rubbery plateau) was observed to increase with increasing temperature for SOL coatings, while the WR coatings exhibited a flat rubbery plateau throughout the experiment. This was most pronounced after 17- and 24-day dry times and less pronounced after the 6-month dry time for SOL coatings. The observation of increased E' vs. temperature is shown in Figure 4 after a 24-day dry time. These data indicated that SOL coatings were not fully cross-linked at dry times less than or equal to 24 days at ambient temperature.

The cross-link density, determined by measuring $\Delta E'$ between plateaus, is significantly increased for SOL coatings after a 6-month dry time, as shown in Figure 5. $\tan \delta$ peak magnitude for the SOL coating was also significantly reduced when allowed to dry at ambient temperature for 6 months. The increase in E' and decrease in $\tan \delta$ peak magnitude strongly suggest a notable increase in cross-link density after a 6-month dry time for the SOL coating. Although WR 5.0 also exhibited increased E' after drying for 6 months, a negligible change in $\tan \delta$ peak magnitude was observed. However, WR 5.0 exhibited a significant increase in T_g after the 6-month dry time. WR 3.5 was not evaluated after 6 months. DMA data for SOL and WR 5.0 coatings is shown in Table 2. The data suggest that, while SOL coatings may undergo

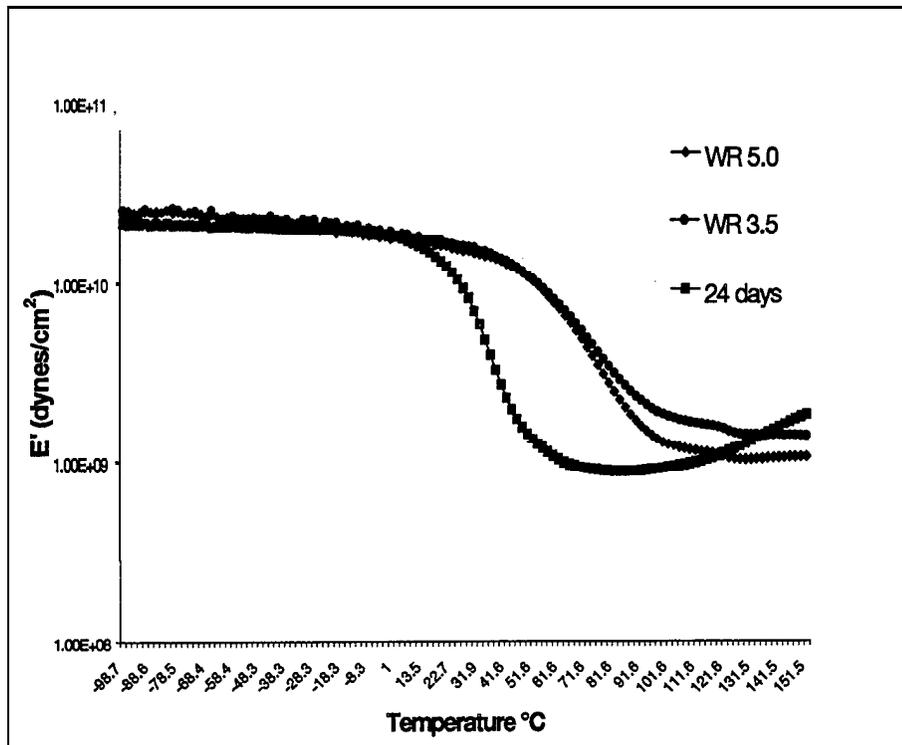


Figure 4. E' at 1.1 Hz of SOL Coatings After a 24-Day Dry Time.

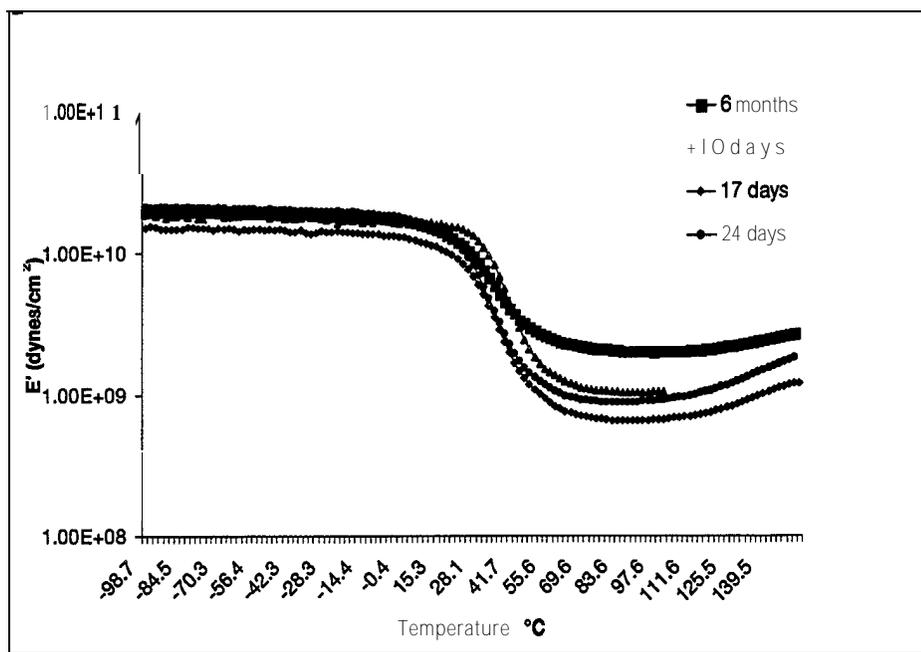


Figure 5. Effect of Dry Time on a Rubbery Plateau Modulus of SOL Coatings.

Table 2. DMA Data Measured at 1.1 Hz of SOL Coatings and WR 5.0 With Respect to Coating Dry Time

Sample	Dry Time	T _g (°C)	Tan δ Peak Magnitude	E' at 100° C (dyn/cm ²)
SOL	10 days	43	0.584	1.06 x 10 ⁹
SOL	6 months	37	0.336	2.00 x 10 ⁹
WR 5.0	10 days	73	0.379	1.13 x 10 ⁹
WR 5.0	6 months	91	0.384	1.96 x 10 ⁹

increased cross-link density with respect dry time, the changes in WR 5.0 are notably different. It is speculated that observed changes in DMA data for WR 5.0 after 6 months may indicate a densification occurring in the coating. The DMA data indicate increased cross-link density of SOL coatings after longer dry times that may actually result in reduced mechanical performance due to brittleness associated with cross-linking. Evidence of densification observed for WR 5.0 may lead to enhanced CAR without further compromise of mechanical properties. Tests on coated panels after longer dry times are necessary to confirm the DMA results. Although the CAR associated with SOL coatings is desirable, CAR is obviously irrelevant if the mechanical properties of the material cannot meet minimum durability requirements.

5. Conclusions

ARL-WMRD has successfully developed a WR chemical-agent resistant coating that meets MIL-C-46168D [6] specifications and exhibits a significant reduction of VOC compared to the currently used SOL polyurethane coating. The WR coating formulation exhibits superior mechanical properties compared to the SOL system. While WR 5.0 meets CAR requirements, WR 3.5 exhibits optimal mechanical properties that are important for long-term durability under broadly varying environmental conditions observed in service. DMA provided insight into the different chemistries associated with film formation of the SOL and water-dispersible formulations, along with the effect of dry time on the dynamic mechanical properties of the coatings. It was determined that the type of cross-linking required for superior CAR is different

for that required to optimize mechanical properties. WR 5.0 may exhibit improved CAR and, therefore, sharper loss transitions compared to WR 3.5 because of the additional cross-linking associated with the excess isocyanate present in the **WR 5.0** formulation. The more highly cross-linked film may exhibit fewer voids that can trap chemical agents. The dynamic mechanical properties of SOL coatings were shown to be much more sensitive to dry time compared to the WR coatings, which may adversely affect coated panel properties of the SOL system in service. Future work is planned to evaluate both WR and SOL coatings on test panels to determine the mechanical properties, such as impact resistance, low-temperature flexibility, and CAR, with respect to aging.

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