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EFFECTS OF SOLVENTS ON CRAZE INITIATION AND CRACK PROPAGATION IN TRANSPARENT POLYMERS

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ABSTRACT

Transparent polymers being considered for use in Army systems must be evaluated for resistance to crack and craze growth in a chemical environment. Current tests for determining craze initiation include environmental stress crazing tests based on ASTM D 790 and ASTM F 484. Results for a series of cast and biaxially stretched poly(methyl methacrylate) (PMMA) materials, as well as several formulations of polycarbonate, show a range of critical strain measurements in crazing solvents. A polyurethane-based thermoset material showed superior resistance to crazing in several of these solvents. The effect of solvents on crack propagation in transparent polymers is demonstrated by a dead weight loading apparatus and compact tension specimens based on ASTM E 399. Samples of PMMA are tested in o-xylene, water, and air. Immersion testing is also used for comparison.

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INTRODUCTION

The U.S. Army Materials Technology Laboratory (MTL) has been working in conjunction with the U.S. Army Chemical Research, Development, and Engineering Center (CRDEC) for the past four years (FY85-88) on evaluating the susceptibility of transparent polymers to failure when exposed to chemicals. This is in response to the NBC Contamination Survivability of Army Materiel Policy, AR 70-71, which requires that all Army system materiel be hardened against degradation in a chemical warfare (CW) environment. This includes the ability to be decontaminated.

Polycarbonate (PC) and poly(methyl methacrylate) (PMMA) are known to craze while under mechanical stress. This behavior is enhanced by exposure to organic solvents.^{1,2} In order to determine the level of degradation in a chemical warfare environment of currently produced transparent materials, environmental stress crazing (ESC) and crack growth studies have been underway at MTL and CRDEC.

This report summarizes much of the work that has been done on solvent-induced craze initiation and crack propagation of transparent polymers, including current studies on the effects of water sorption on crack propagation of a PMMA polymer.

EXPERIMENTATION

Solvents and Materials

Several of the solvents chosen for these studies are considered simulants for chemical agents. Simulants are chosen based on molecular weight, density, vapor pressure, and total solubility parameters similar to the actual agents, GD, HD, and VX. CRDEC has suggested several simulants in its recent report.* MTL has been using diisopropyl methyl phosphonate (DIMP) for GD, and 1,5-dichloropentane (DCP) for HD.

Much work has also been done with the universal decontaminant, DS2, which consists of 70% diethylene triamine (DETA), 28% methyl cellosolve (MECL), and 2% sodium hydroxide (NaOH) by weight. It has been determined that DETA is the most aggressive component of DS2 in environmental stress crazing studies of polycarbonate.³ DETA has also been used for some baseline comparisons between MTL and CRDEC work. MECL has been shown to be aggressive in crack propagation studies.⁴

Crack propagation studies with PC and PMMA have been done with several organic solvents.^{4,5} O-xylene was chosen as a cracking agent for PMMA based on its total solubility parameter and its tendency to crack and craze this polymer. It is also noncorrosive towards the current apparatus being used for these studies.

* LEWIS, R., LIEBMAN, S., ISAACSON, L., SARVER, E. W., and GRASSO, P. S. *Chemical Agent Simulants for Testing Transparent Materials*. Presented at the 1988 U.S. Army CRDEC Scientific Conference on Chemical Defense Research, November 15-18, 1988. To be published.

1. JACQUES, C. H. M., and WYZGOSKI, M. G. *Prediction of Environmental Stress Cracking of Polycarbonate from Solubility Considerations*. J. Appl. Pol. Sci., v. 23, 1979, p. 1153-1166.
2. BURCHILL, P. J., MATIYS, G., and STACEWICZ, R. H. *Analysis and Properties of Some Commercial Poly(methyl methacrylate)-based Materials*. J. Mat. Sci., v. 22, 1987, p. 483-487.
3. LEE, L. H., and VANSELOW, J. J. *Chemical Degradation and Stress Cracking of Polycarbonate in DS2*. U.S. Army Materials Technology Laboratory, MTL TR 87-46, September 1987.
4. HSIEH, A. J., and VANSELOW, J. J. *Environmental Stress Crazing and Cracking of Transparent Polymers*. U.S. Army Materials Technology Laboratory, MTL TR 89-12, February 1989.
5. VANSELOW, J. J., HSIEH, A. J., and BROWN, J. H. *Environmentally Induced Discontinuities in Transparent Polymers*. Presented at the 1988 U.S. Army CRDEC Scientific Conference on Chemical Defense Research, U.S. Army Materials Technology Laboratory, MTL TR 89-11, February 1989.

Samples of materials have been acquired through several sources. These sources are included in the references. Most samples were collected through distributors wishing to gain solvent resistance information.

ESC Test Development

The most widely used test, to this point, has been the static three-point-bend stress crazing test, based on the geometry of the ASTM D 790 flexure test.³ This test is appropriate since it only requires a small amount of liquid. Literature values of this test with PC and several crazing agents are available.¹ The procedure and fixture for this three-point-bend ESC test were developed at MTL and revised by CRDEC to be suitable for chemical agent testing.* CRDEC added the use of a video camera to record results so that constant monitoring in a hazardous environment is not required. CRDEC has also initiated the use of a laser for detecting craze initiation, which promises to be a significant improvement over the visual method.

MTL has also been implementing the ASTM F 484 ESC test, which uses a cantilever beam specimen, for comparison to the three-point-bend test. Results indicate a critical stress level where crazing initiates as opposed to a critical strain level. This test is recommended by MIL-P-83310 for PC, although the ASTM version specifies PMMA with appropriate conditioning requirements. MTL has been using this test for both materials.^{5,6} Again, the cantilever beam ESC test only requires a small amount of liquid and, thus, could be used for agent testing as well. The apparatus, however, is a bit more cumbersome, and larger samples are required. In addition, a material with a relatively low flexural modulus or stiffness cannot be tested with a cantilever beam specimen since the critical stress will be difficult to calculate with a deflection greater than 15 degrees.

Crack Propagation Test Development

In order to determine a material's resistance to crack propagation when exposed to solvents, MTL has been using a dead weight loading apparatus with compact tension (CT) specimens based on ASTM E 399.^{4,6} This test has been improved over conventional studies⁷ by limiting the amount of liquid. Instead of immersing the sample in solvent, the test liquid is added dropwise to only the crack area. This test also has some advantage over the ESC tests since a full set of data showing how the crack increment varies with time can be acquired from one sample. To determine critical strain from the ESC tests, several specimens must be tested. The specimen preparation for the crack growth studies, however, is more complex than the testing bars required for ESC studies. It is important to distinguish the difference between resistance to crack propagation when exposed to solvent, which is determined in this test, and resistance to craze initiation, which is determined in ESC tests. A material can be more or less resistant depending upon which test is used.⁵

* LEWIS, R., LIEBMAN, S., ISAACSON, I., SARVER, E. W., and GRASSO, P. S. *Chemical Agent Simulants for Testing Transparent Materials*. Presented at the 1988 U.S. Army CRDEC Scientific Conference on Chemical Defense Research, November 15-18, 1988. To be published.

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7. LHYMN, C., and SCHULTZ, J. M. *Environmental Testing of a Glass-Fiber Reinforced Thermoplastic*. *Polymer Eng. and Sci.*, v. 24, no. 13, September 1984.

RESULTS AND DISCUSSION

Table 1 shows critical strain measurements for several formulations of PC samples from MTL and CRDEC work performed during the past few years. This collection reflects the range of values that can occur when different formulations are used, as well as possible differences due to test locations. In the case of Lexan 9034, one critical strain value was determined at MTL, and the other at CRDEC; The references indicate where these results were originally reported.

Table 1. CRITICAL STRAIN MEASUREMENTS FOR POLYCARBONATE

Name	Critical Strain in DETA (%)	Critical Strain in DCP (%)	Critical Strain in DS2 (%)
Lexan 9034	0.24 (Ref. 6), 0.30 (Ref. 8)		
SL3000(GE)	0.34 (Ref. 3)	0.40 (Ref. 3)	0.32 (Ref. 3)
Makrolon	0.30 (Ref. 8)		0.31 (Ref. 9)
Lexan MR5	0.55 (Ref. 8)	0.87*	0.59 (Ref. 9)
PC from Swedlow			0.30 (Ref. 5), 0.27 (Ref. 5)

*LEWIS, R., LIEBMAN, S., ISAACSON, L., SARVER, E. W., and GRASSO, P. S. *Chemical Agent Simulants for Testing Transparent Materials*. Presented at the 1988 U.S. Army CRDEC Scientific Conference on Chemical Defense Research, November 15-18, 1988. To be published.

In order to compare some craze initiation studies which have been done on PMMA samples, critical stress measurements, which result from the ASTM F 484 ESC test, can be converted to critical strain measurements by dividing by the flexural modulus. Table 2 illustrates this by converting critical stress measurements made on a series of PMMA materials⁵ to critical strain values. These PMMA samples, under the trade name Acrivue, are a series of cast (320, 350AC, 351) and biaxially stretched (350S, 352S) acrylics from Swedlow, Inc. Their formulations differ in molecular weight, as well as crosslinking agents, not further described by the manufacturer. In Table 3, the critical strain values for the Acrivue materials are similarly obtained and compared to critical strain measurements made at CRDEC with the three-point-bend apparatus and the laser equipment.* The Polycast samples were acquired from LORAL Systems Division. It should be noted that the variation in critical strain measurements with different formulations of PMMA can be as large as, if not larger than, the variation in those of PC. In addition, the Polycast 76 material appears to have the best resistance to solvent-induced craze initiation of the cast acrylics.

CRDEC also tested GAC 590, a polyurethane-based thermoset from LORAL Systems Division, and found the critical strain measurements to be greater than 3.1% in both DCP and DIMP.* This was the best result of all transparent materials tested.

*LEWIS, R., LIEBMAN, S., ISAACSON, L., SARVER, E. W., and GRASSO, P. S. *Chemical Agent Simulants for Testing Transparent Materials*. Presented at the 1988 U.S. Army CRDEC Scientific Conference on Chemical Defense Research, November 15-18, 1988. To be published.

8. VANSELOW, J. J., HENDERSON, V., and STEVENS, J. *Crazing and Cracking in Transparent Materials*. Proceedings of the 1987 U.S. Army CRDEC Scientific Conference on Chemical Defense Research, CRDEC SP 88013, April 1988, p. 1301.

9. VANSELOW, J. J., and LEE, L. H. *Stress Cracking of Thermoplastics*. Proceedings of the 1986 U.S. Army CRDEC Scientific Conference on Chemical Defense Research, CRDEC SP 87008, June 1987, p. 581.

Table 2. CALCULATED CRITICAL STRAINS FOR PMMA SAMPLES TESTED IN DS2 VIA ASTM F 484

Name	Critical Stress (psi)	Flexural Modulus (psi X 10 ⁻³)	Critical Strain (%)
Acrivue 320	1300	450	0.29
Acrivue 350AC	1400	470	0.30
Acrivue 351	2900	460	0.64
Acrivue 350S	3200	450	0.70
Acrivue 352S	5500	450	1.22

Table 3. CRITICAL STRAIN MEASUREMENTS FOR SEVERAL PMMA FORMULATIONS

Material	Critical Strain in DCP (%)	Critical Strain in DIMP (%)
Acrivue 320	0.13	0.33
Acrivue 350AC	0.28	0.43
Acrivue 351	0.43	0.65
Polycast 76*	0.60	0.87
Acrivue 350S	0.78	1.22
Acrivue 352S	1.22	1.33
Poly 76S (LORAL)*	0.87	1.50

*LEWIS, R., LIEBMAN, S., ISAACSON, L., SARVER, E. W., and GRASSO, P. S. *Chemical Agent Simulants for Testing Transparent Materials*. Presented at the 1988 U.S. Army CRDEC Scientific Conference on Chemical Defense Research, November 15-18, 1988. To be published.

Crack Propagation and Water Sorption Studies of PMMA

Water sorption¹⁰ has been shown to significantly affect ESC measurements of PMMA materials. The samples tested at MTL were in a hood and contained less than 0.1% by weight of water. Table 4 shows the amount of water absorbed by these acrylic materials in 30 days.

Table 4. IMMERSION RESULTS - WEIGHT PERCENT UPTAKE IN (24 HOURS) 30 DAYS

Material	Water	o-Xylene
Acrivue 320	(0.19) 0.91	(-0.04) -0.48*
Acrivue 350AC	(0.27) 1.49	(0.02) -0.13
Acrivue 351	(0.25) 0.90	(0.02) -0.11
Acrivue 350S	(0.45) 1.46	(0.09) -0.02
Acrivue 352S	(0.15) 0.99	(0.28) -0.67*

*Surface crazing

Solubility parameters, δ , can be used to predict a region of crazing based on the difference between the total solubility parameter of the solvent and that of the polymer.¹ The total solubility parameter of water is 23.4 (cal/cm³)^{1/2}.¹¹ PMMA or acrylic materials have a range of total solubility parameters of 8.9 to 12.7 (cal/cm³)^{1/2}.¹¹ The large difference between the δ of water and that of PMMA suggests that crazing and cracking would not occur. The δ of o-xylene is 8.8 (cal/cm³)^{1/2}.¹¹ The closeness of the δ 's of o-xylene and PMMA suggest that crazing and cracking in PMMA will occur when exposed to this solvent.

10. BURCHILL, P. J., and STACEWICZ, R. H. *Effect of Water on the Crazing of a Crosslinked Poly(methyl methacrylate)*. *J. Mat. Sci. Let.*, v. 1, 1982, p. 448-450.

11. BRANDUP, J., and IMMERGUT, E. H. *Polymer Handbook*. 2nd ed., John Wiley and Sons, New York, 1975.

In Figure 1, the effect of o-xylene on the craze/crack propagation in a PMMA sample is illustrated. The designation craze/crack is used since this test cannot distinguish between the two phenomena. The three samples of Acrivue 320 are tested at initial stress intensity factors (K_I) of 0.60 to 0.68 MPa·m^{1/2} which correspond to 45 to 50% of its fracture toughness.⁵ Although water is known to plasticize PMMA and, thus, reduce its resistance to solvent-induced stress crazing,¹⁰ when used independently in this craze/crack propagation test, it does not act as a crazing or cracking agent. A follow-up study is suggested, which tests a moist sample of PMMA, which is subsequently exposed to a crazing agent such as o-xylene.

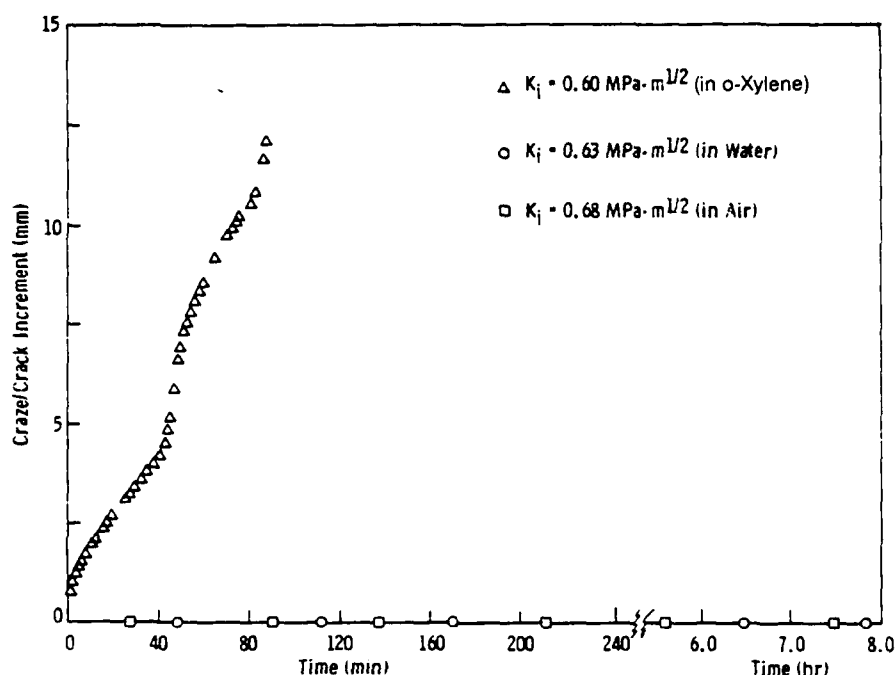


Figure 1. Craze/crack growth curves for Acrivue 320 in o-xylene, water, and air.

Immersion results show a significant weight gain in water of PMMA samples, however, no surface crazing occurs. In o-xylene, weight loss is demonstrated in four out of five of the samples. This indicates more aggressive behavior than in water due to dissolution. The fifth sample, Acrivue 352S, shows less weight gain than in water, but the surface crazes which result indicate aggressive behavior on the part of o-xylene. Acrivue 320 was most affected by the solvent, since significant surface crazing was seen, as well as a higher weight loss. Previous craze/crack propagation studies verify the higher susceptibility of Acrivue 320.⁵ The crack propagation studies demonstrate the aggressiveness of o-xylene in a much shorter time frame than that required for detection of surface crazing.

It should be noted that the range of values for solubility parameters for PMMA and PC [9.5-10.5 (cal/cm³)^{1/2}],¹¹ as opposed to a single value, is due to the large number of formulations in which these polymers exist. This is illustrated by the variation in ESC results shown in Tables 1 through 3.

SUMMARY

The results which have been summarized here identify some of the concerns that must be recognized when conducting different types of tests for chemical resistance. This includes careful identification of nominally similar samples, as in the case of PMMA and PC, and also includes specific identification of the property desired, such as resistance to craze initiation or to crack propagation. Future studies are necessary to continue to assess newly developed transparent polymers being considered for use in Army systems. In addition, modifications should be made to the crack propagation apparatus to prepare it for chemical agent studies.

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