

MTL TR 89-12

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ENVIRONMENTAL STRESS CRAZING AND CRACKING OF TRANSPARENT POLYMERS

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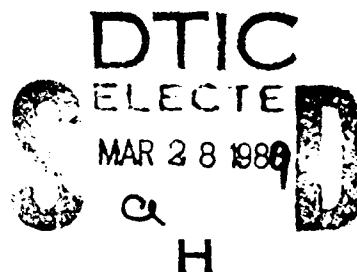
ALEX J. HSIEH and JANICE J. VANSELOW
POLYMER RESEARCH BRANCH

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ABSTRACT

The resistance to crack and craze growth in polycarbonate and poly(methyl methacrylate) is evaluated with several surface active solvents including a component of the universal chemical warfare decontaminant, DS2. A static dead weight loading apparatus is used for experimentation, and linear elastic fracture mechanics (LEFM) is used to interpret craze initiation and crack propagation via compact tension specimens. Results reflect relationships based on solubility parameters of the solvents and the polymers. Various structural effects due to environmentally induced degradation of these amorphous thermoplastics are illustrated.

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INTRODUCTION

Polymeric matrices and polymer-based composites are finding increased use in military applications. Designing for improved damage tolerance and durability of structures has become an important issue over the last few years. A disadvantage of many polymeric materials is the tendency to craze or crack when exposed to solvents, including those in a chemical warfare (CW) environment. When this failure is enhanced by a simultaneous stress, either mechanically induced or from a residual stress due to manufacturing, the behavior is called environmental stress crazing or cracking (ESC). Aggressive surface active agents can cause ductile materials to fracture brittly.

The purpose of this work is to assess the resistance of the amorphous polymers, polycarbonate (PC) and poly(methyl methacrylate) (PMMA), to craze initiation and crack propagation in various solvents, including a component of the decontaminant DS2. This includes the investigation of environmentally-induced failure mechanisms.

BACKGROUND

Polycarbonate, known as a ductile thermoplastic, has been widely used for transparent armor. Despite its good mechanical toughness, PC is susceptible to crazing by many solvents. One of these solvents includes a solution of particular military interest, DS2, which is a chemical warfare (CW) agent decontaminant. DS2 is composed of 70% diethylene triamine (DETA), 28% methyl cellosolve (MECL), and 2% NaOH by weight. In Lee's work, DETA was shown to be the most active component in DS2 (Figure 1) in causing the most rapid PC weight loss.¹ The evidence suggests that chain cleavage, as a result of the nucleophilic attack of DETA on the carbonate group of PC, was responsible for the loss of physical integrity.

The critical strain for the resistance to surface crazing of PC has been shown (Table 1) to be higher in MECL than in DETA,¹ confirming that PC is more resistant to MECL than to DETA in surface degradation. The subsequent question is whether the resistance to crack propagation of PC in MECL follows the same pattern as the resistance to craze initiation.

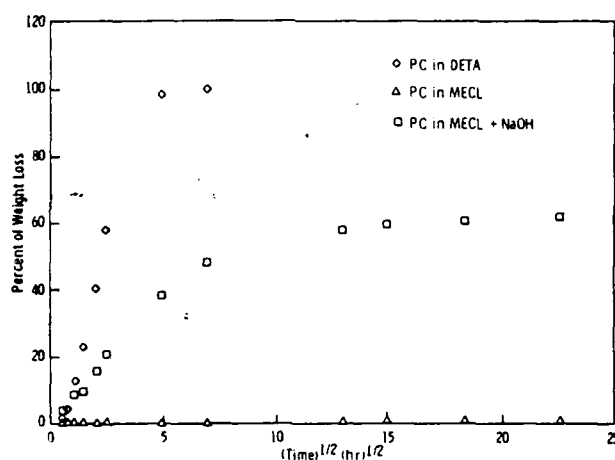


Figure 1. Immersion test results of PC in DETA, MECL, MECL + NaOH.

1. 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.

Table 1. CRAZING RESISTANCE OF POLYCARBONATE

Chemical	Critical Strain (%)
DS2	0.32
DETA	0.34
MECL	0.68

EXPERIMENTAL

The polycarbonate (PC) used in this study was Lexan MR5 from GE Plastics, and the poly(methyl methacrylate) was Plexiglas-G from Rohm and Haas.

The craze/crack growth studies were performed using a static dead-weight loading apparatus.² Compact tension (CT) specimens, following ASTM E399, were used.^{2,3} The specimens were initially notched with a slotter, and the notch tip was sharpened with a razor blade. In most of the tests, the specimen was immersed in the solvent. Later, as a means of reducing the solvent volume, the specimen was subjected to the droplets of solvent pumped through a liquid chromatography pump. Craze/crack length measurements were made as a function of time with either a traveling microscope or a camera.

RESULTS

PC in MECL

Although PC withstands crazing in MECL, it showed poor resistance to crack propagation in this solvent at loads well below fracture. Sample #PC1 broke into two pieces in one hour. The fractured surfaces showed irregular and macroscopic roughness with a hazy white appearance. Due to the swelling and possible yielding at the notch while loading, the two broken pieces did not fit back together. Sample #PC2, which was initially loaded at a lower load, still fractured at a moderate propagation rate with less roughness and less swelling. However, the hazy white appearance near the notch tip was still clearly seen. This is the result of extensive crazing which has concentrated on the surface due to the solvent.

The poor crack resistance of PC in MECL can be attributed to solvent stress cracking. It has been well established that organic agents having solubility parameters, δ , similar to that of a polymer, can act either as solvents or as cracking agents.⁴ As shown in Table 2, the solubility parameter of PC is 9.5 to 10.5 (cal/cm³)^{1/2}, which is similar to that of MECL, 10.5 (cal/cm³)^{1/2}.^{5,6} As a result, MECL can be regarded as a good cracking agent for PC under stress, even though it does not cause a weight loss of PC as previously indicated (Figure 1).

2. VANSELOW, J. J., and HSIEH, A. J. *Evaluation of Transparent Polymers for Chemically Hardened Army Systems*. U. S. Army Materials Technology Laboratory, MTL TR 88-24, July 1988.
3. ASTM, *Standard Method of Test for Plane-Strain Fracture Toughness of Metallic Materials*. ASTM E399-83.
4. KAMBOUR, R. P. *A review of Crazing and Fracture in Thermoplastics*. General Electric Report No. 72CRD285, October 1972.
5. BRANDRUP, J. *Polymer Handbook*, 2nd edition, John Wiley & Sons, New York, 1975, p. IV 340 - IV 354.
6. JACQUES, H. M., and WYZGOSKI, M. G. *Prediction of Environmental Stress Cracking of Polycarbonate from Solubility Consideration*. *Journal of Applied Polymer Science*, v. 23, 1979, p. 1153-1166.

Table 2. SOLUBILITY PARAMETERS OF SOLVENTS AND POLYMERS
(References 5 and 6)

	δ (cal/cm ³) ^{1/2}	H Bonding Tendency
MECL	10.5	Moderate
Ethanol	13.0	Strong
Hexane	7.3	Poor
PC	9.5 - 10.5	
PMMA	8.9 - 12.7	

PC in Ethanol

Ethanol, with a solubility parameter of 13.0 (cal/cm³)^{1/2}, is significantly different from the δ of PC so that ethanol is considered a crazing agent. Figure 2 shows a smaller growth rate in ethanol than in MECL, even at the same load level. This is as expected. The results of craze growth profiles in ethanol as a function of load are shown in Figure 3. The concept of linear elastic fracture mechanics (see Appendix) is utilized to interpret the data. The higher the initial stress intensity factor, (K_i), the faster the initial growth rate. A plot of craze increment versus time on the logarithmic basis is shown in Figure 4.

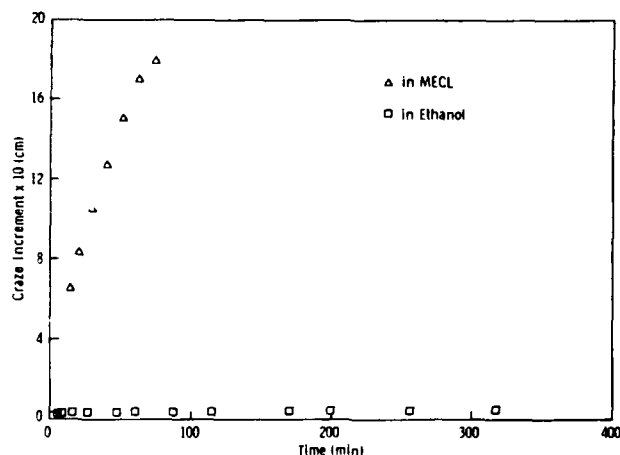


Figure 2. Comparative data for craze growth for polycarbonate in MECL and in ethanol.

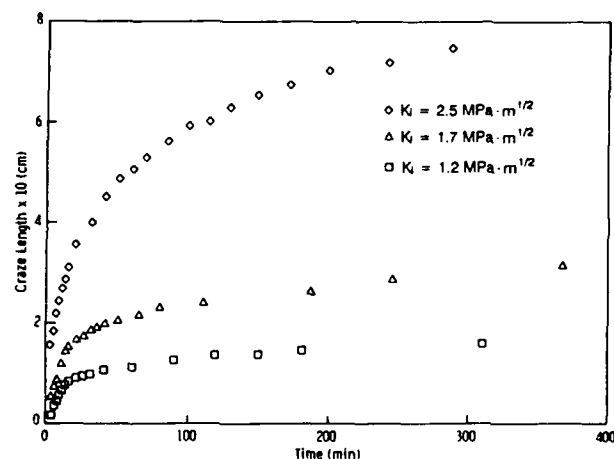


Figure 3. Craze growth curves for PC tested in ethanol at various K_i levels.

The latter portion of the curves are linear with the average slope determined as 0.24. The lines in Figure 4 were extrapolated to determine the intercepts. Plotting the intercepts of the curves in Figure 4 versus K_i on a logarithmic scale results in a straight line as shown in Figure 5. The slope, which is a measure of the dependence of the craze increment on the initial stress intensity factor, was calculated as 1.8. This is similar to 2 in the relation derived by Williams and Marshall⁷ for relaxation controlled craze growth behavior:

$$(\text{craze displacement}) \propto k_i^2 (1-m)^2 (t)^{2m}$$

where t is time, and m is the relaxation parameter. The latter is defined as a measure of the time dependent response of the yield stress. Their approach for environmental stress

7. WILLIAMS J. G., and MARSHALL G. P. *Environmental Crack and Craze Growth Phenomena in Polymers*. Proc. R. Soc., London, A342, 1975, p. 55-77.

crazing/cracking was based upon crack opening displacement in conjunction with simple flow analysis which describes the movement of the liquid environment within the craze. The value of m , equal to half of the average slope in Figure 4, is determined as 0.12 in this study. Williams and Marshall⁷ reported m as 0.04 and 0.11 for PC and PMMA in air at 20°C respectively.

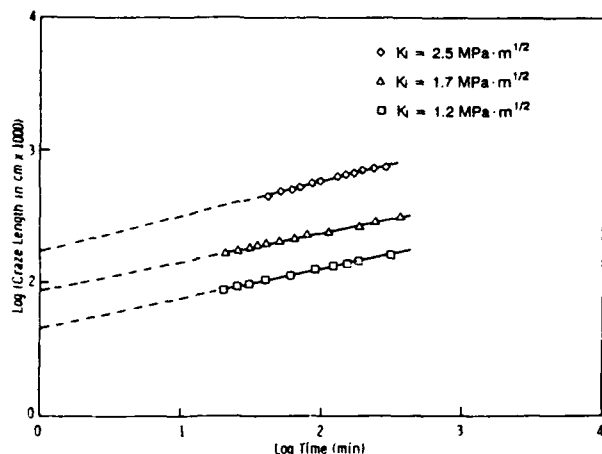


Figure 4. Linear regression of the curves from Figure 3 on a logarithmic plot.

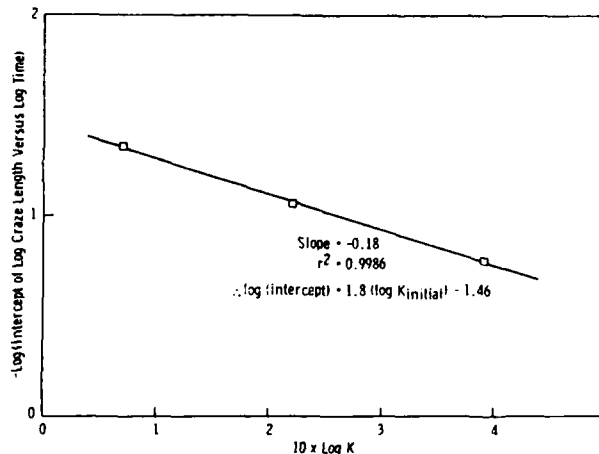


Figure 5. Determination of the dependence of the craze displacement on K_i for PC tested in ethanol.

For all PC specimens tested in ethanol and MECL, an assembly of crazes was seen progressing outward from the initial notch tip. At the beginning, many of these crazes advanced at various angles from the direction of the initial notch, but eventually, after some distance, they tended to grow parallel to the initial notch. The presence of multiple crazing might blunt the notch tip and subsequently influence the craze/crack propagation. For example, the craze growth of PC in ethanol eventually stopped after some period of time without any fracture.

The influence of a sustained load on craze initiation was further studied by choosing a range of lower K_i values. The craze increment profiles are shown in Figure 6. As found in Figure 3, the initial craze growth rate increases as K_i increases. For $K_i < 0.36 \text{ MPa}\cdot\text{m}^{1/2}$ (where m designates meters), there was no obvious craze growth seen. However, for $K_i > 0.64 \text{ MPa}\cdot\text{m}^{1/2}$, multiple crazing was seen immediately after ethanol reached the notch tip of the stressed PC specimens. Therefore, a critical value of the initial stress intensity factor, below which no crazing occurs, will be within the range of $0.36 < K_i < 0.64 \text{ MPa}\cdot\text{m}^{1/2}$.

PC in Hexane

The effect of the solvent on craze initiation and growth was further studied with hexane, which has a δ value of $7.3 \text{ (cal/cm}^3)^{1/2}$. This has about the same $\Delta \delta$ as that of ethanol with respect to PC. With the same $\Delta \delta$, the same rate of crazing would be expected. However, the results in Figures 6 and 7 showed faster rates in ethanol than those in hexane. The functional groups in these solvents result in different tendencies. Ethanol has a much stronger tendency towards hydrogen bonding which results in a greater tendency to craze PC.

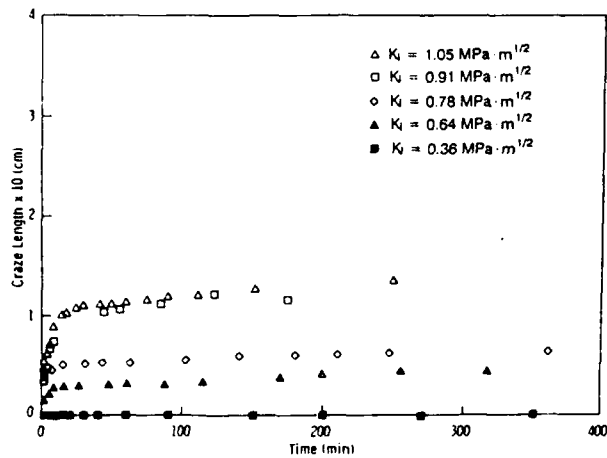


Figure 6. Variation of craze length with time for PC tested in ethanol at various K_I levels.

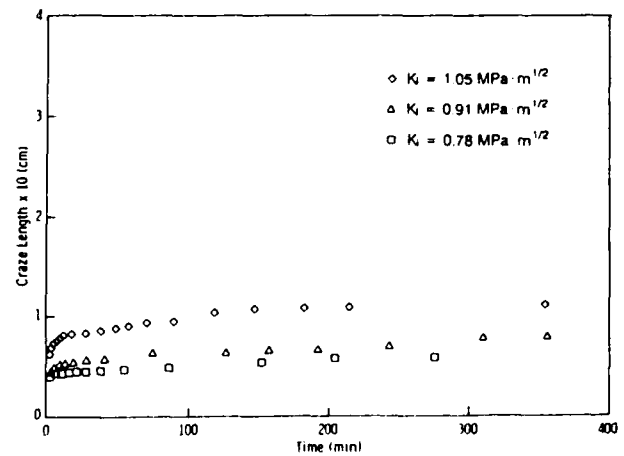


Figure 7. Variation of craze in length with time for PC tested in hexane at various K_I levels.

In addition to the results of the craze increment profiles, the following observations were made:

1. The sizes of the deformation zone ahead of the crack tip in hexane were smaller than those in ethanol under the same load level (Figure 8).
2. The critical load for craze initiation in hexane was higher than that in ethanol.
3. Multiple crazing was still seen, but to a lesser extent, in the crazing of hexane.

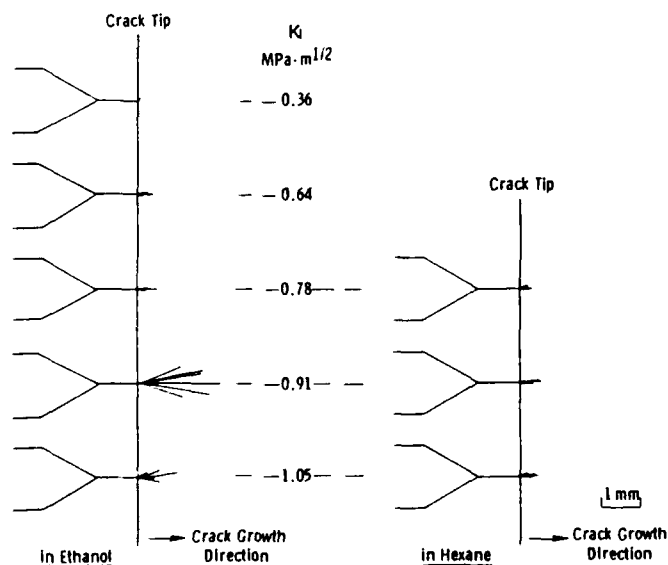


Figure 8. Sketch of the craze profiles near the notch chip for PC tested in ethanol and in hexane.

PMMA in Ethanol

The craze growth rate of PMMA in ethanol (Figure 9) was much faster than that of PC at about the same percent K_I value relative to its critical load of fracture in air, K_{Ic} . K_{Ic} for PC was determined as $2.7 \text{ MPa}\cdot\text{m}^{1/2}$, and that for PMMA as $1.1 \text{ MPa}\cdot\text{m}^{1/2}$. The three curves in Figure 9 of PC craze growth profiles are identical to those in Figure 3. PMMA, at a K_I of 65% of its K_{Ic} , and PC at a K_I of 93% of its K_{Ic} , had about the same initial growth rate, but the rate of growth of PC fell continuously to zero, while the PMMA specimen eventually broke into two pieces. This may be due to the presence of multiple crazing in PC, which blunts the crack tip and slows down the craze growth.

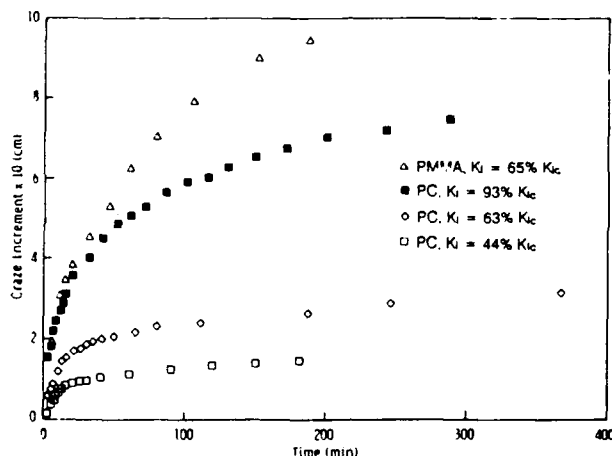


Figure 9. Comparative data for craze growth for PC in PMMA in ethanol.

CONCLUSIONS

By using selected surface active agents with PC, solubility parameters can be used to attempt to predict the initiation and propagation of crazes and cracks. The role of the different functional groups of the solvents must be considered. The results of this work have shown that the higher the initial load, the faster the craze/crack propagation rate in both PC and PMMA. There was no obvious craze growth seen for PC in either ethanol or hexane at stresses below some critical value. At stresses above that critical value, the rate of craze initiation and propagation of PC in ethanol is higher than that in hexane. This suggests that hydrogen bonding plays a significant role in solvent crazing. The presence of multiple crazing, which causes blunting at the crack tip, is responsible for the slower crack growth of PC compared to that of PMMA in ethanol. Continued work in this area should include the use of solvents with solubility parameters of greater variation from the polymer than those selected here.

APPENDIX

In LEFM, a single parameter, K , the stress intensity factor, is used to relate the intensity of the elastic stress-strain field near the crack tip to the loading and geometry of the body. Crack extension will occur when K reaches a critical value, K_{Ic} . The K_{Ic} , named plane strain fracture toughness, is expected to be a material parameter, provided that the plastic zone size adjacent to the crack tip is small compared to the relevant specimen dimensions of crack length, thickness, and width.

The plane strain fracture toughness can be determined according to the relationship between the maximum load and the geometry of the compact tension specimens given in ASTM E399:

$$K_{Ic} = P.(Bw^{1/2}) [29.6(a/w)^{1/2} - 185.5(a/w)^{3/2} + 655.7(a/w)^{5/2} - 1017(a/w)^{7/2} + 638.7(a/w)^{9/2}]$$

where P is the maximum applied load (kN), B is the thickness of the specimen (cm), a is the crack length (cm), w is the width of the specimen (cm), and K_{Ic} is in MPa ($m^{1/2}$).

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2	ATTN: SLCMT-TML
2	Authors

U.S. Army Materials Technology Laboratory
Watertown, Massachusetts 02172-0001
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Alex J. Hsieh and Janice J. Vanselow

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