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Environmental stress cracking resistance. Behaviour of polycarbonate in different chemicals by determination of the time-dependence of stress at constant strains

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Abstract

The mechanism of Environmental Stress Cracking (ESC) of amorphous polycarbonate in isopropyl alcohol, ethylene glycol monomethyl ether and methanol were studied. The three point bending method and the variation of stress with the immersion time at constant strain were used to evaluate the chemical attack. The results show that the variation of the stress with the immersion time can be a very good indicator of how a polymer will behave in specified environment and strain conditions after exposure to various chemicals. The resistance of the polymer was monitored as a function of time at different given strain-values and different initial stresses. The time to ESC was defined as the point where the resistance of a polymer immersed in a given solvent deviates from the equivalent measure of the polymer sample in air. The results showed that the critical strain of polycarbonate is 1.21±0.06% when immersed in isopropyl alcohol, 1.74±0.12% when immersed in methanol, and 0.29% when immersed in ethylene glycol monomethyl ether. (The 1.74% value of methanol is lower than the value given in the literature). The results show that all three liquids give massive reductions in the strength of the polycarbonate. This is especially true in the polycarbonate-ethylene glycol monomethyl ether combination, where the specimens showed complete failure after about 22 s. The images obtained with optical microscopy showed that the cracking propagates from the edge to the center of the specimen and reaches typically about 5 μm below the surface. The diffusion rates of the different solvent molecules in the polymer were further determined by absorption measurements in an attempt to establish a correlation with the environmental stress cracking resistance. The results showed that methanol has the fastest diffusion rate, followed by ethylene glycol monomethyl ether. Polycarbonate immersed in isopropyl alcohol showed very limited weight gain within the given time of monitoring.

Keywords: Polycarbonate; Environmental stress cracking resistance; Constant strain; Time-dependence of stress

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1. Introduction

Failure in polymeric materials is often caused by environmental stress cracking, ESC, which accordingly has enormous industrial and economic implications. The mechanism of ESC has therefore recently attracted significant scientific and applied interests [1–5]. It is believed that the basic mechanism of ESC is related to main-chain motion that leads to the formation of small (<30 nm) voids [6], however, the details of craze initiation are still rather unclear. The nm-sized voids coalesce into planer bands that finally become crazes. The mechanical failure of polymers is highly sensitive to environment agents as well as the stressed condition. As a result, polymers fracture often at stress-values considerably lower than the normal yield stress, with significant reduction in available service time as a consequence.

It is generally accepted that ESC occurs due to a combination of several effects. First, molecules diffuse from the environment into the polymer material and cause plasticization of the near-surface layer [7–12]. The plasticization leads to easier craze formation [8,13,14]. Once formed, these crazes grow due to passage of environmental molecules along the craze, leading to further plasticization of the craze tip and weakening of the craze fibrils [15]. The growth is in particular

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enhanced when the polymer is subjected to stress, and can cause unexpected and often catastrophic brittle failure.

It has experimentally been found that crazing occurs fairly rapidly where the strain is greater than some characteristic critical value, $\varepsilon_{\text{crit.}}$, while crazing remains effectively absent below this value, even for very long testing times. The value of $\varepsilon_{\text{crit.}}$ can therefore be used as a convenient measure of the hostility of the environment [17]. This critical strain concept is widely used in conjunction with bend tests methods, with the attempt to specify and to compare the severity of ESC for a range of environments [2,13,15,16].

The reason that the critical strain concept holds for the case of bend tests is that under these conditions of stress relaxation, the applied stress, which is the driving force for crazing, will decay relatively quickly. This is particularly true where swelling and plasticization occur as the environment diffuses into the surface and as crazes open up. Under these situations, a critical strain criterion is a useful approximation [13].

Environmental stress cracking in polymers involves both solubility and absorption rate phenomena, as well as the state of stresses in the polymer material. Previous work has established that the value of $\varepsilon_{\text{crit.}}$ decreases as the solubility parameters of the polymer and the environment approach one another, i.e. as they become more compatible [9,12,18,19]. The degree of plasticization depends primarily on degree of swelling and the latter depends primarily on the solubility parameters of polymer and swelling agent [20,21].

The critical strain values are normally determined by applying different strains to strips of a specimen immersed in a given chemical, leaving the strips for a certain time in the test equipment and then examining them to establish the strain-limit of craze formation. Common strain tools are a three-point bend tester or an elliptical form.

Environmental stress cracking can also be studied by measuring creep or creep rupture [22–24]. Creep measurements are useful for indirect determination of compatibility with solvents or plasticizers. The time to rupture determines the influence of microcrazes, crazes, or cracks resulting from ESC.

An important fact is that ESC defined as an essentially physical process that does not involve any chemical degradation [17].

The data in the literature lack the studies on the polymer resistance to bending with time to environmental stress cracking at constant strain. Therefore, in the present study, we developed the three-point bending method not only to measure the maximum strain at the mid-point of the specimen, but also to determine the required initial stress to this constant strain. Simultaneously, the variation of the polymer strength is monitored as a function of the time. The maximum strain at the mid-point of the specimen does not change with the time during the test and remains constant because the deflection at the mid-point is constant. By this method, we studied the behaviour of polymer resistance to ESC with time and in the same time to determine the critical strain needed for the initiation of crazes. The critical strain for crazing may be essentially determined in three ways: by extrapolating the craze width versus applied strain such that craze width = 0, by applying the craze width to three point bending theory to yield a theoretical value as shown below in 2.2 or by an iterative method, reducing the applied strain until only one craze is found [6,15,18,25]. General concept, that the tests monitoring the change in strength or stress would be a good indicator of chemical compatibility. We have studied ESC with respect to a few well-defined chemicals.

2. Experimental

2.1. Materials and chemicals

The material used in the present study is Polycarbonate-Lexan, type No. 121R. The multi-purpose test specimens were made by injection moulding into a size and form complying the ISO-3167 standard as shown in Table 1. The multipurpose test specimen has the basic shape of a 4 mm thick tensile ‘dog bone’, 150 mm long, with the center section 10 mm wide and 80 mm long.

Residual stress tests on polycarbonate specimens carried out by using a simple polarscope (the tests are nondestructive, requiring no chemicals or layer removing and fast results). The results show that the annealed specimens (heating at 120 °C for 1 h then slow cooling) have much smaller residual stress compared with the unannealed specimens.

Solvents used in the study were standard laboratory reagents: isopropyl alcohol ($CH_3CHOHCH_3$), ethylene glycol monomethyl ether ($CH_3OC_2H_4OH$), and methanol (CH_4O).

2.2. Test equipment

A prototype test equipment following ISO 178:1993 standard was developed to estimate the variation of ESC of polycarbonate in a chemical environment by

Table 1
Dimensions of the test specimen and apparatus

Length	ℓ	80±2.0 mm
Width	d	10±0.2 mm
Thickness	t	4±0.2 mm
Striking edge radius	R_1	5±0.1 mm
Supports shall radius	R_2	5±0.2 mm

continuously monitoring the stress value versus time. The schematic diagram of the test device is illustrated in Fig. 1. The striking edge radius is $R_1 = 5.0 \pm 0.1$ mm. The radius of the supports is $R_2 = 5.0 \pm 0.2$ mm (Table 1). (This is for the test specimens with thickness $t > 3$ mm. For test specimens with thickness $t \leqslant 3$ mm, $R_2 = 2.0 \pm 0.2$ mm is used.)

The specimen is bent using a three-point bending jig mounted in a chemical environment chamber kept at 23±1 °C. After 1 min, which is enough time to stabilize the loading condition, the chemical is rapidly introduced into the chamber under the stressed specimen. The variation of the stress was recorded using a sensitive load cell (0–100 Newton) connected to multimeter that is recorded every 2 s for 72 h.

The flexural stress value σ on the bottom surface of the specimen is determined using the bending equation [1]:

$$\sigma = 3FL/2dt^2 \tag{1}$$

where F is the applied force measured in Newtons, L is the span length measured in mm, d is the width in mm of the test specimen, and t is the thickness in mm of the test specimen. The flexural stress σ is expressed in Mega Pascals.

When the stressed polycarbonate is exposed to a chemical environmental, the polymer resistance will vary with time. Since microcrazes, crazes, or cracks weaken the chemically exposed polymer, it will tend to have both a shorter time to rupture and a lower stress value, compared to the values of a control specimen measured in dry atmosphere.

The maximum flexural strain value, $\varepsilon_{\max}$, at the mid-point of the tensile surface of a test specimen is determine by [6,16]:

$$\varepsilon_{\max.} = 6\delta t/L^2 \tag{2}$$

where δ is the deflection in mm, and measured by using an ±0.02 mm accurate electronic digital caliper. The critical strain $\varepsilon_{\text{crit.}}$ calculate by [6,18,25]:

$$\varepsilon_{\text{crit.}} = ((L - W)/L)\varepsilon_{\max.} \tag{3}$$

where W is the craze or crack width (width of the damage zone) given in mm and determined using an optical microscope as illustrated in Fig. 2 [16]. Eq. (3)

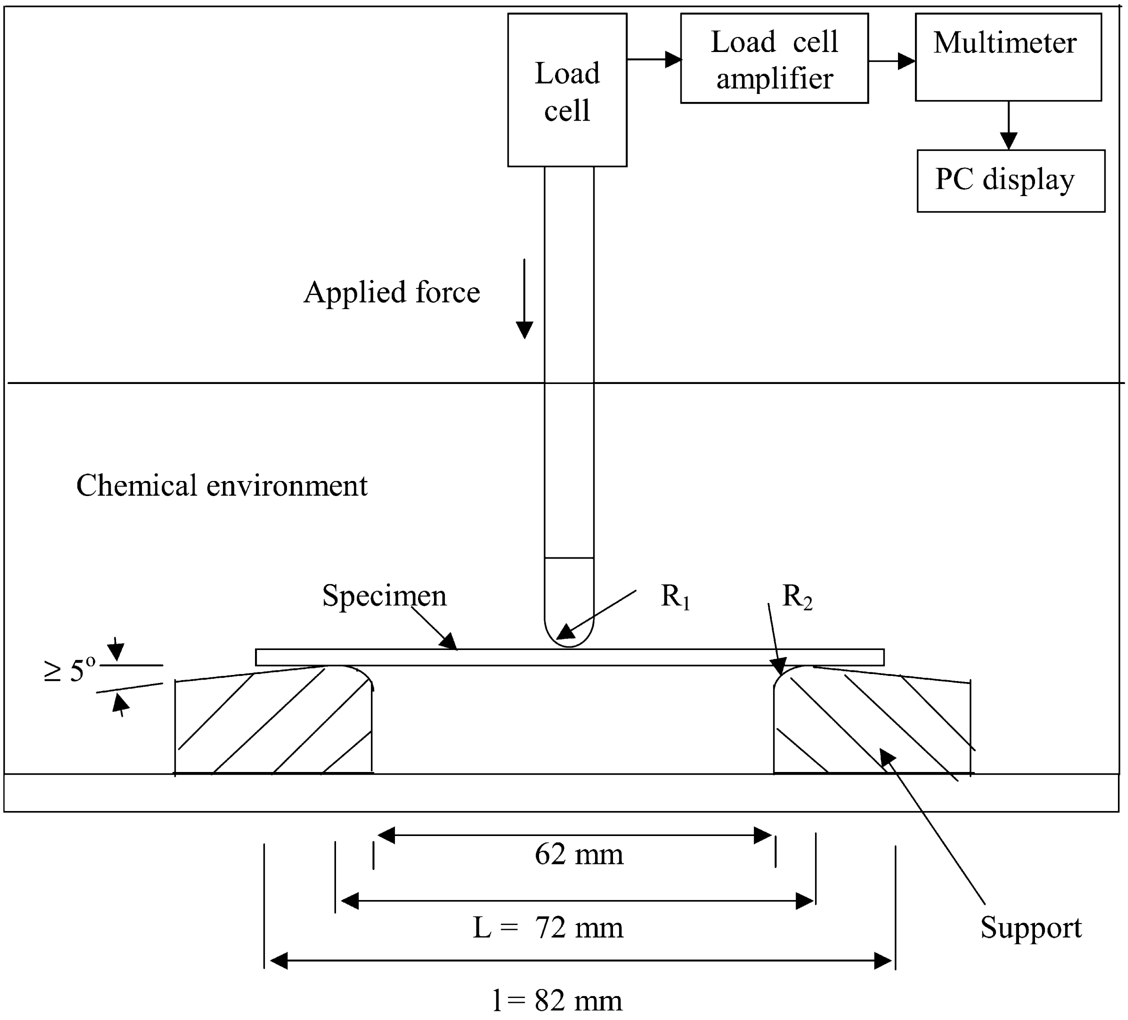

Fig. 1. Schematic diagram of the test device for the ESCR of polymer.

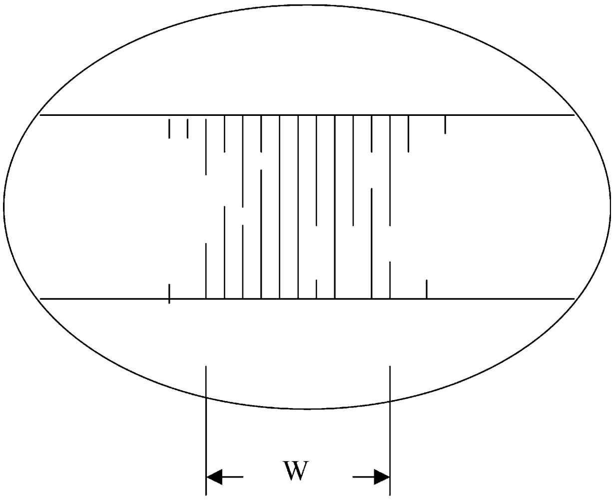

Fig. 2. Crazed sample showing craze width (width of the damage zone), *W*.

has been used for three-point bending tests, because the damage zone appears at the mid-point and increase in the strain increases the width of damage zone. Hence, there is a relationship between the width of the damage zone and the strain at the mid-point. But, in the case of uniform strain as in the bent strip method—determination of resistance to environmental stress cracking (ISO 4599-1986) or in EIA standard-test method for chemical compatibility of polycarbonate by stress crazing evaluations (EIA-564), the damage zone appears along the specimen and there is no relationship between the width of damage zone and the strain.

2.3. Absorption tests

The absorption rates for each of the chemical environments were measured. Rectangular specimens with dimensions 20×20×4 mm^3 were immersed into the chemical environment at 23±1 °C. The weight was measured regularly and rapidly using an ±0.01 mg accurate balance. The measurements were continued to up to 170 days. Before weighing a specimen, excess chemical was removed from its surface with absorbent paper. This procedure was repeated until equilibrium was reached.

3. Results

The variation of the stress with the immersion time and three-point bending method have been used to evaluate the chemical compatibility of polymers and the related environmental stress cracking.

3.1. Polycarbonate–isopropyl alcohol combination

Fig. 3 shows the time dependent variation of the stress of polycarbonate immersed in isopropyl alcohol up to 72 h and exposed to strain values between 0.79 and 1.85%. The corresponding initial stresses needed for the given strains were between 19 and 38 MPa. The figure also includes, for comparison, the equivalent tests made in air. The deviation points between the curves in isopropyl alcohol and in air represent the time where crazes are initiated.

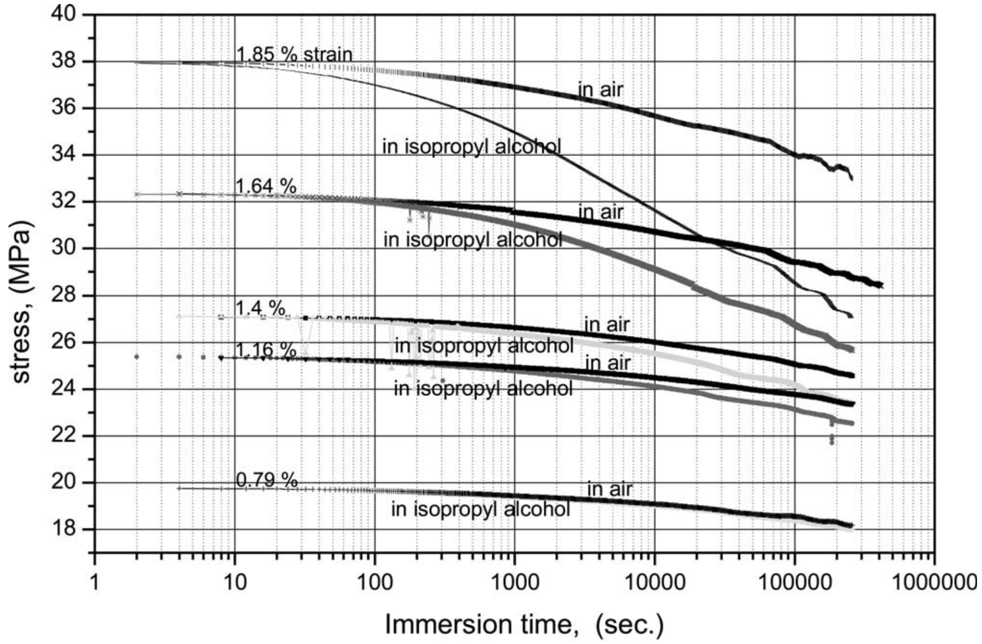

Fig. 3. Variation of the stress of polycarbonate exposed to isopropyl alcohol for 72 h at different strains and compared in air.

At the 0.79% strain level, there are no deviations in the resistance of polycarbonate in isopropyl alcohol as compared to that in air. This indicates that there is no solvent induced cracking with this strain value. Optical microscope inspections agree with these results, showing no signs of cracks or crazes. Increasing the applied strain will decrease the resistance of polycarbonate when embedded in chemicals, and craze initiation occurs when the strain reach as a critical strain.

Absorption measurements, presented in Fig. 4a, show that there is very little weight gain for polycarbonate when immersed in isopropyl alcohol, indicating that ESC can be seen with environments, where there is minor or no measurable solubility or diffusion over reasonable timescales, as in isopropyl alcohol into polycarbonate. Thus, there is very little or no solvent induced softening or plasticization at the time when crazes appear. The mechanism of ESC does not appear to have an origin like that usually ascribed to ESC, and give above. It can be speculated that in this case, the dominant mechanism related to craze formation from minute voids close to the surface, while the diffusion of the environment into the layers very near to the surface will still have an effect. Once these voids have formed,

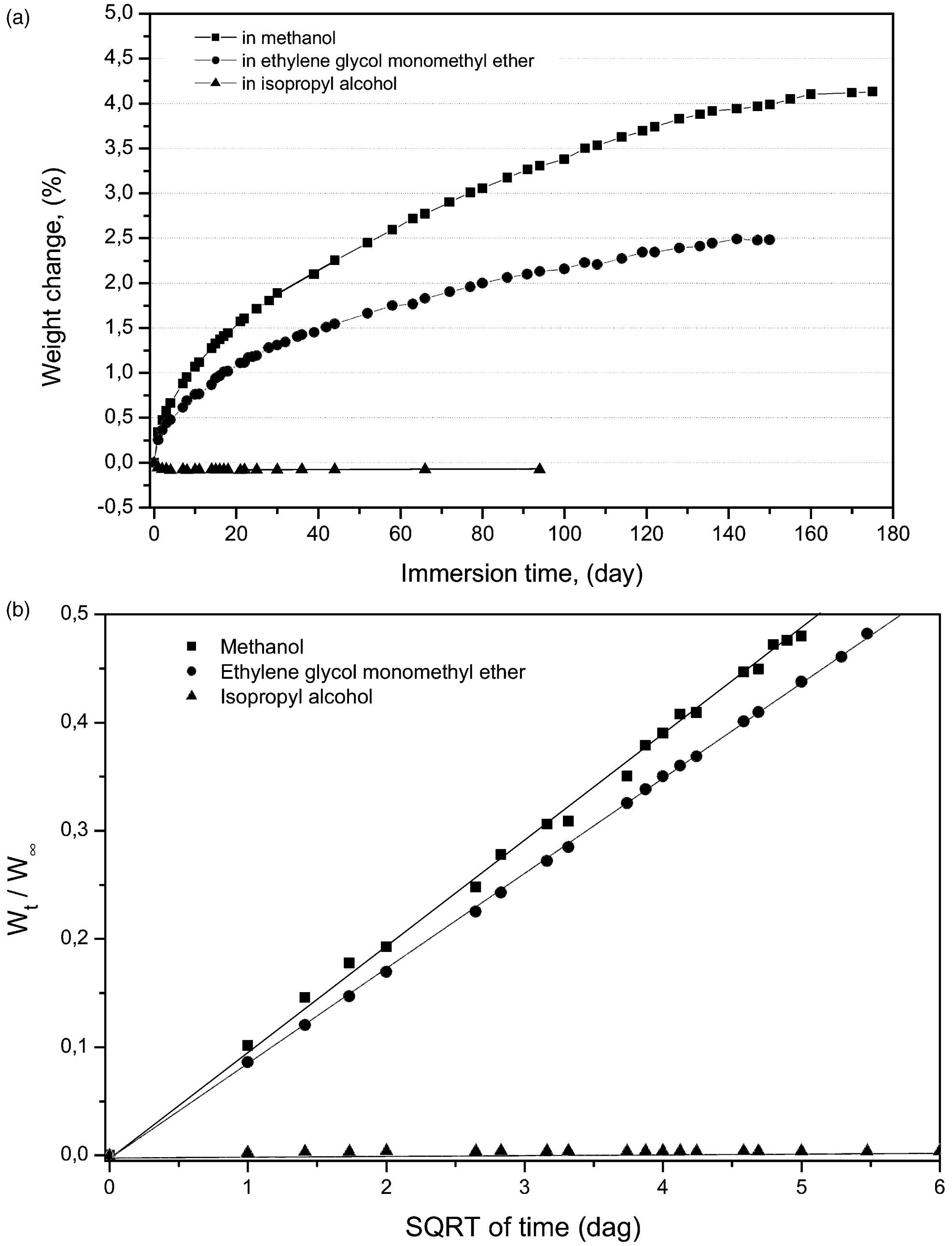

Fig. 4. Absorption behaviour of polycarbonate in isopropyl alcohol, ethylene glycol monomethyl ether and methanol: (a) the weight change versus time, (b) relationship between the ratio of weight increase at time *t* and at equilibrium with sqrt of time.

they can then act as easy diffusion paths for the environment, thus promoting craze formation. Evidence to support this comes indirectly from work on very slow diffusing combinations such as lubricating oil in phenolics [8,17].

Table 2 shows the crazing data of polycarbonate obtained in different environments. The crazing data of polycarbonate in isopropyl alcohol showed that the craze depth and width of the damage zone increase significantly with strain amplitude. The craze depth increases about three times, from 0.59 to 1.6 mm, upon increasing the strain from 1.16 to 1.85%, while the craze width increases about 20 times from 1.02 to 22.5 mm for the same increase in strain. The critical point of strain found from these values, according to Eq. (3), is about 1.21±0.06%.

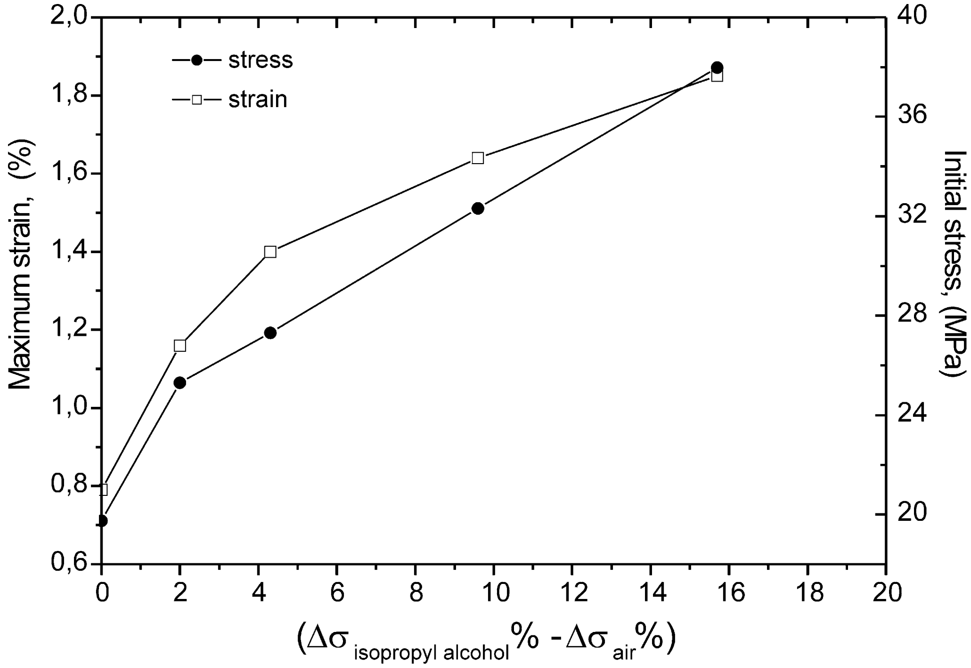

Fig. 5. Variation of ($\Delta\sigma_{\text{isopropyl alcohol}}\%-\Delta\sigma_{\text{air}}\%$) of polycarbonate as a function of maximum strain and initial stress.

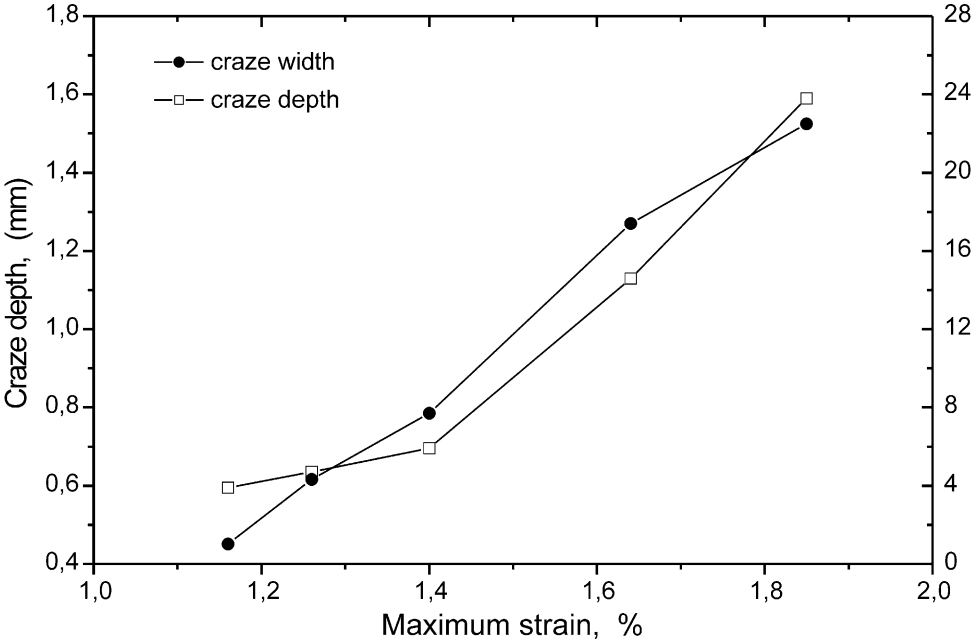

Fig. 6. Relationship between the maximum strain at the midspan versus the craze depth and the craze width of polycarbonate exposed to isopropyl alcohol for 72 h.

Table 2
Cracking data for polycarbonate in isopropyl alcohol, ethylene glycol monomethyl ether, and methanol

Combination	Initial stress (MPa)	Maximum strain ($\varepsilon_{\max.}$%)	Craze depth (mm)	Craze width (mm)	Critical strain ($\varepsilon_{\text{crit.}}$%)	Notes
Polycarbonate–isopropyl alcohol	19.8	0.79	–	–	–	A
	25.3	1.16	0.595	1.02	1.143	E
	27.3	1.4	0.695	7.7	1.25	F
	32.3	1.64	1.13	17.4	1.244	F
	38	1.85	1.59	22.5	1.271	F
Polycarbonate–ethylene glycol monomethyl ether	7.5	0.28	–	–	–	A
	8.6	0.33	B (11.8 h)	–	–	C
	9.5	0.37	B (4.5 h)	16.48	0.29	C
	10.5	0.48	B (12 min)	–	–	C
	11.5	0.55	B (22 s)	–	–	C
Polycarbonate–methanol	27.3	1.4	–	–	–	A
	32	1.64	1.72	1.01	1.62	E
	38	1.85	2.06	7.67	1.65	F
	44	2.2	2.5	10.7	1.87	F

A, no cracking; B, breaking; C, cloudy color; E, edge cracking; F, full width cracking.

Fig. 5 shows a plot of the decrease of ($\Delta\sigma_{\text{isopropyl alcohol}}\%-\Delta\sigma_{\text{air}}\%$) versus maximum strain in % and initial stress of polycarbonate when it is exposed to isopropyl alcohol and air, respectively, for 72 h. The resistance of polycarbonate decreased with both increasing maximum strain and increasing initial stress applied on the specimen. Fig. 6 shows that increasing the maximum strain applied on the specimen will enhance both craze depth and width of the cracks.

An example of a light microscopy image of polycarbonate exposed to isopropyl alcohol for 72 h at 1.26% strain, showing clear details of single crack is given in Fig. 7. Fig. 7a shows the middle of a 1 μm-width crack at the surface of the specimen. Fig. 7b shows the same crack, but at the end of crack. The crack extends about 5 μm under the surface of specimen. This indicates that the cracks propagate under the surface of the specimen.

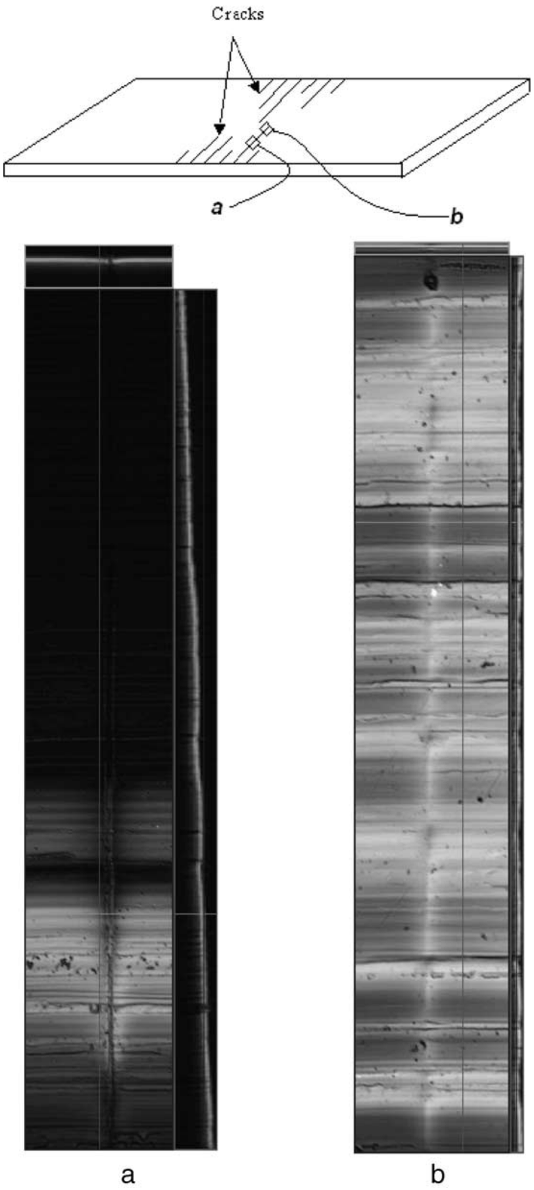

Fig. 7. Microscopic images of polycarbonate exposed to isopropyl alcohol for 72 h at 1.26% strain: (a) middle of the single crack, (b) end of the same crack. Dimensions: 31×181 μm.

3.2. Polycarbonate–ethylene glycol monomethyl ether combination

ESC measurements of polycarbonate exposed to ethylene glycol monomethyl ether revealed marked failure under very low strains and stresses as compared to polycarbonate in isopropyl alcohol. The range of the initial stress applied to the specimen was between 7.5 and 11.5 MPa, and the corresponding maximum strain at the center pin was between 0.28 and 0.55%. The results showed that the ESC resistance of polycarbonate in ethylene glycol monomethyl ether at 0.55% strain decreased within a few seconds, and the sample broke completely after 22 s as shown in Fig. 8.

When the strain decreased to 0.48%, the time to break and failure increased to 700 s. With 0.37% strain the breaking time increased to 16,000 s (4.5 h), with 0.33% strain the breaking time increased to 42,000 s (11.8 h) and with the strain decreased to 0.28% no craze and crack initiation was detected.

Absorption tests showed an increase in weight that reaches about 2.5% at equilibrium (Fig. 4a). A linear relation was obtained between the uptake and the square root of time, where the uptake is defined as the ratio W_t/W_∞ between the weight change at time t (W_t) and the weight change at equilibrium after absorption (W_∞). This uptake is shown in Fig. 4b. The linear relation $W_t/W_\infty \sim \text{sqrt}(t)$ holds for weight uptake W_t/W_∞

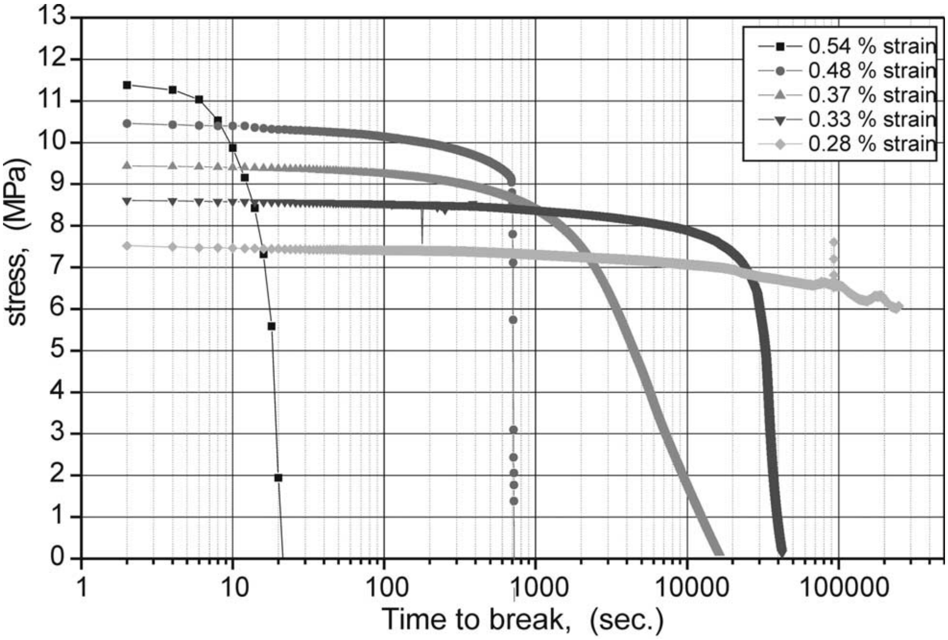

Fig. 8. Variation of environmental stress cracking resistance of polycarbonate exposed to ethylene glycol monomethyl ether at different strains.

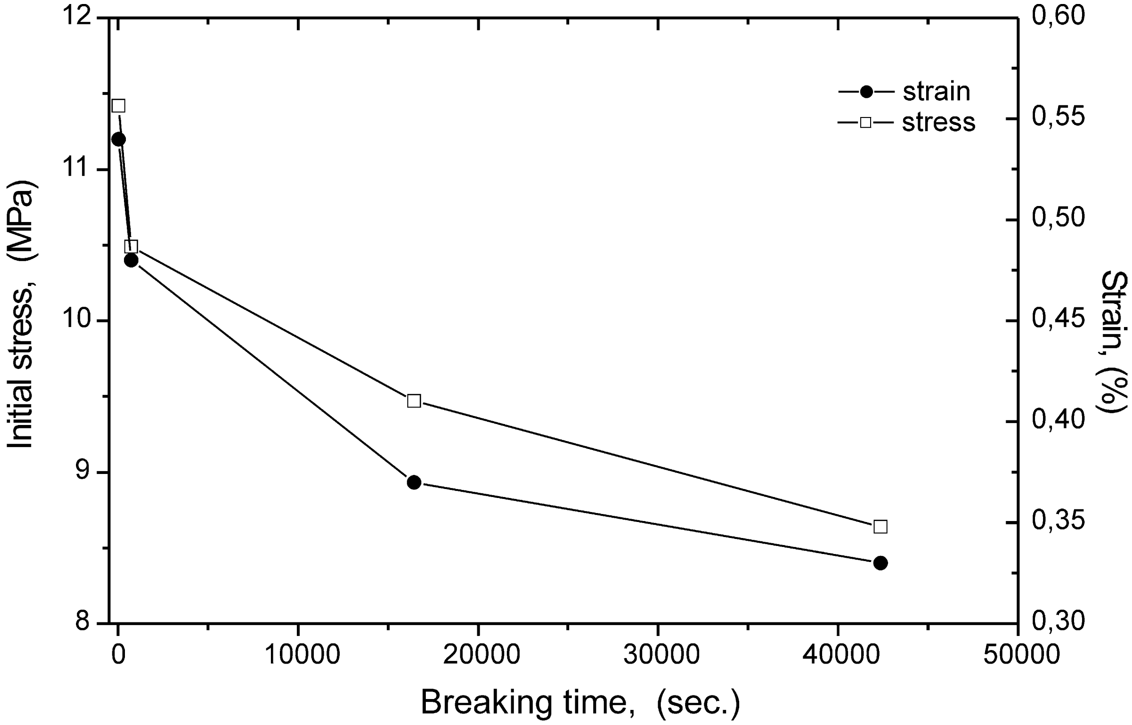

Fig. 9. Relationship between the breaking time of polycarbonate exposed to ethylene glycol monomethyl ether against maximum strain and initial stress.

in range from 0 to 0.5, i.e. when half of the quantity is absorption. The relationship is in agreement with Ficks second law and indicates thus that the uptake is dominated by simple bulk diffusion with no specific surface resistance.

The crazing data obtained (Table 2) for the PC–ethylene glycol monomethyl ether combination showed craze width equal to 16.48 mm at the 0.37% strain. According to Eq. (3) this gives the critical strain value equal to 0.29%. From the visual observations, it appears as if there is changing in the transparency from totally transparent to a somewhat opaque specimen. Fig. 9 represents the relationship between the breaking time against maximum strain (%) and initial stress of polycarbonate exposed to ethylene glycol monomethyl ether. It is clear that the breaking time increases with decreasing maximum strain and initial stress.

3.3. Polycarbonate–methanol combination

Polycarbonate samples exposed to methanol show that the initial applied stress for a given deformation decays relatively quickly with time. This is shown in

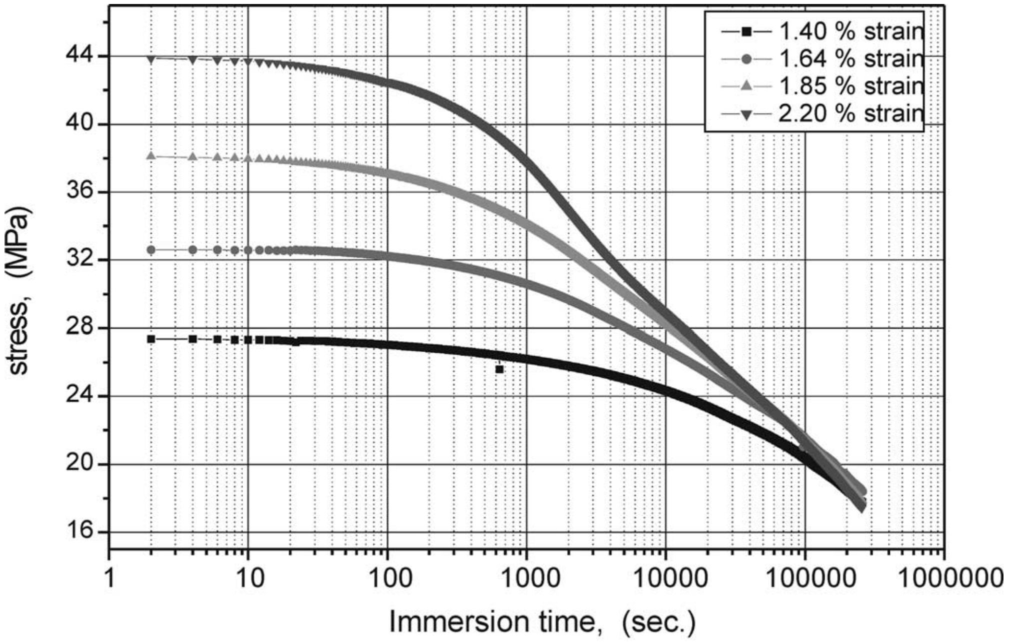

Fig. 10. Variation of the stress of polycarbonate immersed in methanol for 72 h and at different strains.

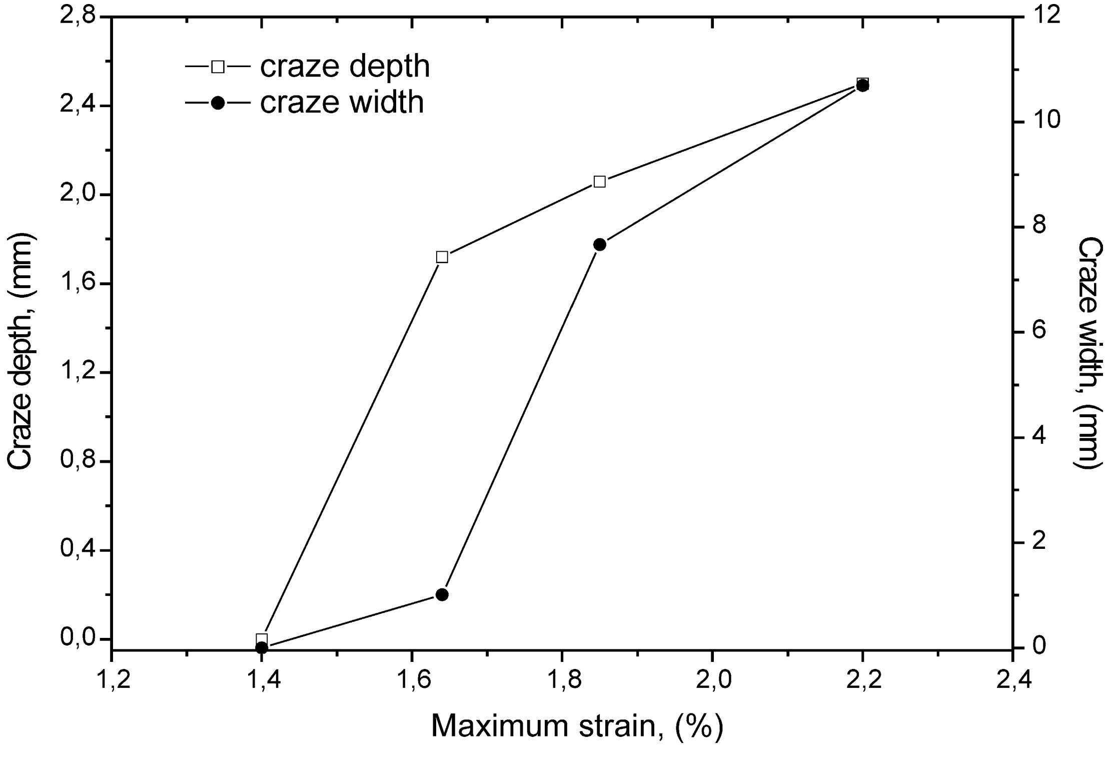

Fig. 11. Relationship between the maximum strain at the midspan versus the craze depth and the craze width of polycarbonate exposed to methanol for 72 h.

Fig. 10. This is a result of the quick diffusion of methanol into polycarbonate, leading to plasticization near the surface.

The time dependent absorption of methanol into polycarbonate is shown in Fig. 4a. The relative weight change reaches about 4.2%. As for the polycarbonate immersed in ethylene glycol monomethyl ether, discussed above and shown in Fig. 4b, a linear relationship is obtained between the relative weight uptake, W_t/W_∞, and the square root of time, i.e. there is dominating bulk diffusion with no specific surface resistance.

The range of initial applied stress was between 27 and 44 MPa, corresponding to strain ranges between 1.4 and 2.2%. No cracks were observed for strain levels up to 1.4–1.6%, while crack formation was observed above these values.

Rapid solvent absorption can to some extent prevent ESC, because of induced softening and thereby stress relaxation. This may be the explanation that cracks begin at relative high initial stress and high strain values for polycarbonate exposed to methanol.

From the crazing data of polycarbonate in methanol, given in Table 2, the critical strain value is about 1.74±0.12%. The increase of the craze depth with increasing strain is higher for exposure to methanol compared to that of isopropyl alcohol; however, the

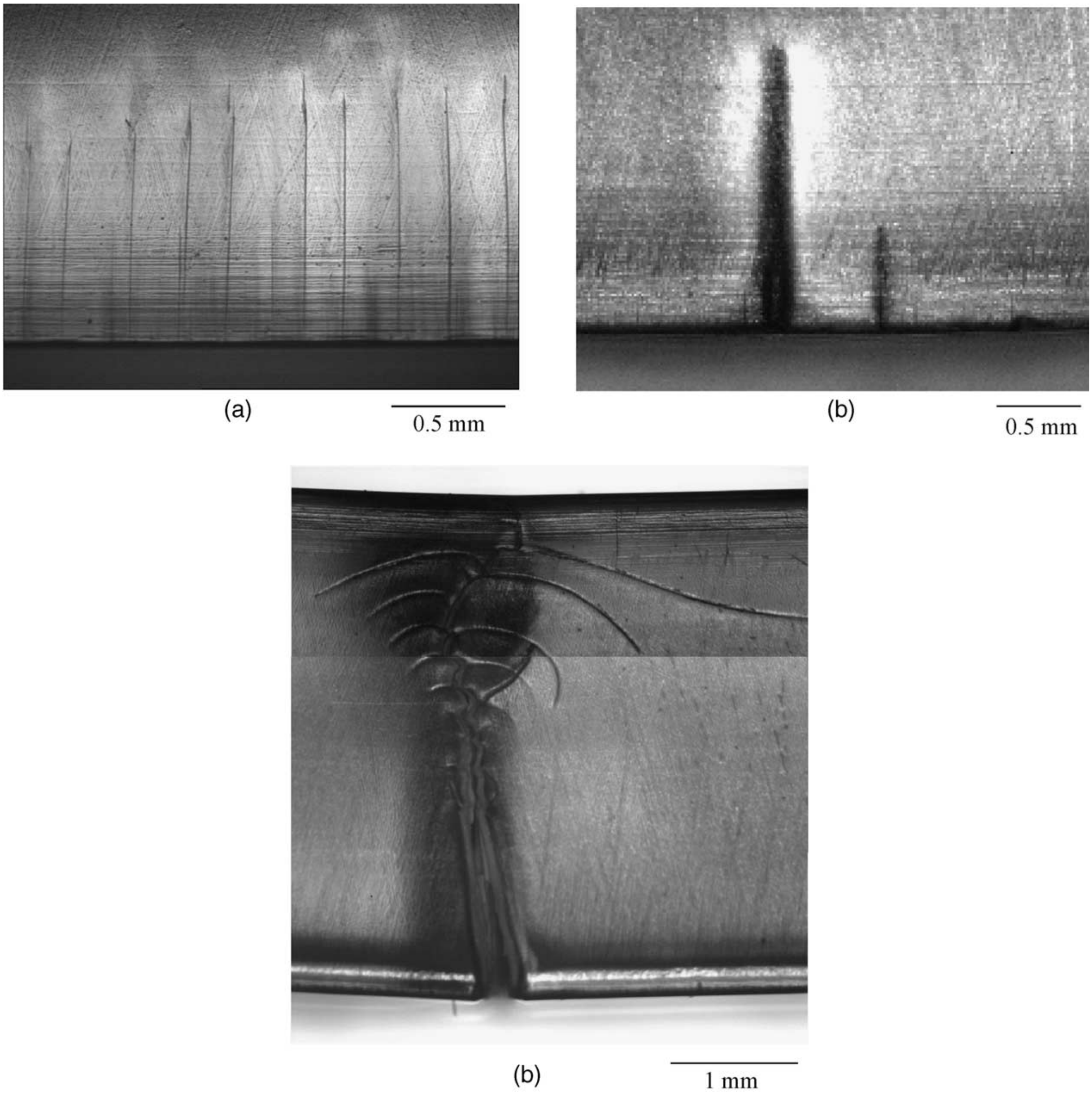

Fig. 12. Optical microscopy photos of cracking in polycarbonate: (a) in isopropyl alcohol for 72 h at 1.64% strain, (b) in methanol for 72 h at 1.64% strain, and (c) in ethylene glycol monomethyl ether for 4.5 h at 0.37% strain.

increase of width of damaged zone (craze width) is lower than that of isopropyl alcohol. Fig. 11 shows the relationship between the maximum strain at the mid-span against the craze depth and craze width of polycarbonate immersed in methanol for 72 h.

Optical microscopy photos shown in Fig. 12 prove that the cracking nature of polycarbonate differ from one environment to another. Fig. 12a shows that thin cracks (about 1 μm) appear on the surface, and the crazes form from minute voids close to the surface. The cracks propagate in parallel to the applied stress. Fig. 12b shows thick cracks (more than 10 μm) on the surface and into the polymer due to the bulk diffusion of methanol into the polycarbonate and there is no surface resistance. The cracks propagate in parallel to the applied stress. Fig. 12c shows the very sudden and catastrophic failure of polycarbonate in ethylene glycol monomethyl ether. The cracks propagate in two direction; both parallel and perpendicular to the applied stress.

4. Conclusions

The studies of polycarbonate exposed to different solvents prove different types of mechanisms can dominate in environmental stress cracking. Our studies of solvent uptake, stress–strain curves and properties of cracks show at least two different classes. The results show that immersion of polycarbonate in all three liquids studied produces ESC. The most dramatic case is in the environment of ethylene glycol monomethyl ether, where the specimens shows complete failure after only few seconds.

The main cause of ESC in polycarbonate immersed in isopropyl alcohol related to craze formation from voids close to the surface without any softening and plasticization. This is supported by the experimental findings that there is no weight gain in this case. Polycarbonate exposed to methanol and ethylene glycol monomethyl ether, on the contrary, have ESC driven by the solvent uptake via bulk diffusion. The solvent uptake is in accordance with Ficks second law of diffusion. The crack effect inside the polymer is therefore higher than the effect on the width of damage zone, with no specific surface resistance, and bulk diffusion.

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