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Stress cracking of nylon polymers in aqueous salt solutions

Part 1 *Stress-rupture behaviour*

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The susceptibility of nylon polymers to environmental stress cracking in aqueous salt solution was investigated. The time to fail (complete rupture) was recorded for razor-cut precracked samples which were totally immersed in water, sodium chloride, or calcium chloride at various initial stress intensity levels and temperatures. Ductile failures were observed in both water and aqueous sodium chloride at all temperatures. However, in saturated aqueous calcium chloride the nylon failure mode changed from ductile tearing, at room temperature, to crazing and cracking at elevated temperatures (50 to 100° C). Lithium and magnesium chlorides were also observed to be potent cracking agents at the elevated temperatures. The temperature interval over which cracking occurred corresponded to the onset of the glass transition temperature, T_g, of the nylon. Thus, nylon 6, having a lower T_g, was more susceptible to cracking than nylon 6,6 at any given temperature. On the other hand, nylon 11, having a lower ratio of amide to methylene groups, was not susceptible to salt-induced cracking. For the nylon 6 and nylon 6,6, the failure kinetics in salt solutions were similar, regardless of whether dry samples or previously moisturized samples were examined. Thus a simple temperature–moisture content equivalence does not apply for salt-induced stress cracking in aqueous solutions.

1. Introduction

Both organic liquids and inorganic salt solutions can cause environmental stress cracking of nylon polymers [1–5]. The role of polymer–liquid solubility in the stress cracking of nylon 6,6 by organic liquids has also been reported [6]. Rapidly sorbed liquids were observed to plasticize the surface, allowing homogenous stress relaxation to occur in competition with craze growth. To minimize this surface plasticization effect, the present study of nylon cracking in aqueous salt solutions employed a fracture mechanics approach whereby the growth of crazes at razor-cut precracks was investigated under conditions of constant load and temperature.

In this study emphasis was also placed on aqueous salt solutions which have not been reported to cause stress cracking of nylons at room temperature. For example, calcium chloride was reported to cause stress cracking at room temperature only when dissolved in methanol, but not when dissolved in water [2]. Calcium chloride is an example of a Type II salt following the classification given by Dunn and Sansom [2–5]. Type I salts, such as zinc chloride, are extremely aggressive cracking agents for nylon even at room temperature and are known to disrupt crystalline as well as amorphous regions of the nylon. Reimschuessel and Kim [7] and Burford and Williams [8] have reported on the morphology and mechanism for stress cracking of nylon by zinc chloride, while Burford and Harraver [9] examined the diffusion of salt solutions into nylon.

It is well known that salt ions interact strongly with nylon and, in the absence of water, can cause an increase in the glass transition temperature of the amorphous phase [10, 11]. Such interactions have been observed for a variety of salts including both Type I and Type II varieties. Based upon the study by Kim and Harget [10] a series of salts were selected for the present investigation of salt-induced stress cracking. The series included lithium, sodium, calcium and magnesium chlorides. Only aqueous solutions were examined, with the specific goals being to define the temperature dependence of stress cracking and to elucidate the mechanism for salt-induced crazing and cracking.

2. Experimental procedure

2.1. Materials

The nylons used throughout the stress cracking analysis were commercially available materials. This included nylon 6,6 (Zytel 101 and ST 801 (supertough)) from E. I. duPont, Wilmington, Delaware, nylon 6 (Capron 8202) from Allied Corporation, Morristown, New Jersey, and Nylon 11 (BMNO Grade) from Rilsan Corporation, Glen Rock, New Jersey.

Analytical reagent grade salts were used for the stress cracking and weight gain analysis. Table I summarizes the molar concentrations and the weight percentage of the saturated salt solutions used. Saturation of the solutions refers to the initial preparation at room temperature. For solutions which were 50% saturated, published values of the saturation

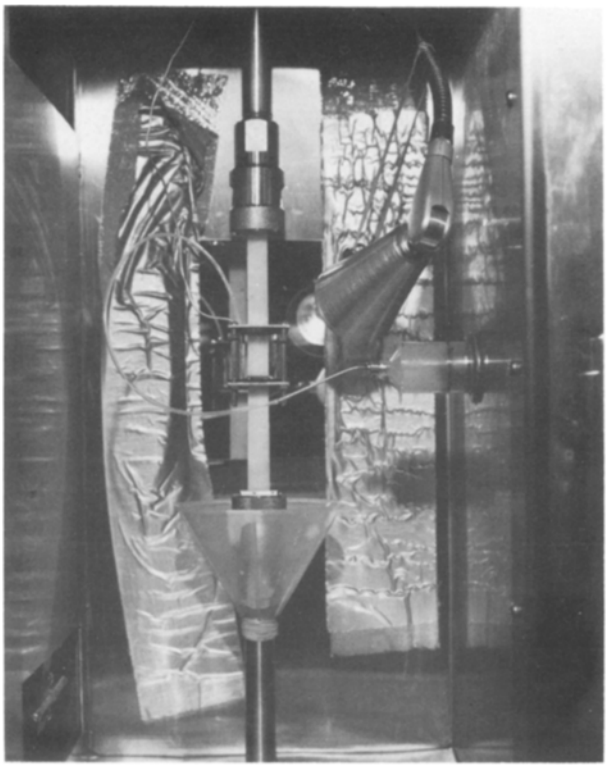


Figure 1 View of a nylon test sample, with attached liquid cell, mounted in a constant-load apparatus.

levels at room temperature were used. No attempts were made to account for the increased solubility of the salts at higher temperatures.

2.2. Processing

Nylon 6,6 and nylon 6 samples were injection-moulded using standard procedures to provide Type I (ASTM D-638) tensile bars. The wider portions on the ends of the tensile bars were milled using a Tensile-Kut® (Seiburg International Inc., Waterbury, Connecticut) apparatus to yield samples with a constant 1.27 cm width over their total length. This permitted attachment of a liquid cell fixture to the specimen (see Fig. 1). After milling, samples were redried in a vacuum oven with desiccant for 24 h at 60° C, and stored in a desiccator to prevent moisture absorption prior to testing.

2.3. Moisture conditioning

Nylon samples were conditioned and tested at 50 and 100% relative humidity (R.H.) to assess the influence that absorbed water had on the stress cracking behaviour. Accelerated conditioning at 50% R.H. was accomplished by heating samples to 121° C in a solution containing 125 parts by weight of potassium acetate to 100 parts by weight of water. Weight gains were followed to ensure a 2.5% water uptake similar to a 50% R.H. moisturized sample [12]. These samples were subsequently placed in a 52% R.H. atmosphere which was controlled by using a saturated aqueous magnesium nitrate salt solution. Samples conditioned at 100% R.H. were boiled in water until the nylon became saturated, i.e. until no increase in weight gain was observed. This corresponded to 8.5% of water sorbed by nylon 6,6. These samples were stored in an atmosphere maintained at 100% R.H. until tested.

TABLE I Concentrations of saturated aqueous salt solutions used in stress cracking

Salt	Concentration	
	(M)	(wt %)
Sodium chloride	6.1	26.3
Calcium chloride	6.7	42.7
Zinc chloride	31.7	81.2
Lithium chloride	15.0	38.9
Magnesium chloride	5.7	35.2

2.4. Stress rupture experiments

The nylon samples used in the stress cracking analysis were all tested under conditions of constant tensile load using the following procedure. A 0.1 cm deep razor-cut precrack was made on both sides of each sample. For this double edge notched sample the initial stress intensity factor at the crack tip is given [13] by

$$K_{\mathrm{I}} = YA^{1/2}\sigma$$

where

$$Y = 1.98 + 0.36\left(2\frac{A}{W}\right) - 2.12\left(\frac{A}{W}\right)^2 + 3.42\left(2\frac{A}{W}\right)^3$$

in which A = crack length (0.1 cm), W = specimen width (1.27 cm) and σ = stress.

Fig. 2a shows schematically the sample geometry while Fig. 2b is a photograph of the actual sample with crazes growing from the precracks.

A Satec Systems Model P1 (Grove City, Pennsylvania) creep-rupture apparatus was employed to provide a constant load. Temperature was controlled using a Model TT-1.1-6-X2S environmental chamber made by Cincinnati Subzero Products, Inc. Cincinnati, Ohio. A thermocouple was placed into the liquid

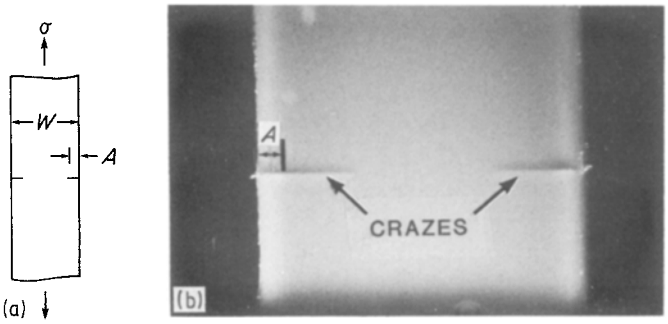


Figure 2 (a) Schematic representation of a double edge notched sample with razor-cut precracks, (b) view of actual sample showing crazes growing from precracks (× 3.3). The length of the precrack is indicated by dimension *A* in this figure and subsequent figures.

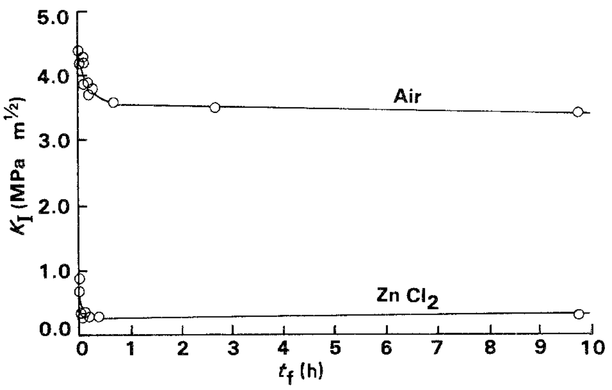


Figure 3 Initial stress intensity factor, K_I, against time to fail, t_f, in air and in an aqueous zinc chloride solution for dry nylon 6, 6 at 22° C.

cell to independently monitor the temperature of the experiment. Sample deformation was observed through a glass window on the chamber using a stereozoom microscope with attached Polaroid camera.

Salt solutions were held in a syringe which passed through the wall of the temperature chamber (see Fig. 1). Once the sample and liquid attained the necessary temperature, the liquid was injected into the cell and the sample was loaded after a one-minute period. The time for complete rupture of the sample was measured for various initial stress intensity values. In this way a characteristic curve of K_I against the time to fail, t_f, was obtained.

2.5. Dynamic mechanical analysis

Measurements were made using 1.9 cm long samples cut from the centre portion of the injection-moulded tensile bars. A duPont (Wilmington, Delaware) 980 Dynamic Mechanical Analyzer was employed. Samples were tested in a vertical mode with a heating rate of 5° C min^{-1}.

3. Results

3.1. Room-temperature measurements

The stress rupture characteristics of nylon 6,6 were initially examined both in air and in a saturated aqueous zinc chloride solution at room temperature. Fig. 3 compares these results and demonstrates the dramatic influence of the stress cracking fluid on nylon 6,6. Whereas extremely high stress intensity levels are required to initiate fracture in air, very low levels are needed to initiate and propagate sharp cracks across the sample in the salt solution.

In addition to the stress levels required to initiate fracture, the mode of fracture is also different in air compared with the cracking fluid. No significant craze growth across the sample is observed in air. The sample ruptures catastrophically once crack initiation occurs with no evidence of slow crack growth. In aqueous zinc chloride the growth of a crack is always preceded by the initiation and growth of a sharp craze across the sample (Fig. 4). The crack initiates within the mature craze structure or at the craze–matrix interface. Though crazes initiated at both precracks, the formation and growth of a large crack leading to failure occurred randomly on one side only in the zinc chloride solution.

In either air or aqueous zinc chloride the time to fail increased rapidly as K_I was decreased and thus minimum or critical values of K_I could be determined below which fracture would not occur at short times. For nylon 6,6 in air or in zinc chloride the K_I values corresponding to failure times greater than ten hours are 3.4 and 0.26 MPa m$^{1/2}$, respectively. This demonstrates the severity of the zinc chloride as a stress cracking agent.

Fig. 5 shows the K_I–t_f data for 50% R.H. equilibrated nylon 6,6 immersed in pure water or in saturated aqueous solutions of sodium or calcium chloride. The data can be represented by a single curve indicating that these salt solutions have no deleterious effect on the nylon, over and above the effect of pure water. The water, acting as a plasticizer, lowers the critical K_I value for nylon 6,6 compared to the behaviour of dry nylon in air. However, the rupture in water, or in sodium or calcium chloride, is characterized by highly ductile tearing as shown in Fig. 6. There is still a relatively localized deformation zone in advance of the blunted crack tip, but no sharp crazes or cracks are observed. The effect of water is discussed further in a later section.

3.2. Effect of temperature

The influence of higher temperature (75° C) on the failure characteristics of nylon 6,6 in aqueous solutions is shown in Fig. 7. Water and sodium chloride provide similar results with lower K_I at this higher temperature. On the other hand, the calcium chloride

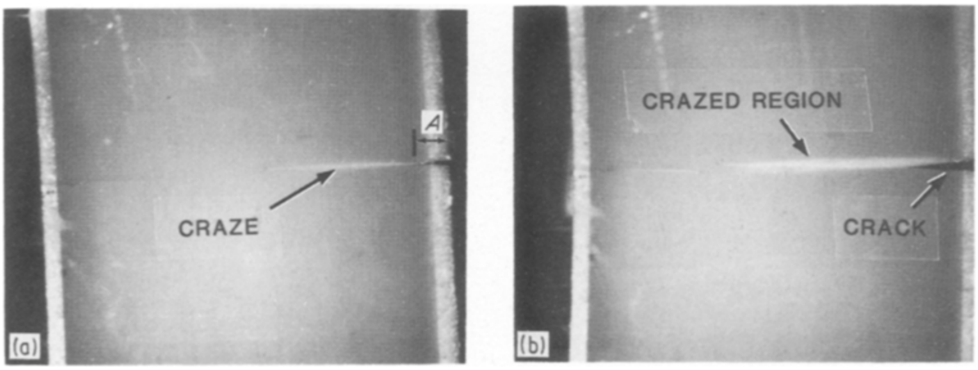


Figure 4 Failure of nylon 6, 6 in saturated aqueous zinc chloride at 22° C by (a) formation of a crazed region and (b) growth of a crack at the craze–matrix interface (× 4.0).

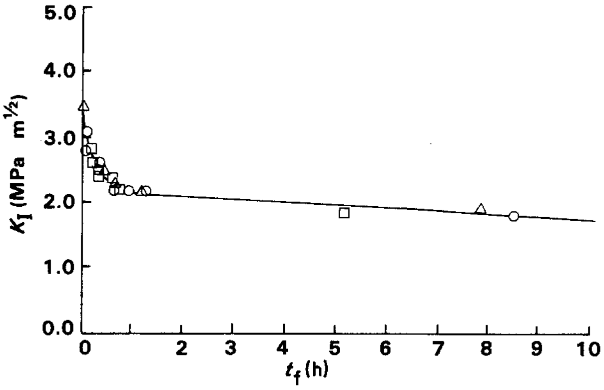


Figure 5 Initial stress intensity factor, K_I, against time to fail, t_f, for nylon 6, 6 (equilibrated at 50% R.H.) (○) in water, and in saturated aqueous solutions of (△) sodium chloride and (□) calcium chloride.

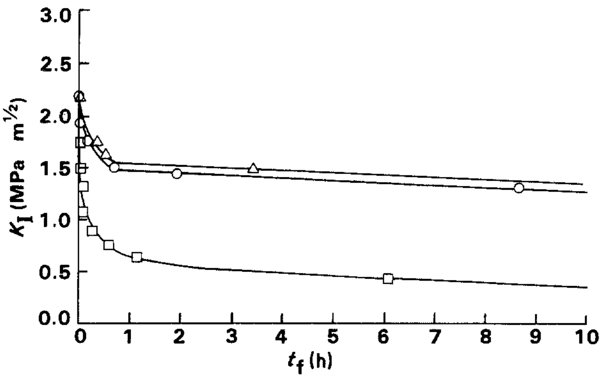


Figure 7 Initial stress intensity factor, K_I, against time to fail, t_f, of nylon 6, 6 (equilibrated at 50% R.H.) (○) in water, and in saturated aqueous solutions of (△) sodium chloride and (□) calcium chloride at 75° C.

has become a potent cracking agent at 75° C with a dramatic reduction in the K_I level required to cause fracture of the nylon 6,6. A lesser effect was noted at 50° C and a very pronounced effect at 100° C. In fact at 100° C the calcium chloride was as severe a cracking agent for nylon 6,6 as zinc chloride was at room temperature with brittle fractures even at K_I = 0.1 MPa m$^{1/2}$. The sodium chloride solution showed no indication of being an active cracking agent at 50 to 100° C. The nylon 6,6 ruptured at lower K_I in aqueous sodium chloride at elevated temperatures; however, this can be attributed to the combined effects of temperature and water absorption, as shown by the data for water immersion (Fig. 7). Furthermore, the ultimate rupture in water or sodium chloride occurs in a ductile manner with considerable drawing and tearing of the sample at all values of K_I.

In the aqueous calcium chloride solution, crazes appear on the surface of the samples at high K_I (see Fig. 8a), even away from the razor-cut precracks. The ultimate failure at the higher K_I involves both ductile drawing (as noted by the expansion of the precracks) and localized craze/crack growth. The mode of failure is much more brittle at the lower K_I levels (Fig. 8b). Failure occurs by a two-step process involving first, the growth of a craze, and second, the initiation and growth of a crack. Thus, the rate-limiting process for failure to occur is the growth of the craze, and the events leading to a breakdown of the craze structure to form a crack. In other words crack propagation occurs in seconds, whereas craze propagation can occur over several hours.

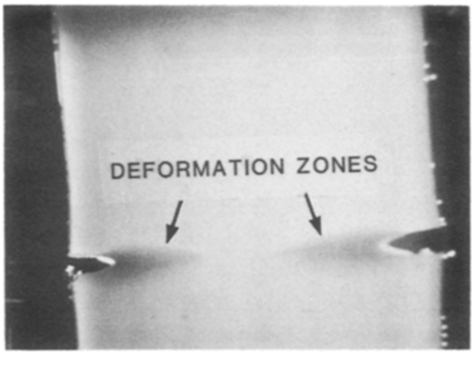


Figure 6 View of the failure of nylon 6, 6 in water at an initial K_I of 1.9 MPa m$^{1/2}$. Diffuse deformation zones precede ductile tearing of the highly drawn precracks (× 3.6).

In attempting to understand the observed effect of increasing temperature on crazing of nylon, it is interesting to note that in this temperature interval (50 to 100° C) the amorphous phase of the nylon 6,6 undergoes a transition from the glassy to the rubbery states. The glass transition, or T_g, is shown by dynamic mechanical analysis of the dry nylon 6,6 in Fig. 9. Comparison of these data with the stress–rupture results indicates that susceptibility to cracking by calcium chloride occurs coincident with the onset of T_g (above 50° C). This suggests that an interaction is occurring between the salt solution and the amorphous phase of the nylon. The nature of this interaction is discussed further in Part 2 of this paper [14].

3.3. Effect of moisture

The plasticizing effect of absorbed water on the mechanical properties of nylon polymers is well known [12]. The effect of this absorbed water on the stress-rupture characteristics of nylon was first examined by testing previously equilibrated samples in air.

Fig. 10 shows that the absorbed moisture significantly lowers the level of K_I at which rupture occurs. In spite of the lower K_I values for failure, the moisturized samples fail in a completely ductile manner as expected. This was true at elevated temperatures as well as at room temperature. For dry nylon, tested in water, the failure is much more localized with a diffuse deformation zone at the crack tip similar to that previously observed in alcohol [6]. However, unlike the salt solutions, under no conditions did the water induce a crazing type of failure mode.

Previous investigations have noted that the lowering of the T_g of nylon by absorbed water is equivalent in many ways to an increase in temperature for the dry nylon [12]. In view of the strong influence of temperature alone on the stress cracking behaviour, it was of interest to examine the combined effects of moisture content and temperature. Fig. 11 shows the results for both dry and 50% R.H. equilibrated nylon 6,6 immersed in saturated aqueous calcium chloride. Though some effect is noted at 50° C, very little difference is noted in the K_I–t_f data at 75° C. In addition,

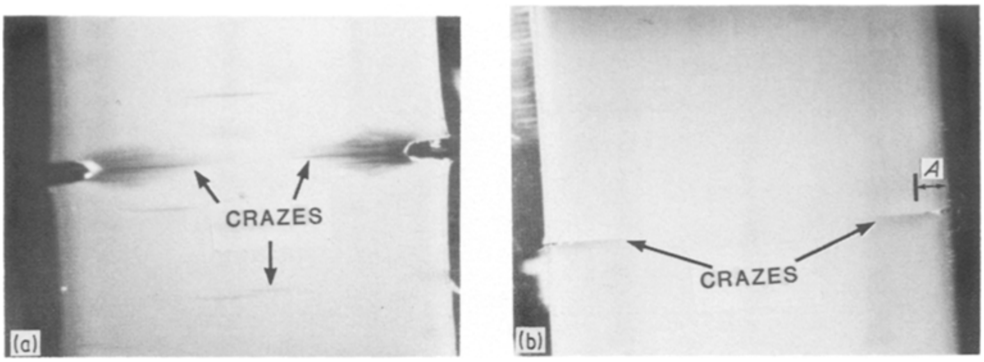


Figure 8 Views of nylon 6, 6 in saturated aqueous calcium chloride at 75° C showing (a) combination of ductile drawing and crazing at high K_I (1.75 MPa m$^{1/2}$) and (b) crazes at low K_I (0.55 MPa m$^{1/2}$) (× 4.0).

no effect of absorbed water was measurable at 100° C. Thus, the reduction in T_g by absorbed moisture, and an increased temperature for a dry sample, are not equivalent, nor are they additive in accelerating stress cracking by the salt solution.

In spite of the similarity in failure times at a given K_I, some differences in the mode of failure were noted for the moisturized samples compared with dry nylon. For example, the craze/crack initiation and growth was highly localized for the dry sample. Evidence of extensive ductile drawing was noted in moisturized samples, particularly in the final stages of fracture. Also, many crazes initiated away from the precracks for moisturized samples.

3.4. Nylon type

Stress rupture results for nylon 6 in aqueous calcium chloride at various temperatures are shown in Fig. 12. Comparing these data with the previous curve for nylon 6,6 (Fig. 7), it is noted that the K_I values to induce failure are significantly lower for the nylon 6. At 75° C, for example, it is not possible to define a minimum acceptable K_I for nylon 6 where cracking would not occur.

It was of interest to determine whether the different temperature dependence for stress cracking of nylon 6 was related to its glass transition behaviour. Dynamic mechanical data for a dry injection-moulded test bar are shown in Fig. 13. Compared to Fig. 9 it is noted the nylon 6 T_g (damping peak) does occur at a lower temperature (71° C) compared with 86° C for the nylon 6,6. Thus, the results for both nylon 6 and nylon 6,6 indicate that the onset of T_g and the onset of significant salt-induced crazing are coincident.

The influence of absorbed moisture on the stress rupture of nylon 6 was also examined. Fig. 14 demonstrates that at 50° C, dry and moisturized samples give similar results, except at high K_I (short times). As in the case of nylon 6,6 the appearance of the samples was affected by moisture content since the ultimate rupture was more ductile with the moisturized sample. Dry samples at high K_I exhibited surface stress cracks and multiple crazing at the highly drawn precracks (Fig. 15a). At lower K_I only a single craze initiates at each precrack and these grow across the entire cross-section prior to crack initiation (Figs 15b and c).

The effect of aqueous calcium chloride at 75° C on nylon 11 is shown in Fig. 16. Similar results were obtained for nylon 11 in pure water. In this case, there is no evidence of stress cracking activity. Thus, the nylon 11 would be an excellent candidate for applications where resistance to calcium chloride-induced cracking is needed. On the other hand, the use of rubber-modified or supertough nylon 6,6 (Zytel ST801) offers no advantage in stress cracking resistance as is also shown by the data in Fig. 16.

3.5. Effect of salt type

The stress cracking of nylon 6,6 in saturated aqueous solutions of calcium, magnesium, and lithium chlorides at 100° C is compared in Fig. 17. It is noted that both the calcium and lithium chloride solutions are

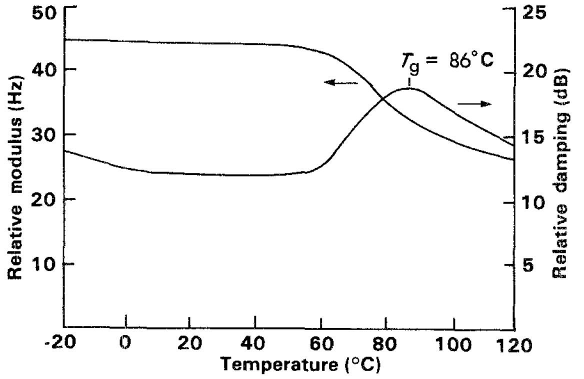


Figure 9 Dynamic mechanical analysis of dry injection-moulded nylon 6, 6 showing the occurrence of the glass transition temperature, T_g.

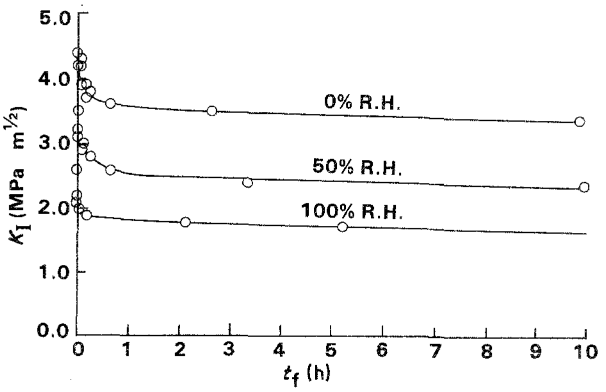


Figure 10 Effect of preconditioning at various relative humidities on the failure of nylon 6, 6 in air at room temperature.

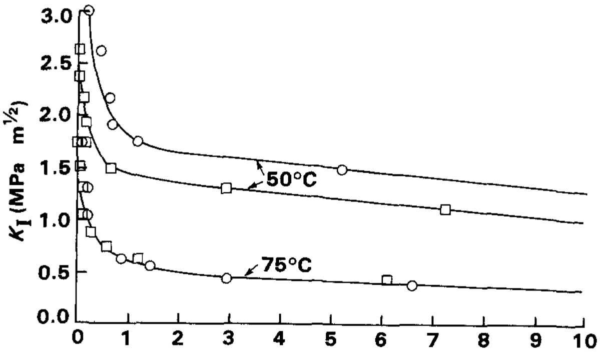


Figure 11 Initial stress intensity factor, K_I, against time to fail, t_f, for (○) dry and (□) 50% R.H. equilibrated nylon 6, 6 in saturated aqueous calcium chloride at 50 and 75° C.

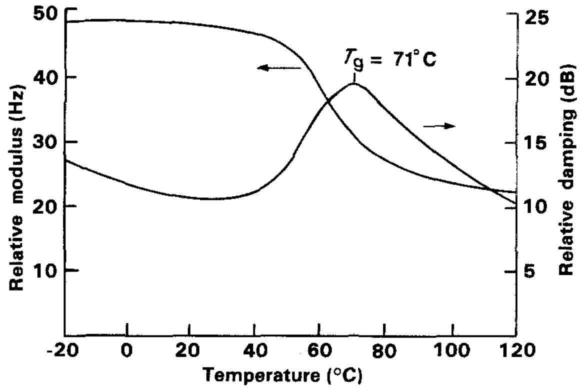


Figure 13 Dynamic mechanical analysis of dry injection-moulded nylon 6.

severe cracking agents with correspondingly low K_I levels. For magnesium chloride more complex behaviour was exhibited, since at high initial K_I ductile failures occurred and the observed times to complete rupture were similar to those observed for nylon 6,6 immersed in water. However, in the magnesium chloride solution some crazes were observed to initiate on the edges away from the precrack. Also, at the lower K_I levels, the magnesium chloride, unlike water alone, caused a complete rupture of the nylon after several hours. The failure mode was also brittle under these conditions with sharp crazes growing out from the precracks. Thus, in spite of the unusual crack blunting behaviour at high K_I, the results at lower K_I demonstrate that the magnesium chloride is a potent stress cracking agent, like the calcium and lithium chlorides.

Stress rupture data were also obtained in equimolar solutions of the three salts by diluting the calcium and lithium chloride solutions to 5.7 M, equivalent to the saturated magnesium chloride. The equimolar lithium chloride solution was much less detrimental than the saturated solution, and in fact was similar to pure water, as shown in Fig. 18, with ductile tearing and no failures below $K_I = 1.2\,\mathrm{MPa\,m^{1/2}}$. The somewhat less concentrated calcium chloride remained a potent cracking agent. This indicates that calcium and magnesium chlorides are the more potent stress cracking agents, though the available data do not indicate which of these two is more severe.

For a 50% saturated calcium chloride solution the K_I–t_f curve is intermediate between those for pure water and the fully saturated solution (Fig. 18). These results, along with the results for equimolar solutions, indicate that fairly concentrated solutions are required to cause stress cracking of the nylon with the implication that dilute solutions should pose no problem.

4. Discussion

The results of this investigation indicate that both nylon 6 and nylon 6,6 are susceptible to stress cracking by saturated aqueous calcium chloride at temperatures above the respective glass transition temperatures of the polymers. Sodium chloride showed no stress cracking activity at all temperatures examined, whereas both lithium and magnesium chlorides did cause cracking at 100° C. The available data indicate that fairly concentrated solutions are required to induce crazing and cracking of nylons at elevated temperature. For example, the 50% saturated solution of calcium chloride showed stress-rupture behaviour intermediate between pure water and the fully saturated solution. Similarly, the less concentrated lithium chloride solution showed no tendency to cause cracking. Sorption measurements are currently underway to possibly explain the observed effects of salt type and temperature and to define the nature of the nylon–salt–water interaction.

In the absence of water, inorganic salts such as calcium, lithium, and magnesium chlorides have been shown to modify the melting, crystallization, and glass transition temperature of nylon by the formation of a chemical complex between the salt cations and the

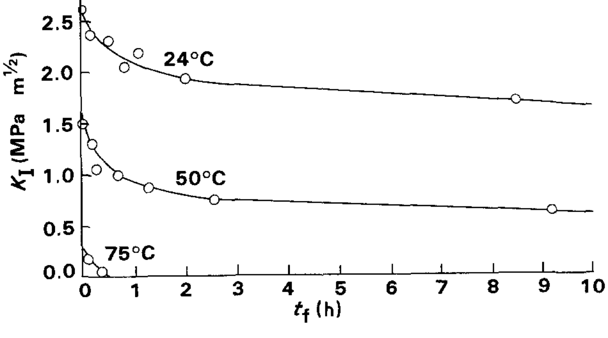


Figure 12 Effect of temperature on the failure of nylon 6 in saturated aqueous calcium chloride.

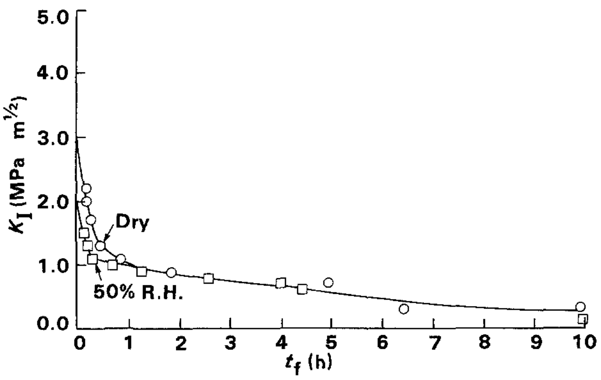
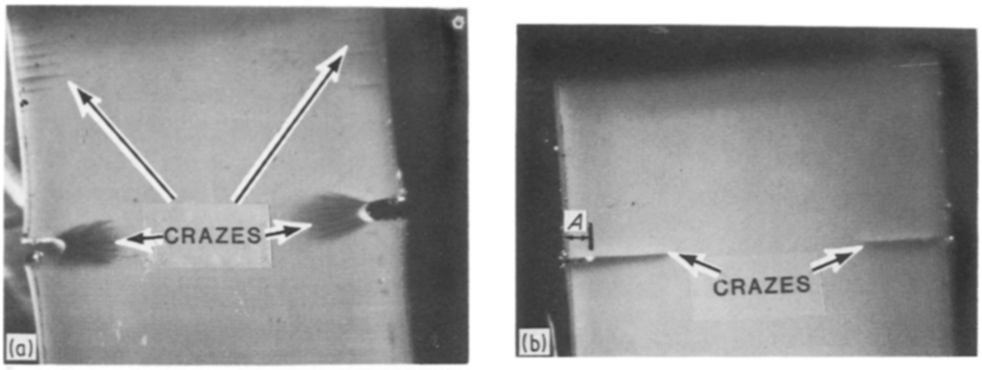


Figure 14 Initial stress intensity factor, K_I, against time to fail, t_f, for (○) dry and (□) 50% R.H. equilibrated nylon 6 in saturated aqueous calcium chloride at 50° C.



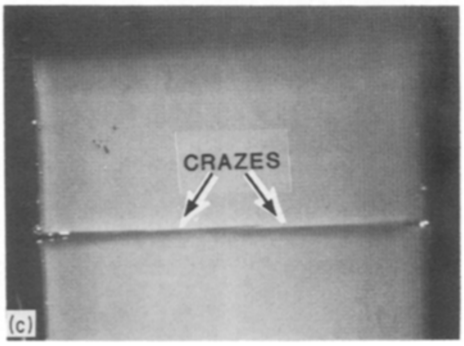


Figure 15 Failure of dry nylon 6 in saturated aqueous calcium chloride at 50° C showing (a) ductile drawing and surface crazing at $K_I = 1.75\,\text{MPa}\,\text{m}^{1/2}$, (b) and (c) growth of single crazes at $K_I = 0.66\,\text{MPa}\,\text{m}^{1/2}$ which eventually propagate across the entire sample cross-section (× 4.0).

carbonyl group of the amide linkage [10, 11]. The action of aqueous salt solutions has received less study, although a similar complex formation of hydrated ions might be anticipated. In fact, a solvation type of mechanism has been proposed [2, 7, 8] for salt-solution cracking, although strongly temperature-dependent phenomena (such as that reported here) have not been investigated in detail.

The results concerning nylon type can also be partially interpreted based upon a solvent crazing/cracking mechanism. For example, the lack of effect of calcium chloride on nylon 11 can be understood in terms of the reduced number of amide linkages in this polymer. Similarly, the use of rubber toughened nylon 6,6 may offer no advantage since the solubility of the salt solution in the nylon matrix may be similar to that in unmodified nylon 6,6. In addition to the number of amide linkages, the degree of crystallinity of the nylon should also contribute significantly to sorption and consequent stress cracking activity. However, these factors are not strongly temperature-dependent, and taken alone, cannot explain the differences in stress cracking at room temperature compared with higher temperatures as reported here. In a subsequent paper (Part 3 of this work, to be published) the craze growth kinetics will be described in order to further elucidate the mechanism of crazing of nylons by aqueous salt solutions.

The role of absorbed water on the stress cracking of nylon is somewhat surprising since no acceleration of cracking is apparent from the K_I–t_f curves. The presence of absorbed water would be expected to enhance the sorption of a salt–water complex, or in some way modify the failure process in the more ductile moisturized nylon. A possible explanation for the lack of an effect, particularly at the longer failure times, is that in either dry or moisturized nylon the crazed region and crack tip have sufficient time to reach the same sorption equilibration in the salt solution prior to actual rupture. In spite of the lack of confirmed explanation, the available results clearly show that the lowering of the nylon T_g by absorbed water is not as detrimental to the stress cracking susceptibility of the nylon as an increase in temperature.

5. Conclusions

1. Calcium, lithium, and magnesium chlorides in aqueous solution are potent stress-cracking agents for nylon at elevated temperature. Aqueous sodium

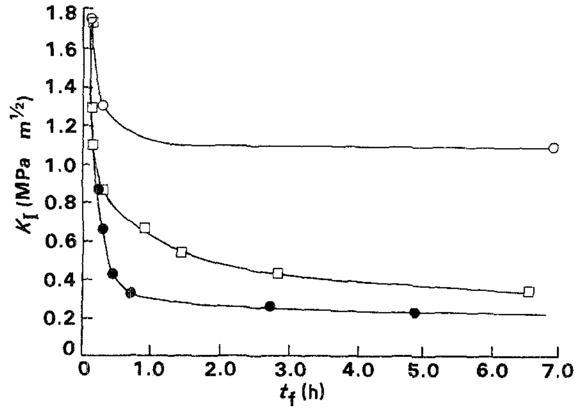


Figure 16 Comparison of the effect of saturated aqueous calcium chloride at 75° C on various nylon materials: (○) nylon 11, (□) nylon 6, 6, (●) super-tough nylon 6, 6.

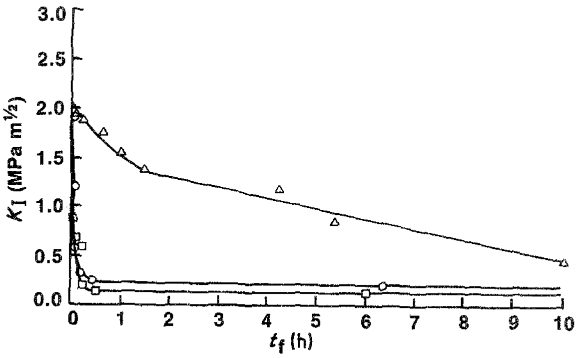


Figure 17 Stress intensity factor, K_I, against time to fail, t_f, for nylon 6, 6 immersed in various saturated aqueous salt solutions at 100° C: (△) $MgCl_2$, (□) LiCl, (○) $CaCl_2$.

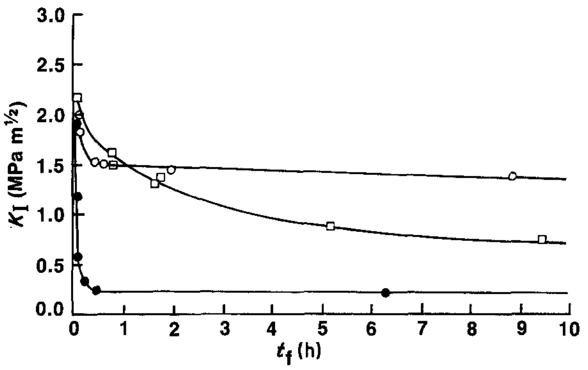


Figure 18 Stress intensity factor, K_I, against time to fail, t_f, for nylon 6, 6 immersed in (○) water or calcium chloride solutions at 100° C: (□) 50% saturated, (●) saturated.

chloride showed no propensity to induce crazing or cracking.

2. The temperature interval over which cracking occurs corresponds to the onset of the glass transition temperature, T_g, of the nylon.

3. Absorbed moisture, which lowers the T_g of the nylon, does not accelerate stress-cracking behaviour, especially at the lower K_I levels.

4. The failure kinetics of nylons immersed in aqueous salt solutions are dictated by craze growth rather than crack growth.

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