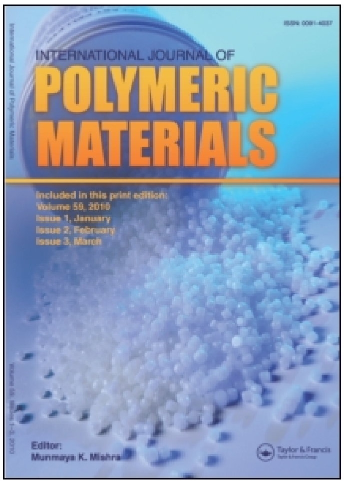

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# Crazing Characteristics of Polymers: The Use of the Slope-Discontinuity Method for Glassy Polymers Containing Rigid Spherical Inclusions

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# Crazing Characteristics of Polymers: The Use of the Slope-Discontinuity Method for Glassy Polymers Containing Rigid Spherical Inclusions

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Stress–strain curves of glass bead filled glassy polymers show a slope-discontinuity related to crazing in the matrix. The slope-discontinuity method is verified in this paper regarding its reliability for the prediction of crazing characteristics provided that orientation-free samples filled with the proper size and concentration of untreated glass beads are used. The effects of orientation, temperature and thickness of the tensile specimens on the discontinuity characteristics are also studied. A stress concentration factor $K$ is found to be independent of bead volume fraction and size, temperature and orientation. A good agreement is usually found between calculated crazing values by the slope-discontinuity method and directly measured values carried out in the present work or reported in the literature.

## INTRODUCTION

Glassy thermoplastic polymers filled with uncoupled glass beads were studied by Nicolais and Narkis.[1,2] These authors found that the stress–strain curves of the particulate systems exhibit a knee, or a slope-discontinuity point, which is absent on the curves of the glassy unfilled polymers as shown in Figure 1. $\sigma_D$, the stress at which the slope-discontinuity occurs is independent of the bead concentration $\phi$ for volume concentrations up to about 35 percent. It

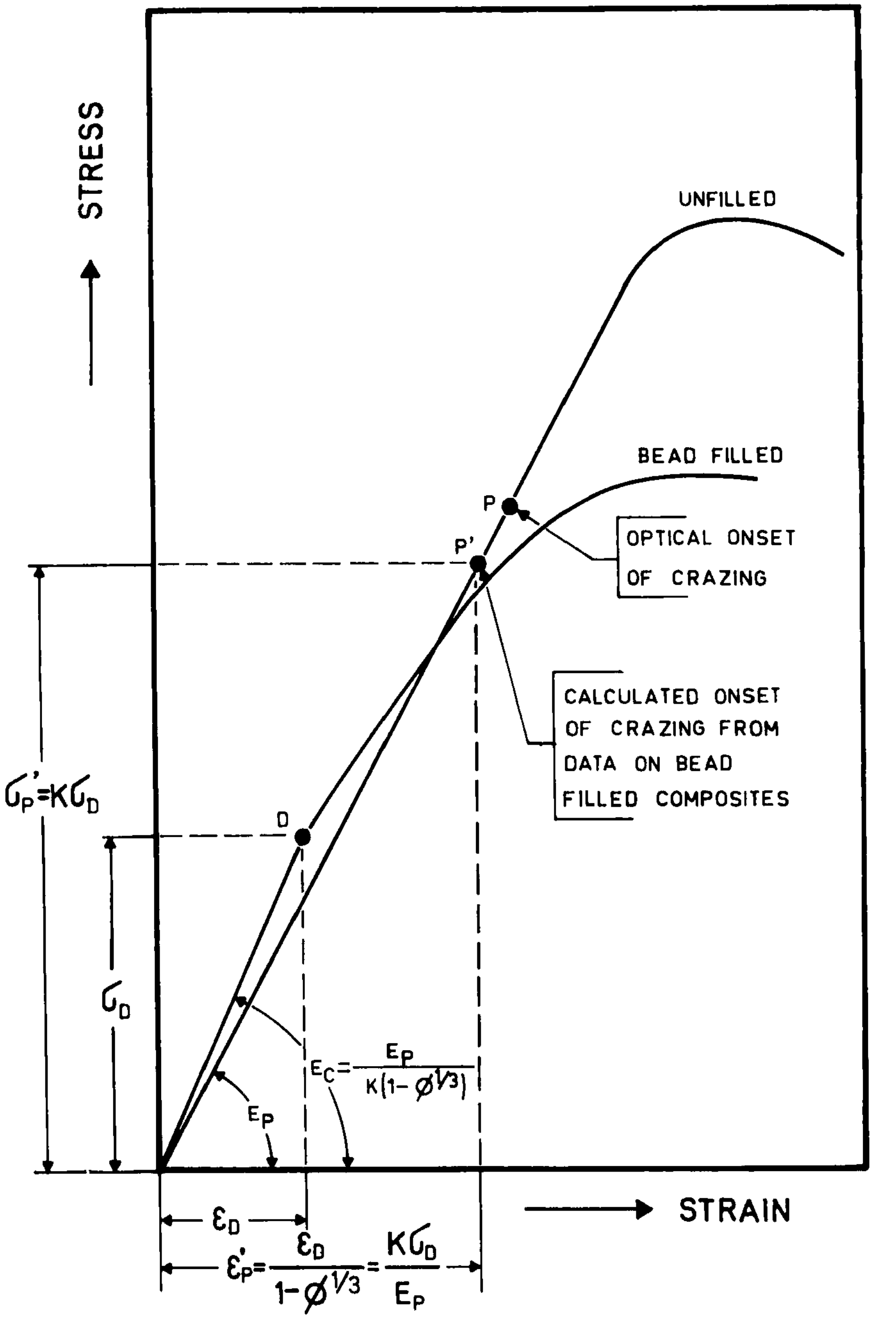

FIGURE 1 Stress–strain curves of unfilled and bead filled glassy polymer.

varies, however, with the specific polymer being tested and is temperature dependent, decreasing linearly with increasing temperature. $P'$ is a theoretical point located on the curve of the unfilled polymer and is calculated from values of the slope-discontinuity point $D(\epsilon_D, \sigma_D)$, shown in the figure. $\epsilon_p'$, the strain in the polymer[3] at the discontinuity point, is calculated from experimental $\epsilon_D - \phi$ data and has been found to be practically independent of the bead volume fraction.[2]

The slope change on the stress–strain curves is accompanied by stress whitening over the entire gage length of the sample.[4,5] This phenomenon, taken together with several other arguments, has led to the conjecture that crazing is involved in the change of the slope.[4] Significant support for the role of crazing as a cause for the slope-discontinuity has been gained from the good agreement found between calculated $\epsilon_p'$ values and literature data on critical strains.[6] These critical strains for the onset of crazing were measured directly on unfilled polymers by several researchers.

In the present work the reliability of the slope-discontinuity method is further studied for glassy polymers including polymethyl-methacrylate, polysulfone and polycarbonate. The effects of the following parameters were investigated: bead content and diameter, thickness, temperature and orientation (injection versus compression molding) of the tensile specimens.

## EXPERIMENTAL

Glass bead filled composites were prepared with polymethyl-methacrylate (PMMA, Diakon MH-253, ICI), polysulfone (PSF, Bakelite P-1700 clear and Bakelite P-1710 opaque, Union Carbide), polystyrene (PS, Lustrex HF-55, Monsanto), styrene-acrylonitrile copolymer (SAN, Kostil, Montecatini Edison) and polycarbonate (PC, Lexan 101, General Electric). Two sizes of glass beads (Ballotini CPO2) were used, with diameter ranges of 4–44 μ and 149–250 μ. Unless stated otherwise, glass beads referred to in this paper were the 4–44 μ size. Coupling agents were removed by burning and the beads were further cleaned by refluxing with isopropyl alcohol. The procedure for preparing compression molded specimens is described elsewhere.[4] Specimens were also injection molded using an Ankerwerk injection molding machine. All specimens were ASTM D-678 type 1. Filler content was determined by ashing small pieces of broken samples. Tensile tests (five replicates in each case) were performed using an Instron universal tester (crosshead speed 0.2 cm/min), at various temperatures and all strains were monitored with a strain gage extensometer. A special bending jig in the form of a quarter section of an ellipse was built for measurements of critical crazing strains of the unfilled polymers.[7,8] Such a form has a varying radius of curvature which yields a continuous spectrum of strains in a sample clamped to it.

## RESULTS AND DISCUSSIONS

The anticipated slope-discontinuity phenomenon was observed in the stress–strain curves of new systems of particulate composites comprising PMMA, PSF or PC filled with glass beads. Kambour[6] reports two strain values, 0.8 and 1.3 %, for the initiation of crazing in PMMA (data of different researchers). In the present work agreement with the 1.3 % value was found as will be shown below.

A summary of tensile and crazing characteristics for PMMA/glass bead composites is given in Table I as function of bead content and specimen thickness. An average value of 1.61 is found for $K$ which is well within the common range of $K$ values for glassy polymers. The average calculated critical strain for the onset of crazing in PMMA (Diakon MH-253) is $\epsilon_p' = 1.22\%$ by the slope-discontinuity method. Exactly the same value was found in a direct measurement on the unfilled polymer using the elliptical bending form. Two other PMMA polymers were studied with the bending form and values of 1.3 % (Resarit 840, Resart, W. Germany) and 1.4 % (Resarit 810, Resart, W. Germany) were found. These results show that the

TABLE I

Summary of tensile results and calculated crazing parameters for PMMA (Diakon MH-253)/glass bead (4–44 μ) composites

| $\phi$ | Thickness mm | $E_c$ kg/cm$^2$ | $\epsilon_D$ % | $\epsilon_p'$ % | $\sigma_D$ kg/cm$^2$ | $K$ | $E_c/E_p$ |
|---|---|---|---|---|---|---|---|
| 0 | 3.4 | 31,800 | — | 1.22[a] | — | — | 1.00 |
| 0.096 | 3.7 | 36,700 | 0.75 | 1.38 | 273 | 1.60 | 1.15 |
| 0.103 | 1.5 | 36,200 | 0.61 | 1.15 | 219 | 1.65 | 1.14 |
| 0.103 | 3.7 | 34,000 | 0.68 | 1.28 | 231 | 1.76 | 1.07 |
| 0.106 | 5.7 | 40,100 | 0.59 | 1.12 | 236 | 1.51 | 1.25 |
| 0.108 | 1.6 | 37,100 | 0.57 | 1.09 | 211 | 1.63 | 1.17 |
| 0.184 | 1.6 | 46,900 | 0.54 | 1.25 | 250 | 1.57 | 1.47 |
| 0.189 | 3.6 | 44,700 | 0.55 | 1.29 | 260 | 1.67 | 1.41 |
| 0.203 | 3.5 | 46,000 | 0.50 | 1.21 | 233 | 1.68 | 1.45 |
| 0.222 | 3.2 | 53,700 | 0.48 | 1.22 | 259 | 1.50 | 1.68 |
| 0.222 | 2.4 | 53,700 | 0.52 | 1.31 | 278 | 1.50 | 1.68 |
| 0.222 | 1.6 | 52,300 | 0.44 | 1.12 | 231 | 1.54 | 1.64 |
| 0.262 | 3.6 | 53,600 | 0.44 | 1.23 | 239 | 1.65 | 1.68 |
|  |  |  | Average | 1.22±0.09 | 244±22 | 1.61±0.08 |  |

[a] Direct measurement on the unfilled polymer with the elliptical bending form.

three PMMA types are characterized by critical strains for initiation of crazing which conform to the upper 1.3% value reported by Kambour.[6] Table I also shows that $\sigma_D$ is almost independent of the glass bead volume fraction (in agreement with previous results for other glassy polymers) and sample thickness in the ranges studied. An estimate of the onset stress for initiation of crazing in an unfilled polymer is given by $\sigma_p'$ where $\sigma_p' = K\sigma_D$. An average value of about 400 kg/cm$^2$ is calculated for PMMA from Table I. Comparison of this value with literature results is difficult due to the small number of reported values and their high scatter. In addition, Gotham[9] has shown that the critical stress is time-dependent and that a high time-dependency is exhibited by PMMA. However, by ranking PMMA and other polymers in accordance with their calculated $\sigma_p'$ values, a qualitative agreement is found with Gotham's ranking.

Results on two grades of polysulfone (PSF) are summarized in Table II. The table shows that the two grades have almost the same $K$ and $\sigma_D$ values. PSF P-1700 has a critical strain of $\epsilon_p' = 1.6\%$ as calculated from the slope-discontinuity curve data. This value is in excellent agreement with the value of 1.57% found for the unfilled polymer using the elliptical bending form. Kambour[6] reports a value of 2.5% for PSF and no other data are available from the literature. Table II also shows that $\sigma_D$ is practically independent of the glass bead volume fraction and sample thickness in the ranges studied.

TABLE II

Summary of tensile results and calculated crazing parameters for polysulfone/glass bead (4–44 μ) composites

| Grade | $\phi$ | Thickness mm | $E_c$ kg/cm$^2$ | $\epsilon_D$ % | $\epsilon_p'$ % | $\sigma_D$ kg/cm$^2$ | $K$ | $E_c/E_p$ |
|---|---|---|---|---|---|---|---|---|
| P-1710 | 0 | 3.3 | 25,100 | — | — | — | — | 1.00 |
| | 0.103 | 1.5 | 31,600 | 0.87 | 1.64 | 274 | 1.50 | 1.26 |
| | 0.122 | 3.0 | 33,000 | 0.73 | 1.45 | 249 | 1.51 | 1.31 |
| | 0.193 | 3.1 | 39,100 | 0.64 | 1.51 | 264 | 1.52 | 1.56 |
| | 0.193 | 1.9 | 44,100 | 0.57 | 1.36 | 253 | 1.35 | 1.76 |
| | 0.193 | 1.4 | 41,000 | 0.62 | 1.46 | 253 | 1.45 | 1.63 |
| | 0.207 | 3.2 | 39,500 | 0.63 | 1.54 | 265 | 1.56 | 1.57 |
| | 0.287 | 3.0 | 53,000 | 0.41 | 1.21 | 254 | 1.39 | 2.11 |
| | | | | Average | 1.45±0.14 | 259±9 | 1.47±0.08 | |
| P-1700 | 0 | 3.3 | 24,500 | — | 1.57[a] | — | — | 1.00 |
| | 0.203 | 3.3 | 40,000 | 0.67 | 1.63 | 268 | 1.49 | 1.63 |
| | 0.203 | 3.3 | 39,500 | 0.64 | 1.56 | 253 | 1.50 | 1.61 |
| | 0.203 | 1.5 | 40,500 | 0.67 | 1.62 | 280 | 1.47 | 1.65 |
| | | | | Average | 1.60±0.04 | 267±14 | 1.49±0.02 | |

[a] Direct measurement on the unfilled polymer with the elliptical bending form.

The low standard deviations found for $K$, $\sigma_D$ and $\epsilon_p'$ are also noteworthy and strengthen the theory and the quantitative approach to the slope-discontinuity phenomenon.

The authors have not been able to produce good compression molded samples of polycarbonate/glass bead composites. Compression molding of the unfilled polymer, however, is straightforward. Injection molding of PC/glass bead samples is easily performed but the samples produced are non-isotropic with preferred orientation in the flow direction. A summary of results on injection molded polycarbonate/glass bead composites is given in Table III. The table also shows data on PMMA composites produced by injection as well as compression molding. Unfilled PC samples were investigated with the elliptical bending form and a value of 1.6% was typically found for the critical strain of compression molded samples. No observable crazing was found in the injection molded, unfilled, PC samples up to 2.5% strain. The upper strain limit of the elliptical bending instrument. This comparison between compression and injection molded samples clearly shows the importance of orientation on crazing behavior. The values for PC and PMMA composites shown in Table III indicate that higher critical strains and stresses characterize oriented samples, in the direction of orientation, than compression molded, orientation-free, samples. Biaxial orientation, which improves the isotropy of the material, is sometimes recommended in practice for enhancing the resistance to crazing. Surprisingly, $K$ is found to be practically independent of the state of orientation. This unexpected finding requires additional work aimed at further assessing the physical meaning of $K$ ($K$ has been assumed to describe a stress concentration factor).

TABLE III

Summary of tensile results and calculated crazing parameters of injection and compression molded specimens. Samples comprising PC and PMMA (Diakon MH-253) filled with glass beads (4–44 μ)

| Process | Polymer | $\phi$ | $E_c$ kg/cm$^2$ | $\epsilon_D$ % | $\epsilon_p'$ % | $\sigma_D$ kg/cm$^2$ | $K$ | $E_c/E_p$ |
|---|---|---|---|---|---|---|---|---|
| Injection | PC | 0 | 23,700 | — | > 2.50[a] | — | — | 1.00 |
| | | 0.112 | 29,400 | 1.11 | 2.15 | 327 | 1.56 | 1.24 |
| | | 0.201 | 36,700 | 0.95 | 2.30 | 338 | 1.57 | 1.55 |
| Injection | PMMA | 0 | 32,400 | — | — | — | — | 1.00 |
| | | 0.108 | 37,800 | 0.62 | 1.25 | 252 | 1.63 | 1.17 |
| | | 0.184 | 49,800 | 0.72 | 1.66 | 356 | 1.51 | 1.54 |
| Compression | PMMA | 0 | 31,800 | — | 1.22[a] | — | — | 1.00 |
| | | 0.108 | 37,100 | 0.57 | 1.09 | 211 | 1.63 | 1.17 |
| | | 0.184 | 46,900 | 0.54 | 1.25 | 250 | 1.57 | 1.47 |

[a] Direct measurement on the unfilled polymer with the elliptical bending form.

A summary of crazing characteristics is given in Table IV for several polymers. The table shows very good agreement between critical strains calculated using the slope-discontinuity method and directly measured strains, using the elliptical bending form, for PMMA, PSF and PS. Somewhat poorer agreement is found for SAN copolymers. The table also summarizes the critical strains for the same polymers as reported in the literature (different grades, however) and compares these with the results of the present work. Gotham[9] measured critical stresses for the onset of crazing and found a time-dependent phenomenon whereby longer periods of time are required to initiate crazing as the stresses are decreased. In some instances, a stress threshold is found below which crazing will not show up even over very long periods of time. Gotham compares his polymers by using the stress value needed to initiate crazing at $10^5$ sec as a criterion for craze resistance. This time scale is unsuitable for comparing his results with the results of the present work based on stress–strain measurements. Using a crosshead speed of 0.2 cm/min, sample crazing and breakage occur after several tens of seconds which implies that a time scale of 100 sec would be appropriate for comparisons. The critical stresses needed to initiate crazing were read from Gotham's figures and are given in the last column of Table IV. As mentioned earlier $\sigma_p'(=K\sigma_D)$ has been suggested as an estimate of the critical stress needed to initiate crazing. Agreement between Gotham's 100 sec stresses, measured on unfilled polymers, with the slope-discontinuity criterion $\sigma_p'$ is surprisingly good for PMMA, PS, SAN and PC. Gotham's value for PSF is appreciably higher than the corresponding $\sigma_p'$ value calculated in the present work.

The effect of bead size was studied by comparing the results obtained using large glass beads, 149–250 μ, with those obtained using the standard 4–44 μ

TABLE IV

Summary of crazing characteristics (discontinuity, elliptical form and literature)

| Polymer | Trade name and grade (This work) | Critical strain $\epsilon p'$ (%) | | | $\sigma p'$ $=K\sigma_D$ kg/cm$^2$ | Gotham[9] $t=100$ sec kg/cm$^2$ |
|---|---|---|---|---|---|---|
| | | Elliptical form | Discontinuity | Literature reference | | |
| PMMA | Diakon MH-253 | 1.22 | 1.22 | 1.3,[6] 0.8–0.9[14] | 390 | 370 |
| PMMA | Resarit 810 | 1.40 | — | | | |
| PMMA | Resarit 840 | 1.30 | — | | | |
| PSF | Bakelite P-1700 | 1.57 | 1.60 | 2.5[15] | 400 | 700 |
| PS | Lustrex HF-55 | 0.71 | 0.72[13] | 0.75,[16] 1.0,[17]<br>0.35,[18] 0.36[19] | 170 | 190 |
| SAN | Kostil | 0.57 | 0.81[13] | 0.49[6] | 260 | 280 |
| PC | Lexan 101 | 1.60 | — | 1.80[6] | 520[a] | 580 |

[a] Injection molded sample.

beads. A summary of results on PMMA and PSF composites containing the large glass beads is given in Table V. The independence of $\sigma_D$ on $\phi$ is no longer true and $\sigma_D$ exhibits a significant decrease with increasing filler content. Since the calculated $\epsilon_p'$ values depend on $\phi$, the model as described in Figure 1 fails and the slope-discontinuity method becomes inapplicable for the larger glass beads. The stress concentration factor $K$ is again unaffected (similarly to the orientation effect discussed earlier) and attains numerical values similar to those describing small bead systems. As shown in Figure 1, $K$ does not actually depend on the slope-discontinuity characteristics and is determined by the moduli ratio, $E_c/E_p$ and $\phi$. $E_c/E_p$ values are practically independent of the bead size in the ranges studied and thus the values for $K$ are similarly independent. These conclusions are in agreement with Goodier's elastic analysis[10] according to which the maximum stress concentration due to an inclusion depends on the nature of the latter and on its geometrical form, but not on its absolute size or concentration.

The effect of temperature on the moduli, slope-discontinuity characteristics and parameters derived from them is summarized in Table VI. $E_c$ and $E_p$ decrease with increasing temperature and follow a similar temperature dependence. This is shown by the fact that the moduli ratio $E_c/E_p$ is almost independent of temperature (last column in the table). A direct conclusion is that the stress concentration factor $K$ (determined by $E_c/E_p$) is also non-temperature dependent and this is verified in the table. Examination of the role of temperature on the behavior of $\sigma_D$ shows clearly that the latter decreases rather markedly with temperature and a substantially linear relationship can be observed by plotting the data. These results for PMMA and PSF in the present work agree with previous results for SAN,[1] PMMA,[11]

TABLE V

Summary of tensile results and calculated crazing parameters for PMMA (Diakon MH-253) and PSF (P-1710) composites filled with glass beads (149–250 μ)

| Polymer | $\phi$ | Thickness mm | $E_c$ kg/cm$^2$ | $\epsilon_D$ % | $\epsilon_p'$ % | $\sigma_D$ kg/cm$^2$ | $K$ | $E_c/E_p$ |
|---|---|---|---|---|---|---|---|---|
| PMMA | 0 | 3.5 | 31,800 | — | — | — | — | 1.00 |
| | 0.091 | 3.5 | 36,200 | 0.70 | 1.27 | 252 | 1.60 | 1.14 |
| | 0.193 | 3.5 | 44,400 | 0.47 | 1.12 | 210 | 1.70 | 1.40 |
| | 0.275 | 3.5 | 51,900 | 0.33 | 0.95 | 150 | 1.75 | 1.63 |
| PSF | 0 | 3.2 | 25,100 | — | — | — | — | 1.00 |
| | 0.085 | 3.2 | 29,500 | 1.18 | 2.11 | 351 | 1.52 | 1.17 |
| | 0.192 | 3.2 | 35,600 | 0.57 | 1.35 | 214 | 1.67 | 1.42 |
| | 0.261 | 3.1 | 46,300 | 0.36 | 0.99 | 168 | 1.50 | 1.84 |
| PSF | 0.117 | 1.6 | 32,400 | 0.98 | 1.93 | 316 | 1.52 | 1.29 |
| | 0.298 | 1.7 | 47,500 | 0.53 | 1.60 | 247 | 1.59 | 1.89 |

TABLE VI

Effect of temperature on tensile properties and crazing characteristics. PMMA (Diakon MH-253), PSF (P-1710), glass beads (4–44 μ)

| Polymer | Temperature | $\phi$ | $E_c$ kg/cm² | $E_p$ kg/cm² | $\epsilon_D$ % | $\epsilon_p'$ % | $\sigma_D$ kg/cm² | $K$ | $E_c/E_p$ |
|---|---|---|---|---|---|---|---|---|---|
| PMMA | 24 | 0.103 | 34,000 | 31,800 | 0.68 | 1.28 | 231 | 1.76 | 1.07 |
| | 45 | 0.103 | 25,700 | 25,000 | 0.69 | 1.30 | 177 | 1.76 | 1.03 |
| | 65 | 0.103 | 22,600 | 21,000 | 0.67 | 1.26 | 151 | 1.75 | 1.08 |
| | 85 | 0.103 | 19,000 | 17,100 | 0.50 | 0.94 | 96 | 1.69 | 1.11 |
| PMMA | 24 | 0.203 | 46,000 | 31,800 | 0.50 | 1.21 | 233 | 1.68 | 1.45 |
| | 45 | 0.203 | 34,300 | 25,000 | 0.54 | 1.32 | 190 | 1.77 | 1.37 |
| | 65 | 0.203 | 29,200 | 21,000 | 0.48 | 1.18 | 144 | 1.75 | 1.37 |
| | 85 | 0.203 | 24,000 | 17,100 | 0.42 | 1.02 | 100 | 1.73 | 1.40 |
| PSF | 24 | 0.207 | 39,500 | 25,100 | 0.63 | 1.54 | 265 | 1.56 | 1.57 |
| | 50 | 0.207 | 37,200 | 24,500 | 0.62 | 1.51 | 231 | 1.61 | 1.52 |
| | 100 | 0.207 | 36,000 | 23,000 | 0.52 | 1.29 | 205 | 1.57 | 1.56 |
| | 150 | 0.207 | 34,000 | 20,400 | 0.37 | 0.92 | 125 | 1.47 | 1.67 |

PPO[4] and PS.[12] The rate of decrease of the critical stress ($K\sigma_D$) for initiation of crazes with temperature is an intrinsic property of the polymer and the following series is obtained in a descending order (absolute slope values): PS 4.37,[12] PMMA 3.75,[9] PMMA 3.44 (this work), SAN 1.56,[1] PSF 1.38 (this work) and PPO 0.4 (kg/cm²)/°C.[4] A very good agreement is found for PMMA between Gotham's results (unfilled polymer, critiacl stress at 100 sec) and the present results (stress-discontinuity method, filled polymer). It is likely from the data that the temperature sensitivity of the critical stress increases with increasing the polymer chain flexibility.

The calculated critical strain $\epsilon_p'$ seems to be less temperature dependent than $\sigma_D$. $\epsilon_p'$ does not follow a linear relationship with temperature and it apparently begins to drop significantly upon approaching the polymer glass transition temperature.[12]

In conclusion, the slope-discontinuity method has been further verified regarding its reliability for the prediction of crazing characteristics provided that orientation-free samples filled with the proper size and concentration of clean glass beads are used. Over the experimental ranges studied, the stress concentration factor $K$ has been shown to be independent of bead content, bead size and sample orientation and temperature for composites having glassy thermoplastic matrices. $K$ is also known to be independent of the bead surface treatment (coupling agent) while the slope-discontinuity method is actually inapplicable in coupled systems.

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