Influence of processing and subsequent annealing on the ductile-brittle transition in PC

M.C.G. van Zuilichem
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Coaches:

Dr.Ir. L.E. Govaert
Ir. T.A.P. Engels

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Introduction ductile/brittle failure

Failure of a material can happen in several ways. A stress-strain diagram gives a good indication about the way failure occurs. Such a diagram can be obtained from a tensile-test. In a tensile-test a specimen is subjected to a constant strain-rate. When the stress which is measured is plotted versus the strain a lot of mechanical properties can be seen directly. The image below shows an example of a stress-strain diagram for a tensile-test. With increasing strain the stress increases linear. During this linear viscoelastic part of deformation the material deforms elastic (after the stress is removed, the specimen returns to its original state). At a certain strain the line leaves linearity, this part is the non-linear viscoelastic part. When the stress reaches a critical value, the yield stress, the deformation changes from elastic to plastic. The result of plastic deformation is permanent strain. The stress decreases until failure occurs at a much larger strain. The energy which is required to break the material can be obtained by integrating the area under the stress-strain curve. The amount of energy is an indication for the toughness.[1]

![Stress-strain curve of PC.](image)

Figure 1: Stress-strain curve of PC.

The macroscopic response in a tensile test can be very different, even for one material. Failure can occur in more than one way. The image above shows an increasing stress with increasing strain. When the yield stress is reached plastic deformation occurs. The plastic deformation causes a neck to grow in the middle of the specimen. This local plastic zone grows over the entire length of the specimen. The stress remains constant until the neck has grown over the entire length after which the stress starts to increase again until the material breaks. This deformation mechanism is called ductile failure. The image below shows a different stress-strain diagram. Again the stress increases with increasing strain which generates elastic deformation but this time the material breaks at a critical stress value. Necking doesn’t occur and there is no plastic deformation on macroscopic scale. This type of failure is called brittle failure.
so which deformation mechanism is favored over the other? What causes a material to fail ductile or brittle? To understand this we have to look more closely to brittle failure. Macroscopically it looks like no plastic deformation occurs but when we look at the material on microscopical scale it appears plastic deformation does occur. Right before the material breaks a material dependent number of so-called "crazes" are formed. Long elongated voids are created within the material which appear visually to be much like conventional cracks. They differ from cracks, however, in that the broad faces of the crazes are spanned by a great many elongated fibrils that have been drawn from the polymer as the craze opens. These fibrils can handle stress so elsewhere in the material other crazes can be formed. When the stress increases further the fibrils are brought to rupture and the crazes transform into fatal cracks. The deformation is so strongly localized that the material fails in a brittle way. Ductile failure has moderate localization and the neck can grow macroscopical.
Polycarbonate is a typical ductile polymer but the deformation mechanism can be influenced and changed to brittle failure. A factor that influences the deformation mechanism is the stress state, i.e. the loading geometry. To investigate the influence of the stress state a four-point bending test was done by I. Narisawa. In that test samples with a v-notch were used. V-notches influence the fracture behavior in a few ways. The V-notch acts as a stress magnifier and also enhances the rate of strain near the notch. Therefore the notch speeds up failure of the specimens. Another important influence of the notch is that it changes the stress system at the notch. Where the stress normally is uniaxial, the notch changes this system to a biaxial or triaxial one. Which system is chosen depends on the dimensions of the sample near the notch. The change from a uniaxial to a triaxial stress state has a big influence on the deformation mechanism. The test Narisawa did will show what this influence is. To obtain more insight, some of his work will be summarized below.

Shear deformation dominate the deformation mechanism that is observed in Polycarbonate. Under extreme conditions crazing can dominate the deformation mechanism as well. Crazing leads to brittle failure while shear deformation produces ductile failure. During the test the influence of the stress state on the deformation and fracture behavior ahead of the notch tip was examined. Two modes were examined: a tensile mode (mode I) and a shear mode (mode II). During the test both stress states were examined as well a combined stress state of both modes. The deformation mechanisms that were observed were different for the different stress states. Mode I resulted in brittle failure. As predicted this brittle failure was caused by the nucleation of an internal craze. While the stress state was slowly changed from mode I to mode II, the deformation mechanism changed from brittle failure to a mixed brittle-ductile failure. Further, the internal craze disappeared when the mode II component reached a critical value. Instead of an internal craze, stable crack growth appeared at the notch tip. The influence of the stress state obviously is big, but why do different stress states result in different deformation mechanisms?

To understand why the material breaks in mode I failure we first have to understand how crazes are formed. In the tensile mode the stress will increase with increasing strain. Craze initiation starts when plastic deformation occurs near a stress concentration. This plastic deformation zone will cause a triaxial stress state because it grows rapidly while the surrounding material deforms elastic. There is a big difference between uni- and biaxial stress states on the one hand, and triaxial stress states on the other hand. Uni- and biaxial stress states can grow stable because material can flow from one or two directions to the direction(s) that needs the material to deform. A triaxial stress state can not grow stable because material has to flow to all directions. There is no direction where material can flow from. The material is subjected to a hydrostatic stress in all directions. When this hydrostatic stress reaches a critical value micro voids are formed in the plastic zone.

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1 T. Kuriyama, I. Narisawa, Fracture behavior of ductile polymer under mixed mode loading, submitted
The ligaments between these micro voids are strengthened enormously and form the fibrils. With increasing strain the crazes will grow in both length and width due to the growth of the fibrils and the creation of more micro voids and fibrils. Eventually the fibrils will fail and the craze transforms into a fatal crack. So the initiation of a crack is affected by two factors: (1) the forming of a local plastic zone (2) the forming of a micro void in the plastic zone.

The forming of a local plastic zone is dependant on the post-yield behavior: the behavior of the yield stress. The forming of a micro void requires the hydrostatic stress to reach a critical value; the critical hydrostatic stress. This critical hydrostatic stress is dependant on nucleations and the entanglement density. The value of the hydrostatic stress will be highest near nucleations while a higher density gives more resistance against the forming of micro voids.

When the stress state was changed from the mode I to mode II a difference in crack growth was observed. Mode II showed stable crack growth, leading to ductile deformation, while mode I showed unstable crack growth, resulting in brittle failure. When a mixed mode was applied the results were dependent on the ratio of shear to tensile components. While this ratio was larger than 0.67 stable crack growth occurred and the results were quiet similar to that of pure mode II failure. However, when the ratio was lowered under 0.3, two things happened. One was that the value of the load peak was lowered and the other thing was that both brittle and ductile failure occurred. The local deformation shows more than one types in this transition zone. Not only does shear deformation occur, there also formed a internal craze ahead of the localized yield zone. The direction in which the craze was formed was perpendicular to the maximum principal stress direction. At the notch tip appears a third deformation type, a ductile crack is being initiated. While the load was increased it appeared ductile deformation took over brittle failure because the ductile crack propagated in a stable fashion and the local shear bands initiated ahead of the internal craze at the same time. Therefore brittle failure was suppressed and the fracture behavior again approached to that of the mode II loading case.

Figure 3: triaxial stress state.
The author concluded that the hydrostatic stress produced by the stress triaxiallity ahead of a localized yield zone nucleates the internal craze. The stress triaxiallity ahead of a crack tip decreases with an increasing applied shear stress. The change of triaxiallity in the mixed mode is shown in the figure above. The stress triaxiallity is plotted against the ratio of an applied stress components. The material property is plotted as a horizontal band. The figure shows that increasing stress triaxiallity, which generates hydrostatic stress, results in a transition from ductile to brittle failure and is therefore an important factor to determine the fracture behavior of ductile polymers under mixed mode loading.
Conditions that influence the ductile-brittle transition

The ductile-brittle transition has been investigated for a few decades now. In the 70s research on polycarbonate was done by D. Legrand. Polycarbonate is an exceptional thermoplastic because unlike most thermoplastics it shows ductile failure when it is subjected to a uniaxial tensile test. Under certain conditions however, polycarbonate can fail in a brittle way. To understand this transformation a lot of different conditions were inquired by Legrand. One situation in which polycarbonate deforms in a brittle manner is when a sample is annealed for a long time at a temperature just below the glass temperature. The sample shows a lot less Izod impact strength compared to normal samples. There is a certain annealing time after which the deformation mechanism changes from ductile to brittle. At the same time the energy that is required to break the material is reduced enormously. Results from the annealing experiment show that there is a energy level for ductile failure and a much lower energy level for brittle failure. This drop in energy is the ductile-brittle transition. The results from this experiment are visible in the figure below.

![Impact data as function of annealing time](image_url)

**Figure 5:** Impact data as function of annealing time, from [3].

During these experiments certain conditions were altered. An increase in molecular weight moved the ductile-brittle transition to a longer annealing time. Another condition that was altered is the annealing temperature. The results of this test is that the higher the annealing temperature is, the faster the ductile-brittle transition will occur. Finally Legrand changed the molding pressure to investigate the influence of production conditions. He did not get good results on this test but another production condition, the molding temperature, might be interesting to investigate. Legrand’s conclusion was that most thermoplastics deform in a brittle manner because fracture or crazing prevent the material from yielding. He also stated that fracture or crazing could be preventing by lowering the yield stress under a critical stress for fracture. Since the value of the yield stress is affected by the strain rate and the temperature this adds another two conditions that influence the ductile brittle failure. Summarizing we found five conditions that might influence the deformation mechanism.

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D. Legrand, Crazing, Yielding and Fracture of Polymers. I, Ductile Brittle Transition in Polycarbonate, submitted
• Subsequent annealing (higher temperature gives faster transition)
• Molecular weight (increase in molecular weight gives faster transition)
• Molding temperature
• Strain rate (increase in strain rate gives increase in yield stress and therefore faster transition)
• Temperature (increase in temperature gives decrease in yield stress and therefore slower transition)
Outline of investigation

We have an adequate constitutive model which accurately describes plastic deformation of glassy polymers, however the model only describes ductile failure. As suggested brittle failure is dependent on hydrostatic stress. To include brittle failure in the constitutive model we will start with an investigation of the ductile-brittle transition. We will try to find the critical hydrostatic stress for the ductile-brittle transition. Therefore we first have to investigate the conditions that influence the ductile-brittle transition. These conditions were pointed out in the previous chapter. A summary of the constitutive model is given below.

Constitutive approach, from Klompen [4]

The basis of any 3D constitutive model for solid polymers is the split of the total stress in two contributions.

\[ \sigma = \sigma_s + \sigma_r \]  

(1)

here \( \sigma_r \) denotes the strain hardening contribution that is attributed to molecular orientation of the entanglement network, modeled using a Neo-Hookean elastic expression. The contribution \( \sigma_s \) accounts for the rate-dependent plastic flow response, attributed to intermolecular interactions on a segmental scale, and represented by a non-linear Maxwell model. Table 1 summarizes the equations of the constitutive model.

<table>
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<th>Description</th>
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<tr>
<td>( \sigma = \sigma_s + \sigma_r )</td>
<td>Total stress split into strain hardening and rate-dependent plastic flow components</td>
</tr>
<tr>
<td>( \sigma_r = G_r \tilde{B}_e^d )</td>
<td>Strain hardening contribution</td>
</tr>
<tr>
<td>( \sigma_s = K(J - 1) + G \tilde{B}_e^d )</td>
<td>Rate-dependent plastic flow component</td>
</tr>
<tr>
<td>( J = \text{Jtr}(D) )</td>
<td>Trace of deformation rate tensor</td>
</tr>
<tr>
<td>( \tilde{B}_e = (D^d - D_p) \cdot \tilde{B}_e + \tilde{B}_e \cdot (D^d - D_p) )</td>
<td>Effective deformation gradient tensor</td>
</tr>
<tr>
<td>( D_p = \frac{\sigma_s^d}{2 \text{Jtr}(\sigma_s, \tau, p, S)} )</td>
<td>Plastic deformation rate tensor</td>
</tr>
<tr>
<td>( \tilde{\tau} = \sqrt{\frac{1}{2} \text{tr}(\sigma_s^d \cdot \sigma_s^d)} )</td>
<td>Effective stress tensor</td>
</tr>
<tr>
<td>( \dot{\tilde{\tau}}_p = \sqrt{2 \text{tr}(D_p \cdot D_p)} )</td>
<td>Plastic deformation rate tensor</td>
</tr>
<tr>
<td>( \eta(\tilde{\tau}, T, p, S) = \eta_{0,r}(T) \frac{\tilde{\tau}/\tau_0}{\sinh(\tilde{\tau}/\tau_0)} \exp \left( \frac{\mu p}{\tau_0} \right) \exp(S(t, \tilde{\tau}_p)) )</td>
<td>Strain hardening contribution</td>
</tr>
<tr>
<td>( \eta_{0,r} = \eta_{0,r,\text{ref}} \exp \left( \frac{\Delta U_a}{R} \left( \frac{1}{T} - \frac{1}{T_{\text{ref}}} \right) \right) )</td>
<td>Reference strain hardening rate</td>
</tr>
<tr>
<td>( \tau_0 = \frac{RT}{V_s} )</td>
<td>Reference stress</td>
</tr>
<tr>
<td>( S(t_{\text{eff}}(t, T, \tilde{\tau}), \tilde{\tau}<em>p) = S_a(t</em>{\text{eff}}(t, T, \tilde{\tau})) \cdot R_a(\tilde{\tau}_p) )</td>
<td>Strain hardening rate</td>
</tr>
<tr>
<td>( S_a(t_{\text{eff}}(t, T, \tilde{\tau})) = c_0 + c_1 \cdot \log(t_{\text{eff}}(t, T, \tilde{\tau}) + t_a) )</td>
<td>Strain hardening rate</td>
</tr>
<tr>
<td>( R_a(\tilde{\tau}_p) = \frac{(1 + (\alpha_0 \exp(\tilde{\tau}_p))^r)^{\frac{\tau_0 - 1}{rt}}}{(1 + \alpha_0 t_a)^{\frac{\tau_0 - 1}{rt}}} )</td>
<td>Strain hardening rate</td>
</tr>
<tr>
<td>( t_{\text{eff}}(t, T, \tilde{\tau}) = \int_0^t a_T^{-1}(T(t')) a_{\sigma}^{-1}(\tilde{\tau}(t')) dt' )</td>
<td>Effective time</td>
</tr>
<tr>
<td>( a_T(T) = \exp \left( \frac{\Delta U_a}{R} \left( \frac{1}{T} - \frac{1}{T_{\text{ref}}} \right) \right) )</td>
<td>Temperature dependence</td>
</tr>
<tr>
<td>( a_{\sigma}(\tilde{\tau}) = \frac{\tilde{\tau}/\tau_0}{\sinh(\tilde{\tau}/\tau_0)} )</td>
<td>Strain hardening rate</td>
</tr>
<tr>
<td>( \tau_0 = \frac{RT}{V_s} )</td>
<td>Reference stress</td>
</tr>
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Table 1: Summary of the constitutive model employed [?, ?]
Under isothermal conditions the nonlinearity of the model is completely governed by a stress-, pressure- and state-dependent viscosity \( \eta \), defined as [4]:

\[
\eta(\bar{\tau}, p, S) = \eta_0, r \frac{\bar{\tau} / \tau_0}{\sinh(\bar{\tau} / \tau_0)} \exp \left( \frac{\mu p}{\tau_0} \right) \exp \left( S(t, \bar{\gamma}_p) \right)
\]  

(2)

The parameter \( \eta_{0, r} \) denotes the zero-viscosity for the completely rejuvenated state. The part marked (I), with \( \bar{\tau} \) the equivalent stress, represents the stress dependent part of the viscosity governed by the parameter \( \tau_0 \). Part (II) is the pressure dependent part, governed by the pressure dependence \( \mu \) and the hydrostatic pressure \( p \). The combination of part (I) and (II) gives a rate dependent plastic flow response according to the pressure modified Eyring flow expression. Finally, part (III) represents the dependence of the viscosity on the state of the material expressed by the parameter \( S \).

In time and upon deformation two processes occur, a) physical aging, leading to an increase in yield stress, and b) strain softening, leading to a decrease in yield stress. In our model approach it is assumed that these two are fully decoupled, or:

\[
S(t, \bar{\gamma}_p) = S_a(t) \cdot R_{\gamma}(\bar{\gamma}_p)
\]  

(3)

The parameter \( S_a \) can be regarded as a state parameter that uniquely determines the current state of the material. It is the evolution of this parameter that enables us to capture changes in mechanical properties with physical aging.

The value of \( S_a \) can in principle be determined directly from the yield stress measured in a uniaxial tensile test according to: or vice versa: where is the yield stress measured at a strain rate, is the strain hardening modulus and the draw ratio at the yield point.

This constitutive model describes plastic deformation accurately, however the model only describes ductile failure. The next thing we want to do is implement brittle failure and the ductile-brittle transition. Since the stress triaxiality, which generates hydrostatic stress, is an important factor to determine the fracture behavior of ductile polymers we have to find the critical hydrostatic stress which defines the ductile-brittle transition. There are several properties that influence the ductile-brittle behavior. After we look at these conditions we can find a suitable experimental method to investigate the transition.
Experimental investigation

To investigate the ductile-brittle transition in polycarbonate a lot of experiments have to be done. It’s important to keep the big picture in mind. The results from the experiments are going to be used to add an extra part to the constitutive model so the results have to be very reliable. The first plan is to do the experiment under moderate conditions.

The most important thing to obtain reliable results is to keep errors and standard deviations very low. To achieve this the strain rate during the experiments will be low; 10 mm/s. There is a limit on the data points per second so a high strain rate might generate too little data for accurate results. Therefore the strain rate will be low. Another important thing we must keep in mind is that the experiments have to be translated to numerical simulations easily. That’s why the notch is round instead of v-shaped. A round notch will give less localization of the plastic zone but can be implemented in a numerical model far easier. The notch used in the first experiment has a radius of 1.5 mm.

The first experiment will be an investigation on subsequent annealing. When a material is "quenched" (quickly cooled) from the rubber state, the material will have a relatively high specific volume. In the stress-strain curve for such a material the stress will not drop after the yield stress is reached. Since the material is not in thermodynamical equilibrium the specific volume will decrease in time which is called physical ageing. The density of the product will increase, which will increase the yield stress. This influence is shown in the stress-strain curve below.

![Figure 6: True stress-strain curves measured in uniaxial compression on PC in the quenched and annealed state (1 week, 120°C).](image)

At high strain something remarkable happens: the influence of the physical ageing (thermal history) is removed with increasing plastic deformation. The drop of the stress after the yield stress is called strain softening and this strain softening is the reason the thermal history is removed. The fact that the yield stress increases due to physical ageing also means that this property can be influenced with heat treatments. One of these heat treatments is annealing. The material is heated to a temperature below the glass temperature. This heating speeds up the physical ageing and so increases the yield strength and strain softening. Annealing has a negative influence on the toughness of a material. The increase in strain softening causes the deformation behavior to be a
lot more unstable. Even very small stress-concentrations will lead to the initiation of local plastic zones. Another negative influence on the toughness of polycarbonate is that the increase of yield stress and strain softening causes the strain to be higher before the stress reaches the level of the yield stress. The plastic zone has to deform further before stable growth is possible. So annealing has a negative influence on the toughness of polycarbonate because it will make the deformation more unstable which eventually will lead to brittle failure.

First experiment: annealing

The samples of Lexan polycarbonate used for the annealing experiment have a length of 110 mm, a width of 10 mm and a thickness of 5 mm. The notch was created with a standard milling operation and has a radius of 1.5 mm. The specimens with a notch were heated in an oven at 120 degrees Celsius together with normal specimens without a notch which will be used as reference. After a half hour the first two specimens were taken out together with two normal specimens. Specimens were taken out at 1 hour, 3 hours, one day, two days, four days and a week, each time in couples and each time 2 notched samples and 2 normal samples. The specimens were subjected to a uniaxial tensile test with constant strain rate. The results of the tensile tests are visible in the image below. The horizontal axis shows the annealing time while the vertical axis shows the yield stress. The normal samples clearly give a linear connection between the yield stress and the logarithmic time. The notched samples also show an increase in yield stress.

![Figure 7: a)Results annealing experiment: stress-strain curves. b)Results annealing experiment: stress vs strain, different annealing time.](image-url)

Unfortunately the ductile-brittle transition is not reached after a week of annealing. These results are a bit disappointing. The experiment strategy has to be changed and the focus is changed from moderate conditions for reliable experiment data to big results because that is more interesting for this project. For the remainder of the experiment different samples will be used. The notches are changed from a round form to a V-shape. This will increase the localization of the plastic zone which will speed up our investigation of the ductile-brittle transition.
Second experiment: higher strain rate

The first condition we are going to change is the strain rate. An increase in strain rate will give a higher yield stress which will lead to a more unstable deformation. The V-notch has a radius of 0.45 mm, a top angle of 35 degrees and the depth of the notch is 4 mm. The samples are tested at different temperatures. The experiment starts at room temperature. Three samples are tested at a strain rate of 10 mm/s and 3 other samples are tested at a strain rate of 1000 mm/s. After this the temperature is lowered to zero degrees and again 6 samples are tested. The temperature will be lowered with steps of 20 degrees and at every step 6 samples are tested until the temperature of -100 degrees is reached. The results of this experiment are visible in the image below. The vertical axis shows the energy that is required to break the material, the horizontal axis shows the temperature from -100 degrees up to room temperature. The blue line defines the strain rate of 1000 mm/s while the red line defines the strain rate of 10 mm/s.

![Figure 8: Different strain rates.](image)

As was pointed out by Legrand the lines consist of two energy levels which are reasonable linear and a steep drop from the high to the low level at a certain temperature, this is the ductile-brittle transition. For both strain rates the ductile-brittle transition was found within the reach of the temperature. When we compare the two lines it is obvious the ductile-brittle transition takes place at a much lower temperature for the lowest strain rate. The difference in temperature is 60 degrees. As predicted the yield stress is higher for the highest strain rate but when we look at low temperatures for the high strain rate something awkward happens.
Instead of a linear line for the brittle failure there seems to be an extra drop in the energy level at -80 degrees. A look at the cross sections for the samples at -80 degrees gives an extra indication that an extra transition took place. The samples with a much lower energy level show a very smooth surface. It looks like the samples were cut with a knife. Cross sections for every temperature step are shown below. At 80 degrees both the normal brittle and the “smooth” brittle samples are shown to make the difference visible. This extra transition might be interesting to investigate in another project.

![Cross sections for every temperature.](image)

**Figure 9:** Cross sections for every temperature.

Third experiment: Processing history

The next condition that is going to be examined is the processing history, more specific, the molding temperature. Samples are produced at 120 degrees Celsius and at 30 degrees Celsius. Again samples with a V-notch with a radius of 0.45 mm and a depth of 4 mm are used. The samples again will be tested at different temperatures beginning with room temperature. Three samples that were produced at 120 degrees and three samples that were used at 30 degrees are tested at every temperature. Further both types of samples are tested at different strain rates to investigate if there is a difference. The temperature will be lowered to zero degrees and the same amount of tests will be performed. The temperature will be lowered with steps of 20 degrees until the temperature of -100 degrees is reached. The results of this experiment are visible in the images below. The vertical axis shows the energy that is required to break the material while the horizontal axis shows the temperature from -100 degrees up to room temperature. The blue line defines the samples produced at 30 degrees while the red line defines the samples produced at 120 degrees. The picture to the left shows a strain rate of 10 mm/s while the picture to the right shows a strain rate of 1000 mm/s.
Figure 10: a) Energy line for both molding temperatures tested at a strain rate of 10 mm/s. b) Energy line for both molding temperatures tested at a strain rate of 1000 mm/s.

The results from this experiment are a bit disappointing. There is no real difference between the two temperatures, especially at a high strain rate. For both molding temperatures the ductile-brittle transition occurs at virtually the same temperature. The only difference is that for both the high and the low strain rate the variation at the ductile-brittle transition is bigger for the higher molding temperature (120 degrees). Concluding the molding temperature has little influence on the ductile-brittle transition.
Fourth experiment: Molecular weight

According to Legrand, the molecular weight plays a role in the ductile-brittle transition. Legrand used different molecular weights in his annealing experiment and concluded that a higher molecular weight resulted in a longer annealing time before the ductile-brittle transition occurred. In this fourth experiment the influence of the molecular weight on the ductile-brittle transition will be tested for normal tensile tests. Like the two experiments previously done samples with a V-notch (radius 0.45 mm and depth 4 mm) are used. The two types of Lexan polycarbonate used are Lexan-141 and Lexan-101. Lexan 101 having the higher molecular weight. The complete range, starting at room temperature and going down to -100 degrees, will be tested with three samples at every temperature. Again temperature-steps of 20 degrees are made. The strain rate is 1000 mm/s for this experiment. The results of this experiment are visible in the picture below.

![Figure 11: Different molecular weights at a strain rate of 1 m/s.](image)

The results show quite a difference for both molecular weights. Like Legrand predicted the samples with the higher molecular weight required a lower temperature for the ductile-brittle transition. Both ductile-brittle transitions were found within the reach. The D-B transition for Lexan 101 is about 20 degrees lower than that of Lexan 141. It is also very obvious that Lexan 101 has a higher yield stress than Lexan 141.

Fifth experiment: Annealing

The first annealing experiment was somehow not very satisfying. That's why we decided to do this experiment over searching for big results. We use the V-notch from the 2nd to the 4th experiment and we use a much higher strain rate. This will enhance errors and will be negative for the reproducibility but finding big effects is more important. The strain rate will be 1000 mm/s and the V-notch has a radius of 0.45 mm and a depth of 4 mm. The samples are heated in an oven at 120 degrees Celsius and are taken out after six days. The first three samples are tested at room temperature and according to the results the temperature will be lowered or increased. The first three samples showed smooth brittle fracture so it appeared we had already reached the ductile brittle transition. To check this the temperature was lowered to 0 degrees and after that to -20
degrees. Because we only found smooth brittle fracture we decided to increase the temperature to 40 degrees and keep increasing the temperature by 20 degrees until the ductile-brittle transition was found. The results from this experiment are shown in the figure below.

![Figure 12: Results annealing experiment.](image)

The result is one linear line and thus one energy level. Together with the fact that all cross sections looked smooth brittle the conclusion is that the ductile-brittle transition is not found for this experiment. In fact, by changing the experimental conditions to find big effects, we ended up far beyond the ductile-brittle transition and so a big effect was found. Another big difference that was found was the yield stress for this experiment. The difference with the unannealed energy line for a strain rate of 1000 m/s was 11 MPa.
Conclusion and recommendation

Five different conditions that could influence the ductile-brittle transition were investigated. The results varied a lot for the five conditions. The molding temperature, on one hand, barely showed any influence on the ductile-brittle transition, while subsequent annealing showed a huge change in fracture behavior, even moving the ductile-brittle transition beyond the borders of the experiments done. The other three conditions, molecular weight, strain rate and temperature, also had quiet some impact on the fracture mechanic that occurred. Molecular weight showed a difference of 20 degrees in the ductile-brittle transition temperature while multiplying the strain rate by a factor 100 resulted in a shift of 60 degrees of the ductile-brittle transition temperature. To implement a new part in the constitutive model regarding brittle failure we now know which conditions should be investigated more closely. The experiments that have been done showed big standard deviations and therefore a lot more samples have to be tested at every temperature or strain rate. Another interesting subject to be tested are the smooth surfaces that occurred at very low temperatures for high strain rates and during the second annealing experiment. It looked like there is another transition to a lower energy level. This transition obviously has an influence on the fracture mechanic and therefore might be interesting to investigated further.
Bibliography


