Yielding of Polymers Filled with Large-Diameter Particles

ARTHUR P. METZNER

Code 4410G, Naval Surface Warfare Center, Indian Head Division, Department of the Navy, 101 Strauss Avenue, Indian Head, Maryland 20640-5035

ABSTRACT: The yield stress of a polymer filled with rigid filler particles much larger than 5 μm is affected by the single-particle debonding stress at large volume fractions of filler. This relationship holds up to a limiting modulus based on the size of the polymer chains of the binder. An elastic yield energy, or resilience, is defined. Within the limits of the yield stress equation, this yield energy is constant over a wide range of strain rates and temperatures at high volume fractions of filler. © 2002 Wiley Periodicals, Inc. J Appl Polym Sci 85: 455–466, 2002

Key words: composites; mechanical properties; fillers; yielding; adhesion

INTRODUCTION

Polymers are frequently blended with rigid fillers such as talcum, calcium carbonate ($CaCO_3$), and mica to improve their mechanical properties. If an energetic filler is used, such as ammonium perchlorate or cyclotrimethylene trinitramine (RDX), a rocket motor propellant or plastic-bonded explosive can be created.

When the filler particles are very small, the debonding strength of the composite may exceed the strength of the polymer.[1] For spherical particles, this reinforcement[2] may occur with particles as large as 5 μm. For polymers filled with poorly adhering spherical particles much larger than 5 μm, debonding[3] of the filler particles causes the material to yield, lowering its strength. Therefore, a completely different relation is needed to describe the yield strength of polymers filled with large ($\geqslant$5 μm) particles. A number of mathematical descriptions[3–9] of the debonding of large filler particles have been published. These allow the debonding stress to be described in terms of the modulus and Poisson's ratio of the binder, the work of adhesion between the binder and the filler, and the particle size of the filler. However, not all the equations are in agreement.

It is shown here that the debonding stress is affected by a limiting modulus. Also, the debonding process can be characterized by the elastic energy expended to deform the filled polymer to the debonding stress. Within limits, this energy is independent of the strain rate and temperature of the sample.

BACKGROUND

One of the first debonding stress relations was derived by Nicholson.[4] A small, rigid sphere, surrounded by a much larger sphere of an elastic matrix material, is debonded by a uniform radial stress applied at the outer surface of the matrix sphere. Under this triaxial loading, the debonding stress is

$$\sigma_d = \sqrt{\frac{4(\gamma)(E)(1 + \nu)}{9r(1 - \nu)^2}} \qquad (1)$$

where γ is the energy absorbed in debonding a unit area of the interface, E is the elastic modulus of the matrix sphere, ν is Poisson's ratio of the matrix sphere, and r is the size of the rigid filler particle.

A slightly different debonding relation was derived by Gent.[5] In this analysis, a small particle in uniaxial tension in an elastic matrix has a small debonded region already existing. The stress required to make this defect grow and debond the particle is

$$\sigma_d = \sqrt{\frac{4\pi(\gamma)(E)}{3r[\sin(2\Theta)]}} \qquad (2)$$

where Θ is the angle from the center of the particle to the boundaries of the debonded region.

Another relation for debonding stress was derived by Pukanszky and Voros[6] for uniaxial tension:

$$\sigma_d = -\sigma_T + \frac{2(\gamma)G^m 1}{C_1 \sigma_T r}$$

where σ_T is the thermal stress applied to the particle, G^m is the shear modulus of the matrix, and C_1 is a constant. Because

$$G^m = \frac{E}{2(1 + \nu)}$$

their expression may be written

$$\sigma_d = -\sigma_T + \frac{(\gamma)(E)1}{C_1(1 + \nu)\sigma_T r} \qquad (3)$$

An alternate equation has been proposed for when σ_T values are very small:

$$\sigma_d = -\frac{\sigma_T}{2} + \sqrt{\frac{2(\gamma)G^m}{C_1 r}}$$

Therefore, in this case

$$\sigma_d = -\frac{\sigma_T}{2} + \sqrt{\frac{(\gamma)(E)}{C_1(1 + \nu)r}} \qquad (4)$$

Equation (4) is similar to eq. (1) when σ_T is 0, except for the terms of Poisson's ratio.

Pukanszky and Voros[6] predicted debonding stresses for $CaCO_3$ particles in poly(vinyl chlo-

Table I   Relations from Ref. 6 Debonding Predictions

Matrix	C_2	n	R^2
PVC	26.413	0.281	0.9898
PP	17.267	0.2154	0.9729
LDPE	4.5965	0.2781	0.987

ride) (PVC), polypropylene (PP), and low-density polyethylene (LDPE) matrices for three $CaCO_3$ particle sizes: 60, 3.6, and 1.3 μm.

The estimated debonding stresses can be fit to the following form:

$$\sigma_d = \frac{C_2}{r^n}$$

from the ref. 6 estimates with the Microsoft Excel trendline function. The resulting values for C_2 and n are given in Table I, along with the R^2 values for the fit. The values of n are different from the expected values of 1 and 0.5 , suggesting a theoretical dependence on particle size close to 0.25.

A relation for the yield stress of a composite, which is similar in form to the Nicholson relation, was derived by Vollenberg et al.[3] It does not contain any dependence on Poisson's ratio. The yield strength of a polymer containing a large volume fraction of filler should be related to the debonding stress if the debonding stress is lower than the yield stress of the polymer alone. The Vollenberg relation is

$$\sigma_y = \frac{\sigma_T}{a} + \frac{k}{a}\sqrt{\frac{(\gamma)(E)}{r}} \qquad (5)$$

where σ_y is the yield stress of the composite, a is a stress concentration factor, and k is a nearly constant parameter that has a slight dependence on the moduli of the polymer and the filler. The authors presented data for polystyrene and PP composites filled with glass beads that showed that σ_y was proportional to $r^{(-0.5)}$.

Another method for calculating the yield strength involves calculating the yield strain. The yield stress can then be estimated from the modulus, with a linear relation assumed between stress and strain before debonding occurs. An equation for yield strain was derived by Vratsanos and Farris.[7,8] If there is no superimposed

outside pressure, the yield strain of the composite in uniaxial tension is

$$\varepsilon_c \propto \sqrt{\frac{\gamma}{\left(\dfrac{dE_c}{d\phi_f}\right)r}}$$

where ϵ_c is the yield strain of the composite and $dE_c/d\phi_f$ represents the change in the modulus of the composite as a function of the change in the volume fraction of particles that remain bonded.

If the stress–strain trace of a composite is linear up to the yield stress, the form of the yield stress equation would be as follows:

$$\sigma_c \propto \sqrt{\frac{(\gamma)E^2}{\left(\dfrac{dE_c}{d\phi_f}\right)r}} \tag{6}$$

Summarizing eqs. (1–6) shows a fair amount of agreement concerning the particle size (r) and debonding energy (γ). There is less agreement on the role of the binder modulus, and no agreement on the role of Poisson's ratio of the matrix.

EXPERIMENTAL

A series of plastic-bonded explosive mixes with a constant volume fraction of RDX filler was made for sensitivity testing.[10] A constant ratio of 150- and 20-μm particles was used for each mix. The volume fraction of the filler was within the range typical for castable plastic-bonded explosives (67–82%). The polymer chains of the polyurethane binder consisted of polypropylene glycol (PPG) molecules linked together by a mixture of isocyanate molecules. The polymer chains were diluted with a plasticizer, isodecyl perlargonate. Varying the type and amount of the isocyanate curative molecules produced samples with tensile strengths ranging from 0.115 to 0.466 MPa. The moduli ranged from 0.447 to 12.933 MPa. The mechanical properties were measured with 12.7-mm-thick, dog-bone-shaped test specimens with 68.5-mm gage lengths. The temperature of the tensile tests was 25°C, and the strain rate was 0.7407 min^{-1}.

The yield strengths of these mixes were not measured. However, there should not be a large difference between the tensile strength and yield strength for such highly filled composites. Once

the large filler particles debond, there is only a small amount of polymer still carrying the tensile load. As a result, tensile stresses much greater than the yield stresses should not be possible.

The tensile strengths of the explosive mixes are plotted versus the square root of modulus in Figure 1. There is a reasonable linear fit to most of the data. This suggests that some of eqs. (1,2,4 or 5) may be applicable. It is possible that eq. (6) is applicable, depending on the form of $dE_c/d\phi_f$. However, the linear relationship between the tensile stress and the square root of the modulus only holds up to a limiting modulus.

Each mix contained the same plasticizer and filler contents. Therefore, differences in the modulus from mix to mix should largely be caused by differences in the size and branching of the polymer chains.

A simple model of the polyurethane binders of the explosive mixes explains their mechanical properties. For simplicity, assume that all the polyurethane polymer chains of a given mix have the same average molecular weight (M). In reality, the size of the polymer chains fits some narrow distribution around M. However, the polymer chains that are larger than M will be balanced out to some extent by the ones that are smaller than M.

M is related to the degree of reaction by Carothers' equation:[11]

$$R = \frac{2}{f} - \frac{2}{fM} \tag{7}$$

where R is the reacted fraction of the PPG reactive groups and f is the functionality of the mix.

f is defined as the number of reactive groups divided by the number of molecules. The PPG molecules contained two reactive groups per molecule. Therefore, a mix formulated to have 10 PPG molecules per branch point would have 20 reactive groups from the PPG molecules and 1 from the branch point. f would be

$$f = \frac{20 + 1}{10} = 2.1$$

This can be written as

$$f = \frac{2P + 1}{P} \tag{8}$$

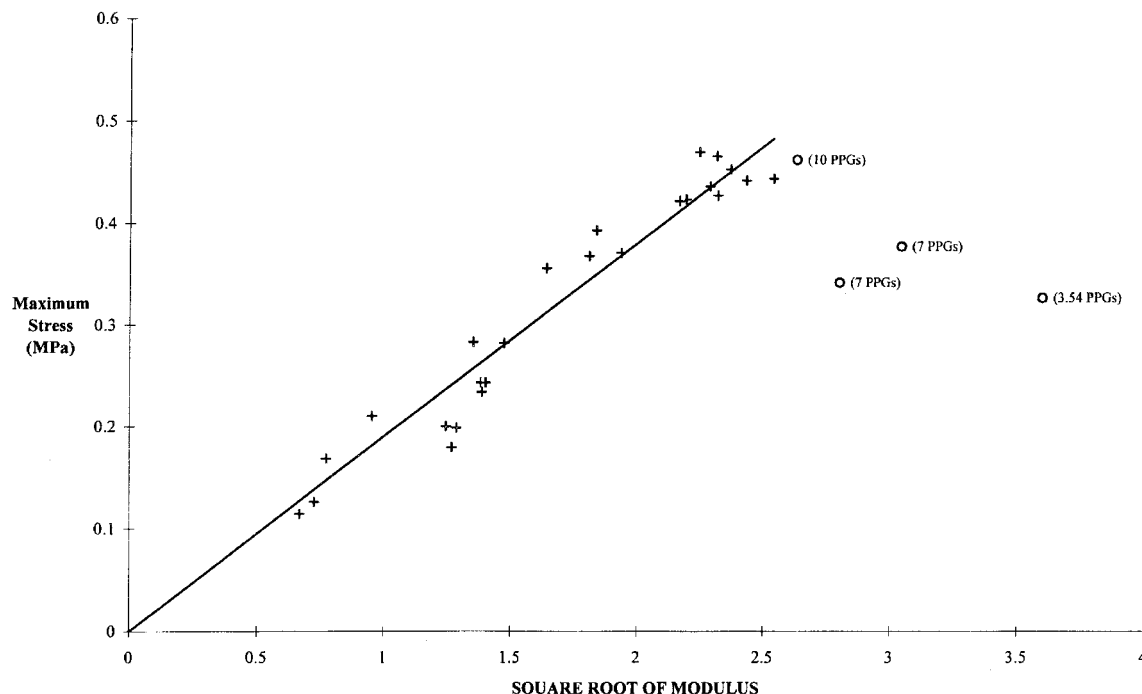where P is the number of PPG molecules per branch point.

Figure 1   Tensile strength of an RDX-filled polyurethane binder.

Equation (7) can be rewritten to solve for M for fR values less than or equal to 2:

$$M = \frac{2}{2 - fR} \qquad (9)$$

The value of R is not known. However, an estimate can be made, and the resulting values of the molecular weight can be compared to a related material property such as tensile strength. P is known from the formulation for each mix.

The molecular weights calculated from eq. (9) are plotted for three reaction fractions in Figure 2. Each curve becomes asymptotic at two points. The horizontal asymptote represents the value of M without any branching, that is, at a mix functionality of 2. The vertical asymptote represents the number of PPG molecules per branch at which M becomes infinite. At this point, $fR = 2$, and the binder becomes one giant polymer chain.

The tensile strengths of all the mixes that had the same curative ratio are plotted as a function of the number of PPG molecules per branch point in Figure 3. From right to left, from 100 to 10 PPG molecules per branch point, the tensile strengths of the mixes increase. To the left of 10 PPG molecules per branch point, the tensile strengths sharply decrease.

The 75 and 90% reaction curves of Figure 2 would be very hard to fit to the tensile stress data. Both curves increase to the left of 10 PPGs per branch point, whereas the tensile stress decreases. A much better correlation occurs with the 95% reaction curve.

The vertical asymptote of the 95% reaction curve occurs at 9.5 PPG molecules per branch point. The 95% reaction curve lies to the right of 9.5 PPG molecules per branch point, so it is increasing over the same range in which the tensile stress increases. This reaction percentage is possible because the curative ratio is greater than 1.0.

If the reaction percentage actually was 95%, then the molecular weights calculated by eq. (9) should relate to the tensile stress data. Polymers with a narrow molecular weight distribution fit the following relation:[12]

$$\sigma = \frac{C_3(M - M_T)}{M} = C_3 - C_3 M_T\left(\frac{1}{M}\right) \qquad (10)$$

where σ is the maximum tensile stress, C_3 is a constant, and M_T is the threshold molecular weight required to produce a positive stress. Plotting the tensile stress data as a function of $1/M$
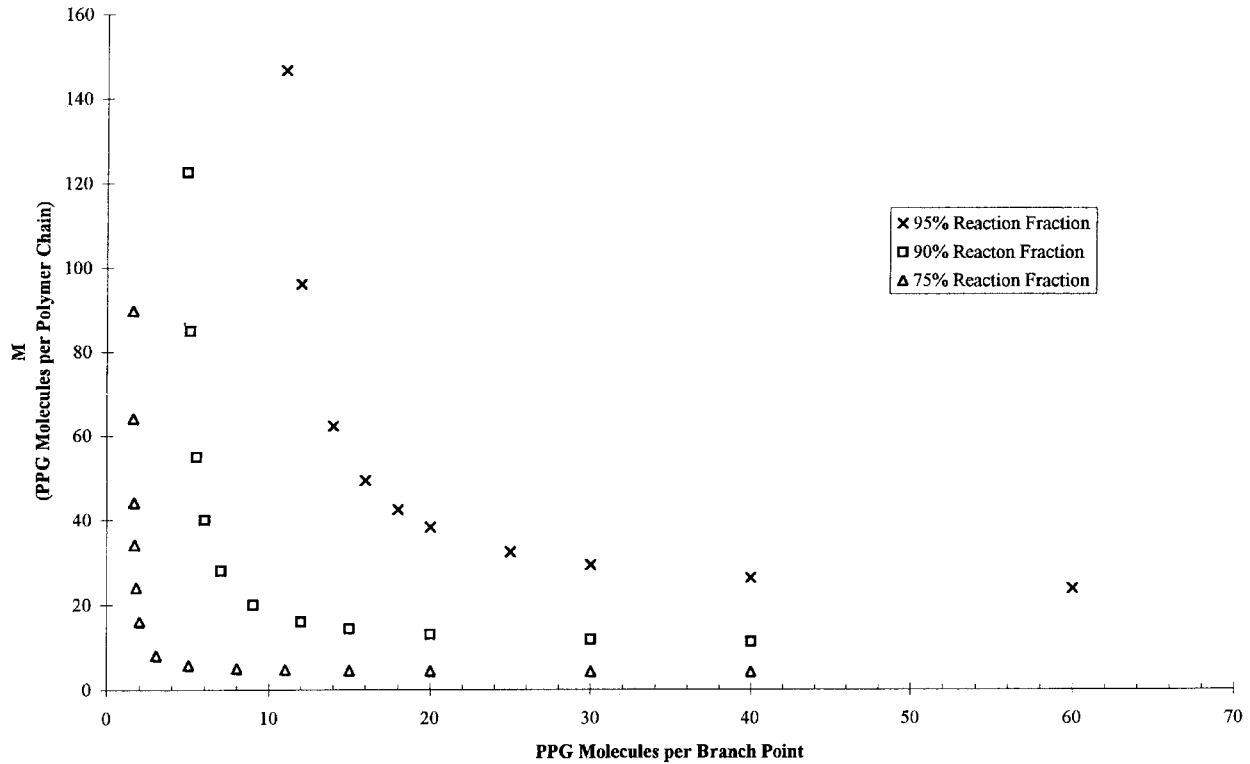
Figure 2   Estimated values of M.

should produce a linear relation with an intercept of C_3 and a slope of $-C_3 M_T$. As Figure 4 shows, the relationship is not linear over the entire range of $1/M$ values.

Equation (10) is not valid as the molecular weight of the polymer chains approaches infinity, even if the true molecular weight of the polymer chains is measured and used for M. It predicts that a bigger mix of an identical formulation would have a higher tensile strength because, if the binder was all cured up into one giant polymer chain, the bigger mix would have a higher molecular weight polymer chain. It is known from experience that a 1-gallon mix produces a material identical in tensile strength to that from a 150-gallon mix. Therefore, eq. (10) can only be used to predict the tensile strength for polymer chains considerably smaller than those created when $fR = 2$.

A reasonable linear relationship exists in Figure 4 for $1/M$ values of 0.033 and greater. By the application of a linear fit to the data, it is determined that $C_3 = 0.9651$ MPa and $M_T = 17.508$ PPG molecules per polymer chain.

Figure 5 shows that the predicted tensile stress with these values for C_3 and M_T is quite accurate

for molecular weights of 30 PPG molecules per branch point and greater. This suggests that the 95% reaction estimate is in fact correct over that data range.

At this point, the reaction fraction of some of the mixes has been calculated, and the functionality of the mixes is known from formulation. This allows the calculation of the size of the polymer chains from eq. (9), which will help explain why the linear relation in Figure 1 has a limiting modulus.

ANALYSIS

The binder becomes essentially one giant polymer chain at 95% reaction and 9.5 PPG molecules per branch point, as shown by eq. (9). This assumes that the reaction percentage continues to be close to 95% as the number of PPG molecules per branch decreases from 30 PPG molecules per branch.

Figure 1 shows that 9.5 PPG molecules per branch point marks the boundary between the mixes, which obey a linear relation between the
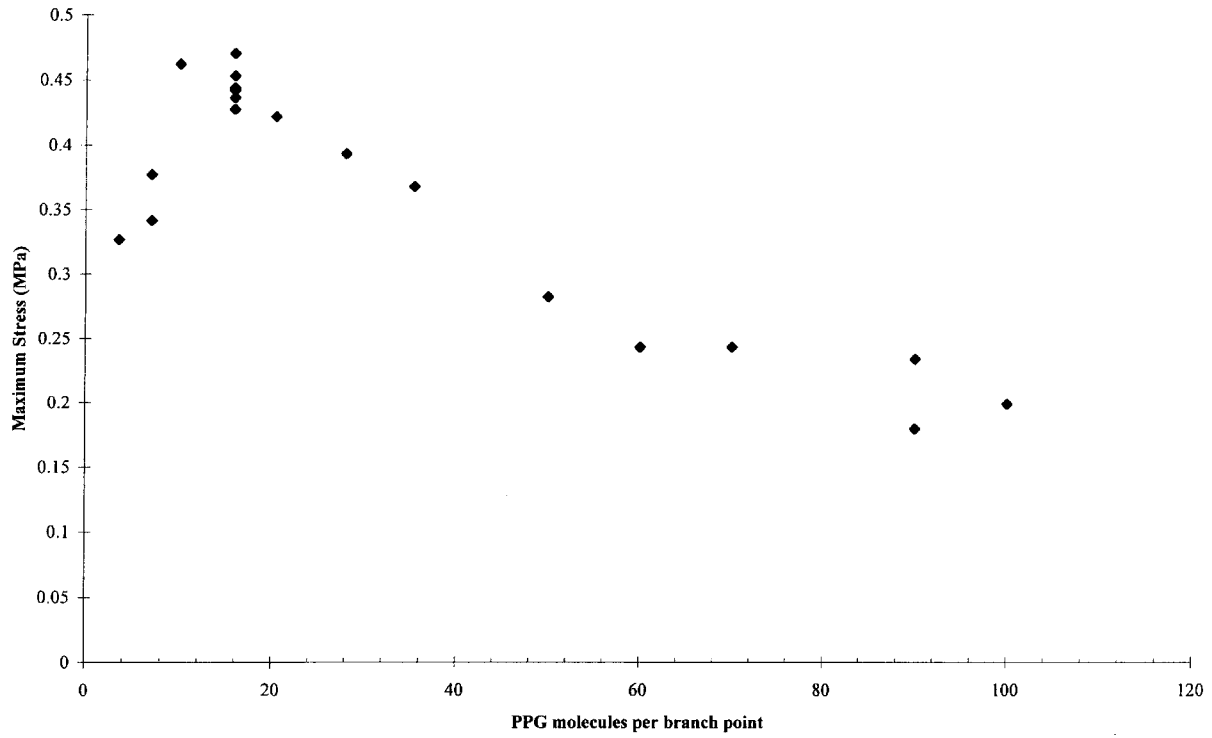
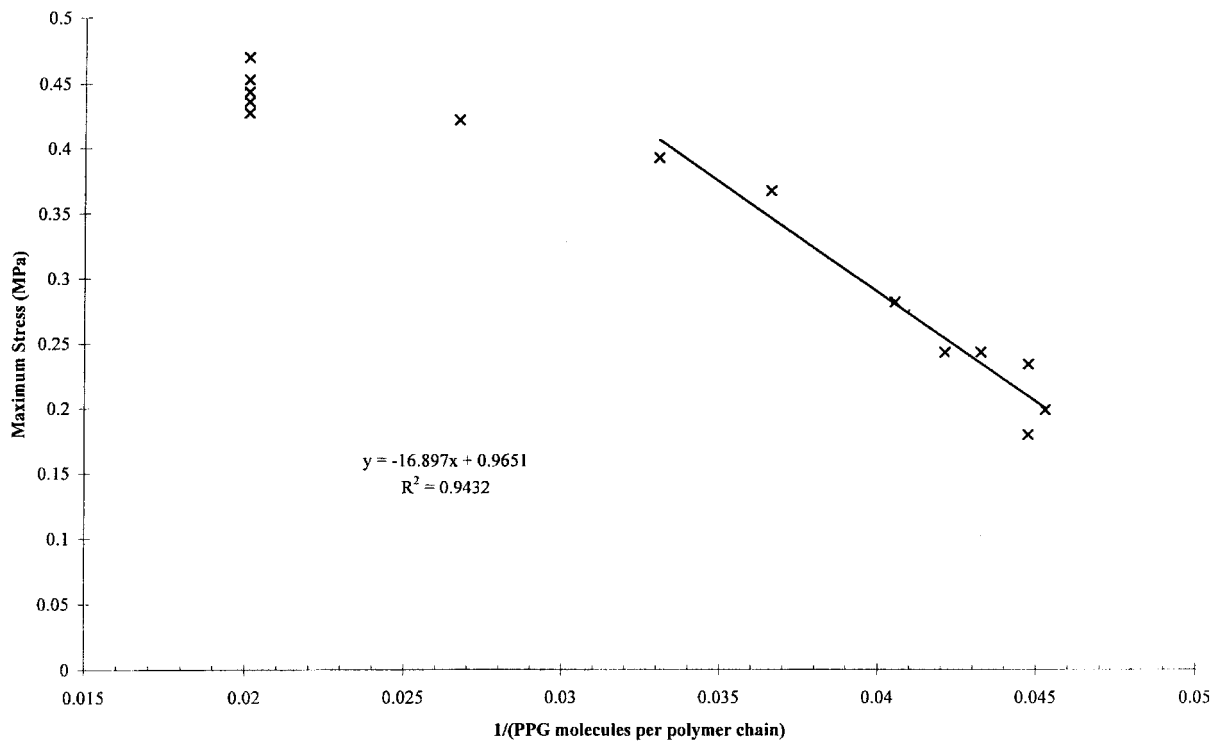**Figure 3** Tensile strength versus branching.

$$y = -16.897x + 0.9651$$
$$R^2 = 0.9432$$

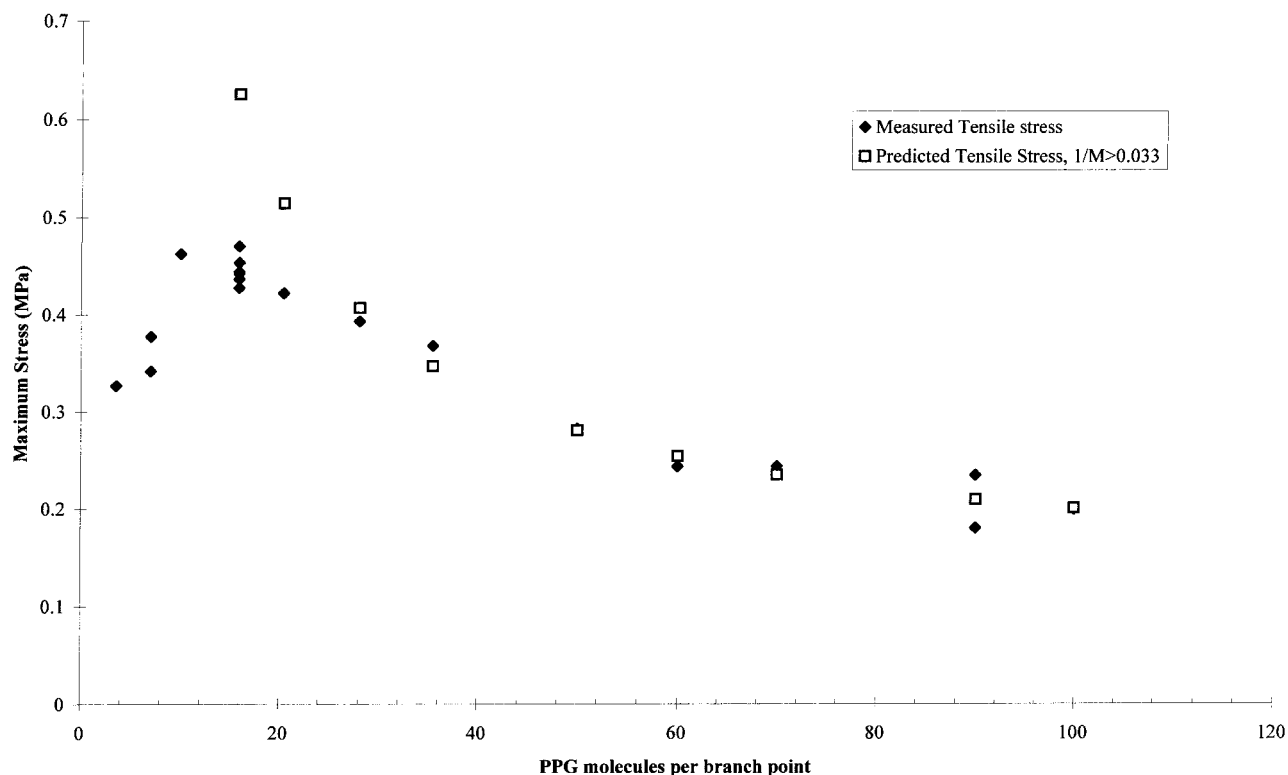**Figure 4** Tensile strength versus reciprocal polymer chain size.

Figure 5 Measured and predicted tensile strength versus branching.

tensile stress and the square root of the modulus. At 10 PPG molecules per branch point, the data point is possibly within the accuracy of the linear relation. At 7 PPG molecules per branch point, the data point is well below the expected linear relation. At 3.5 PPG molecules per branch point, the tensile stress is even further below expected values. Therefore, a decrease in tensile strength as modulus increases occurs when the value of fR in eq. (9) exceeds 2, and the binder becomes essentially one giant polymer chain.

This effect does not necessarily occur if large particles are absent. Data from Liu and Bui[13] on castor-oil-based polyurethane polymers and Eroglu[14] on polybutadiene-based polymers confirm this. In both cases, the tensile strength continued to increase as the modulus increased for values of fR well above 2. Also, data from Haska et al.[15] showed that the tensile strength also increased at fR values above 2 for polybutadiene-based polyurethanes filled with 25-nm carbon black particles. Therefore, the decreasing tensile strengths as the moduli increased in Figure 1 were probably caused by an effect related to both the sizes of the polymer chains of the binders and the size of the RDX filler particles.

The mixes in Figure 1 were cured at 60°C. Because the mixes were liquid when put into the curing oven, they should have been stress-free at that temperature. The actual stress-free temperature of the cured solid mixes may have been slightly higher than 60°C because of a slight cure shrinkage during the transition from a liquid state to a solid state.

The tensile tests were run on samples that were held at 25°C for at least 1 day. The linear coefficient of thermal expansion for the mixes was approximately 10×10^{-5} cm/cm/°C. However, the linear coefficient of thermal expansion of the RDX filler was approximately 6.4×10^{-5} cm/cm/°C. Because the volume fraction of the binder was smaller than the volume fraction of RDX, the binders of the mixes must have had significantly higher coefficients of thermal expansion than the RDX filler. As a result, thermal stresses must have developed around each filler particle.

The mixes that clearly fit the linear relation in Figure 1 had PPG molecules per branch point values ranging from 15.89 to 100. As can be seen in Figure 2, the M values of their polymer chains ranged from 50 to 22 PPG molecules. These polymer chains should be able to disentangle and flow
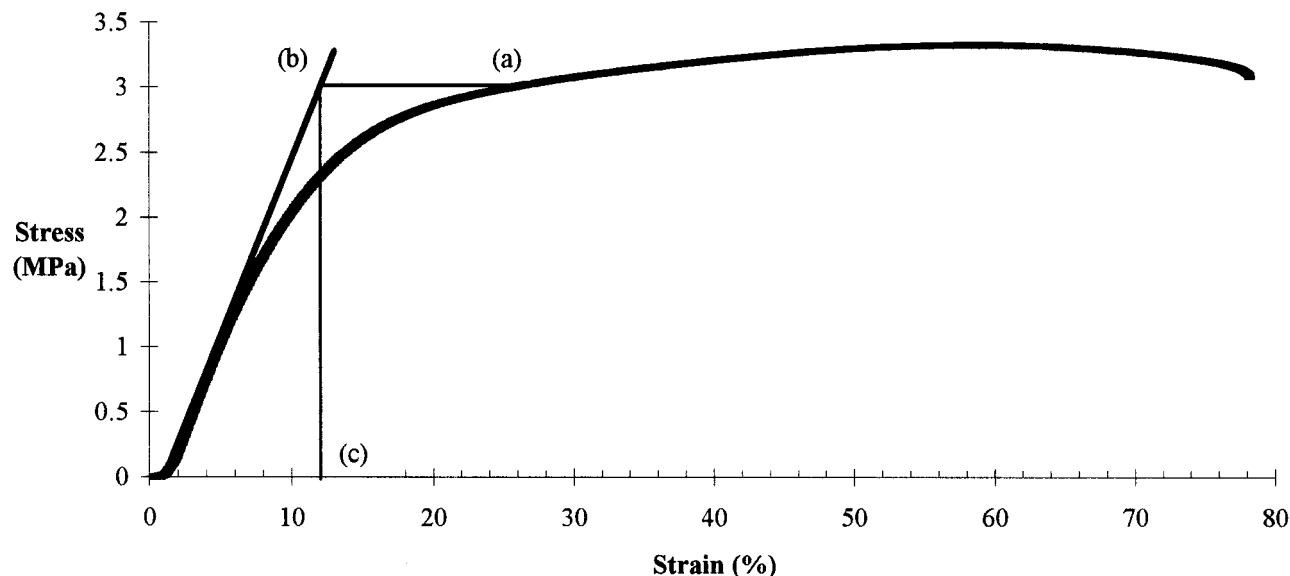
Figure 6 Properties of a composite propellant ($-54°C$, 0.7407/min): (a) the yield stress, (b) an artificial yield point with the same yield stress, and (c) the strain at the artificial yield.

to relieve the thermal stresses generated during cooling from 60 to 25°C. However, mixes formulated with 3.5 and 7 PPG molecules per branch point would have few, if any, discrete polymer chains. Their ability to flow in response to a stress would be limited to the molecular weight between crosslinks. At 3.5 PPG molecules between crosslinks, the largest thermal stresses would remain, and the binder would be prestressed the most. Therefore, that formulation has the lowest tensile strength of the nonlinear mixes. At 7 PPG molecules per crosslink, more thermal strain can be relieved, and the tensile strength is less degraded. At 10 PPG molecules per branch point, the polymer chains can deform enough to relieve essentially all the thermal stresses.

Therefore, up to a limiting modulus, which depends on the size of the polymer chains of the binder, the yield strength of a polymer highly filled with large particles should be proportional to $E^{(0.5)}$. As demonstrated experimentally by ref. 3, it should also be proportional to $r^{(-0.5)}$). The relation of the yield strength to γ, the energy absorbed in debonding a unit surface area of the filler, has not been experimentally demonstrated here. However, eqs. (1,2,4,5) and possibly eq. (6), which have the correct form for the modulus, suggest that the yield strength is proportional to $\gamma^{(0.5)}$.

THEORETICAL DEVELOPMENT

A typical stress–strain curve for a composite rocket motor propellant is shown in Figure 6. The initial part of the curve is almost linear. The slope of the tangent to this part of the stress–strain curve is the modulus of the propellant.

At some point, the rubbery propellant binder starts to detach from the large filler particles. The curve bends away from the modulus line as the large filler particles detach because they no longer can carry a load. As more of the large filler particles debond, the slope of the stress–strain curve decreases. At some point, all the large filler particles have debonded. At this point, if there is not another mechanism to affect the slope of the curve, it will remain relatively unchanged until another slope-changing mechanism comes into play. This point on the stress–strain curve, at which the slope has temporarily stopped decreasing, is defined here as the yield point. The stress and strain at that point are defined as the yield stress and yield strain, respectively.

After the yield point, the curve will usually resemble the top of a plateau. In this section of the curve, small tears in the binder are beginning to grow. Medium-sized filler particles, if present, may possibly detach in this portion of the curve. Eventually, the small tears develop into large fissures, and the sample ruptures.
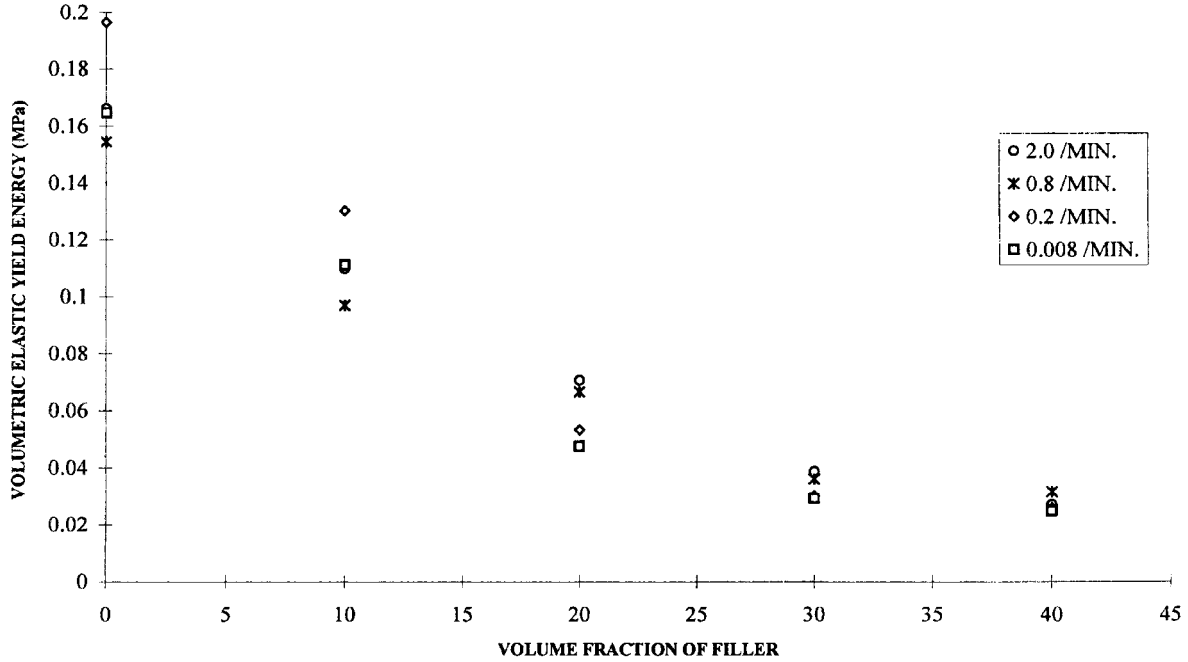
Figure 7 Energy to linear yield strain for HDPE/CaCO$_3$ composites (data from ref. 16).

As concluded in the previous section, up to a limiting modulus, the yield strength of a polymer filled with a large volume fraction of large ($\geqslant$5 μm) particles should be proportional to

$$\sigma_y \propto \sqrt{\frac{\gamma E_c}{r}} \qquad (11)$$

$$E_c < E_\infty$$

where γ is the work of adhesion between the binder and filler, E_c is the modulus of the composite material, r is the particle size of the filler, σ_y is the yield stress (approximately equal to the maximum stress), and E_∞ is the modulus of the composite at $fR = 2$. There may also be some dependence on Poisson's ratio.

The elastic energy required to yield the composite propellant can be approximated by the creation of an artificial yield point on the modulus line. In Figure 6, the yield stress of the sample [Fig. 6(a)] is used to locate this point [Fig. 6(b)] on the modulus line. The linear yield strain is the strain [Fig. 6(c)] at that point on the modulus line at which the stress is equal to the yield stress. Then,

$$\varepsilon_{ly} = \frac{\sigma_y}{E_c}$$

where σ_y is the yield stress [Fig. 6(a)] and ϵ_{ly} is the linear yield strain [Fig. 6(c)].

The area under the triangle origin-b-c represents the work done in traveling from the origin to point b, divided by the stressed volume of the sample. The work done is

$$V = \frac{1}{2}\,\sigma_y\varepsilon_{ly} = \frac{\sigma_y^2}{2E_c} \qquad (12)$$

where V is the elastic yield energy per unit volume. This elastic yield energy is also known as the modulus of resilience.

If γ and (perhaps) ν are not strongly dependent on the strain rate and temperature, $\sigma_y^2/2E$ should not depend strongly on the strain rate and temperature for polymers with large volume fractions of filler with some adhesion to the polymer.

Yield properties were measured for high-density polyethylene (HDPE) filled with CaCO$_3$.[16] The elastic yield energy, $\sigma_y^2/2E$, is plotted in Figure 7. At 0 and 10% volume fractions of filler, the elastic yield energy forms a similar pattern, with the lowest and highest strain rate data points falling between the 0.2- and 0.8-min^{-1} data. At a 20% volume fraction of filler, the elastic yield energy is strain-rate-dependent, with a large variance between the lowest and highest strain rates. At higher volume fractions of filler, where
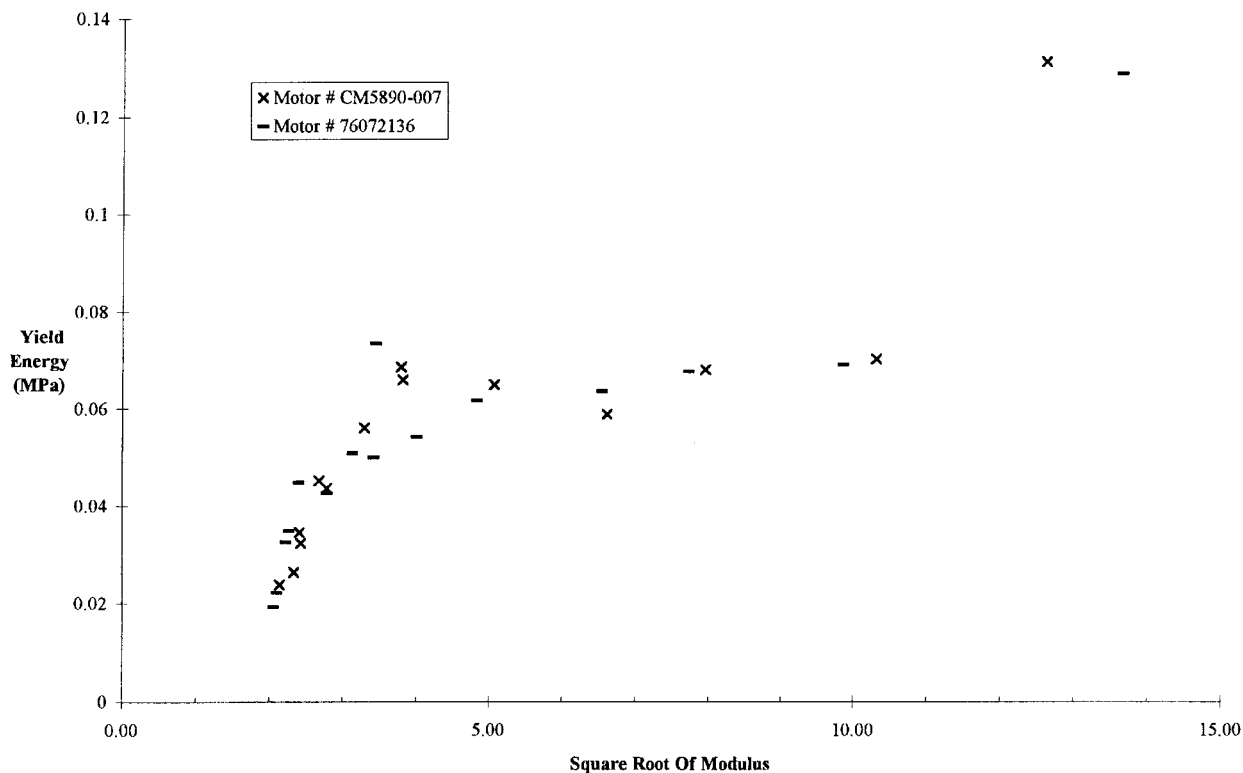
Figure 8 Volumetric elastic yield energy for a composite propellant.

debonding of the filler particles affects the yielding mechanism, the elastic yield energy varies less with the strain rate. This is shown as follows, where the difference between the highest and lowest elastic yield energies, divided by the average yield energy, is listed for each volume fraction of filler, ϕ.

ϕ (%)	$\dfrac{V_{\max} - V_{\min}}{V_{\mathrm{avg}}}$ (%)
0	24.6
10	29.6
20	38.9
30	27.8
40	25.2

Other data show that at a very high volume fraction of the filler, the elastic yield energy is constant within a certain range. Figure 8 plots the elastic yield energies of samples taken from two composite propellant rocket motors. The elastic yield energy is constant for square root of modulus values between 3.75 and 10. Outside this range, the elastic yield energy is not constant.

A potential explanation for the reduced yield energy is suggested in Figure 9, which displays the ratio of the elastic yield energy to the total energy under the stress–strain curve. At very low moduli, the energy ratio approaches 50%. This means that the area of the elastic yield triangle is almost half the area under the whole curve. The area of the triangle is $(1/2)\sigma_y \epsilon_{ly}$, whereas the area under the rest of the curve is roughly $\sigma_y(\epsilon_{\mathrm{rupture}} - \epsilon_{ly})$. This assumes that σ_y is approximately the same as the maximum stress and that the curve is a flat plateau. For similar areas, $\epsilon_{\mathrm{rupture}}$ must be quite close to ϵ_{ly}. If the rupture point is only slightly after the linear yield point, it is probable that rupturing processes started in the yielding portion of the stress–strain curve.

The elastic yield energy was calculated under the assumption that debonding of the filler preceded rupturing of the propellant. When the energy ratio approaches 50%, this assumption may no longer be true. When the square root of modulus in Figure 9 reaches 3.75 and the volumetric elastic yield energy becomes constant, the energy ratio is approximately 20%. For this composite material, the yield process is only independent of rupturing when the elastic yield energy is less than 20% of the total energy.
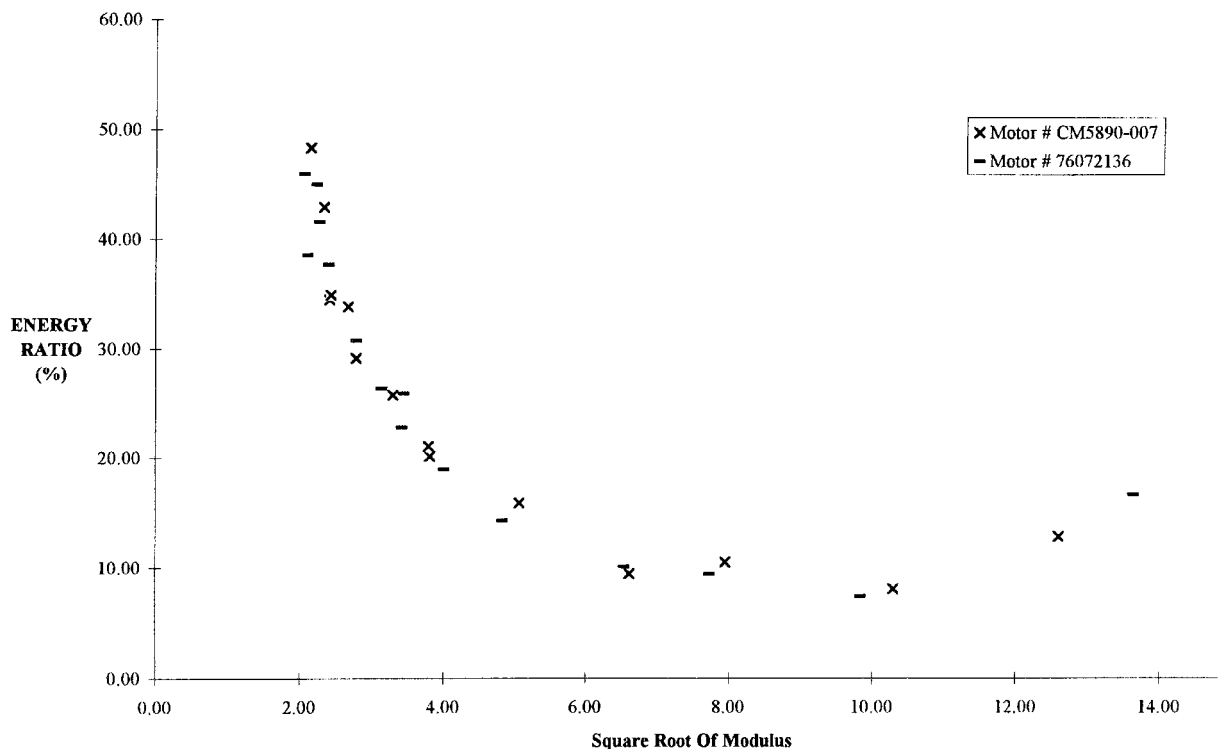
Figure 9 Ratio of the elastic yield energy to the energy to break for a composite propellant.

Two data points on the right side of Figure 8 also do not fit on the constant yield energy plateau. When the square root of modulus approaches 12.5, there is a great increase in the yield energy. These two data points were taken at $-40°C$ at a strain rate of 68.5 min^{-1}. In comparison, the Figure 1 data were taken at 0.7407 min^{-1}, the standard rate for lot acceptance testing of propellants. At such a cold temperature and such a high strain rate, the propellant may no longer be rubbery. It might be in the leathery zone between the glass transition and rubbery behavior. Equation (11) was derived under the assumption that the binder is an elastic material. This assumption may no longer be true at $-40°C$ and 68.5 min^{-1}.

A second factor may be that maximum stress data, not yield stress data, were used to calculate the yield energies for Figure 8. At low temperatures and high strain rates, the yield stress may be below the maximum stress.

CONCLUSIONS

Up to a limiting modulus, the yield stress of a polymer filled with a large volume fraction of rigid filler particles much larger than 5 μm is related to the single-particle debonding stress. The debonding stress is proportional to $(E/r)^{(0.5)}$. It is likely also proportional to $(\gamma E/r)^{(0.5)}$.

The limiting modulus occurs when the polymer chains of the binder are nearly all linked up into one giant molecule. The reaction fraction and functionality required to form this polymer can be calculated by Carothers' equation.[11] The difference between the coefficients of thermal expansion of the polymer and filler, the temperature, the size of the filler particles, and the molecular weight between crosslinks of the binder affect the tensile strength of the composite material when the modulus exceeds the limiting modulus.

The elastic energy, required to yield a polymer filled with a very large volume fraction of rigid filler particles much larger than 5 μm, is constant over a wide range of strain rates and temperatures for mixes within the modulus limit. Therefore, $\sigma_y^2/(2E)$ is constant over that range of strain rates and temperatures. If either the modulus or the yield stress is known as a function of the strain rate and temperature, the other can be calculated at any strain rate or temperature in the applicable range.

The range over which the elastic yield energy is constant has two boundaries. At low moduli, rupturing of the polymer may occur simultaneously with the debonding of the filler, lowering the yield energy. At cold temperatures and very high strain rates, the yield energy may be higher than that predicted by the debonding equations.

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