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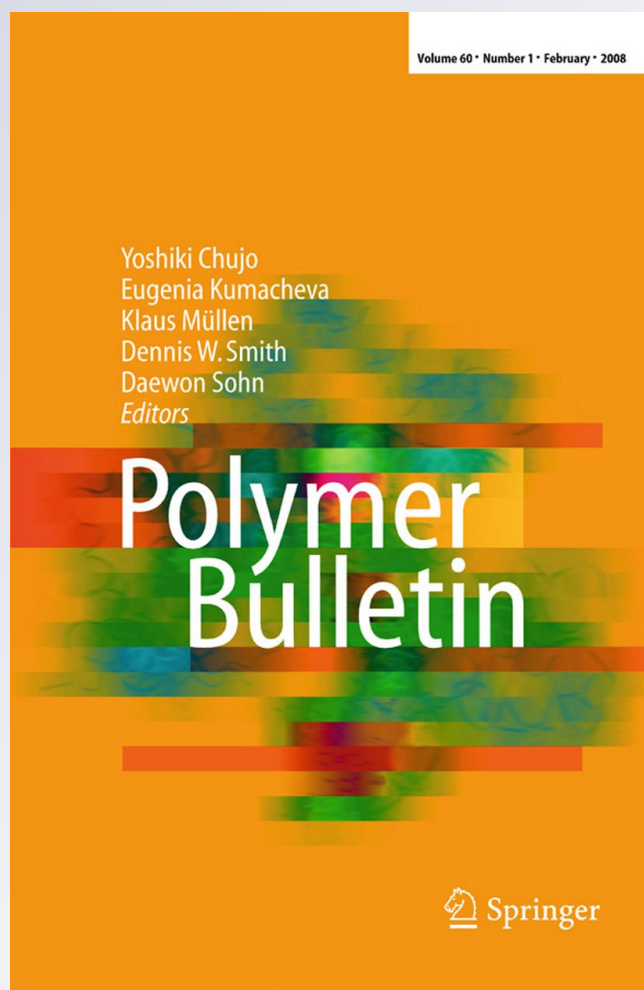
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The concept of materials brittleness and its applications

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Abstract Brittleness is a significant property considered in product design and the research and development of materials. However, for a long time the methods to determine brittleness have been largely “hand-waving” arguments or else circumferential properties—in other words describing numerous properties related to brittleness but not actually quantifying brittleness itself. We have defined brittleness of polymeric materials quantitatively with applications to multiple areas. Relationships between brittleness and both tribology and mechanics have been discovered and are described. Moreover, the definition has been applied in the development of multilayer composite materials; structural integrity of the composites decreases with increasing brittleness. Other applications and the fact that toughness is not an inverse of brittleness are also discussed.

Keywords Mechanics · Tribology · Composites · Brittleness · Impact strength

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Background

The terms such as ‘brittleness’, ‘hardness’, or ‘strength’ are applied by scientists, engineers, and laymen alike to characterize appearance and mechanical behavior of materials [1, 2]. While thus the term ‘brittleness’ is familiar and in frequent use, for a long time there was no widely accepted quantitative definition. In 1976 Gordon [3] said: “Most metals and timbers, and also Nylon, Polythene, fiber-glass, bones, teeth, cloth, rope, and jade are tough. Most minerals, glass, pottery, rosin, Bakelite, cement, and biscuits are brittle, and so is ordinary table jelly as one can readily prove by propagating a crack in it with a spoon and fork. It is not at all easy to see what it is that makes one thing tough and another brittle because the substances in each of these lists seem to have little enough in common. The distinction is a very real one however”. Clearly the concept of brittleness is important not only to Polymer Science and Engineering (PSE) but to all of Materials Science and Engineering (MSE). Reviewing the literature, one finds that more than 30 years ago a conference was convened specifically to address brittleness and toughness of plastics with the results compiled in a book on that topic [4]. The body of work from that 1974 symposium still defined the quantity brittleness largely by visual assessment of fractures and related properties. For example, Yee et al. [5] described the ductile-to-brittle transition by electron micrographs of fracture patterns and strain behavior modulations. Importantly, tensile elongation is noted for its relationship to brittleness early on by Matsuoka [6] and later also by Menges and Boden [7].

A 1992 report by Wu [8] described connections between toughness—determined by impact testing—and chemical composition and chain structure. However, toughness cannot be equated with the reciprocal of brittleness, a point further addressed in a later section of this article. These works among others provide reasons why we observe so-called brittle fracture in certain materials but not in others. A quantitative index for brittleness appeared in 1997 [9], developed by Quinn and Quinn for ceramic materials. Because the brittleness parameter described by Quinn and Quinn assumed certain features of *elasticity*, the index cannot be applied to polymer-based materials (PBMs) which are always viscoelastic. We note that metals also exhibit viscoelasticity—although less than PBMs, a phenomenon discovered long ago [10] and confirmed by more recent results as well [11].

Brittleness defined

In 2006, we have developed a quantitative definition of brittleness that is not limited to elastic materials and thus applicable to polymers and composites [12]. The brittleness B is defined by the following equation:

$$B = 1/(\varepsilon_b E') \quad (1)$$

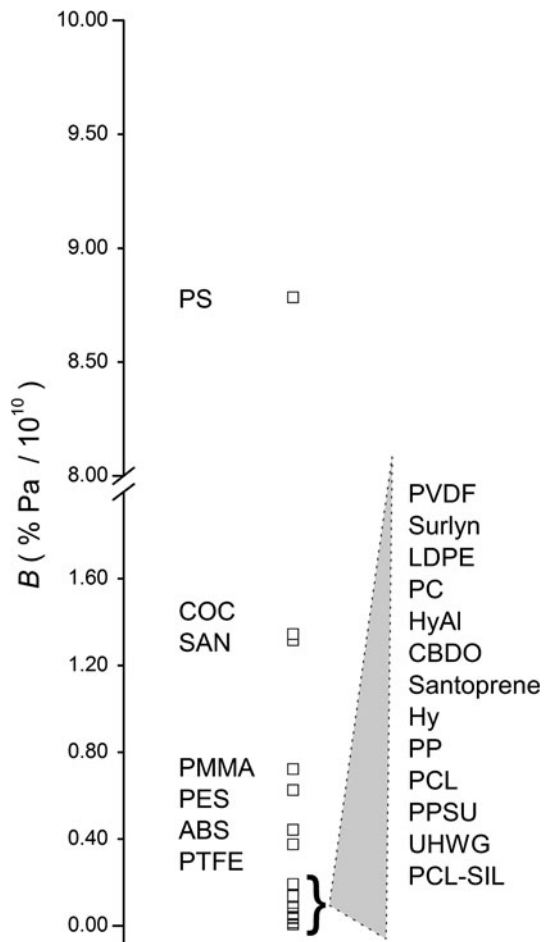
where ε_b is the tensile elongation at break and E' is the storage modulus determined at 1 Hz and the temperature of interest (such as 25 °C) by dynamic mechanical analysis (DMA). The significance of elongation with respect to brittleness has

already been mentioned. What is more, the ϵ_b term in the denominator in our definition of brittleness takes into account *large deformations* of a material. On the other hand, the storage modulus accounts for *repetitive loading or fatigue*—so important in service. This is an essential aspect as it relates to viscoelasticity of PBMs. The DMA technique is well described in the literature [13–16] and distinguishes by mechanical testing the solid-like (storage modulus E') and liquid-like (loss modulus E'') behaviors of PBMs.

Brittleness as defined by Eq. 1 has been evaluated for neat thermoplastics including homopolymers, copolymers, and elastomers as well as for polymer composites including those with ceramic fillers [12, 17–19]. This is shown on a one-dimensional scale in Fig. 1, including also examples of polymer + ceramic and polymer + metal composites. In the following we shall discuss various already known and potential applications of this concept.

Figure 1 shows a big gap between polystyrene and all other materials, whether polymers or composites. In general, one is developing new materials with the

Fig. 1 Brittleness of polymers and certain composites, with B calculated according to Eq. 1. Materials are: polycarbonate (PC); cycloolefin copolymer (COC, [25]); polypropylene (PP); polystyrene (PS); polytetrafluoroethylene (PTFE); styrene/acrylonitrile copolymer [Luran®] (SAN); Santoprene™ (Santoprene); acrylonitrile/butadiene/styrene (ABS); Surlyn® 8149 [ethylene/methacrylic acid copolymer] (Surlyn); polyethersulfone (PES); low-density polyethylene (LDPE); poly(methylmethacrylate) (PMMA); polyphenylsulfone (PPSU); polyvinylidene fluoride (PVDF); Hytel® (Hy); Hytel + aluminum (HyAl); polycaprolactone (PCL); polycaprolactone + silica (PCL-SIL); ultrahigh molecular weight polyethylene [UHMWPE] (UH); UHMWPE + white graphite (UHWG); a copolyester (CBDO)



requirement that they will be strong [20–24]. PS is an exception since in some applications brittleness is either required or unimportant. An example of the former is easily collapsible coffee cups; here the high B value is an advantage. An example of the latter are PS sheets used in building insulation; there are no moving parts, hence no danger of wear and also no variable loads are expected.

Brittleness and sliding wear

One can determine scratch resistance in single scratch testing [20–22]. Using the same micro scratch tester, one can determine sliding wear by repetitive scratching along the same groove [20, 23]. In either single scratch testing or in any run in sliding wear determination, there is an instantaneous (penetration) scratch depth R_p . For viscoelastic materials, following a rest period after scratching, there is a recovery of the groove to a shallower (healing, recovery) depth R_h . This viscoelastic recovery f can be quantified [24], with higher values corresponding generally to lower wear:

$$f = [(R_p - R_h) \cdot 100\%]/R_p \quad (2)$$

We have demonstrated a correspondence between B and viscoelastic recovery f [12, 17] for a variety of polymers with different chemical structures as well as for polymer-containing composites. The relationship is:

$$f = 30.6 + 67.1e^{-B/0.505} \quad (3)$$

Accordingly, materials with low brittleness have higher recovery in sliding wear and vice versa. The sliding wear recovery is further related to the free volume, as described in detail elsewhere [12]. Thus, we have connected Mechanics with Tribology.

While we stress polymers in this article, for a wider perspective let us note some results for thick film copper compositions used in thermoelectric device fabrication [11]. Two materials were studied, one with small grains (C1) and one with large grains and also large intergranular spaces (C2). The small grain samples have much smaller microindentation areas and much higher hardness than large grain samples, a consequence of very small intergranular spaces and thus high cohesion in the former. Both penetration and residual depths in scratch testing are smaller in C1. As we now expect, this situation is reflected also in brittleness. The brittleness value for the small grains material is much lower than for the large grains material; the actual numbers are $10^{10}B/(\%Pa) = 0.0368$ for C1 and in the same units 0.131 for C2, a factor close to 3.5 [11].

Brittleness related to impact strength

Brittleness ought to be related to impact resistance, and developing a correspondence between the two would provide an element for predicting product behavior. Since the common measures of impact strength are the Charpy impact energy (U_C ,

mostly used in Europe) and the Izod impact energy (U_I , mostly used in the US), those values have been determined for a set of the materials shown in Fig. 1 [18]. For both Izod and Charpy values and in obvious notation, a relationship to brittleness was found and can be described by the following equations [18]:

$$U_C = a_c + 1/\tanh(b_c B) \quad (4)$$

$$U_I = a_I + 1/\tanh(b_I B) \quad (5)$$

Again a body of data for different polymers was used. The values of the parameters are $a_c = -0.640$, $b_c = 1.63$, $a_I = -0.660$, and $b_I = 2.29$.

Consider the situations when $B > 1$; polystyrene is an important example. Then a further increase in B does not bring about any further lowering of U_C or U_I . Any material therefore presents some resistance to impact, even a very brittle one.

Equations 3–5 have been obtained on the basis of data for a variety of materials and composites—already seen in Fig. 1. Thus, as far as we can tell, we have ‘universal’ curves for polymers and polymer-containing composites. When a new polymer is made and its B determined, these equations should allow calculation of its viscoelastic recovery in scratch testing, its Charpy impact strength and Izod impact strength.

Brittleness applied to blends and multilayer composites

A combined Italian and Czech team has investigated blends of linear low-density polyethylene (LLDPE) with a cycloolefin copolymer (COC) [25]. The latter is a rigid material with low elongation at break ε_b . Addition of COC to LLDPE results in stronger materials, the moduli increase with increasing COC concentration. However, the decrease of ε_b with increasing COC concentration is *faster* than the modulus increase. As a result, brittleness increases when more COC is added.

Shen and coworkers in Sichuan have studied blends and multilayered composites of propylene–ethylene copolymer (PPE) + ethylene 1-octene copolymer (POE) [26]. The composites were prepared by a microlayered coextrusion system. The results were interpreted among others using our Eq. 1. We need to consider separately their results for blends and for layered composites.

The Sichuan group has varied in their PPE + POE blends the concentration of the elastomeric component, which is POE. When they increased 5-fold the POE concentration, they have found a nearly 3-fold increase in the tensile elongation at break ε_b . At the same time, the dynamic storage modulus E' had decreased, but at a rate slower than the elongation at break increase. This led to a decrease in B , not large but quite clear.

For the 64-layer samples, the elongation at break remained approximately constant when increasing the concentration of the elastomer. Shen and coworkers attributed this situation to parallel alignment of POE layers along the direction of the external force. E' decreased with increasing POE content. Consequently, the brittleness B has *increased*. Clearly the simple reasoning “we add more elastomer

which is flexible, therefore B of the composites should decrease” does not apply. The Sichuan group explains their results as follows: the B increase is a consequence of poorer adhesion between layers at higher POE contents. This is a significant result. Brittleness not only characterizes single phase materials. *It also characterizes structural integrity* of multiphase composites.

We recall the results for copper pastes with intergranular spaces—noted at the end of “[Brittleness and sliding wear](#)” section. We also recall that Kopczynska and Ehrenstein [27] have stressed the importance of interfaces in multiphase materials.

Brittleness is not an inverse of toughness

A question was directed at us: does not the inverse of toughness serve the same purpose as brittleness? First of all, there are several definitions of toughness. If one uses the impact strength to represent toughness, our Eqs. 4 and 5 show that the relationship is *not* a simple inverse proportionality. One uses also the area under the force versus displacement curve in an impact event to represent toughness—as advocated by Adams and Wu [28]. These authors point out: “the deficiency of the conventional single-point extrapolation procedure is apparent, because the energy-to-break is neither linear with fracture area nor extrapolates through the origin”. Thus, even if one would decide that toughness should be defined in terms of results of impact testing, more than one option exists.

Still other quantities in use; Nielsen and Landel [29] say that “the concept of toughness can be defined in several ways, one of which is in terms of the area under a stress–strain curve”. Raab and Nezbedova [30] discuss fracture toughness, also known as the J -integral. This is a still different quantity defined in *yield* fracture mechanics; it represents the energy required to initiate slow crack growth. There are also further quantities related to the J -integral such as the critical energy release rate but they are based on *linear elastic* fracture mechanics which is not applicable to PBMs in general.

Koning et al. [31] who have investigated poly(cyclohexane carbonate) (PCHC) state that “The low elongation at break, in spite of a reasonably high molecular weight, implies that PCHC is a brittle polymer, although a low temperature relaxation is present, which is quite often related to toughness”. We have actually located that article after having formulated Eq. 1 and now we find in it support for our assumption that ε_b is a factor involved in brittleness. Moreover, the phrase “often related” is one more manifestation of the lack of a single accepted definition of toughness.

Apart from ambiguity in “toughness”, there is a flaw that existing definitions of toughness seem to have: they pertain to a single loading event. The situation of any component in service is different. There are repetitive loadings, therefore, gradually *fatigue* ensues—not taken into account at all in toughness, no matter how defined. Our definition by Eq. 1 has thus an important advantage briefly noted earlier: fatigue is taken care of by the storage modulus E' term.

Aging and other processes affecting brittleness

It is expected that we and others will continue to discover valuable applications of the definition of brittleness provided by Eq. 1. Equations 3–5 are examples. We note that in general ε_b is a function of the strain rate. A ductile material may become brittle at a high strain rate, the critical strain rate [32]. Thus, we assume that the ε_b values used in Eq. 1 are obtained at strain rate levels below the critical strain rate. Actually, the very existence of the critical strain rate provides an indication on the nature of brittleness. Materials with internal structures unable to adapt to the change of shape and size imposed by external rapidly acting force respond to that force in a brittle way.

PBMs are known to undergo aging. As stated by Robertson and Kim [33] “over time at any given temperature, one finds increases in density, elastic modulus, and yield strength and decreases in enthalpy and compliance” and “the material tends towards brittleness”. Aging processes have been investigated for several decades [34, 35]—including environmental stress cracking which is a special type of aging [36].

Data needed to calculate B as a function of aging are limited at this time; some available results were already discussed in our first article [12]. It turns out that aging causes a decrease of ε_b faster than the simultaneous increase of E' . Thus, brittleness increases—confirming the qualitative opinion prevailing before Eq. 1 was formulated.

Irradiation such as by gamma rays or electron beams causes changes in polymer properties [37–42] and is also sometimes called aging. Long-term irradiation effects appear during shelf life [43]. Changes in B in such cases deserve further investigation.

Other areas of possible activity are thin films or coatings. Glass transition temperatures are different than in bulk materials [44], as are melting temperatures [45]. The film thickness and the nature of the substrate affect transition temperatures, crystal orientation, and morphology.

A separate area of surface studies is chemical modification of surfaces. For instance, direct fluorination of polymers—through treating surfaces with gaseous fluorine mixtures—is sought to enhance barrier properties, gas separation properties, adhesion, printability, or mechanical properties. Kharitonov and Kharitonova [46] report data on the time required to form a 1 μm thick fluorinated layer on a variety of polymers, some of which are the same as those investigated by the present authors and listed in Fig. 1. A simple comparison suggests that that fluorine penetrates faster into a brittle material. Since there are important cost and environmental concerns associated with direct fluorination—and the time variation of the reported materials is some 700 min—validation of such a relationship could enable one to predict in advance the time needed for fluorination of a given material. Polymer surfaces are modified also by selective etching [47] or else by plasma treatment [48]; effects of such treatments on B have not been investigated so far.

We have noted briefly above that metals also show viscoelasticity [10, 11]. Thus, the concept of brittleness can be useful for metals also. Kommel et al. [49, 50] have subjected copper to severe deformations. They report a decrease of ε_b as a result of those deformations. Chicot et al. [51] report viscoelastic behavior including creep of magnetite coexisting with hematite in a natural dual-phase crystal. In such materials E' can be determined and brittleness calculated.

There is a variety of materials reinforced by a filler with high hardness. Thus, Dec et al. [52] have studied wear of several heat-treated metal alloys using a turntable device moving at 0.04 m/s. The counter specimen was a slag conditioner (65 wt%) with calcium aluminate (25%) and hard granular alumina (10%). One assumes that the material to be investigated will undergo wear—rather than the counter material. What happened was chipping off little pieces of alumina; those pieces increased the abrasion and wear of both partner surfaces significantly [52, 53]. Therefore, the idea “we shall include a filler with high hardness and thus reinforce our material” might bring results *just opposite* to those expected. A high hardness filler can lower the elongation at break while not increasing E' much—unless good adhesion to the matrix prevails.

On the other hand, Assouline et al. [54] report that addition of carbon nanotubes (CNTs) to isotactic polypropylene (iPP) results in formation of the α phase of PP. CNTs act here as a nucleating agent [55, 56] while fibers instead of spherulites are formed. A number of polymer properties can be varied by inclusion of CNTs [57–63]. Thus, there is maneuverability of properties of composites with fillers—while B could be a measure of success of achieving desired properties. In other words, it is likely that brittleness may be particularly helpful in distinguishing the performance within a particular class of materials—much like the story about multilayer composites, above. As one seeks to develop new materials with a given set of properties, determining the brittleness of a series of blends containing different amounts of a filler could lead to deeper understanding of the materials behavior and thus more effective enhancement of desired properties.

Brittleness reflects changes in PBMs also in situations other than those named above. For instance, addition of Ni nanopowder to a thermoplastic elastomer results in an increase of B since cohesion or structural continuity in the polymer is perturbed by the added filler [64]. Crosslinking of the same elastomer lowers the elongation at break and thus increases B . By contrast, when both Ni powder is added and crosslinking performed on the same elastomer, B decreases [64]. Metal nanoparticles go either into existing free volume pockets in the elastomer or create new such pockets. In the former case, there is an enhancement of mechanical properties including E' ; in the latter case there is an increase of the elongation at break—also lowering B .

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