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IRRADIATION EFFECTS ON THE MECHANICAL PROPERTIES OF COMPOSITE ORGANIC INSULATORS*

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Abstract

Four kinds of cloth-filled organic composites (filler: glass or carbon fiber; matrix: epoxy or polyimide resin) were irradiated with 2-MeV electrons at room temperature, and were examined with regard to the mecahnical properties. Following irradiation, the Young's (tensile) modulus of these composites remains practically unchanged even after irradiation up to 15,000 Mrad. The shear modulus and the ultimate strength, on the other hand, begin to decrese after the absorbed dose reaches about 2,000 Mrad for the glass/epoxy composite and about 5,000 ~ 10,000 Mrad for the other composites. This result is ascribed to the decrease in the capacity of load transfer from the matrix to the fiber due to the radiation-induced debonding at the interface. As to the fracture behavior, the propagation energy increases from the beginning of irradiation. This result is attributed to the radiation-induced decrease in the bonding energy at the interface. The same study was made also for these composites and an alumina fiber-epoxy composite irradiated with fast neutrons at room temperature and 5 K.

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1. Introduction

Magnetic fusion reactors will rely heavily on the stability of mechanical and electrical properties of magnet insulators under irradiation [1-3]. Leading candidates for this material are composite organic insulators because these are superior to inorganic insulators in terms of cost and processing [4]. Organic composites, however, may suffer serious radiation damage due to fast neutrons and concomitant γ-rays when used at low temperature in fusion magnets. For this reason, several studies have been done recently on the low-temperature irradiation effects of composite organic insulators [5-8].

In spite of these studies, however, questions still remain about the mechanism of radiation-induced degradation in mechanical properties even for room temperature irradiation as well as low temperature irradiation. In the present work, we tried to elucidate the mechanism based on the dose dependence of the mechanical properties at room temperature. For this purpose, an electron accelerator was used since it can give a desired dose in a relatively short time. The present paper mainly describes the dose dependence of mechanical properties such as Young's and shear moduli, ultimate strength, and

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fracture propagation energy. The dose dependence is interpreted and formulated based on the mechanics of composite materials and the target theory used in radiation biology. Irradiation effects due to fast neutrons at room temperature and 5 K are also presented, and are compared with the electron irradiation effects.

2. Experimental

Four kinds of organic composites were especially prepared by Sumitomo Bakelite Co., Ltd., using E-glass fiber cloth (KS-1210) or carbon fiber cloth (Torayca #6142) as reinforcing filler and epoxy (Sumiepoxy ELM-434) or polyimide as matrix resin. The 2.0 mm-thick laminate sheets of these composites were cut into specimens of 6.4 x 2.0 x 70 mm^3 dimensions. An epoxy matrix composite reinforced with unidirectional alumina fibers was provided and cut into specimens of 3.2 x 1.6 x 70 mm^3 dimensions by Sumitomo Kagaku.

Electron irradiations were carried out at room temperature with a Cockcroft-Walton type accelerator. The accelerator power was set at 2.0 MV and 5 mA during the irradiation. The absorbed dose measurements were made by Tanaka et al. using the CTA (cellulose triacetate) film dosimeter [9]. Fast neutron irradiations were carried out at room temperature and 5 K with the Intense Pulsed Neutron Source (IPNS) installed at Argonne National Laboratory [10]. The absorbed dose was determined by measuring the radioactivity of the nickel wire attached to each specimen or its container.

The mechanical properties were examined by performing three-point bend tests at room temperature and at a crosshead speed of 0.5 mm/min. The details are described elsewhere [11].

3. Results

3.1 Young's and Shear Moduli

According to the Bernoulli-Euler theory [12], the total deflection arising from tensile and shear stresses in a rectangular specimen loaded at three points is given by

$$\Delta = \left[\frac{(\ell/h)^3}{4bE} + \frac{0.3(\ell/h)}{bG} \right] P \qquad (1)$$

where Δ is the total deflection, P is the applied load, ℓ is the span length, b is the specimen width, h is the specimen depth (thickness), E is the Young's (tensile) modulus, and G is the shear modulus. This equation indicates that the ratio of tensile to shear deflection depends on the span length. If therefore three-point bend tests are performed at various span lengths for each specimen, both E and G can be determined by solving the resulting simultaneous equations. For this purpose, the bend tests were made at span lengths of 63.5, 50.8, 38.1, 25.4, 19.05, and 12.7 mm.

The Young's modulus thus determined for electron irradiated composites is plotted as a function of absorbed dose in Fig. 1, and the shear modulus is plotted in Figs. 2-5 for each composite. The Young's modulus of the control specimens is seen to increase in the order of the glass/polyimide, glass/epoxy, carbon/polyimide, and carbon/epoxy composites. This result reflects the higher Young's modulus of carbon fibers and epoxy resins compared with that of glass fibers and polyimide resins, respectively. Following irradiation the Young's modulus of these composites is seen to remain practically unchanged even after irradiation up to 15,000 Mrad.

The shear modulus of these composites, on the other hand, is seen to have a dose dependence. For the glass/epoxy composite (Fig. 2), the shear modulus remains unchanged up to about 2,000 Mrad and then decreases with increasing absorbed dose. For the other composites (Figs. 3-5), the shear modulus does not change appreciably until the absorbed dose exceeds about 10,000 Mrad, and after that it begins to decrease.

3.2 Failure Strength

According to the Bernoulli-Euler theory on a rectangular specimen loaded at three points [12], the maximum tensile (or compressive) stress, σ_{max}, and the maximum shear stress, τ_{max}, exist at the surface of the specimen and at the neutral axis, respectively, and are given by

$$\sigma_{max} = 3P(\ell/h)/2bh \qquad (2)$$

$$\tau_{max} = 3P/4bh. \qquad (3)$$

From a simplistic standpoint, the specimen is regarded to fail in a tensile (or compressive) mode or in a shear mode depending on which of σ_{max} and τ_{max} becomes critical at the failure [12]. If therefore the load at failure, P_f, varies inversely proportional to the ℓ/h ratio, the failure is a tensile mode, and if P_f is independent of ℓ/h, the failure is a shear mode. The failure tests made at ℓ = 50.8, 25.4, and 12.7 mm showed that at ℓ/h = 12.7/2.0 all of the specimens studied here fail in a tensile mode except the glass/epoxy composite irradiated up to 15,000 Mrad and the carbon/epoxy composite. Accordingly, the ultimate strength of a composite, σ_{cu}, was calculated by using eq. (2) in the present work, with the recognition that although the σ_{cu} value is equal to the ultimate tensile strength for a tensile mode, it becomes the ultimate shear strength times 2(ℓ/h) for a shear mode.

The ultimate strength thus calculated for electron irradiated composites is plotted as a function of absorbed dose in Figs. 2-5 by the solid point (●) which is the average value of three tests made at the span length of 12.7 mm. Comparison of the ultimate strength with the shear modulus (o in the same figure) suggests that they are correlated with each other. The correlation is, in fact, confirmed by the plot of the ultimate strength vs. the shear modulus shown in Fig. 6.

3.3 Fracture Propagation

The fracture initiation energy was calculated by integrating the load-deflection curve over Δ from zero to the point of failure [13]. The plot of the initiation energy vs. absorbed dose was found to be essentially the same as that described for the ultimate strength (see Figs. 2-5), and hence the plot is not shown in this paper.

The fracture propagation energy, on the other hand, was calculated by integrating the load-deflection curve from the failure point to infinity [13]. The plot thus obtained for electron irradiated composites is shown in Fig. 7, where each data point is the average value of three tests made at the span length of 12.7 mm and is normalized by dividing the specimen cross-sectional area. The propagation energy is seen to increase with absorbed dose from the beginning of irradiation for the glass/epoxy (●) and glass/polyimide composites (o). For the carbon/polyimide composite (x), the propagation energy appears to decrease at first and then increases with absorbed dose. As for the carbon/epoxy composite, the plot is not shown here because the exact evaluation is made impossible by the fact that the load does not approach zero even at a large deflection after the failure. It can be said, nevertheless, that the propagation energy for this composite is much larger than that for the other composites. This is also the case for the glass/epoxy composite irradiated up to 15,000 Mrad.

3.4 Fast Neutron Irradiations

The same composites as described above and an alumina/epoxy composite were irradiated with fast neutrons at room temperature and 5 K. The mechanical properties were tested at room temperature. The results are collected in Table 1. Within the absorbed dose range so far covered, the four kinds of cloth-filled composites appear to maintain their initial values of Young's modulus, shear modulus, and ultimate strength. This result means that not only electrons but also fast neutrons have an incubation dose before these mechanical properties begin to degrade. As for the alumina/epoxy composite, a slight decrease in the shear modulus and the ultimate strength is observed for 5 K irradiation up to 465 Mrad.

The fracture propagation energy, on the other hand, begins to change even at low doses, as is also true with electron irradiated composites. For the glass/epoxy and glass/polyimide composites, the change in this energy with absorbed dose seems to be larger for room temperature irradiation than for 5 K irradiation. For the 5 K irradiation, even a decrease in this energy is observed for the glass/polyimide composite. These findings suggest that at low temperature the degradation of matrix polymers is retarded and/or the crosslinking of these polymers becomes predominant at least at low doses. As for the carbon/polyimide composite, on the other hand, the propagation energy is decreased by irradiation at both temperatures. This decrease is observed at low doses of electron irradiation also (see x in Fig. 7).

4. Discussion

4.1 Young's and Shear Moduli

The Young's modulus of a composite can be represented by the rule of mixtures: $E_c = \alpha E_f V_f + E_m V_m$, where E is the Young's modulus, V is the volume fraction, α is the coefficient dependent on the form of fillers (ca. 0.5 for cloth-filled composites and 1.0 for unidirectional fiber composites), and the subscripts c, f, and m stand for the composite, fiber, and matrix, respectively [14]. This equation predicts that the E_c value does not change as long as both E_f and E_m remain unchanged. Accordingly, the unchanged Young's modulus shown in Fig. 1 strongly suggests that the epoxy and polyimide resins as well as the glass and carbon fibers maintain their initial Young's modulus even after the irradiation up to 15,000 Mrad.

The shear modulus of a composite, on the other hand, may be approximated by the inverse rule of mixtures: $1/G_c = V_f/G_f + V_m/G_m$, where G is the shear modulus [14]. It is apparent, however, that this equation can not explain the dose dependence of the shear modulus shown in Figs. 2-5, because both G_f and G_m are supposed to be unchanged up to 15,000 Mrad owing to the relation: $G = E/2(1 + \nu)$, where ν is the Poisson's ratio. Nevertheless, it should be pointed out that the underlying principle of the inverse rule of mixtures is that the shear modulus is actually determined by the weakest part in the composite. The weakest part will therefore be the fiber/matrix interface. In addition, the interface appears to be the most radiation-sensitive part in the composite, because separation or debonding between fiber and matrix is optically observed in irradiated composites even before the mechanical tests [15]. From these considerations, it is reasonably concluded that the dose dependence of the shear modulus should be interpreted in terms of radiation damage at the fiber/matrix interface.

4.2 Failure Strength

The linear relationship found between the shear modulus and the ultimate strength (Fig. 6) strongly suggests that the dose dependence of the ultimate strength should also be interpreted in terms of radiation damage at the fiber/matrix interface. The radiation damage at the interface and the resulting debonding will decrease the capacity of load transfer from the matrix to the fiber, thus decreasing the ultimate strength and the shear modulus. This idea will hold true irrespective of the tensile and shear failure modes, because in both cases the failure of a composite is considered to occur when further increases in load could not be transmitted to the fibers due to the upper limit of the shear stress at the interface.

Taking into consideration these points, we derived the following formula to describe the dose dependence of the ultimate strength of composite materials, σ_{cu}:

$$\sigma_{cu} = \alpha\sigma_{fu}V_f\eta + \sigma_m^* V_m \tag{4}$$

with

$$\eta = \eta_b^o S + \eta_i \quad (\eta_b^o + \eta_i = 1) \tag{5}$$

$$S = \exp(-kD) \sum_{i=0}^{n-1} (kD)^i / \; i! \tag{6}$$

where σ_{fu} is the ultimate tensile strength of the fibers, σ_m^* is the matrix stress at the composite failure, η is the load transfer capacity normalized by its initial value, η_b^o is the initial contribution due to the chemical bonding, η_i is the contribution due to the frictional shear stresses at the fiber/matrix interface and/or between fibers [16], S is the probability of the debonding not taking place, D is the absorbed dose, k is the radiation-sensitivity parameter, and n is the number of hits in the target theory [17]. The details of these equations are described elsewhere [11].

The fitting procedure to the experimental data was made for various values of each parameter so as to determine the parameters which give the best fit between the calculated and observed plots. A reliable determination, however, was practically impossible at the present time for lack of experimental data for doses greater than 15,000 Mrad. Nevertheless, it was found that eqs. (4)-(6) can describe the dose dependence of the ultimate strength for the glass/epoxy and carbon/polyimide composites, as shown by the solid curves in Figs. 2 and 5, respectively. In these figures, the shear modulus is also fitted by using eqs. (4)-(6) and the linear relationship shown in Fig. 6. As to the carbon/epoxy and glass/polyimide composites, the fitting procedure was not made because eq. (6) fails to explain the slight increase in the ultimate strength which is observed for these composites (see Figs. 3 and 4). This increase may be associated with a radiation-induced increase in σ_{fu} in eq. (4). If this idea is true, and furthermore if the dose dependence of σ_{fu} is known, then the fitting procedure using eqs. (4)-(6) would be possible even for these composites.

4.3 Fracture Propagation

The dose dependence of the fracture propagation energy shown in Fig. 7 should also be interpreted in terms of radiation damage at the fiber/matrix interface. In fact, the propagation energy is formulated by taking into account the bonding energy at the interface by Outwater and Murphy [18]:

$$G_I = \frac{V_f d}{12 E_f \tau_i} \, [\sigma_{fu}^3 - (8E_f G_{II}/d)^{3/2}] \qquad (7)$$

where G_I is the propagation (or fracture) energy, G_{II} is the bonding energy, i.e., the energy required to debond the fiber from the matrix during fracture, τ_i is the frictional shear stress at the interface, and d is the fiber diameter. Although this equation is for unidirectional fiber composites under pure tensile stress, it will be valid even for cloth-filled composites under tensile and shear stresses if used for qualitative purposes. It predicts that the G_I value increases with a decrease in G_{II}. Accordingly, the increasing propagation energy with absorbed dose (Fig. 7) is most likely ascribed to the decrease in the bonding energy due to the radiation damage at the fiber/matrix interface. As to the carbon/epoxy composite, the bonding energy appears to be relatively small even before irradiation, thus making the propagation energy too large to be measured by the present three-point bend test. This will also be true with the glass/epoxy composite irradiated up to 15,000 Mrad.

It is worth noting that the propagation energy (Fig. 7) increases from the beginning of irradiation, whereas the shear modulus or the ultimate strength (Figs. 2-5) begins to decrease only after some accumlation of absorbed dose. From this finding it can be speculated that the decrease in the bonding energy is caused by each scission of the chemical bonds at the interface, whereas the debonding during irradiation is caused by some accumulation of these scissions. A detailed discussion of this point, however, would require a higher experimental accuracy as well as data from a wider dose range than is presented in the present work.

4.4 Fast Neutron Irradiations

When aromatic substances such as benzene and biphenyl are irradiated with various types of radiation, the decomposition efficiency increases with increasing LET (linear energy transfer, or stopping power) [19]. This LET effect is interpreted by the mechanism involving the competition between the deactivation of an excited molecule by eq. (8) and the radical formation by eq. (9):

$$M^* + M \rightarrow 2M \tag{8}$$

$$M^* + M^* \rightarrow \text{radicals} \tag{9}$$

where M^* and M stand for the excited molecule and a ground state molecule, respectively. High LET radiations such as fast neutrons produce these excited molecules more closely to one another than low LET radiations such as electrons and γ-rays, thus increasing the yield of radicals by the bimolecular reaction of excited molecules (eq. 9) at the expense of the competing unimolecular reaction (eq. 8). This mechanism applies also for polymers containing aromatic rings. In fact, the degradation efficiency of polystyrene for fast neutron irradiation in vacuo is 8 ~ 11 times larger than that for γ-ray irradiation [20]. It is therefore expected that the degradation efficiency of epoxy and polyimide resins is also larger for fast neutron irradiation, leading to an enhanced degradation efficiency of composite materials containing these matrix resins.

The results of fast neutron irradiations listed in Table 1, however, demonstrates that this expectation can not be confirmed at the present stage, because both the shear modulus and the ultimate strength do not degrade due to an incubation dose. Nevertheless, the expectation may be checked by looking at the fracture propagation energy for the glass/polyimide composite irradiated at room temperature. The rate of increase is estimated to be about 1.2×10^{-3} and 1.6×10^{-4} kg/mm/Mrad for fast neutron and electron irradiations, respectively (Table 1, Fig. 7). This result suggests that the degradation efficiency of polyimide resin is about 7 times larger for fast neutron irradiation and, consequently, the shear modulus and the ultimate strength of polyimide matrix composites may be much more sensitive to this type of radiation. Additional irradiations with fast neutrons are now being made to confirm this suggestion and to check the applicability of eqs. (4)-(6) to neutron irradiations.

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Figure Captions

Fig. 1. Plot of the Young's modulus vs. the absorbed dose of the matrix resin for the glass/epoxy (●), carbon/epoxy (+), glass/polyimide (o), and carbon/polyimide composites (x) irradiated with electrons.

Fig. 2. Plot of the shear modulus (o) and the ultimate strength (●) vs. the absorbed dose of the matrix resin for the glass/epoxy composite irradiated with electrons. The solid curves are the results of fitting procedure made by using eqs. (4)-(6) with $n=2$ and $k=2.86 \times 10^{-4}$ Mrad^{-1} (see text).

Fig. 3. Plot of the shear modulus (o) and the ultimate strength (●) vs. the absorbed dose of the matrix resin for the carbon/epoxy composite irradiated with electrons.

Fig. 4. Plot of the shear modulus (o) and the ultimate strength (●) vs. the absorbed dose of the matrix resin for the glass/polyimide composite irradiated with electrons.

Fig. 5. Plot of the shear modulus (o) and the ultimate strength (●) vs. the absorbed dose of the matrix resin for the carbon/polyimide composite irradiated with electrons. The solid curves are the results of fitting procedure made by using eqs. (4)-(6) with $n=10$ and $k=5.60 \times 10^{-4}$ Mrad^{-1} (see text).

Fig. 6. Correlation between the shear modulus and the ultimate strength. See the caption for Fig. 1 for the symbols of data point.

Fig. 7. Plot of the fracture propagation energy vs. the absorbed dose of the matrix resin for the composites irradiated with electrons. See the caption for Fig. 1 for the symbols of data point.

Table 1
Mechanical Properties of Composites Irradiated with Fast Neutrons

Composite	Irradiation temperature	Absorbed dose[a] (Mrad)	Young's modulus (t/mm^2)[b]	Shear modulus (kg/mm^2)	Ultimate strength (kg/mm^2)	Propagation energy (kg/mm)
glass/epoxy	—	0	3.27	273	77.0	0.222
	room temp.	46	3.28	269	77.7	0.283
	room temp.	347	3.42	281	83.4	0.355
	5 K	293	3.43	279	80.0	0.217
	5 K	413	3.32	299	81.4	0.262
carbon/epoxy	—	0	5.90	224	67.7	--
	room temp.	47	5.78	236	65.3	—
	room temp.	353	5.81	238	64.8	--
	5 K	286	5.87	239	60.4	—
	5 K	370	5.71	262	68.1	--
glass/ polyimide	—	0	2.50	184	50.6	0.252
	room temp.	43	2.53	215	51.3	0.248
	room temp.	297	2.57	198	53.3	0.598
	5 K	239	2.57	234	53.7	0.206
	5 K	429	2.51	220	51.6	0.210
carbon/ polyimide	—	0	5.17	186	74.7	2.073
	room temp.	45	5.13	213	74.5	1.896
	room temp.	333	5.19	206	77.4	0.902
	5 K	286	5.28	202	78.3	0.528
	5 K	351	5.10	209	76.7	0.745
alumina/ epoxy	—	0	11.27	349	131.1	1.181
	5 K	465	11.47	318	116.3	1.237

[a] Absorbed dose of the matrix resin

[b] $t = 10^3$ kg.

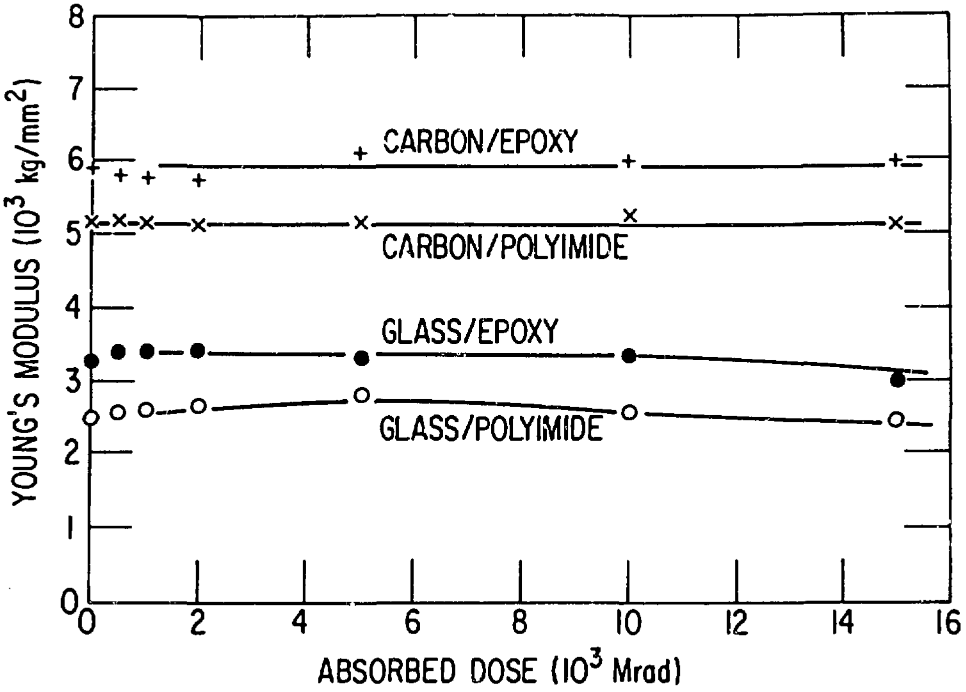

Fig. 1. Plot of the Young's modulus vs. the absorbed dose of the matrix resin for the glass/epoxy (●), carbon/epoxy (+), glass/polyimide (o), and carbon/polyimide composites (x) irradiated with electrons.

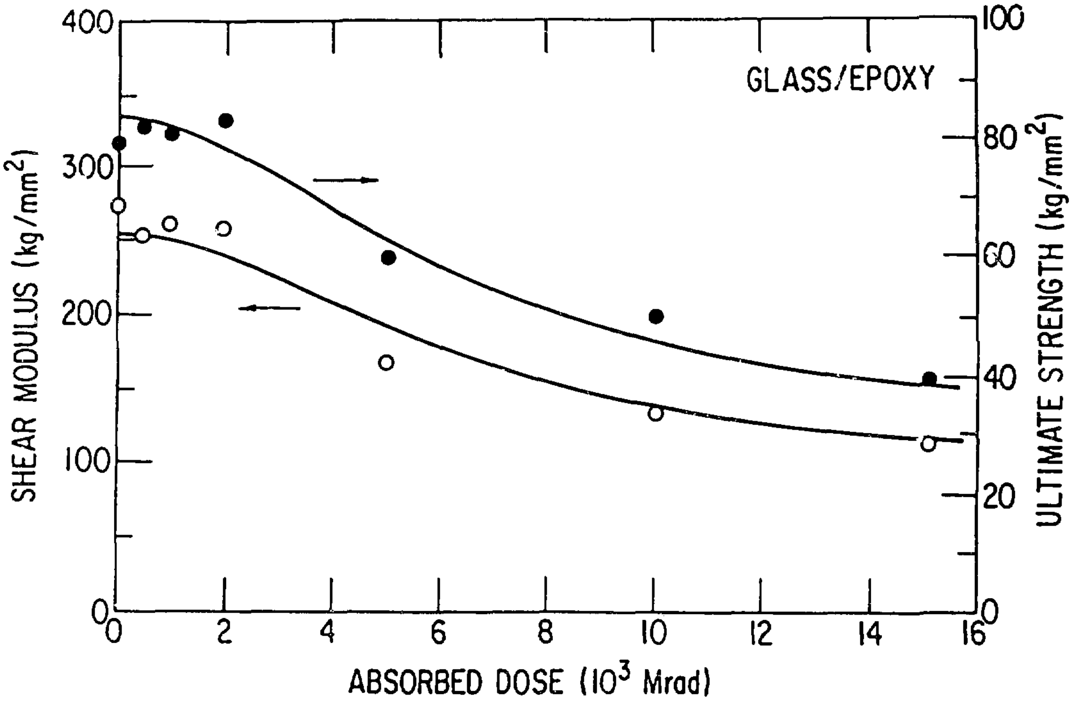

Fig. 2. Plot of the shear modulus (o) and the ultimate strength (●) vs. the absorbed dose of the matrix resin for the glass/epoxy composite irradiated with electrons. The solid curves are the results of fitting procedure made by using eqs. (4)-(6) with $n=2$ and $k=2.86 \times 10^{-4}$ Mrad^{-1} (see text).

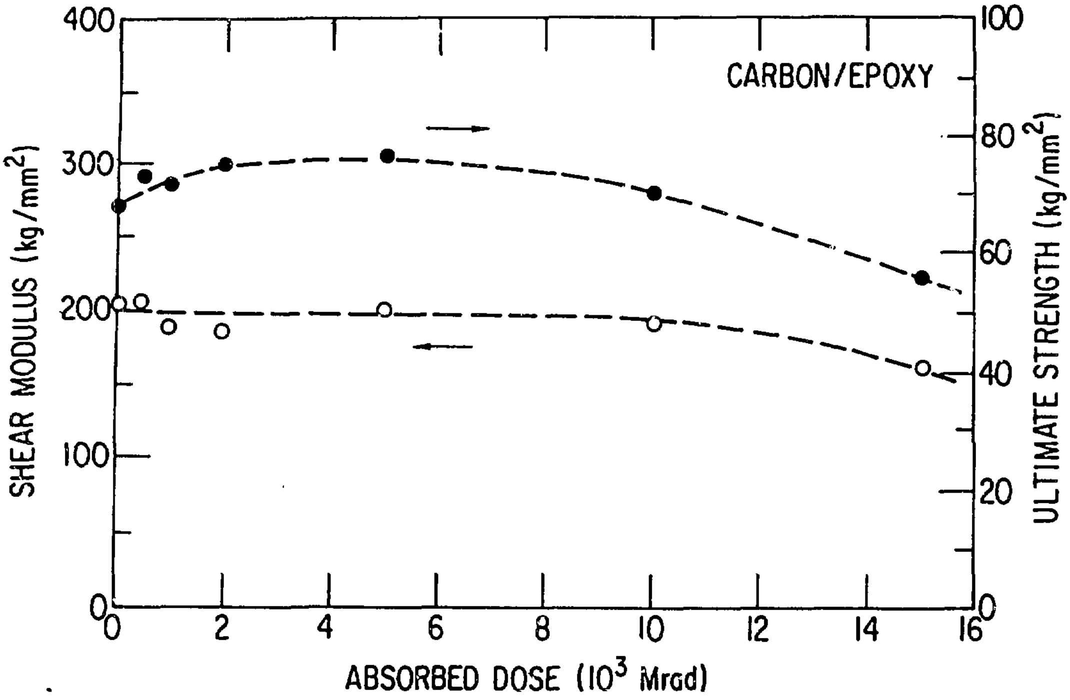

Fig. 3. Plot of the shear modulus (o) and the ultimate strength (●) vs. the absorbed dose of the matrix resin for the carbon/epoxy composite irradiated with electrons.

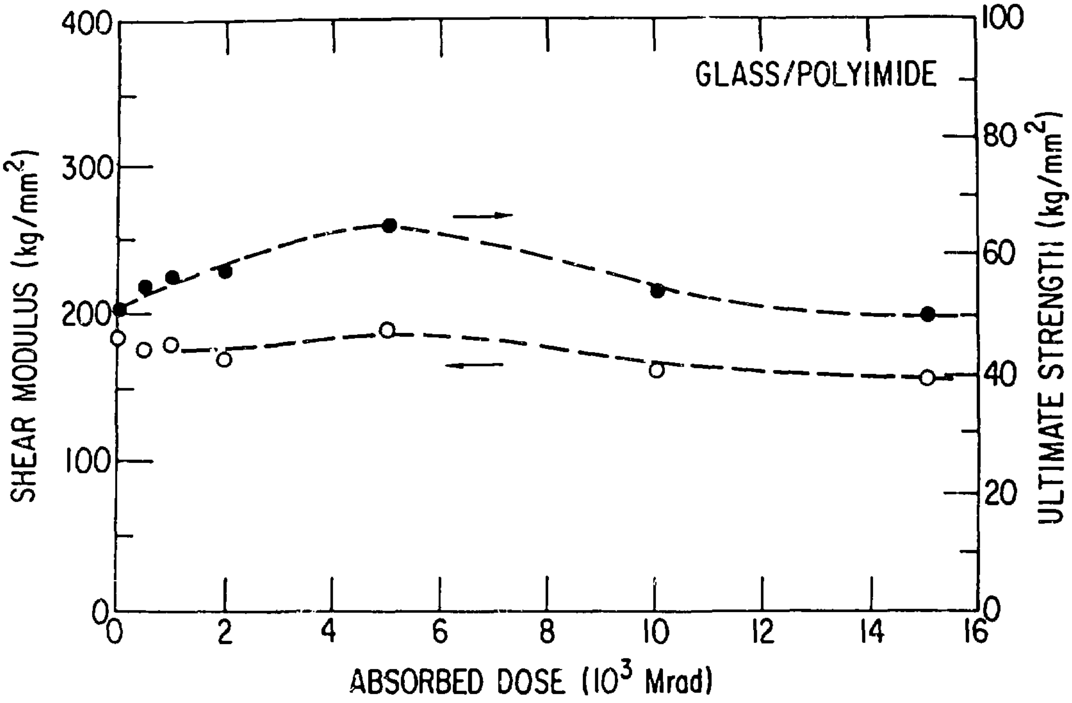

Fig. 4. Plot of the shear modulus (o) and the ultimate strength (●) vs. the absorbed dose of the matrix resin for the glass/polyimide composite irradiated with electrons.

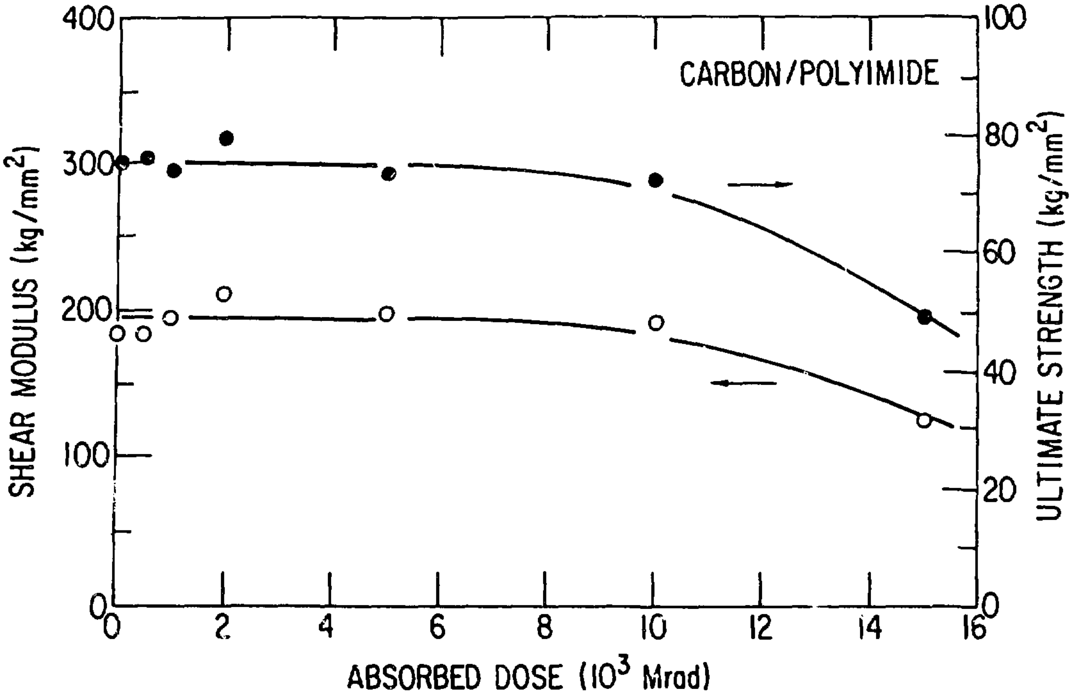

Fig. 5. Plot of the shear modulus (o) and the ultimate strength (●) vs. the absorbed dose of the matrix resin for the carbon/polyimide composite irradiated with electrons. The solid curves are the results of fitting procedure made by using eqs. (4)-(6) with n=10 and $k=5.60 \times 10^{-4}$ $Mrad^{-1}$ (see text).

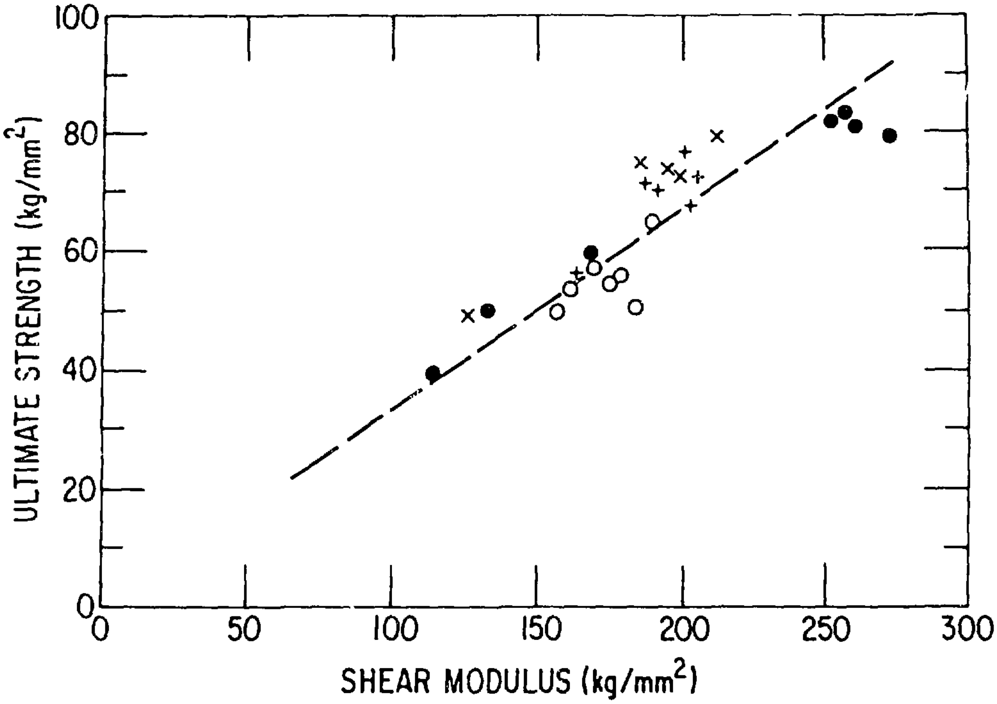

Fig. 6. Correlation between the shear modulus and the ultimate strength. See the caption for Fig. 1 for the symbols of data point.

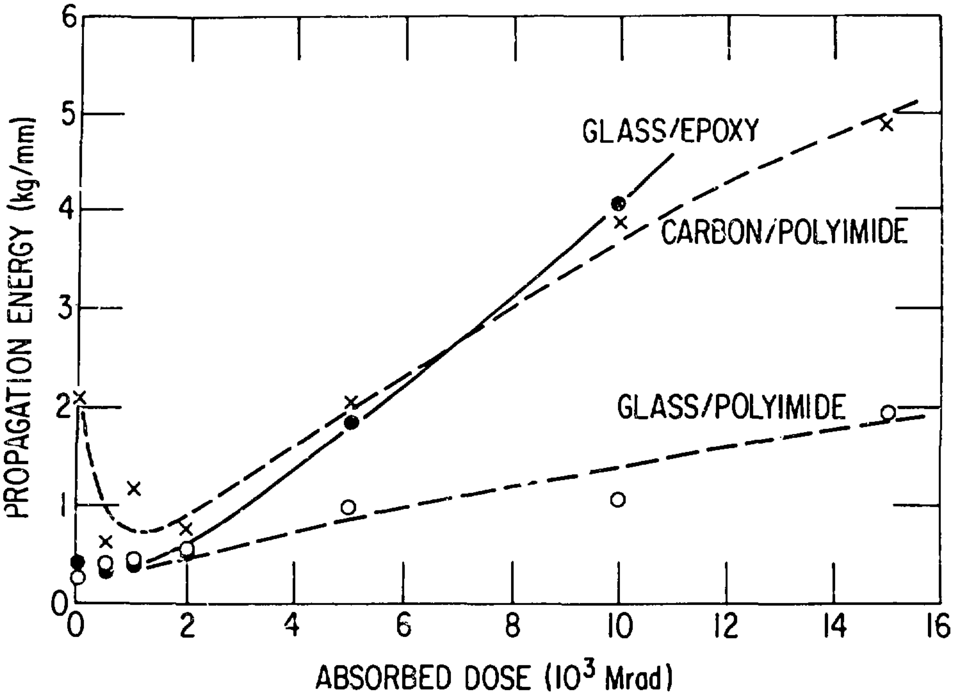

Fig. 7. Plot of the fracture propagation energy vs. the absorbed dose of the matrix resin for the composites irradiated with electrons. See the caption for Fig. 1 for the symbols of data point.