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**A PRELIMINARY ASSESSMENT OF THE EFFECTS OF RADIATION
ON POLYMER PROPERTIES**

**EVALUATION PRELIMINAIRE DES EFFETS DU RAYONNEMENT
SUR LES PROPRIETES DES POLYMERES**

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Pinawa, Manitoba R0E 1L0

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RÉSUMÉ

On a examiné brièvement la bibliographie relative aux effets du rayonnement sur les propriétés de divers polymères et complexes dans le but d'identifier les polymères qu'on pourrait irradier pour améliorer leur efficacité. L'irradiation des polymères pourrait conduire à une réticulation ou à des réactions de scission en chaîne selon la nature chimique du polymère. La réticulation conduit généralement à l'amélioration des propriétés mécaniques du polymère. La scission en chaîne conduit à la détérioration des propriétés mécaniques. En outre, les propriétés des polymères irradiés sont fonction du degré de cristallinité et des conditions d'irradiation dont le débit de dose et la présence d'oxygène, les agents de réticulation et d'autres additifs. On peut obtenir une augmentation de 30% de la résistance à la traction par réticulation au rayonnement dans des conditions appropriées.

Société de Recherche de l'Énergie Atomique du Canada, Limitée
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ABSTRACT

The literature on the effects of radiation on the properties of various polymers and composites has been briefly reviewed for the purpose of identifying polymeric materials that could be irradiated to improve their performance. Radiation treatment of polymers may lead to cross-linking or chain scission reactions, depending on the chemical nature of the polymer. Cross-linking generally leads to an improvement in the mechanical properties of the polymer. Chain scission leads to deterioration in mechanical properties. The properties of irradiated polymers also depend on the degree of polymer crystallinity and the irradiation conditions, including dose rate and the presence of oxygen, cross-linking agents and other additives. A 30% increase in the tensile strength of many polymers may be obtained by radiation cross-linking under appropriate conditions.

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1. INTRODUCTION

The literature on the effects of high-energy radiation on the properties of polymeric materials has been reviewed by a number of authors (Charlesby 1960, Chapiro 1962, Nikitina et al. 1963, Dole 1972 and 1973, Wilson 1974). Many of the publications in this field focus on radiation damage to plastics used in nuclear power plants (Bruce and Davis 1981, Harrington 1956, Parkinson 1969, Phillips 1978, Spadaro et al. 1984a and 1984b, Van de Voorde 1970, Wilski 1987, Wuendrich 1985). The effect of radiation on the properties of a polymer depends on the relative importance of cross-linking and chain scission reactions in that polymer. Cross-linking generally results in increases in tensile strength, hardness, softening temperature, solvent resistance, abrasion resistance, dimensional stability and adhesion, and a decrease in elongation at break (Huglin 1966, Bruce and Davis 1981). Chain scission usually leads to reduced tensile strength, hardness and softening temperature, and increased solubility and elongation. Some polymers that undergo chain scission ultimately form a liquid at high radiation doses. Cross-linking and scission usually occur simultaneously in a polymer and the overall change in properties depends on which process predominates.

Radiation-induced cross-linking of thermoplastic materials increases the softening temperature and, in this respect, the properties of cross-linked thermoplastics are more similar to those of elastomers than to the properties of the original thermoplastic. A schematic diagram of the tensile modulus as a function of temperature for these materials is shown in Figure 1 (Singleton and Claburn 1986). The strength of the thermoplastic is much reduced above its melting temperature, but the cross-linked thermoplastic maintains significant strength at the higher temperatures, as do the elastomers.

In Table 1 polymers are classified according to their response to high-energy radiation (Wilson 1974, Singleton and Claburn 1986). The determining factor in setting up the table was whether or not an insoluble gel is formed by irradiating the polymer under vacuum. The presence of alkyl-substituted carbon atoms in the polymer chain increases the probability that the polymer will undergo chain scission reactions during irradiation. For example, polyethylene cross-links, polyisobutylene degrades and polypropylene may either cross-link or degrade depending on the irradiation conditions. Similarly, the polyacrylates cross-link but the polymethacrylates undergo chain scission. Increasing degrees of halogenation of the monomer increase the tendency of the polymer to degrade during irradiation. For example, polyvinylidene fluoride and chlorinated polyethylene cross-link, polyvinyl chloride has an intermediate response, and polytetrafluoroethylene, polytrifluoroethylene and polyvinylidene chloride degrade. Obviously, those polymers which undergo radiation-induced cross-linking reactions are the best candidates for commercial radiation applications.

The relative amounts of cross-linking and scission in polymers is affected by the degree of crystallinity of the polymer (Wilson 1974). Increased crystallinity in the polymer reduces cross-linking between polymer chains and may enhance intramolecular cross-linking. Increased cross-linking also reduces the degree of crystallinity of the polymer because the three-dimensional molecules produced by cross-linking do not

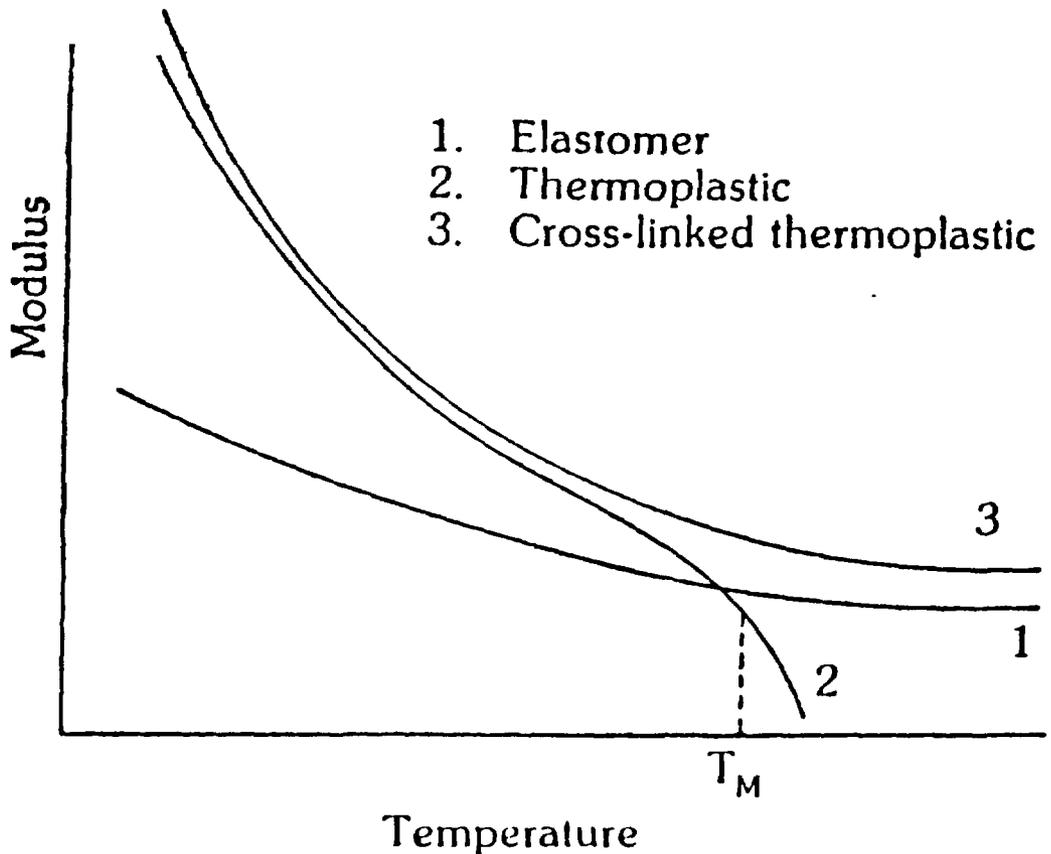


Figure 1: Schematic Diagram of the Effect of Temperature on the Tensile Modulus of an Elastomer, a Thermoplastic and a Cross-Linked Thermoplastic (from Singleton and Clabburn 1986)

crystallize easily. The effect of radiation on the strength of a polymer is moderated because irradiation increases the amount of cross-linking at the expense of the crystallinity, and both increased cross-linking and increased crystallinity normally increase the strength of a polymer.

TABLE 1
PREDOMINANT PROCESSES IN IRRADIATED POLYMERS
(WILSON 1974, SINGLETON AND CLABBURN 1986)

Cross-linking	Intermediate*	Scission
Polyethylene	Polypropylene	Polyisobutylene
Polyvinylidene fluoride	Polyvinyl chloride	Polytetrafluoroethylene
Polystyrene	Polyethylene oxide	Poly- α -methylstyrene
Polyacrylates		Polymethacrylates
Polyacrylamide		Polymethacrylamide
Polyamides		Polyvinylidene chloride
Polyesters		Cellulose
Polyvinylpyrrolidone		Cellulose acetate
Natural rubber		Polytrifluorochloroethylene
Synthetic rubbers		Polymethacrylic acid
Polysiloxane		Poly- α -methacrylonitrile
Polyvinyl alcohol		Polyethylene terephthalate
Polyacrolein		
Polyacrylic acid		
Polyvinyl alkyl ethers		
Polyvinyl methyl ketone		
Polymethylene		
Chlorinated polyethylene		
Chlorosulphonated polyethylene		
Polyacrylonitrile		
Sulphonated polystyrene		

* Cross-linking or scission may predominate for the polymers in the intermediate category, depending on the irradiation conditions.

The irradiation conditions that most affect the relative amounts of cross-linking and chain scission in polymers are the dose rate, and the presence of oxygen, additives and solvents (Wilson 1974). Chain scission is enhanced in polymers irradiated at low dose rates in the presence of oxygen or air (Wilski 1987). This effect is much reduced at dose rates greater than about 10 kGy/h, since there is insufficient time for oxygen to diffuse into the polymer prior to the occurrence of radical recombination reactions. The reaction of oxygen with polymer radicals produced during irradiation reduces the amount of cross-linking in the polymer. It also contributes to long-term degradation through the formation of peroxide, carboxylic acid, aldehyde, ketone and alcohol functional groups which act as initiating sites for autooxidation and degradation reactions induced by ultraviolet light.

Residual free radicals remaining in the polymer after radiation cross-linking will react with atmospheric oxygen over a period of months and cause a slow degradation of polymer properties. This effect may be minimized in some polymers either by heating to the crystalline melting temperature to allow the residual free radicals to recombine, or by including antioxidants in the polymer formulation (Bruce and Davis 1981, Roediger and Du Plessis 1986).

Most additives are likely to affect the response of polymeric materials to irradiation. Two types of additives are especially important in this regard, multifunctional monomers and inorganic fillers (Wilson 1974). The multifunctional monomers, such as allyl methacrylate and divinylbenzene, promote cross-linking by reducing the radiation dose required to form a three-dimensional network. Even some polymers that are susceptible to radiation degradation can be cross-linked in the presence of multifunctional monomers. Inorganic fillers, small particles dispersed in a polymer prior to radiation cross-linking, enhance the tensile strength and elastic modulus of the product. This effect may be due either to a chemical bond formation between the polymer and the filler particle, or to the reduced extension permitted by the physical presence of the filler particle inside the polymer network.

2. RADIATION EFFECTS ON SPECIFIC POLYMERS

Some data on the effect of radiation on the tensile strength and elongation at break of various polymers is presented in Table 2. Only data that showed an increase in either property as a result of radiation treatment was selected for inclusion in the table.

2.1 POLYETHYLENE

The literature on the effects of radiation on the properties of polyethylene is probably more extensive than that for any other polymer (see Mukherjee et al. (1986) for a recent review). Since polyethylene has a very simple chemical structure, it is the prototypical polymer for radiation cross-linking studies. Harrington (1956) performed an extensive study of the effect of gamma-ray irradiation in air on the mechanical properties of various polymeric materials, including several polyethylenes. The radiation dose range for this study was from 0 to 1.5 MGy and the dose rate was about 1.0 kGy/h. A dose of 100 kGy gave the largest increase in tensile strength (about 35%) in most of the polyethylene samples studied. The elongation at this dose increased for some samples and decreased for others, depending on which additives were present. The properties of previously irradiated low density polyethylene (LDPE) formulated for cable insulation decreased on further irradiation. Lawton et al. (1958) reported an increase in the tensile strength of Marlex-50 polyethylene from 18.6 to 34.5 MPa as a result of electron-beam irradiation in a nitrogen atmosphere. The higher strength of the irradiated Marlex-50 would make it suitable for use in applications that normally require polypropylene. Roediger and Du Plessis (1986) have shown that the tensile strength of polyethylene containing 5% triallylcyanurate, a chemical cross-linking agent, may be increased from 23 to 31 MPa by irradiating to a dose of 50 kGy. Lyons (1983) has

TABLE 2
CHANGES IN TENSILE STRENGTH AND ELONGATION PROPERTIES OF VARIOUS
POLYMERS INDUCED BY IRRADIATION

Polymer	Radiation Dose (kGy)	Tensile Strength (MPa)	Elongation (%)	Atmosphere	References
Polyethylene (LDPE)	0	12.4	-	air	Harrington 1956
	100	16.8 (+35%)*	(+46%)		
Polyethylene (HDPE)	0	23.7	20	air	Harrington 1956
	50	26.6 (+12%)	45 (+125%)		
Polyethylene (Marlex-50)	0	18.6	100	nitrogen	Lavton et al. 1958
	-	34.5 (+85%)	900 (+800%)		
Polyethylene (5% triallylcyanurate)	0	23	-	-	Roediger and Du Plessis 1986
	50	31. (+35%)	-		
Polypropylene (stabilized)	0	30.4	190	vacuum air	Begazy et al. 1986
	15	41.2 (+35%)	230 (+21%)		
	25	35.3 (+16%)	50 (-74%)		
Polyvinyl chloride (rigid)	0	32.	160	air	Zahran et al. 1985
	100	32. (0%)	240 (+50%)		
Polyvinyl chloride (plasticized)	0	17.9	310	air	Harrington 1956
	50	20.4 (+14%)	420 (+36%)		
Polyvinyl chloride (plasticized)	0	10.	-	-	Roediger and Du Plessis 1986
	50	23. (+130%)	-		
Epoxy (cycloaliphatic)	0	59.8	4.0	vacuum vet air	Spadaro et al. 1984a Spadaro et al. 1984b
	1500	66.0 (+10%)	4.0 (0%)		
	1500	66.0 (+10%)	4.0 (0%)		
Acrylate ester (fillers, additives)	0	6.69	275	air	Harrington 1956
	100	9.37 (+40%)	181(-34%)		
Polyurethane (fillers, additives)	0	29.4	530	-	Nikitina et al. 1963
	50	>48.0 (>+60%)	475 (-10%)		
Polystyrene	0	32.0	0.75	-	Nikitina et al. 1963
	100	34.5 (+8%)	0.99 (+30%)		
Styrene-Acrylonitrile Copolymer	0	12.3	230	air	Harrington 1956
	100	13.2 (+7%)	245 (+7%)		

- indicates information not given in original reference

* Values given in parentheses are the percent change (+for increase, - for decrease) in the value of the property induced by radiation treatment.

determined that the tensile modulus of irradiated high density polyethylene (HDPE) increases linearly with dose from 50 kGy to 400 kGy. The tensile moduli of HDPE samples with lower crystallinity (fast quench from melt) prior to irradiation are higher than those for samples of higher initial crystallinity.

There is a transient increase in the electrical conductivity of polyethylene during irradiation, but there is little permanent effect at

doses lower than those required for degradation of mechanical properties (Phillips 1978). Radiation cross-linked polyethylene shows a higher resistance to high voltage breakdown than normal polyethylene (Nikitina et al. 1963); the dielectric strength of the cross-linked polymer is about 10^5 V/mm compared to 2 to 4×10^4 V/mm for the normal polymer. Irradiated polyethylene is slightly coloured with the colour depending on the nature of the additives present.

2.2 POLYPROPYLENE

When polypropylene is irradiated in air, the tensile modulus increases, but both the tensile strength and the elongation at break decrease (Mukherjee et al. 1986). The tensile strength of the polypropylene increases by about 10% when the irradiation is performed under vacuum (Mukherjee et al. 1986). Both the glass transition temperature and the crystalline melting temperature of polypropylene decrease by 10 to 20°C at a radiation dose of 400 kGy. These effects are substantially different from those observed for irradiated polyethylene, because of the high degree of crystallinity of commercial polypropylene. Hegazy et al. (1986) observed a 35% increase in the tensile strength of stabilized polypropylene after irradiation to a dose of 15 kGy under vacuum. Sawasaki and Nojiri (1987) showed that irradiation of polypropylene in the presence of multifunctional monomers gave a product with increased tensile modulus (at 120°C) and higher impact strength with little change in other properties.

2.3 POLYVINYL CHLORIDE

Polyvinyl chloride (PVC) is cross-linked by high-energy radiation in the absence of oxygen or in large objects where oxygen cannot diffuse through the relatively impermeable PVC. Fine powders of PVC, however, are degraded by irradiation in the presence of air (Nikitina et al. 1963). The main chemical effect of radiation on PVC is the release of hydrogen chloride. The HCl trapped in the PVC matrix leads to an increase in the electrical conductivity of the polymer. If the HCl is produced from two neighbouring polymer chains (intermolecular process), cross-linking results, and intramolecular HCl production leads to double bond formation. When a double bond is formed in a polymer chain, the barrier to the release of neighbouring chlorine atoms is reduced, and conjugated double bonds are formed. Irradiated PVC is often strongly coloured because of the absorption of visible light by the conjugated double bonds.

Zahran et al. (1985) observed no change in the tensile strength of rigid PVC irradiated to 100 kGy, but the elongation of the sample increased by 50%. Harrington (1956) tested the effects of radiation on two different plasticized PVC formulations. One formulation showed a 36% increase in elongation and a 14% increase in tensile strength with little change in elasticity and hardness at a radiation dose of 50 kGy. The other formulation showed only minor property changes up to a dose of 100 kGy. Roediger and Du Plessis (1986) reported a plasticized PVC formulation which increased in tensile strength by 130% at a dose of 50 kGy.

2.4 POLYAMIDES

Fibres of aliphatic polyamides, such as nylon-6 and nylon-6/6, are degraded by relatively low doses of radiation in the presence of air (Bruce and Davis 1981), but several additives which improve their radiation stability are known. In contrast, the aromatic polyamides have much better radiation and oxidation resistance. Well known trade names for aromatic polyamides are Kevlar and Nomex. One study (Bernstein et al. 1965) has shown that increased gel fractions at lower radiation doses may be obtained when nylon-6/6 is swollen with allyl methacrylate prior to irradiation. But no information on changes in mechanical properties was obtained.

2.5 EPOXIES

The epoxies are among the polymers most resistant to damage by high-energy radiation (Bruce and Davis 1981, Van de Voorde 1970). Many epoxies show little decrease in mechanical and electrical properties at doses up to 40 MGy. Tilmans and Krokosky (1971a) and Fornes et al. (1981) have shown that both the tensile and flexural strengths of epoxy resins are increased by exposure to ionizing radiation. Spadaro et al. (1984a, 1984b) have shown that the tensile strength of a cycloaliphatic epoxy resin increased by about 10% at a dose of 1.5 MGy. There was no difference between the tensile strengths of the epoxy samples irradiated under vacuum and those of the samples irradiated in the presence of moisture-saturated air.

2.6 ACRYLATES

In general, the properties of acrylates improve with moderate radiation doses, but the properties of the methacrylates degrade (Bruce and Davis 1981, Van de Voorde 1970). Polymethyl methacrylate irradiated to a dose of 40 kGy in an inert atmosphere loses about 15% of its tensile strength (Parkinson 1969). The tensile strengths of many acrylates increase by about 10% at a dose of 50 kGy (Harrington 1956, Nikitina et al. 1963).

2.7 POLYURETHANES

Polyurethane elastomer formulations irradiated to doses of 50 to 100 kGy have significantly increased tensile strength (Nikitina et al. 1963). One sample irradiated to 50 kGy increased in tensile strength from 29.0 MPa to greater than 48.0 MPa with minimal change in other properties.

2.8 POLYSTYRENE

Polystyrene is quite resistant to irradiation under vacuum, but when irradiated in air at a dose of 1 MGy significant degradation of its tensile and flexural strengths is noted (Bruce and Davis 1981). Some increase in tensile strength is noted at a dose of about 100 kGy (Nikitina et al. 1963). Styrene-acrylonitrile and acrylonitrile-butadiene-styrene copolymers show increases in tensile strength at a dose of about 100 kGy (Bruce and Davis 1981, Harrington 1956).

2.9 POLYTETRAFLUOROETHYLENE

Polytetrafluoroethylene (PTFE) is readily degraded by high-energy radiation, especially in the presence of air (Bruce and Davis 1981). Only the impact strength shows a slight increase (25%) at 3.6 kGy. At this dose the other mechanical properties are reduced by >25%. A copolymer of fluoroethylene and perfluoropropylene is about 10 times more resistant to radiation than PTFE itself. Harrington (1956) showed that PTFE irradiated to a dose of 50 kGy in air has 41% less tensile strength and 93% lower elongation than virgin PTFE.

2.10 POLYESTERS

Polyesters are produced by condensation (esterification) reactions of polyhydric alcohols with difunctional acids. One common polyester is polyethylene terephthalate (PET), made by the reaction of ethylene glycol with 1,4-benzenedicarboxylic acid. PET is quite resistant to radiation (Bruce and Davis 1981). Non-oriented fibres (Dacron) have a damage threshold of about 44 kGy and oriented films (Mylar) have a damage threshold of about 400 kGy. Greater degradation is observed at lower dose rates, but no report of enhanced mechanical properties has been discovered to date.

Unsaturated polyesters are made by the reaction of unsaturated difunctional acids with polyhydric alcohols (Allcock and Lampe 1981). Typical unsaturated acids used in this application are maleic acid and fumaric acid. The unsaturated polyesters may be dissolved in a vinyl monomer, such as styrene, and cross-linked in a mould to produce a thermoset material. The cross-linking of an unsaturated polyester with a vinyl monomer is a vinyl polymerization reaction, and as such, is a good candidate for initiation by high-energy radiation.

2.11 ELASTOMERS

Rubbers, both natural and synthetic, are usually vulcanized, or cross-linked, during processing. Irradiation makes little, if any, additional improvement in their mechanical properties (Allcock and Lampe 1981, Bruce and Davis 1981). Radiation treatment may however be used in place of the normal vulcanization process to increase the strength of the uncured rubber (Mohammed and Walker 1986). The most promising use of radiation in tire manufacture appears to be in the enhancement of green (uncured) strength of the inner tire liner. This increased strength results in greater form stability and retention of shape during assembly, moulding and vulcanization. Increased green strength must be balanced against reduced tack, and Mohammed and Walker (1986) have shown that the optimum radiation doses for chlorobutyl and bromobutyl rubber formulations are 10 to 15 kGy and 10 to 25 kGy, respectively.

2.12 COMPOSITES

The effects of irradiation on the properties of advanced composites are relevant to the use of composites as construction materials in nuclear reactors and spacecraft. Bullock (1972a) proposed irradiating graphite fibre-epoxy matrix composites, to a dose sufficient to substantially reduce the strength of the epoxy matrix and its adhesive bond to

the fibres, in order to study means of making the best use of fibre strength properties in the composite. Fornes et al. (1981) found that irradiation up to a dose of 50 MGy slightly increased the tensile strength and modulus of graphite fibre composites with epoxy, polyimide and polysulphone matrices. Bowles et al. (1986) found that irradiation of graphite-epoxy and graphite-polyimide composites introduced residual strains in the composite, but did not change their coefficients of thermal expansion. Bullock (1972b and 1974) observed increases of 10 to 20% in the transverse flexural strength and 2 to 5% in the interlaminar shear strength of boron fibre-epoxy matrix composites irradiated to about 7 MGy. The transverse flexural strength primarily measures the strength of the resin matrix, and the interlaminar shear strength is indicative of the strength of the fibre-to-matrix bonding. Tilmans and Krokosky (1971b) showed that irradiation increases the strength of bonding between epoxy coatings and both glass and metal substrates. Tennyson and his co-workers (1982, 1983a, 1983b, 1983c) and Mazzio and Huber (1983, 1984) have also made measurements of the properties of irradiated composites. The radiation doses required for significant increases in strength properties of epoxy matrix composites are probably too high to permit their use in an industrial setting.

3. CONCLUSIONS

- (1) Radiation induces both cross-linking and chain scission in polymeric materials.
- (2) Cross-linking leads to improved mechanical properties of the polymer and scission leads to deterioration of mechanical properties.
- (3) Highly crystalline polymers do not cross-link effectively and cross-linked polymers do not crystallize easily. Since both higher crystallinity and higher cross-linking give improved strength, the radiation effects on the strength of crystalline polymers are usually small.
- (4) Higher radiation dose rates favour cross-linking over chain scission, for polymers irradiated in air or oxygen atmospheres.
- (5) Multifunctional monomers absorbed into a polymer matrix prior to irradiation enhance the strength of the product.
- (6) Inorganic fillers increase the strength of radiation-cross-linked polymer formulations.
- (7) The increased mechanical properties of radiation-cross-linked polyethylene and rubber has led to commercialization for radiation treatment of these polymers in the wire insulation and automotive tire industries.
- (8) The tensile strength of many polymers may be increased by 10 to 40% by irradiation under appropriate conditions.

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