Polymer Characteristics and Fracture Morphology of Radiation-Polymerized Styrene-Impregnated Mortar

VIJAY K. BHATTACHARYA, MRINAL M. MAITI, BASUDAM ADHIKARI, and SUKUMAR MAITI, Polymer Division, Materials Science Center, Indian Institute of Technology, Kharagpur 721302, India

Synopsis

Mechanical and durability properties and the fracture morphology of polystyrene-impregnated mortar composites prepared by impregnation of styrene and its *in situ* polymerization by gamma radiation have been studied. Variation of flexural strength of the composites, percent extractable polystyrene, and its molecular weight suggest crosslinking of the impregnated polymer under the influence of prolonged gamma irradiation. Moreover, crosslinking appears to contribute toward the improvement in flexural strength but has little effect on durability properties. SEM micrographs of the composites reveal a very weak cement-polymer interfacial bonding which is believed to be responsible for poor durability properties of the composites.

INTRODUCTION

Impregnation of cement-mortar or concrete with polymers has been found to greatly improve the performance characteristics of the substrate matrix. Among them more important ones include mechanical properties, durability in chemical and hydrothermal environments, and freeze-thaw resistance.¹⁻³ It is suggested that these improvements are primarily due to pore filling by the polymer which acts as continuous, randomly oriented reinforcing network.⁴ The decrease in porosity is one of the factors controlling the properties of the composite; but this alone possibility cannot account for the different performance recorded with different polymers. Additional factors are therefore believed to be involved.⁵ Although a number of reports on the improvement in the properties of polymer-impregnated concrete (PIC) have been published, very little is known about the nature and characteristics of the polymer inside the pores and the correlation, if any, between the properties of the polymer itself and the ultimate properties of PIC.⁶ The molecular weight of the impregnated polymer is important in this respect, for most of the properties of a polymer are governed by its molecular weight. It has been reported that the molecular weight of the polymer obtained by in situ polymerization in cement-matrix is greater than that obtained by bulk polymerization.⁶⁻⁸ Molecular weights of polymethyl methacrylate (PMMA) prepared by thermocatalytic in situ polymerization^{6,8,9} and radiation-induced ones^{7,10} have been reported. However, so far, no work has been reported on the influence of radiation and its overdose on the molecular weight of the impregnated polystyrene and its consequent effects on the ultimate mechanical properties of PIC.

In the present communication, results on the effect of dose of gamma radiation on the flexural strength, the molecular weight of the extractable polystyrene, and durability in dilute acid solution of radiation-cured polystyrene/mortar composites have been presented. Attempts have been made to interpret the results in terms of the radiation chemistry of the monomer and polymer as well as by matrix-polymer interfacial adhesive strength. Scanning electron fractographs of the composites were also utilized to lend support to the interpretations.

EXPERIMENTAL

Specimen Preparation. For preparation of mortar samples ordinary portland cement (ACC, India) and Cossey river bed sand retained between sieves BS-25 and BS-52 were used. The mortar specimens were prepared according to the specifications of ASTM 303-65 with sand/cement ratio of 3.0 and water/ cement ratio of 0.5. The mortar specimens were prisms of $10 \times 2 \times 2$ cm. The mortar slurry was handmixed, and during casting the molds were kept on a vibrating table for 15 min to facilitate removal of air bubbles as far as practicable. Specimens were demolded after 24 h and allowed to cure at ambient temperature under water for 7 days. Finally the specimens were dried in an oven at 130°C for 20 h and stored in a desiccator.

Impregnation and Polymerization. Monomer impregnation was carried out by the method of Villamizar et al.¹¹ Previously weighed specimens were subjected to evacuation in a vacuum chamber for 6 h to 2–4 mm Hg, and, subsequently, the styrene monomer was introduced while maintaining the vacuum. The monomer did not contain any dissolved initiator, catalyst or promoter. The specimens were kept immersed in the monomer for an optimum period of 6 h and then taken out and wrapped with aluminum foil to minimize monomer loss by evaporation during subsequent polymerization by gamma irradiation.

The monomer, commercial grade styrene (SRL, India), was washed free of inhibitor and was distilled under vacuum before use.

The *in situ* polymerization of the impregnated styrene was carried out in a Gamma Chamber 900 unit (BARC, India). The source was Co-60 and had an intensity of 0.1066 ± 0.01 Mrad/h at the time of experiments. Specimens were taken out from the irradiation chamber after irradiation for a predetermined dose. Residual monomer was removed by vacuum heating at 50°C. Percent monomer conversion and percent polymer loading were determined gravime-trically.

Mechanical Properties. Flexural strength of the styrene-impregnated radiation-cured mortar samples [PIC(R)-PS] was determined in a Universal Testing Machine (Fu 10,000e, Veb Thuringer Industrie Werk, West Germany) by center point loading. Only the flexural strength of PICs has been studied, since the stress under flexure would provide both compression and shearing, and thus the interfacial bonding between polymer and mortar would directly come into effect.

Polymer Extraction and Molecular Weight. PIC(R)–PS samples were ground to powder of BSS 100 mesh size in a ball mill. A weighed quantity of the powdered material was subjected to soxhlet extraction for an optimum period of 72 h using cyclohexane (BDH, India) as solvent. Polystyrene was precipitated by methanol (BDH, India), purified by reprecipitation and finally dried under vacuum at 60°C to constant weight.

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Days of	Total dose ^a of radiation	Monomer (styrene)	Polymer loading	Flexural strength	Extractable polymer ^b	wt
irradiation	(Mrad)	polymerized (%)	(0_{0}^{\prime})	(MPa)	(%)	$M_v \times 10^{-3}$
0 (control)	0	0	0	4.51	1	1
1	2.56	7.18	0.92	5.78	74	60.8
2	5.11	$19.44 (20.17)^{c}$	2.24	7.05	78	$103.7 (125.7)^{c}$
с г	7.67	34.56	3.49	11.07	77	145.6
4	10.23	42.20 (42.8)	4.86	10.97	88	155.8(145.6)
5	12.79	57.95	6.62	16.36	88	162.8
9	15.35	84.74 (55.5)	10.35	21.17	77	170.5(255.7)
7	17.90	84.65	10.20	22.62	42	287.8
. 8	-	— (52.6)	I	I	I	- (263.6)
10	25.58	84.90	10.42	23.71	38	236.1
11	28.14	86.10(52.0)	10.50	22.73	45	194.8(224.7)
^a At 0.1066 ± 0	.01 Mrad/h.					

^b Solvent–cyclohexane. ^c Values in parentheses relate to bulk polymerization.



Fig. 1. Variation in percent styrene polymerized, percent polymer loading, and flexural strength of PIC(R)–PS with days of irradiation: (O) % monomer polymerized; (Δ) % polymer loading \times 10; (\Box) flexural strength.

Intrinsic viscosity of the extracted polymer was determined in cyclohexane at 34°C by an Ubbelohde suspended level viscometer. The viscosity average molecular weight (\overline{M}_v) was determined indirectly using equation¹²

$$[\eta] = 82 \times 10^{-5} \,\overline{M}_p^{0.5}$$

Bulk Polymerization of Styrene. Bulk polymerizations of styrene were carried out in small glass ampules under almost identical conditions of gamma irradiation as adopted in the case of PICs. Conversions of the monomer were determined by extracting the irradiated materials by hot cyclohexane followed by precipitation with methanol. The precipitated polymers were reprecipitated twice and dried under vacuum at 60°C to constant weight.

Molecular weights of the polymers were determined viscometrically as described in the earlier section. Amounts of crosslinked gell in each case were determined from the residue left after soxhlet extraction of the soluble fraction of the polymers by cyclohexane.

Durability. Durability of PIC(R)–PS samples in aqueous- H_2SO_4 (5% w/w) solution was determined in terms of cumulative weight loss after 3, 7, and 10 days of immersion in a stagnant environment. The acid solution was changed after 3 and 7 days.

Fracture Morphology. Fracture surfaces of the PIC(R)–PS samples were cut after performing flexural tests and vacuum coated with graphite followed by copper. Scanning electron micrographs were taken in an ISI-60 model within 48 h of mechanical testing.



Fig. 2. Variation in percent extractable polystyrene and its molecular weight with days of irradiation: (O) molecular weight $\overline{M}_v \times 10^{-3}$; (Δ) % extractable polymer.

RESULTS AND DISCUSSION

Flexural Strength

In Table I are presented data on the percent conversion of the monomer (styrene) to polymer, percent polymer loading, and flexural strengths of PIC(R)-PS samples. Percent extractable polymer in cyclohexane and the viscosity average molecular weight (\overline{M}_v) of the extracted polymers are also entered in Table I.

The conversion of monomer to polymer rises exponentially up to 6 days (15.35 Mrad) (Fig. 1). A marginal increase is observed beyond 6 days to a limit of about 86% over a period of another 5 days (12.8 Mrad). The conversion was never 100% due to the loss of monomer by evaporation. Similar results have also been reported by other workers.⁷

So far as polymer loading is concerned, it increases sharply to about 10.35% after 6 days with only a little further increase thereafter up to 11 days of irradiation. Flexural strength of PIC(R)–PS composites also exhibits a parallel trend with a level of 21 MPa attained after 6 days (~15 Mrad) and only 23.7 MPa after 10 days (~ 26 Mrad) of irradiation, which is about five times that of the control (4.5 MPa).

Polymer Extraction

Inside the pores of mortar the polymer may remain in two forms. One fraction of the polymer remains strongly bound to the inorganic cementitious mortar matrix through some sort of interaction¹³ whereas the other fraction acts just

Days of	Total ^a dose	Polymer loading	Percent cumulative weight loss in aqueous-H ₂ SO ₄ (5% w/w) medium after (days)		ive 5% er
irradiation	(Mrad)	(%)	3	7	10
0	0	0	7.8	19.0	29.3
1	2.56	0.92	7.8	25.6	29.4
2	5.11	2.24	7.5	24.3	40.8
6	15.35	10.35	7.7	19.2	24.3
7 .	17.90	10.20	7.3	20.7	25.7
10	25.58	10.42	7.8	20.8	27.8
11	28.14	10.50	7.2	16.7	21.7

 TABLE II

 Durability of PIC(R)-PS Composites in Aqueous-H2SO4 (5% w/w) Medium

^a At 0.1066 ± 0.01 Mrad/h; environment—stagnant; temperature—ambient.

as pore filler without entering into any interaction, chemical or physical, with the matrix. This chemically or otherwise nonbound fraction is extractable with a suitable solvent. The variation of the amount and the viscosity average molecular weight (\overline{M}_v) of extractable polymer from PIC(R)–PS composite against number of days of irradiation are shown in Figure 2.

It is seen from Figure 2 that the amount of extractable polymer increases gradually and attains the maximum value (88%) at about 5 days of irradiation (~13 Mrad) and subsequently decreases with increase in the total dose of irradiation. The initial increase of the amount of extractable polymer is in keeping with the increase in the conversion of monomer to polymer inside the cement matrix with the time of irradiation. However, the decrease in the amount of extractable polystyrene at later period (i.e., after 5 days) of irradiation may be due to onset of crosslinking. When the impregnated monomer is first polymerized and then crosslinked, the polymer cannot be extracted by the solvent. In the crosslinked gel there is a possibility of entanglement of the uncrosslinked fraction of the polymer, which is consequently difficult to be extracted. Under such conditions larger polymer molecules may be retained relatively easily than the smaller ones simulating perhaps an action of size exclusion chromatography.¹³ It may be noted here that a fraction of unextractable polymer is likely to be present chemically bound to the cement matrix.

Characteristics of Bulk Polymerization

Bulk polymerizations of styrene were carried out to ascertain if there could be any difference in the polymerization characteristics of styrene in bulk and in the mortar matrix. Relevant data are entered in Table I. It is quite apparent from the data that the rate of conversion to polymer in either cases is not affected up to 4 days of irradiation. Also, in both the cases, the optimum conversion is observed at 6 days of irradiation. Beyond 6 days, however, conversion is limited to only about 52% in bulk polymerization compared to about 86% in mortar matrix.

This higher rate of polymerization in mortar matrix compared to that in bulk



Fig. 3. Variation in cumulative weight loss in aqueous- H_2SO_4 (5% w/w) of PIC(R)–PS composite with days of immersion. (1) Control; (2) PIC(R)–PS prepared with 15.35 Mrad (6 days); (3) PIC(R)–PS prepared with 28 Mrad (11 days).

has also been reported for methyl methacrylate by other workers.^{2,7} A possible energy transfer effect,² or reduced gel effect⁷ in mortar matrix, has been offered to account for the observation.

The molecular weights of the bulk polymers, however, did not show any appreciable difference from those of the *in situ* polymers. A maximum molecular weight was observed at 8 days of irradiation, beyond which a small decrease in molecular weight up to 11 days has been recorded.

Crosslinking of bulk polymerized polystyrene was observed only after 6 days of irradiation. The extent of crosslinking was rather low, but increased with the increase in the days of irradiation. The values were only 0.40%, 0.73%, and 0.84% of the polystyrene formed after 6, 8, and 11 days of irradiation, respectively.

Molecular Weight

Molecular weight of the extractable polymer with cyclohexane increases steadily and attains the maximum value possibly at about 8 days of irradiation and then decreases (Fig. 2). Although, under the influence of gamma irradiation polymerization, crosslinking and chain scission take place, at initial stages of irradiation, polymerization is the predominant process causing molecular weight of the polymer to increase. At the later stage of irradiation, crosslinking and chain scission would be the main processes due to the lack of sufficient monomer present in the polymerization site. While crosslinking would increase the proportion of polystyrene insoluble in cyclohexane, chain scission would result in the lowering of molecular weight of the extractable fraction of the polymer. It has been reported¹⁴ that the rate of crosslinking, G(c.l.), of polystyrene by gamma irradiation is about 0.04 and the scission to crosslinking ratio, (β/α), is 0.14. Consequently, crosslinking would be predominant. It is also to be noted that only the lower molecular weight fraction which are yet to be crosslinked or obtained as a result of chain scission can escape through the pores of the crosslinked



Fig. 4. SEM fractograph (234×) of PS-mortar prepared with 10 days (~26 Mrad) of irradiation. Entry path of styrene monomer is clearly visible as rootlike network of polymer-filled wide pores with narrower branching. Only a few very fine pores terminate at a spherical microvoid of about 0.06 mm diameter. This is responsible for a limited filling of the microvoid by polymer. Surface of the polystyrene embedded in the pores is smooth and convex, suggestive of a low degree of polymer-mortar interfacial adhesion.

gel whereas the larger molecules are trapped. Presence of an enhanced proportion of the crosslinked polystyrene as well as of the higher molecular weight fraction of the uncrosslinked polystyrene entrapped inside the crosslinked gel is perhaps responsible for the observed trend of a slow but steady increase in flexural strength of the PIC(R)-PS composite (Fig. 1).

Durability

In Table II is presented percent cumulative weight loss after 3, 7, and 10 days of immersion of the specimens in the test solution. Figure 3 shows the typical curves for cumulative weight loss with days of immersion for control and PIC(R)–PS prepared with 6 days (~15 Mrad) and 11 days (~28 Mrad) of irradiation.

It is apparent that the loss in weight of the composites with time follows more or less a similar trend. The durability improved with increased polymer loading, though not very significantly.

These behavior of PIC(R)–PS composites clearly establishes that impregnation of PS into mortars, though it improves strength properties considerably, offers rather little improvement in durability in dilute H_2SO_4 medium. A weaker cement–polymer interaction is believed to be responsible for such behavior. This aspect has been discussed further in the following section.

Scanning Electron Micrograph of Fractured Surface of PIC(R)-PS

SEM fractograph of PIC(R)-PS were taken to collect information about the state of polystyrene in the composites. The following important observations have been recorded.

Filling Up of Microvoid by the Polymer: Extent of monomer impregnation into mortar matrix has been a subject of considerable investigation and is known



Fig. 5. SEM fractograph (156×) of a PS-mortar composite prepared by 6 days (~15 Mrad) of irradiation. A solid polystyrene bead embedded in mortar matrix formed by complete filling of the microvoid through wide entry pores, one passing across the diameter and the other at the upper left side of the photograph. A smooth and convex surface of the bead is an evidence in favor of a weak polymer-mortar compatibility. A brittle fracture pattern of the imbibed polymer is clearly discernible at the sides of the bead.

to depend upon such factors as the viscosity of monomer at the experimental temperature, time of impregnation, initial vacuum attained in the impregnation chamber, external pressure applied on the liquid monomer, as well as the number and dimension of the pores terminating at the voids.¹³ Figure 4 represents a SEM fractograph of a PS-mortar composite prepared with ~26 Mrad of total radiation in which path of entry of monomer into the matrix is clearly discernible as rootlike network of polystyrene-filled wide pores with narrower branching. Also apparent is a spherical macrovoid of about 0.06 mm diameter with a thin film of polymer on the inner surface. Passage of monomer into the void is through very fine micropores terminating at the spherical void. The pores are also very few in number. These two factors together are believed to be responsible for a very limited filling of the void.

In Figure 5 the white line passing along the diameter and the one terminating at the upper left end of the sphere are the monomer entry paths into the void, which due to their larger dimensions have helped complete filling of the void by the monomer. These two fractographs (Figs. 4 and 5) clearly demonstrate that the monomer loading into the microvoids of the matrix is largely governed by, besides other factors, the pore dimension and the number of pores leading to microvoids.

Mechanical and Constitutional Characteristics of the Imbibed Polystyrene: It has already been shown (Table I) that the dose of gamma radiation required for an optimum conversion (~85%) of polystyrene is about 15 Mrad, beyond which the flexural strength of the composites register further improvement with very little or no increase in polymer conversion and loading. The observation has been explained to be due to the formation of crosslinked polystyrene under the influence of high energy gamma radiation. The fractographs of PS-mortar composites (Figs. 6 and 7) reveal a striking coincidence with the above explanation. The fractographs of the composites prepared by 7 days (Fig. 6) and 10 days (Fig. 7) of irradiation bear some well-scattered bright spots which are not found with composites prepared by 15 Mrad (6 days) and lower dose of radiation. These scattered bright spots are possibly the nucleation zones of



Fig. 6. SEM fractograph $(139.8\times)$ of a PS-mortar composite prepared by 7 days (~18 Mrad) of irradiation showing a solid polystyrene sphere embedded in mortar matrix with a smooth and convex surface. Some scattered bright spots possibly indicate the onset of crosslinking of polystyrene. Spots are due to segregation of the crosslinked phases. Brittleness of the polymer is indicated by the fracture pattern at the bottom right of the bead.

crosslinking polystyrene matrix. Due to lack of sufficient segmental motion of the molecules compared to uncrosslinked polymer, these crosslinked phases are more insulating in character and are identifiable as bright spots due to more intense scattering of incident electron beams.

In Figure 7, these crosslinked phases are distributed along the concentric beach marks in the fractograph. A plausible explanation for this localized distribution of crosslinked phases is that the polymers are more abundant along the beach marks, which is quite apparent from the wavy fracture characteristics of polystyrene (PS) at the two middle beach marks. These bright spots are identifiable also in other parts of fractographs in Figure 7 as also in Figure 4.

Ductility/Brittleness of the Imbibed Polystyrene: The fractographs of the composites (Figs. 4, 5, and 7–10) also exhibit a gradual transformation from a ductile to brittle nature of the polymer with an increase in the total dose of radiation. The network of polymer at the fracture surface of composite formed



Fig. 7. Fractograph $(64.8\times)$ of a PS-mortar composite prepared by 10 days (~26 Mrad) of irradiation. Brittle fracture of polystyrene is indicated by steps and stripes with distinct impression of the direction of fracture propagation. A small solid polymer bead at the bottom right and bright spots of crosslinked polystyrene scattered along the concentric beach marks are clearly visible.



Fig. 8. Fractograph of a PS-mortar composite prepared by 2 days (\sim 5 Mrad) of irradiation. Ductile behavior of the impregnated polymer at the fracture surface is clearly indicated by fibrillar impression and flow characteristics of the polymer around a microcrater. Possibly the ductility has been induced by low molecular weight of the polymer.

with 2 days (5 Mrad) of irradiation, as revealed in Figure 8, is a typical of ductile behavior. The ductility has been induced due to low molecular weight of PS formed at lower dose (Table I) of irradiation. However, beyond 5 days (~13 Mrad) of irradiation the nature has totally changed to one of brittle fracture. It may be mentioned here that with polymers a true brittle fracture as encountered with crystalline metals and alloys or amorphous glass are seldom observed. Even at low temperature or with polymers having high glass transition temperature (T_g) ductile deformed films and fibrils become visible at high magnification. Thus the brittleness of polymer as reported here is only a macroscopic feature and is characterized by steps and striped patterns is also by the impressions of the direction of fracture propagation. All these characteristics are clearly visible in Figures 5, 7, 9, and 10.

Figure 7 is particularly interesting in that it provides an elaborate microcharacteristics of many parallel propagation paths from a central point of origin with concentric beach marks lying at right angles to the bands. Occurrence of such a type of fracture path is possibly due to the presence of a very localized zone (or the central point of origin) of high resistance to applied stress, which when overcome triggers a sudden propagation of innumerable fractures at a very fast



Fig. 9. An enlarged view $(247\times)$ of Fig. 7 depicting several concentric beach marks with the divergent impression of the fracture waves propagating at right angles to the beach marks.



Fig. 10. Fractograph $(16.9\times)$ of a PS-mortar composite prepared by 6 days (~15 Mrad) of irradiation. The picture shows a hollow sphere of polystyrene with a big perforation at lower right of the sphere. Some small perforations are also visible. Surface of the sphere is rough. Possibly, this is caused by an easy deformability of the polymer shell because of lower thickness, and does not indicate a stronger PS-mortar interfacial interaction.

rate. The beach marks are believed to originate from the concentric stress accumulation zones at which the velocity of fracture propagation is restricted considerably.

Mortar–Polymer Interfacial Adhesion: Fractographs of the mortar–PS composites also provide some important informations about the strength of mortar–polymer interfacial interaction. It is quite apparent from the fractographs, (Figs. 4–6) that the fracture surfaces of the polymer phase, present either as solid bead (Figs. 5 and 6), or as root like network (Fig. 4), are all virtually smooth and typically convex. Such characteristics of the fracture surface indicate that the fracture waves have predominantly propagated through the mortar–PS interface which have offered minimum resistance to fracture propagation. Obviously, the PS–mortar interfacial interaction is very low and hence much weaker in strength compared to the cohesive strengths of either polystyrene or the mortar matrix.

In Figure 10, however, a very rough surface of a microbead of polystyrene with wide perforations are clearly visible. This roughness of the polymer surface may not be an indication of a stronger interfacial adhesive strength, since the polymer bead under consideration is a hollow one and thin enough to be easily deformed by the applied stress.

A weak interfacial interaction between PS and mortar matrix can also be inferred from the durability behavior of the composites. It has already been discussed earlier that the durability of PS-mortar composites in sulfuric acid medium is not improved to any significant extent over that of the control mortar, although a sharp incease in the mechanical strength could be observed. In a comparative study of the durability of mortar-PMMA and mortar-PS composites in sulfuric acid medium,¹⁵ it has been argued that a poorer durability corresponds to a situation in which the polymer-filled pores allow a relatively easier entry of the corrosive medium. Thus the poor durability properties of mortar-PS composites as observed in the present study, as also in the reported one,¹⁵ are very much suggestive of a weak interfacial interaction and hence a poor interfacial adhesive strength between PS and mortar matrix. As indicated elsewhere, this poor interfacial interaction or adhesive strength possibly originate from the very nonpolar character of polystyrene^{15,16} as well as from the absence of any reactive functional groups which would have offered firm anchorage to the cementitious matrix.¹⁷

CONCLUSIONS

In conclusion, it may be stated that:

Mortars impregnated with styrene and polymerized by Co-60 gamma irradiation have flexural strength about five times that of control mortar at a polymer loading of about 10%.

The amount of polystyrene extractable with cyclohexane as well as the \overline{M}_v of the extracted polymer decreased with longer exposure (>15 Mrad) to gamma radiation. This is believed to be due to increased degree of crosslinking of polystyrene with higher doses of radiation.

Durability of PS-mortar systems, however, is little improved over the control at all level of exposure to gamma radiation up to 26 Mrad.

Improvement in the strength of mortar-PS composites is ascribed to the mechanical properties of the imbibed polystyrene, its loading in the matrix as well as by the polymer-mortar interfacial bond strength.

Improvement in the durability of the composites is determined by the rate of seepage of the aggressive liquid medium into the matrix, which, in turn, is governed more by the interfacial adhesive strength between the polymer and mortar than the loading and strength of imbibed polymer.

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