Effects of γ Radiation on Fiber-Reinforced Polymer Concrete

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Composites were made from 30% unsaturated polyester resin + 70% calcium bentonite and marble as aggregates, as well as 0.3 and 0.4 vol% of nylon fibers. The fiber-containing polymer concretes (PCs) were subjected to 5, 10, 50, and 100 kGy applied radiation doses. The compressive strength values depend on both the fiber concentration and the irradiation dose applied. Moreover, the polyester-based PCs containing two mineral aggregates, calcium carbonate and marble, have lower compressive strength values than those reported earlier for PCs containing only either silica sand or CaCO₃. However, significant improvement of the compressive strain and the compression modulus of elasticity are achieved when nylon fibers are added. Both these properties go symbatically with the radiation dose. Mechanical characteristics can be related to the morphological features observed by scanning electron microscopy. POLYM. COMPOS., 29: 1244–1251, 2008. © 2008 Society of Plastics Engineers

INTRODUCTION AND SCOPE

It is well known that polymer concrete (PC) is three to five times stronger than Portland cement concrete (PCC), but brittle failures of the former have limited its usefulness for load-bearing applications. Thus, improvements in PC toughness and postpeak stress-strain characteristics are highly desirable, particularly in fatigue loading, impact, and earthquake conditions [1, 2]. Such improvements can have far reaching consequences, since cements constitute not only one of the oldest classes of materials developed by mankind but also one of the most used in terms of annual volume around the world [3–5].

The fiber-reinforced composites (FRCs) have a reputation for high stiffness, strength, and creep resistance. A variety of such materials has been developed for a variety of specific applications in structural civil engineering, automotive applications, and high temperature corrosive environments. Nevertheless, there is very limited information on the fiber-reinforced PC systems. Thus, substantial experience and broad knowledge on the optimal compositions, properties, and stress–strain relationships are desirable, in particular with respect to design and quality control.

Some reinforcements have been added to PC, such as glass or organic fibers. These reinforcements have little effect on the precracking behavior but do substantially enhance the postcracking response, improving the toughness and the ductility as well as the tensile, flexural, and impact strength.

Reinforcement fibers have been usually placed along the principal stress directions. For example, glass-fiber-reinforced polymer (G-FRP) rebars used in PC have shown two behaviors in creep strain as consequence of the orientation of the fibers in the loading direction [6]. For unidirectional composites, the changes in the tensile strength may be enhanced by optimization of several parameters, such as length and number of fibers, constituent material properties, including interface, and also features of the microstructure (volume fraction and spatial arrangement of the fibers) [7].

Reis and Ferreira report improvement by 95% in the flexural strength for polyester-based PC system after adding 2% glass fibers and using silane as a coupling agent [2]. In other experiments, both the flexural strength and the fracture behavior of epoxy-based PC materials have been improved by adding 2, 4, or 6 wt% of chopped glass...
fibers (without sizing and soaked in a 2% silane solution); the polymer matrix concentration was between 10 and 18% by weight [8]. Specific characteristics of the chopped glass fibers are needed: 800 bonded glass strands, each 13 μm in diameter, 13 mm long, density 2.55 g/cm³, tensile strength 2.5 GPa, and tensile modulus of 70 GPa [8].

Moreover, to improve the fracture behavior of the epoxy-based PC systems, Reis and Ferreira added 1 wt% of high-performance chopped carbon fibers. The fibers were made from poly(acrylic nitrile) (PAN); their average length was 6 mm [2].

As mentioned before, mechanical improvement depends on the fiber type and the fiber concentration in a PC. For example, Ribeiro et al. report that plain epoxy mortars require 1 wt% of chopped glass fibers or 2 wt% of chopped carbon fibers; both kinds of fibers without sizing and soaked in a 2% silane solution, 6-mm long [9]. They have prepared plain mortar formulations by mixing foundry sand (80 wt%) and an unsaturated orthophthalic polyester resin (20%). The resin was a viscous liquid with a styrene monomer content of 44%; it was preaccelerated by the manufacturer and the methyl ethyl ketone peroxide (MEKP) (2 phr) was used as initiator [9]. The PCs were allowed to cure for 7 days at room temperature and then postcured at 80°C for 3 h [9]. Other formulations have been used for manufacturing PCs, for example polyester-based PCs with unsaturated polyester resin with a styrene monomer content of 43.9% (20 wt%); sand (80%), and MEKP (2.5%) as catalyst [10], as well as our PCs formulations studied recently [11, 12].

The choice of the fine aggregates is quite important to assure good workability of the PCs; that property depends on the number of constituents in the aggregates. The mix design of PC typically uses an aggregate gradation to provide the lowest possible void volume that will require the minimum polymeric binder contents necessary to coat the aggregates and to fill the voids, for example, PC with 15 wt% of resin and 85 wt% of siliceous sand with grain size of 245 or 342 μm [2, 9]. Sometimes, the sand has different grain sizes, for example, 1,350 μm (60%), 120 μm (30%), and 670 μm (10%) [13]; or 850 μm (23%), 425 μm (10.2%), and 90 μm (6.1%) [14]. Apart from the grain size, it is necessary to take into account the composition of the aggregates. For example, when using bentonite, it is recommended to apply sedimentation techniques to separate the clay fraction from the coarse fraction. As we know, bentonite consists of montmorillonite and small amounts of fragments that include plagioclase group, silica group (quartz and its polymorphs tridymite and cristoballite), calcite, and sometimes volcanic glass [15].

Compressive strength values for a number of glass-fiber-reinforced-PCs (G-FRPCs) formulations have been reported [8]. The polyester resin content was typically in the range 10–18 wt%, while the fiberglass in the range 2–6 vol%. The results show that the compressive strength values are between 33 and 83 MPa, and as expected depend on the fiber and the polyester resin content. For unreinforced-PC, the compressive strength values show an increment-decrement-increment behavior (56-68-62 MPa) for 10-14-18% of the resin content, respectively. A similar behavior is found for 2% of fiber: 60-78-66 MPa for 10-14-18% of the resin content. For higher fiber content (4 and 6%), the compressive strength increases when the fiber content increases. For 4% of fiber, the sequence is 42-70-83 MPa for 10-14-18% of the resin content, respectively; and for 6% of fiber it is 33-60-80 MPa. Thus, the maximum compressive strength value (83 MPa) is found for 4% of the fiber and 18% of resin content. Moreover, there is an optimal fiber content (based on maximum strength) for each resin content. If there is a higher fiber concentration then more polymer binder is needed to coat the fibers and the silica sand particles.

For epoxy-based PC with 80 wt% of foundry sand and 20% of epoxy resin, maximum compressive strength values are found according to the fiber kind and contents: without fibers (59 MPa), with 1% of glass fibers (64 MPa); and with 2% of carbon fibers (69 MPa) [2].

Another important mechanical property is of course the modulus of elasticity in compression $E$. For example, in a glass-fiber-reinforced-PC (G-FRPC), the values decrease when the fiber contents increase (from 0 to 6% in volume) [8]. Moreover, for each of the fiber content, $E$ increases along with an increase of the polyester resin concentration (from 10 to 18 wt%). Thus, for unreinforced-PC, a maximum value is obtained for 18% polyester resin (7.1 GPa). The modulus of elasticity values range from 3.5 to 7.1 GPa. Variations of $E$ are probably due to entanglements of some glass fibers, resulting in pores and causing a decrement in the stiffness. Sometimes silane as coupling agent is added to the monomer to improve the bond strength between the resin and the aggregates. However, the main problem arises from the viscoelastic properties of the resin; the polymers usually have a low modulus of elasticity and are flexible, and exhibit creep behavior [16].

For epoxy-based PC with 80% of foundry sand and 20% of epoxy resin (parts by mass), maximum values of the modulus of elasticity in compression were found, according to the fiber added: without fiber (11.3 GPa), with 1% of glass fiber (10.8 GPa); and with 2% of carbon fiber (11.5 GPa) [2].

As for strain at failure, in polyester-PCs, the values increase when the glass fiber concentration increases. For 10% of polyester resin content, the values go from 0.013 to 0.024 mm/mm when the fiber contents increase from 0 to 6 vol%. Values for 14 and 18% of polyester resin are similar: from 0.015 to 0.024 mm/mm and 0.019 to 0.027 mm/mm for 14 and 18%, respectively [8].

Most of the manufactured polyamides such as nylon are used in fiber production. The commercial success of polyamides is due to their outstanding properties and economical advantages. Small amounts of nylon fibers substantially improve the impact resistance of composites due to stretching and pull-out of the fibers, which occurs
at relatively low loads and results in large strains after failure. Nevertheless, the added fibers have very little effect on tensile or bending strength. Thus, it would be advantageous if composites could be designed to support an increasing load after the cracking of the matrix. This situation can be solved by improving the stress transfer from the matrix to the fibers after matrix failure. The transfer will depend on the aspect ratio of the fiber and the interfacial shear strength. Thus, there are possibilities for improving these parameters by modifying the fiber surfaces or the polymeric matrix surface [17].

In some composites, chemical bonding (sometimes referred to as elastic bonding) between the fibers and the matrix is not strong in comparison to frictional resistance along the debonded segment against pull-out. In general, friction plays an important role with increasing fiber size in confining stress [18]. In addition, most fiber deformation processes lead to local mechanical interactions between fiber and matrix and, therefore, may be regarded as a macroscopic “roughening” effect. Two techniques used to modify the interfacial bond between polymeric fibers and cementitious matrixes are the fibrillation and the twisting deformation of the fibers. The fibrillation increases the surface contact area and enhances mechanical anchoring to the matrix, as well as improves the fiber modulus. Both techniques are particularly suitable for polymeric fibers due to their low strength and large strain capacity. In the case of nylon fibers, the pull-out process can be significantly altered by mechanical crimping of the fibers; moreover, the mechanical bond is so strong that pull-out of the fiber is limited by the strength of the nylon fiber [18].

Improvements of the PCs depend on physicochemical properties of both the resins and the mineral aggregates. An alternative is using γ radiation; it has several advantages compared with thermal processes or chemical attack. The advantages include: curing at ambient temperature; no need for additives; better control of part dimensions and elimination of internal stresses, which reduce material strength; shorter curing times; and no emission of volatiles to the environment [19].

γ radiation produces a high rate of the radical formation, in general orders of magnitude higher than with classical initiators. The radiation initiation does not require any activation energy, and the termination reaction is practically always diffusion-controlled. Higher viscosity means higher activation energy of the termination reaction, decreasing the overall activation energy. When using chemical initiators, the decrease of the overall activation energy (by a few kJ per mole) does not compensate for the increased radical concentration and poorer heat transfer. In radiation-initiated polymerization reactions, however, the overall activation energy may fall to very low values, making thermal explosions impossible [20].

When the polymeric resins are irradiated, the chain scissions result in the formation of the free radicals. Clough reports that both radical and cationic crosslinking mechanism can occur in irradiated composites of epoxy resin and carbon or aromatic polyamide [19]. Thus, we have both weakening of the material by scissions and strengthening it by crosslinking. According to Nishiura et al. [21], there is more creep during irradiation than afterward; the authors explain this by chains scissions during irradiation. While that explanation is plausible, we have a different one. Already in 1965, Kubat [22] demonstrated that different materials including metals and polymers have similar stress relaxation curves. He explained this by cluster relaxation, a collective phenomenon. Cluster relaxation has been subsequently seen in stress relaxation simulations of metals and polymers by molecular dynamics [23–26]. Creep is a “twin” to stress relaxation in terms of the variable kept constant and the variable followed [16]. Like stress relaxation, we believe that creep is also a collective phenomenon. The clusters have a size distribution, with clusters containing between 6 and 16 statistical segments more probable than, say, a cluster containing only one such segment [24]. Computer simulation of cluster formation has demonstrated that the cluster size distribution can be represented by an equation. However, the simulation was performed for a system containing individual uncrosslinked polymeric chains [24]. Crosslinking necessarily has to increase the size of clusters undergoing creep. Therefore, more energy per cluster is needed to cause a local creep event, and the number of such events is lower in a crosslinked material.

In unsaturated polyester resins submitted to γ radiation, the reaction runs smoothly and the product is flawless—contrary to badly foamed products obtained when using catalysts. Moreover, impregnation and curing of different substrates with polyester resins at high temperatures are safer in the absence of initiators [20].

γ radiation effects on calcium bentonite depend on the granulometrical fraction (clay and coarse), which in turn reflects differences in the mineralogical composition. For example, thermoluminescence (TL) response of the coarse fraction in calcium bentonite is two orders of magnitude stronger than for the clay fraction, and the concentration of radiation-induced defects increases with increasing dose. As a result, the coarse fraction has a higher concentration in defects (more than one order of magnitude) than the clay fraction. These results are consistent with the fact that the coarse fraction contains minerals (silica and plagioclase groups) that are very sensitive to radiation. In the case of the clay fraction, the γ radiation promotes defects in its crystalline lattice, mainly affecting the stability of the Al—O and Si—O bonds. The defects are holes trapped in the former positions of O atoms in the structure [15].

One of the most important applications of calcium Bentonite is as engineering barrier for long-living radioactive waste materials from the nuclear industry such as soluble salts, aqueous solutions of nitrates, oxides, and glasses [14]. The requirements for acting as an engineering barrier include radiation and thermal stability, and structural
integrity. For example, aqueous nitrates with calcium aluminates (in high early strength mortars and concretes) and impregnated with styrene-divinyl benzene have shown radiation stability up to 100 MGy, which is the total integrated dose expected for 1,000 years exposure [14].

In the present work, we have studied the γ radiation effects on fiber-PC systems consisting of an unsaturated polyester resin, calcium bentonite, marble, and nylon fibers. We focus on mechanical improvement in relation to morphological behavior.

EXPERIMENTAL PROCEDURES

Specimen Preparation

For preparing the polymer concrete specimens, calcium bentonite and marble as fine aggregates from a local company (GOSA™, Tlahnepantla, Mexico) were used. Both aggregates were dried before adding to polymeric resin in an automatic mixer. Then, we have added crystalline nylon 6,12 fibers (DuPont, Wilmington, DE) with approximate length of 5 mm and diameters varying from 30 to 40 μm.

The polymer matrix was a commercial unsaturated pre-accelerated polyester resin (orthophthalic), a viscous liquid resin with a styrene monomer content of 30% (Polylite 32493-00™, Reichhold, Atlacomulco, Mexico). The concentration of methyl ethyl ketone peroxide (MEKP) initiator added to the polymer for starting the free-radical polymerization process was 1 mL/100 g of the resin weight.

Five different polymer concrete lots identified by A, B, C, D, and E were prepared, each one a different day. The proportion of the polyester resin in the polymer concrete was 30% by weight and the fiber contents were 0.3 and 0.4% in volume. Also a 100% polyester resin specimen as a reference was prepared. After mixing, the concrete cubic specimens (5 × 5 × 5 cm³) were placed in a controlled temperature in room at (23.0 ± 3.0) °C up to 72 h.

Mechanical Tests

The compressive tests of the polymer concrete cubic specimens were carried out in an Instron Universal Testing machine model 1125. The charge speed was between 91 and 184 kg/s, and a cross head speed of 0.5 mm/min, holding the charge until reaching the maximum value to assure the reliability of the test.

Morphological Characterization

The surfaces of the nylon fibers, polyester resin, and the manufactured fiber-PCs, before and after irradiation, were analyzed by scanning electron microscopy (SEM) in a JEOL model JSM-5200 machine, in the secondary-electron mode. Previously to the observation, these components were vacuum-coated with carbon (thickness between 3 and 10 nm) in a vacuum pump (E.F. Fullam) at 50 mTorr. The secondary electron mode is preferred in the present case, while for instance in characterization of polymer + metal hybrids, backscattered electrons provide a better contrast [27].

Irradiation Procedure

The nylon fibers and the polymer concrete were exposed to varying γ radiation doses using a 60Co source. The fibers were placed in packets of 50 into a capillarity tube. The experiments were performed in air at the room temperature; the dosages were 5, 10, 50, and 100 kGy at the dose rate of 6.10 kGy/h. The irradiation was provided by a 651 PT Gammabeam Irradiator manufactured by the Atomic Energy of Canada (AECL, Chalk River, Ontario), and located at the Institute of Nuclear Sciences of the National Autonomous University of Mexico.

COMPRESSIVE STRENGTH

In Fig. 1, we see that all irradiated PCs have higher compressive strength values than those nonirradiated. Moreover, independently of the applied radiation dose, all fiber-PCs have higher values than those PCs without fibers.

In general, the values for PC with 0.3 vol% of fiber are higher when comparing to composites with 0.4%, except for 5 kGy. Thus, a higher fiber concentration lowers the compressive strength values. The maximum compressive strength improvements with respect to nonfiber PC are 33 and 51% for 0.3 and 0.4 vol% of the fiber, respectively.

The compressive strength behavior of irradiated-fiber PC shows the following stages: (I) increase for 5 kGy; (II) then a decrease from 5 to 10 kGy; and (III) finally, an increase again from 10 to 100 kGy. This type of behavior has been observed for PC composites containing SiO₂ [11] or CaCO₃ [12]. The minimum values are located at 10 kGy. However, the improvements at this...
dose vary from 4 to 14% with respect to PC without fibers.

The compressive strength necessarily depends on the fiber concentration and the radiation dose while well-defined stages are present (periodical behavior). The fibers do not have a preferential arrangement in the PC and suffer mechanical deformations when the PC is submitted to the compression test; the deformations can vary according to the applied dose. Now consider the tensile stress behavior of irradiated fibers carried out in separate experiments. We have reported before that the maximum values of the tensile stress at yield point for nylon 6,12 fibers (used also in this work) are achieved at 10 and 50 kGy (with improvements of 143% with respect to nonirradiated fibers); for higher doses up to 100 kGy the values go down [28]. Thus, at 10 and 50 kGy, the fibers behave as a hard material, conversely, at 100 kGy, a soft material is obtained. Relating to the compressive strength of the fiber-PCs, we infer that “hard” fibers (at 10 and 50 kGy) cause lowering of the compressive strength. By contrast, at 100 kGy “ductile” fibers provide more compressive strength to the fiber-PCs.

Such mechanical behavior is a consequence of morphological changes produced by γ radiation. We see in Fig. 2a surface deterioration of irradiated nylon fibers at 5 kGy. The deterioration increases when the applied dose increases to 10 kGy (Fig. 2b), at this dose the lowest compressive strength values are found. When the dose increases to 100 kGy, scrap particles emerge from the fibers (Fig. 2c)—a consequence of the formation of oligomers created by scissions of the chains. Since the fibers are blended with the polyester resin and the aggregates, the scrap particles inside the blend serve as fillers (less than 1 μm in length). As a consequence, we achieve higher compressive strength values.

We are dealing with composites containing 70% of calcium bentonite and marble as mineral aggregates, 30% of polyester resin, and 0.3 or 0.4 vol% of nylon fibers. Using calcium bentonite, it was not possible to reach values higher than 67.5 MPa for compressive strength after irradiation. This value can be compared with those reached for polyester-based PCs with other aggregates, for example, with silica sand (from 64.1 to 97.0 MPa) [11] or with CaCO₃ (from 86.4 to 135.0 MPa) [12]. Thus, a combination of calcium bentonite and marble diminish the compressive strength performance.

**COMPRESSIVE STRAIN AT YIELD POINT**

Figure 3 tell us that all irradiated fiber-PCs have higher values than those standard values for polyester-based PC reported (0.01 mm/mm). For nonfiber PC, the compressive strain increases along with the applied radiation dose. The situation for PCs with 0.3 vol% of fiber is similar. However, at 0.4% of fibers, the strain behavior is very different, now three stages are present: (I) an increase for 5 kGy; (II) a decrease for 10 kGy; and (III) an increase again for dose values higher than 10 kGy. Similar periodic behavior has been observed in other polyester-based PC composites [11, 12].

In contrast to the compressive strength, in the case of the compressive strain, the influence of the calcium car-
bonate and marble is large. It is possible to obtain 290% of improvement with respect to the reported values for polyester-based PC [1]. Let us compare our compressive strain results (varying from 0.011 to 0.039 mm/mm) with previous ones for polyester-based PC composites. Differences are notable; for PC with silica sand (SiO_2) as aggregate the values are between 0.0009 and 0.0018 mm/mm [11]; for PC with CaCO_3, the values are from 0.012 to 0.016 mm/mm [12]. We conclude that the combination of two different aggregates and nylon fibers as fillers generate a more ductile material—in contrast to materials, which contain only one aggregate.

Our high compressive strain values can be explained in terms of surface morphology of the PCs and the tensile strain values of the same nylon fibers obtained by us earlier [28]. For irradiated PC at 5 kGy, a heterogeneous morphology is observed, including the aggregate particles covered by polyester resin (Fig. 4a). When increasing the applied radiation dose, a softer surface is seen and the aggregate particles are fully covered (Fig. 4b). This situation is a consequence of crosslinking of the chains in the two polymers involved, the polyester resin and the nylon fibers. Moreover, for higher applied doses, the polyester resin is constrained, the surfaces show more agglomeration regions, what produces the highest compressive strain values (Fig. 4c).

Previously, we have reported a maximum improvement of 26% for the strain of the raw nylon fibers when the applied dose was increasing up to 50 kGy, but a lowering by 58% when one reaches 100 kGy [28]. In the 0–50 kGy interval, there is a partial damage and a possibility of repair of the bonds. Apparently, there is chain reorientation due to the repolymerization of the fibers with an increase in the crystallinity; however, if the radiation process goes on, the damage can be permanent [29]. In the present work, we reach the lowest compressive strain values at 10 kGy, not at 50 kGy as for the fibers, what implies there is an influence of the polyester resin on the nylon fibers.

**COMPRESSION MODULUS OF ELASTICITY**

We do not need to argue the importance of ductility—or brittleness [30] on properties. The ductile behavior effects on strain at the yield point are corroborated by the compression modulus of elasticity $E$ (see Fig. 5). All our materials have $E$ values below the standard ones reported for polyester-based PC composites (6.7 GPa) [1]. In general, the modulus decreases with increasing fiber concentration. For nonfiber PC, a proportional decrement is observed when increasing the radiation dose. However, a different behavior is observed for PC with 0.3 or 0.4 vol% of fibers—where several stages are seen. For 0.3% of the fibers: (I) an increase from 0 to 5 kGy and (II) a decrease from 5 to 100 kGy. More stages are observed for PC with 0.4% of fibers: (I) a decrease from 0 to 5 kGy; (II) an increase from 5 to 10 kGy; and finally (III) a decrease for higher dose (at 50 and 100 kGy). Similar multistage behavior has been observed for polyester-based PC with silica sand (SiO_2) [11] or with CaCO_3 [12].

![FIG. 4. SEM micrograph of irradiated PC with 0.4 vol% of fibers: (a) 5 kGy, (b) 50 kGy, and (c) 100 kGy.](image)

![FIG. 5. Compression modulus of elasticity of nylon-fiber polymer concrete.](image)
After irradiating PC with 0.4 vol% of fibers, a notable result was found: the composite has the lowest modulus of elasticity (1.5 GPa), 77% lower than those reported in the literature for polyester-based PCs. Thus, by using high radiation dose (100 kGy), we have a highly deformable PC, which has a moderate compressive strength value (65.4 MPa).

While the applied radiation affects both the polyester resin and the nylon fibers, apparently the resin is more affected. To understand the changes of the elasticity modulus of the PCs, we have irradiated the neat polyester resin and studied the morphological changes by SEM; see Fig. 6.

For nonirradiated resin, a homogeneous surface is seen. It contains regions not fully polymerized by the MEKP catalyst (Fig. 6a). When we apply 50 kGy irradiation (Fig. 6b), a surface consists of several constraining regions, apparently responsible for the elasticity modulus reported above. At 100 kGy, the γ radiation results in the presence of two morphologies on the resin surface, constrained regions, and voids, what leads to the highest elasticity modulus (Fig. 6c).

Improvement in mechanical properties caused by irradiation is clearly related to morphology of the components and the void volume in the composites. The larger the irradiation dose, the larger are the void volumes, caused by morphology deteriorations suffered by both the nylon fibers and the polyester resin. In the case of the nylon fibers, void volume is related to formation of scrap particles resulting from the chain scissions; these particles take the role of fillers in the composite and interact more with the polyester resin rather than with the nylon fibers. An adequate aggregate gradation can provide low void volumes and thus good mechanical strength. Variations in the γ radiation dose enable modification of the void volume and thus higher compressive strain.

CONCLUDING REMARKS

As expected, mechanical features depend on both the fiber concentration and the applied dose. Compressive strength values are similar to those of the extant PCs. By contrast, notable improvements in the compressive strain and elastic modulus are seen. This is a consequence of the contributions of the two aggregates—calcium carbonate and marble—as well as nylon fibers. Such behavior has not been seen in polyester-based PCs containing only one aggregate [11, 12].

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