Full Paper Transactions, SMiRT 28
Toronto, Canada, August 10-15, 2025

CREEP AND STRESS RELAXATION BASED NANOINDENTATION ON IRRADIATED CEMENT PASTE

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ABSTRACT

The current research utilizes nanoindentation to better understand radiation damage mechanisms in hardened Portland cement paste arising from ionizing and non-ionizing radiation. A Preliminary work at the K-State TRIGA facility produced a profound impact on nanomechanical time dependent properties for the samples studied at dose levels much lower than the nominal thresholds for damage (10^{18} N/cm^2 & 10^{19} Grays). The initial study indicated the need for further investigation at moderate dose rates to enhance understanding of the detrimental effects of neutron and gamma radiation on Portland cement paste. Consequently, in the current study, cement paste samples were irradiated at the Texas A&M Nuclear Science Research Centre (NSRC) with varying total doses and dose rates to evaluate their long-term performance through creep and stress relaxation features. Meaningful degradation of mechanical properties was achieved in the second phase of irradiation experiments conducted at NSRC as measured by statistical creep and stress relaxation nanoindentation. An obvious reduction in the reduced modulus was observed from the results. The results also demonstrate significant influence of varying dose rate on the time dependent properties of cement paste at similar total dose. This indicates the need to study the aging creep and relaxation properties of irradiated cement paste.

INTRODUCTION

Concrete in nuclear power plants and research reactors is exposed to neutron and photon radiation, both of which can cause structural damage over time (Le Pape, Stemberk, Ferreira, & Maruyama, 2021; Pomaro, 2016). Radiation effects for Portland cement concrete have been studied extensively dating back to the seminal work from (Hilsdorf, Kropp, & Koch, 1978). Neutron radiation primarily affects aggregates, leading to radiation induced volumetric expansion (RIVE) due to atomic displacements in mineral structures (Field, Remec, & Pape, 2015). This expansion increases porosity leading to microcracking and damaging the mechanical properties (William, 2013). Alternatively, photon radiation interacts mainly with cement paste, which is responsible for the time dependent properties. The photon radiations targets the hydration products causing radiolysis of water molecules, loss of bound water, and phase changes (Kontani, Ichikawa, Ishizawa, Takizawa, & Sato, 2012). Gamma irradiation alters concrete's time dependent properties uniquely (Rosseel et al., 2016).

Recent interest of the U.S. Nuclear Regulatory Commission (NRC) in extending the operating license of nuclear power plants (NPPs) (Remec, Rosseel, Field, & Le Pape, 2018) has called for in detail study of time dependent properties of cementitious composites. During the extended 80-year operational lifetimes, biological shield walls in light water reactors may accumulate extreme radiation exposure of about 100–200 MGy gamma irradiation and neutron fluxes up to 6×10^{19} n/cm^2 (E > 0.1 MeV) (Baral et al., 2022). Thus, it is vital to study and enhance the long-term performance of irradiated concrete subjected to high dosages of ionizing and nonionizing radiations. The literature exhibits ambiguity in the time dependent properties of cement paste (Lowinska-Kluge & Piszora, 2008) (Hilloulin, Robira, & Loukili, 2018; Hunnicutt, Rodriguez, Mondal, & Pape, 2020) and are irradiated at comparatively low gamma irradiation dose relative to the anticipated dosage levels for the estimated 80-year design life of NPPs (Robira et al., 2018) (Y. Khmurovska et al., 2019) (Rodriguez, Hunnicutt, Mondal, & Pape, 2018; Tajuelo Rodriguez, Hunnicutt, Mondal, & Le Pape, 2020). The present research attempts to fill the gaps by adding to the scant experimental data at accelerated dose rate and dose levels of neutron and photon radiation expected at extended operation.

Preliminary work at the K-State TRIGA facility produced a profound impact on nanomechanical time dependent properties for the samples studied at dose levels much lower than the nominal thresholds for damage (10^{18} N/cm^2 & 10^{19} Grays) (Patil & Jones, 2022). In this preliminary work, the total neutron dose received by the cement paste samples was relatively low (8.43 x 10^{15} n cm^{-2} thermal and 9.97 x 10^{15} n cm^{-2} fast), however the dose rate was relatively high, on the order of 2 x 10^{15} n cm^{-2} second^{-1}. The gamma dose and rate were similar (180 Mrad at 5 x 10^4 rad second^{-1}). Even though the total dose was well below the thresholds reported (Hilsdorf et al., 1978), it is likely that the high dose rates in the preliminary experiment were linked to the profound reduction in reduced modulus and increase in creep. The degradation of the properties of irradiated concrete is a function of core location (Remec et al., 2018). Thus, additional study at more moderate dose rates, combined with the spatial precision was conducted and statistically significant amount of data collected. This will help to better understand the damaging effects of combined neutron and gamma radiation on time viscoelastic properties of Portland cement paste.

Recognizing that creep stress relaxation behavior in concrete originates primarily from cement paste, statistical creep and stress relaxation nanoindentation was performed on the cement paste samples exposed to combined neutron and photon radiation. The study focused on three nanomechanical properties: the reduced modulus (E_r), creep compliance, and relaxation modulus characterizing the time dependent deformation. These irradiated samples were systematically compared with control samples to isolate radiation-induced damage in the microstructure mechanical response at the nanoscale.

SAMPLE PREPARATION AND NANOINDENTATION

Three cement paste specimens were casted using ASTM C150 Type I/II ordinary Portland cement with water-to-cement ratios (w/c) of 0.35, and 0.40. Coin-shaped discs (17 mm diameter × 3 mm thickness) were sectioned from the specimens using an Isomet diamond blade saw, and precision polished on a FEMTO 1100S automated polishing system. The detailed procedure of sample preparation for nanoindentation testing are presented elsewhere (Patil & Jones, 2022). The Hysitron Triboindenter model TI Premier fitted with Berkovich probe was used to perform the nanoindentation testing. Creep and stress relaxation tests were conducted on irradiated samples using a load and depth control feedback mode. A trapezoidal load function of 5s loading and unloading, with 30s holding, was used. The corresponding data of load/depth versus time and the E_r were recorded. The time dependent creep compliance and relaxation modulus curves are plotted to compare the test results between controlled and irradiated cement paste sample using the closed form analytical solution (Patil & Jones, 2024). The statistical nanoindentation tests on the samples have been discussed in detail elsewhere (Patil & Jones, 2023).

Based on the elastic–viscoelastic correspondence principle the creep and stress relaxation function obtained to fit the data is given in Equation 1 and Equation 2 (Sneddon, 1965). The creep compliance function *J(t)* and relaxation modulus *R(t)* are given by Equation 3 and Equation 4 (Patil & Jones, 2024) below

$$h(t) = 0.66\sqrt{P_{max}\, J(t)(1-\vartheta^2)} \tag{1}$$

$$P(t) = \frac{2.28\, h_{max}^2\, R(t)}{(1-\vartheta^2)} \tag{2}$$

where *h(t)* and *P(t)* is time-dependent indentation depth and load, respectively. P_{max} and h_{max} are the maximum load and depth during holding segment and ϑ is the Poisson's ratio of the indented material.

$$J(t) = \frac{1}{E_0} + \frac{1}{E_1}\frac{\left(1-e^{\frac{-t}{t_h}}\right)}{(1-e^{-1})} + \frac{1}{E_2}\frac{\left(1-e^{\frac{-t}{t_h}}\right)}{(1-e^{-1})} + \frac{1}{E_3}\frac{\left(1-e^{\frac{-t}{t_h}}\right)}{(1-e^{-1})} \tag{3}$$

E_0, E_1, E_2, and E_3 = the fit parameters; and t_h = the length of the load holding segment.

$$R(t) = \frac{R_0(R_s(e-1)+R_0(R_2R_3+R_1(R_2+R_3))e^{\frac{eR_0t(R_2R_3+R_1(R_2+R_3))+eR_s(t-t_h)+R_s(t-t_h)}{(1-e)R_st_h}}}{R_s(e-1)+eR_0E(R_2R_3+R_1(R_2+R_3))} \tag{4}$$

where R_0, R_1, R_2, R_3 = fit parameters; and $R_s = R_1R_2R_3$.

NEUTRON AND PHOTON IRRADIATION

Irradiation experiments were conducted in the Texas A&M NSRC. The cement paste samples were irradiated to a 90MRad dose (50% of the dose achieved in the previous experiment at the KSU TRIGA reactor) at differing dose rates and irradiation locations i.e. D-3, A-6, and A-2 (Figure 1 and Table 1). For all experiments the reactor was held at constant power of 450 kW thermal. The experiment setup followed the standard NSCR experiment setup. The samples were loaded into an aluminium can and placed at the bottom of the pressurized irradiation device.

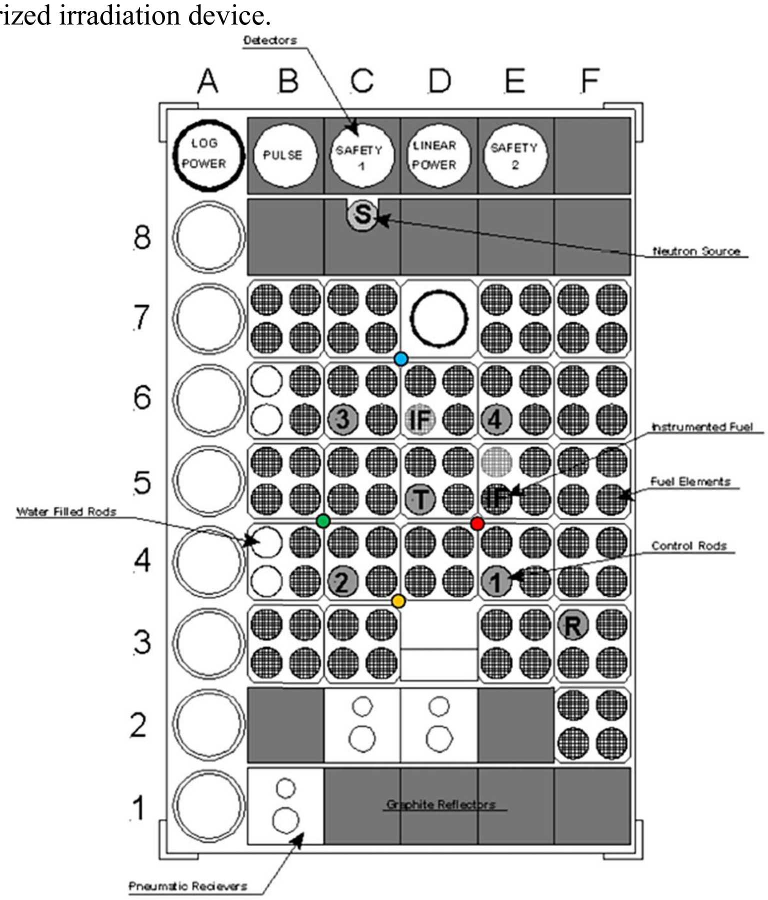

Figure 1: Diagram of the TRIGA reactor showing the core locations (Tyler Gates & Tsvetkov, 2024)

The device consists of a metal tube pressurized with CO2 gas, weighed down by a lead section at the bottom of the tube, and sealed by a threaded top. The irradiation device was then placed in the desired location on the core for irradiation. NSCR operators increased power to achieve the desired dose rates and operate at steady state until the desired total dose was achieved. Once the total dose was achieved, the reactor power was lowered, and the samples removed to be stored for decay. Prior to irradiation, the expected material activation was calculated, and proper decay time was determined. An analysis was performed using Monte Carlo 'N' Particle transport software (MCNP®) to approximate the dose received by the cement samples in each irradiation location. Prior to irradiation, the expected material activation was calculated, and based on the results, a decay period of 5 days was sufficient to get activities well below exempt quantities and able to be safely handled.

Table 1: TAMU NSRC Core Flux and Dose Rates for Intended Irradiation Locations

Core Location	Irradiation Time (minutes)	Total Neutron Flux $(\frac{n}{cm^2 s})$	Total Photon Flux $(\frac{p}{cm^2 s})$	Neutron Dose Rate $(\frac{MRad}{hr})$	Photon Dose Rate $(\frac{MRad}{hr})$	Neutron to Photon Dose Rate Ratio
D-3	28	5.75E12	5.93E6	190	6.1	31.2
A-6	102	1.86E12	2.70E6	50.5	2.7	18.7
A-2	296	6.75E11	1.10E6	17.2	1.1	16.4

RESULTS AND DISCUSSION

The load-controlled nanoindentation test was performed on the irradiated cement paste samples located at three different locations in the core. The resulting values of E_r were recorded and compared with the control sample as shown in Figure 2. The box and whisker plot (Figure 2) shows decrease in the E_r for all the irradiated samples compared with the control sample irrespective of the core location and are consistent with the preliminary work (Patil & Jones, 2022). The plot also depicts that modulus varies linearly with the dose rate and core location, it is seen that the percentage decrease for sample at D3 location is highest, followed by A6 and least reduction at location A2. This trend remains same for 0.35 and 0.40 w/c samples, showing that increase in porosity lowers the stiffness of the paste (Song et al., 2024). The decrease in the stiffness of cement paste can also be defined as function of neutron flux, as degradation of mechanical properties in irradiated concrete is dominated by neutrons. Since, total neutron flux calculated is at least twice that of photon flux, it is evident that neutrons contribute to alteration of the microstructure of the irradiated cement paste producing microcracks.

The nonparametric 'Mann Whitney U' (Patil & Jones, 2022) test indicated a statistically significant difference between the E_r data of irradiated and control samples, with a p-value that is exceedingly small ($p<0.05$) for all samples. The literature offers conflicting data on the influence of gamma radiation on the mechanical properties of irradiated cement paste. While some studies indicate no change, some show an increase in stiffness and others a reduction. (Baral et al., 2022; Biwer, Ma, Xi, & Jing, 2021). In the present study, gamma radiation appears to be somewhat responsible for the relative decrease in E_r observed in cement paste samples with increasing photon dose rate (Figure 2 and Table 1).

The possible drying due to water release by radiation decomposition and gamma heating might target the water phase of cement paste and alter its properties. Also, the products of radiolysis might react with hydration products to change properties of cement paste. The current investigation highlights the influence of neutrons and photon radiation on the water phase of cement paste leading to microstructural rearrangements that detrimentally affect its stiffness. The finding highlights the importance of understanding micro/nano scale interactions of radiations and microstructure to reduce uncertainties in quantifying the impact of concrete irradiation on the mechanical properties.

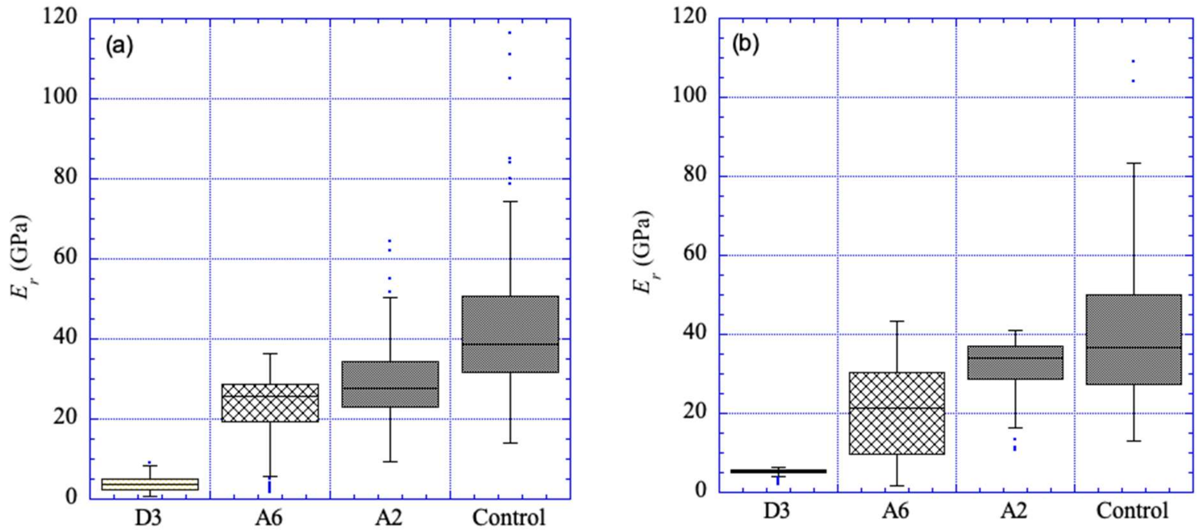

Figure 2: The box and whisker plot for mean E_r of irradiated samples at different core location and control cement paste sample a) 0.35 w/c b) 0.40 w/c

Considering the possible extension of service life of nuclear reactors, it is necessary to understand and characterize the radiation effects on time dependent properties of the cement paste. Thus, in the current work , the load and depth controlled feedback gain was used to compute the creep compliance and relaxation modulus using a closed loop constitutive analytical solution (Patil & Jones, 2024).

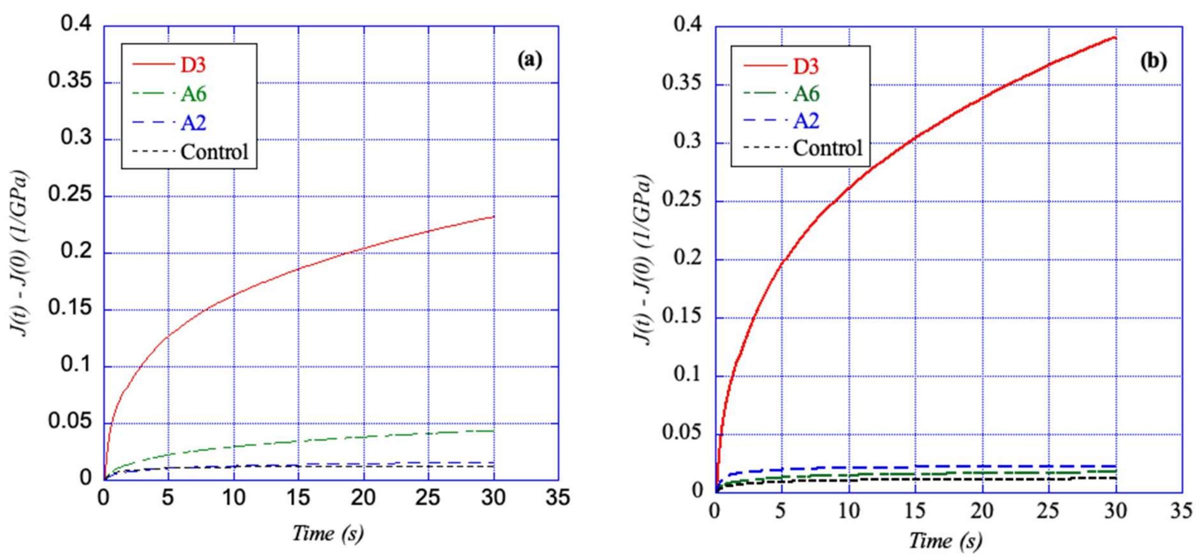

Figure 3: Normalized creep compliance of irradiated samples at different core location and control cement paste samples a) 0.35 w/c b) 0.40 w/c

The results show that creep compliance is considerably higher for all the samples at all three-core locations compared with the control sample (Figure 3) and the data statistically significant. Increase in creep is largest for 0.35 and 0.4 w/c sample at core location D3, followed by A6 and A2. Figure 4 presents the effect of combined neutron and photon irradiation on stress relaxation of the cement paste at different locations. The statistical analysis proves that the relaxation data *R(t)* are significantly different, comparable with the creep results. The reduction in the relaxation modulus aligns with the creep results with respect to core location and dose rate.

The significant percentage difference in the creep and stress relaxation data between core location D3 and other locations confirms that degradation of time dependent properties is a function of core location and dose rate (Figure 3 and Figure 4). Even at lower power (450 kW), dose rate dominates the effect on viscoelastic properties.

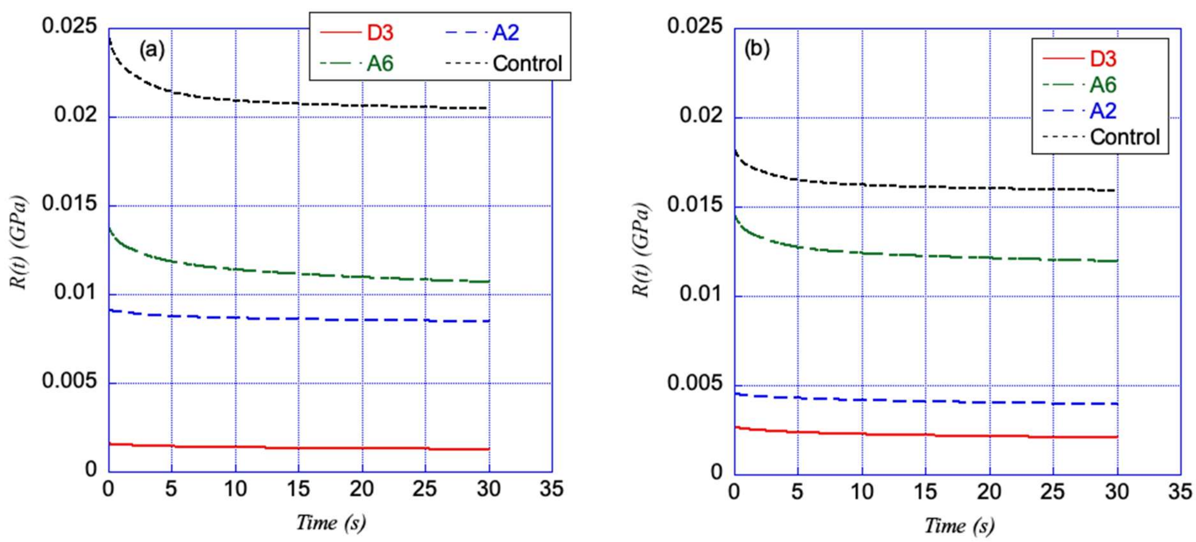

Figure 4: Normalized relaxation modulus irradiated samples at different core location and control cement paste samples a) 0.35 w/c b) 0.40 w/c.

The effect of gamma irradiation on the time viscoelastic properties of concrete are indeterminate. There is limited data on the study on effects of extended gamma and neutron irradiation on creep and stress relaxation of irradiated concrete and cement paste, and the results are contradicting (Fillmore, 2004; Gray, 1972; Hunnicutt et al., 2020; McDowall, 1971). But, in the present study, the creep and relaxation results align with the preliminary study at K-State which shows negative effect on the time dependent properties in comparison with control sample. Due to the ability of nanoindentation to indent shallow depths and increased sensitivity of the sample to degradation, the results signify possible effect of neutron radiation on the solid phase of the cement paste in addition to water phase.

As noted, the E_r of irradiated cement paste sample is lower than that of control sample, suggesting a corresponding increase in creep (Biwer et al., 2021). The higher creep compared to control sample possibly be attributed to gamma induced heating, which promotes water evaporation. Also, radiolytic decomposition of water molecules, releasing gaseous products may further dehydrate the irradiated paste. Similar to drying, irradiation may amplify porosity through formation of microcracks, necessitating detailed microstructural analysis using micromechanical testing like SEM, XRD, TGA.

The effects of gamma and neutron irradiation on time dependent properties of cement paste differ due to their distinct interaction mechanisms. Gamma radiation predominantly causes ionization and radiolysis of water, generating hydrogen gas, microcracking, and moisture loss, all of which can aggravate creep and relaxation properties (Yuliia Khmurovska et al., 2021). In contrast, neutron irradiation triggers atomic displacement damage, leading to the C-S-H gel amorphization, swelling, and internal stresses. These microstructural changes may result in severe degradation of viscoelastic properties—often reducing the elastic modulus by 30–50% and significantly accelerating creep (Field et al., 2015; Yuliia Khmurovska et al., 2021). While gamma radiation impact is predominantly chemical changes in the solid phase of the cement paste such as bond breaking and drying, neutron irradiation produces physical structural damage through displacement cascades, making it more detrimental to long-term mechanical performance.

CONCLUSIONS

The current study examined the combined effects of photon and neutron radiation on cement paste's viscoelastic behavior and E_r. Creep and relaxation tests using nanoindentation were conducted on cement paste samples that were exposed to radiation with varying, controlled dose rate at constant dose. The results are summarized as follows:

- All irradiated samples, irrespective of core location indicated a decrease in E_r as compared to the control samples, with the percentage reduction showing a linear dependence on dose rate. Statistical significance was confirmed for all measured data.
- The observed reduction in the E_r substantiates the detrimental effect of irradiation on the mechanical properties of the cement paste and is consistent with preliminary study at k-State and literature.
- Considering that calculated neutron flux is approximately twice that of photon flux, the study suggests a potential mechanism of neutron-induced damage on the solid phase of hydration products at nanoscale.
- The significant increase in creep and reduction in stress relaxation of irradiated cement paste is a function of controlled dose rate and core location.
- The authors propose that the observed discrepancies in time-dependent properties compared to literature may stem from distinct radiation effects: (1) photon-induced radiolysis predominantly affecting the water phase, and (2) neutron-driven atomic displacement causing chemical modifications in the solid phase. This differential damage mechanism hypothesis warrants verification through phase-separated irradiation experiments

The study demonstrates that while gamma radiation effects on cement paste remain important, neutron irradiation damage constitutes a critical and distinct degradation mechanism that demands equal consideration in irradiated concrete performance. The present work acknowledges some discrepancies in the calculation of the dose rate and neutron and photon flux. Thus, current understanding of radiation damage mechanisms in cement paste remains incomplete, leading to uncertainties in the results. To address these knowledge gaps, we recommend systematic experimental investigations of the porous microstructure to both validate existing models and inform the development of more robust predictive frameworks.

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