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Division I

INVESTIGATION OF VISCOELASTIC PROPERTIES OF IRRADIATED CEMENT PASTE USING STATISTICAL CREEP NANOINDENTATION

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ABSTRACT

The present study investigates the effect of combined neutron–gamma irradiation on cement paste to understand the viscoelastic behaviour of irradiated concrete in nuclear safety structures. Considering the fact that creep originates from calcium silicate hydrate (C-S-H) in cement paste, K-State TRIGA Mark II research reactor licensed to operate at up to 1.25 MW was used to irradiate the cement paste samples. Three samples with water to cement ratio of 0.35, 0.40, and 0.50 were exposed to combined neutron and gamma radiations for one hour at 500 kW thermal power. The reduced modulus and creep compliance of irradiated and the control samples obtained from reference creep nanoindentation test at various locations are presented and explained in detail in this study. To analyse the nanoindentation based creep data of irradiated and controlled samples and to obtain viscoelastic properties under Berkovich indenter, a time-dependent solution developed based on the elastic–viscoelastic correspondence principle is employed. The results demonstrate a considerable drop in reduced modulus of irradiated samples and a significant increase in the creep compared to the control samples.

INTRODUCTION

Due to its structural performance, radiation shielding capacity and irradiation resistance, concrete is widely used in nuclear power plants (Bamonte and Gambarova (2014)). Due to the long-term exposure of such concrete components to radiation and temperature, it is vital to study the degradation of irradiated concrete. Neutron radiation and gamma-ray radiation are the two principal sources of radiation that degrade concrete. It is considered that neutrons interact with subatomic particles, the lattice spacing inside the material may alter as a result of the impact, mostly affecting the aggregates, whereas gamma radiation considerably affects the cement paste through radiolysis (William, Xi, and Naus (2013)). Long-term operation of nuclear power plants has prompted research in long-term aging effects in concrete and metallic structures. Radiation effects on concrete structures is one of the numerous concrete aging degrading mechanisms relevant to long term plant operations (Graves (2014)). Reviewing the literature, it can be seen that the influences of these radiations are studied individually to assess the mechanical properties of concrete (Hunnicutt, Rodriguez, Mondal, and Pape (2020); Khmurovska et al. (2021)). Many studies documented reduction in the strength, and modulus of elasticity of irradiated concrete (Field, Remec, and Pape (2015); Hilsdorf, Kropp, and Koch (1978)), but fail to address the question whether this deterioration is due to temperature or radiation.

It is very well known that concrete is a viscoelastic material that exhibits creep and stress relaxation characteristics with aging (Grasley and Lange (2007); Li (2012)). Creep influences long-term crack growth by relaxing stresses in the cement paste (Rosseel et al. (2016)) altering the damage pattern in the structural component along with substantial deformations and deviations from the projected position of the structure

(Y. Khmurovska et al. (2019)). Recent studies have focused on understanding the creep behaviour of irradiated concrete (Hilloulin, Robira, and Loukili (2018); Hunnicutt et al. (2020); Robira et al. (2018); Tajuelo, Hunnicutt, Mondal, and Le Pape (2019); Tajuelo, Hunnicutt, Mondal, and Le Pape (2018)). However, the existing data is still limited, and part of it is on bulk concrete, some on cement mortar, and some on synthetic calcium silicate hydrate (C-S-H), making it difficult to generalize the phenomena. Some studies showed that the creep of irradiated concrete at macroscopic and microscopic scale is reduced (Hilloulin et al. (2018); McDowall (1971)) and other studies showed increase of creep in irradiated sample (Gray (1972); Y. Khmurovska et al. (2019)), and some no significant change (Tajuelo et al. (2018)). Given the difficulty of controlling specimens and hygrothermal conditions in macroscale creep experiments, the current study employs nanoindentation to perform creep testing on irradiated samples.

The advantage of using nanoindentation to test the irradiated samples is twofold. Firstly, the ability to perform indentation tests at shallow depth (< 3μm), which assures the uniform radiation dosage. Secondly, the ability to obtain sufficient number of statistically significant data fairly quickly. Studying creep deformation at the nanoscale provides insight into macroscale long-term creep behaviour (Vandamme and Ulm (2013)). Owing to the handful of studies and data available to address the effect of neutron and gamma radiation on concrete creep, the present work seeks to measure and understand effects of ionizing and nonionizing radiation on concrete mechanical properties, in particular the instantaneous elastic modulus and short-term creep modulus. Knowing that creep originates from calcium silicate hydrate (C-S-H) in cement paste, statistical nanoindentation was performed on three samples with 0.3, 0.4 and 0.5 water to cement ratio (w/c) irradiated with combined neutron and gamma radiations. The reduced modulus (E_r) which represents elastic deformation both in the sample and the indenter tip and creep compliance were compared with control samples.

MATERIALS AND SAMPLE PREPARATION

Three samples were cast from TYPE I/II cement with w/c (Patil and Jones (2022)) of 0.35, 0.40, and 0.50 for the current investigation. The paste was poured into 45 mm length and 17 mm diameter cylindrical plastic moulds. All of the samples were cured for 28 days in a 100 percent relative humidity condition, and none of the samples were extruded before being placed in the wet room. The specimens were removed from the cured cement paste samples by using a Isomet diamond blade saw at medium speed and water as a lubricant to cut coin size discs. The discs were let to dry in room temperature. The exposed flat surface of the samples size 3mm thick and 17 mm diameter was polished with FEMTO 1100S automated polishing machine using silicon-carbide abrasive discs. Diamond lapping discs were used for further polishing. During this polishing process specimens isopropyl alcohol was used as lubricant to prevent any dissolution of water-soluble hydration products, primarily calcium hydroxide. The polishing machine wheel and the specimen rotated in the same direction at a speed of 200 RPM, at a downforce of 18 to 22 N. The time of polishing on each abrasive size was varied in this (Table 1).

Table 1: Summary of polishing method

Abrasive type (grit)/ Size	180	240	320	400	600	800	1200	3 μm	1 μm	0.5 μm
Time (min)	0.5	0.5	1	1	30	30	120	45	45	45

NEUTRON AND GAMMA IRRADIATION

Each of the three cement samples and iron wires were placed in 4 mil thick LDPE bagging and heat sealed.

A second barrier of heat-sealed LDPE surrounded all samples. The LDPE provided containment of the activated materials and a water barrier. The final arrangement was placed in a HDPE sample holder as shown in Figure 1. Samples were separated to minimize flux depression and non-uniformity. Iron wires were placed between samples to estimate flux variation across the three samples. The reactor was ramped to approximately 500kW thermal power over approximately 1.15 minutes and maintained for 1 hour. Due to cooling system and high-power operation, the reactor pool temperature varied from a low of 22.0˚C to a maximum of 32.2˚C while the samples were in the central thimble. Following a manual SCRAM, the reactor power was reduced to less than 50 kW (10% of steady state) within 0.08 minutes. Samples were removed from the reactor core region within 17 minutes. Thus, the samples were irradiated for approximately 60 +/- 2 minutes. The irradiation dose was estimated from iron co-witness wires. An estimate of the neutron flux during irradiation was determined by analyzing the activation of each iron wire. Thermal neutron flux was calculated based on the activation of Fe-58 to Fe-59. The average thermal neutron flux across the three wires was 2.34 x 10^{12} +/- 1.06 x 10^{11} cm^{-2} sec^{-1}. Using the threshold reaction for Fe-54 to Mn-54 ($E_{threshold}$ = 0.8 MeV, $E_{average}$ =2.8 MeV), the average fast flux for the three wires was 2.77 x 10^{12} +/- 1.93 x 10^{11} cm^{-2} sec^{-1}. For the one-hour irradiation, the thermal fluence was 8.43 x 10^{15} +/- 3.82 x 10^{14} n cm^{-2} and the fast fluence was 9.97 x 10^{15} +/- 6.93 x 10^{14} n cm^{-2}. From the KSU TRIGA Mark II Research Reactor training manual, the gamma dose rate at 500 kW power is 5.0 x 10^4 rad sec^{-1} resulting in a total dose of 180 Mrad for a one-hour irradiation.

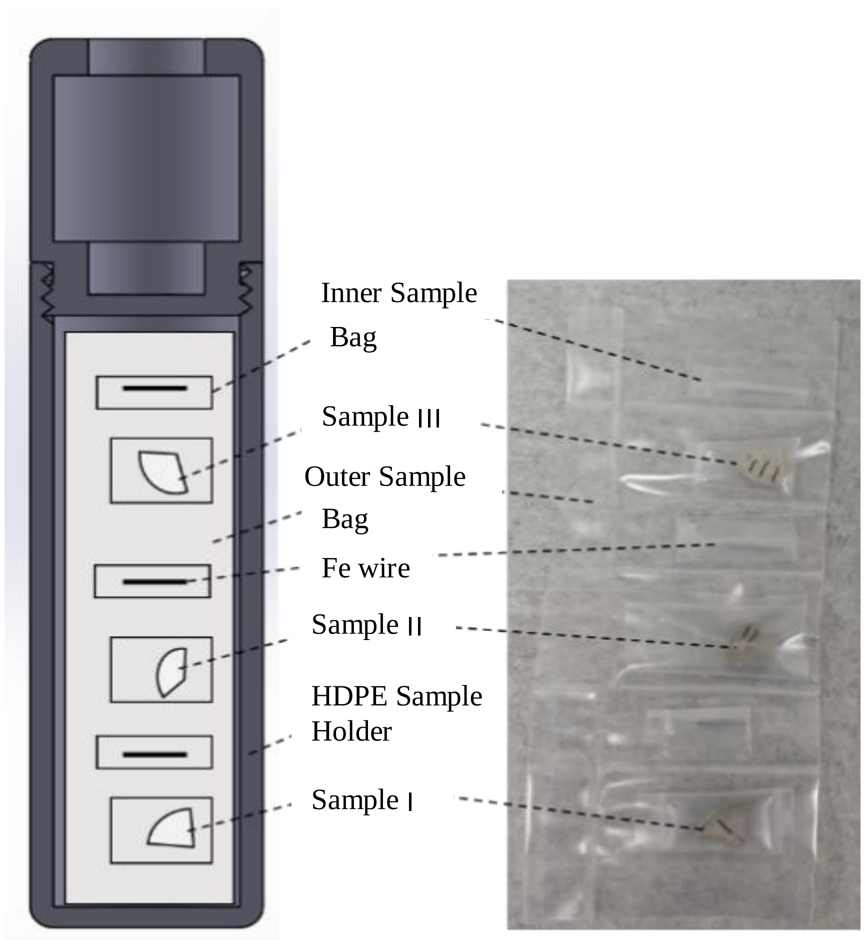

Figure 1: Arrangement of cement samples and iron wires within sample holder. Assembly placed in central thimble of reactor for irradiation.

Prior to irradiation the cement samples elemental mass was estimated based on the cement mill certificate. The resulting quantities of each element were individually assessed for activation using the NIST Database/Tool and a decay period of 60 days was determined. To allow for safe removal of the activated materials, samples were moved to a position in the central thimble outside of the core region (negligible neutron flux) for initial radioactive decay. After decaying in the central thimble for nine days, the samples were stored in a lead box in an administratively controlled cabinet in the K-State Reactor facility. After 60 days the samples were removed and surveyed, and the resulting gamma readings were within the expected levels. The samples were then transferred to the K-State nanoindentation facility. Samples were stored a controlled laboratory environment of approximately 23˚C and 40% RH for the duration of the post-irradiation decay and subsequent nanoindentation testing.

NANOINDENTATION TESTING

A Hysitron Triboindenter model TI Premier was used to perform the nanoindentation testing. A typical Berkovich probe was used to make basic elastic indents with trapezoidal load function of 5s loading, 2s holding and 5s unloading to determine the critical depth and corresponding critical load, the load was varied from 500 µN to 5000 µN (Patil and Jones (2022)). The nanoindentation creep tests were performed using the critical load on control sample (Patil and Jones (2022)). Reference creep tests on the specimen with a step load function in which the second (longer) reference frequency segment will be analysed as the creep. This technique is insensitive to changes in drift rates and allows tests to run over long durations of time. Creep tests with minimum critical load of 3225 µN to a maximum load 5000 µN were performed on irradiated samples using a load control dynamic feedback gain with achieving maximum load in 5s, maintaining it steady for 30s, and unloading for 5s. Statistical creep nanoindentation with 10 x 10 grid size and 3 µm spacing was employed, and the corresponding data of depth versus time and the E_r was recorded. The E_r of the material was evaluated from the unloading part of the load–displacement curve obtained from nanoindentation (Oliver and Pharr (1992)).

Sneddon's well-known quadratic P–h relation for conical indentation on a fully elastic body (Jones and Grasley (2011)) invites the application of the quadratic P–h relationship to materials that range from perfectly elastic to perfectly plastic and has been utilized to analyse the nanoindentation creep data. Sneddon's elastic solution (Sneddon (1965)) in time regime for a conical indenter with the inclined face angle β = 24.7^o is given by Equation 1 below

$$h^2(t) = \frac{\pi}{2\ \cot\beta}\,\frac{P(t)}{E'} \qquad (1)$$

where $h(t)$ and $P(t)$ are time dependent indentation depth and load respectively, and E' is the elastic modulus defined by $E\,/\,(1-\nu^2)$ using Young's modulus E and assuming Poisson's ratio ν of the material under study to be constant and with approximation of ν=0.25. Based on the elastic–viscoelastic correspondence principle (Jones and Grasley (2011)) the creep function obtained to fit the data is given in Equation 2. The creep compliance function $J(t)$ in the form Kelvin chain is given by Equation 3 below

$$h(t) = 1.21\sqrt{P_{max}\,J(t)\ \tan\beta} \qquad (2)$$

$$J[t] = \frac{1}{E_0} + \frac{1}{E_1}\,\frac{\left(1-e^{-t/t_h}\right)}{(1-e^{-1})} + \frac{1}{E_2}\,\frac{\left(1-e^{-t/t_h}\right)}{(1-e^{-1})} + \frac{1}{E_3}\,\frac{\left(1-e^{-t/t_h}\right)}{(1-e^{-1})} \qquad (3)$$

Where, E_0, E_1, E_2, E_3 are fit parameters, t_h is the length of load holding segment. The experimentally obtained data was fitted using Equation 2 using Mathematica.

RESULTS AND DISCUSSIONS

The strength, stiffness, and creep behaviour of the cement paste are determined by the hydrogen bonding of the water inside the layers with C-S-H hydration product. Thus, the E_r and the short-term creep compliance obtained from creep based statistical nanoindentation of irradiated with combined neutron and gamma radiations and control cement paste samples are compared. Figure 2 presents the box and whisker plot for the E_r of irradiated and control cement paste samples. It can be clearly seen that the E_r value is significantly reduced for all three irradiated cement paste sample compared to the control (Table 2).

In contrast to prior research (Hilloulin et al., (2018); Hunnicutt et al. (2020)), the results demonstrate a 61 % drop in E_r value in 0.35 w/c, 65 % in 0.40 w/c, and 75 % in 0.50 w/c in irradiated samples as compared to control samples. The data was tested for normality using the Kolmogorov-Smirnov test (Aslam (2019)), and it was discovered that the data was not normally distributed. Mann Whitney U test (Hart (2001)), which is used when the data are not normally distributed was employed with a confidence level of 95% to test the significance of the data obtained. The nonparametric test showed that the difference between the E_r data of irradiated and control sample is statistically significant with p-value vanishingly small ($p<0.05$) for all the samples. The decrease in the stiffness of irradiated samples can be related to the somewhat influence of neutrons on the water phase of cement paste even (Fillmore (2004)). Despite the fact that the neutron fluence (1.95×10^{16} n cm^{-2}) was less than the reference level of 1×10^{19} n cm^{-2} (Hilsdorf et al. (1978)), the considerable loss in E_r reported may be attributed to the sample's uniquely elevated sensitivity to damage as a result of pre-polishing prior to irradiation and small specimen size. It should be noted that neutron penetration depth is shallow in bulk concrete as the concrete acts as an effective shield. Thus, nanoindentation appears to be an efficient tool to study the neutron effect on the elastic modulus as it is noted that the first layer of concrete (10–50 cm) is where thermal neutrons generate heat (Abdo and Amin (2001)) and NI is capable of indenting shallow depth (<3µm).

A number of studies attributed reduction in concrete stiffness to the neutron irradiation (Fillmore, 2004; Hilsdorf et al. (1978); Kontani, Ichikawa, Ishizawa, Takizawa, and Sato (2012)). When a neutron collides with a nucleus, the neutron loses energy while the nucleus emits dispersed gamma rays and ejects a neutron (William et al. (2013)). Since in the present study the samples are irradiated with both neutron and gamma radiation, the decrease in E_r can be explained majorly by the radiolysis caused by gamma radiations. Also, cement hydrates undergo amorphization because of gamma radiation absorption, which causes them to disintegrate leading to decreased modulus. It is noted in the literature (Herman Graves (2014)) that nuclear heating occurs due to attenuation of radiation by sample and this increased temperature changes the pore structure of the sample producing microcracks which adversely affect the mechanical properties. Unhydrated cement may be further hydrated by gamma heating, accelerating the production of C-S-H. However, in the current investigation, the maximum temperature observed during irradiation was 32.2˚C, implying that the combined neutron and gamma radiation is the primary contributor to the deteriorating impact on the modulus of the cement paste sample.

Table 2: Reduced Modulus (E_r) of irradiated and control cement paste sample

	Mean Reduced Modulus, E_r (GPa)		
Sample (w/c)	**0.35**	**0.40**	**0.50**
Irradiated	19.06	16.50	8.22
Control	48.87	47.83	32.39

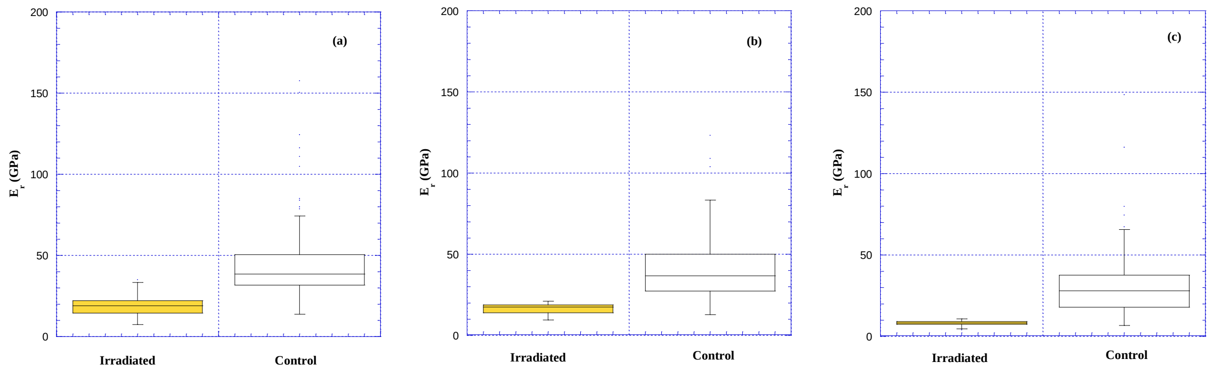

Figure 2: The box and whisker plot for mean E_r of irradiated and control cement paste sample a) 0.35 w/c b) 0.40 w/c c) 0.50 w/c

The radiolysis of water in cement paste due to gamma radiation influences the microstructure of C-S-H and the change is majorly observed at nanoscale (Giorla, Pape, and Dunant (2017)). Thus, the data obtained from statistical reference creep nanoindentation tests on the irradiated and control cement paste samples are analysed and presented in the study. The creep compliance *J[t]* of irradiated and control sample at 30 second holding time obtained from 100 indents is presented in the form box whisker plot as shown in Figure 3. The results indicated that the creep compliance of irradiated samples is higher than the control samples and the trend is consistent across all the samples (Figure 3). The statistical significance of the non-normal creep data at 30 second holding time was tested using Mann Whitney U (Hart (2001)) test with confidence level of 5% and it was evident from the test that data from irradiated and control samples are significantly different.

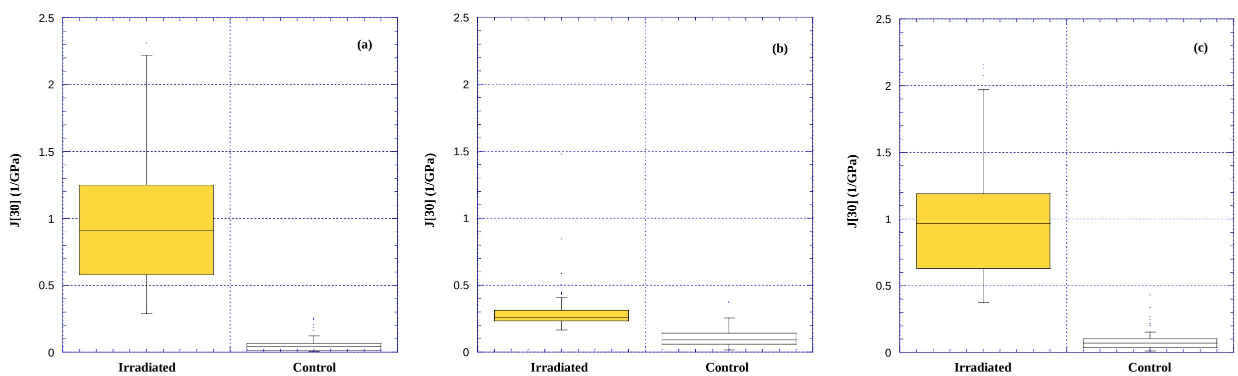

Figure 3: Box and whisker plot of creep compliance for irradiated and control cement paste samples at 30 seconds a) 0.35 w/c b) 0.40 w/c c) 0.50 w/c

The fitted creep compliance (*J [t]- J [0])* curve and the corresponding one standard deviation (upper and lower bound) curves for irradiated and control cement paste samples is shown in Figure 4. The one standard deviation curve was calculated as a function of holding time *(t)* from the large dataset of 100 creep indents. For 0.35 w/c sample, the normalized creep compliance is significantly higher than the control sample, and it can also be observed that upper and lower bound of SD (Figure 4(a)) for the two samples do not fall within each other indicating the considerable increase of creep in irradiated sample. Similar pattern of increased creep compliance is noted in 0.40 w/c irradiated sample (Figure 4(b)). The sample with 0.5 w/c displays higher creep than the control sample (Figure 4(c)), here, the lower bound of the standard deviation curve is not presented since the values were less than zero and therefore invalid. It should be noted that the creep compliance is reduced with increase in w/c (Figure 4). Study of influence of irradiation on creep (Fillmore, 2004) ascertain that creep is expected to increase with increasing levels of exposure. But many studies have reported contradictory results (Hunnicutt et al. (2020); McDowall (1971); Tajuelo et al. (2019)).

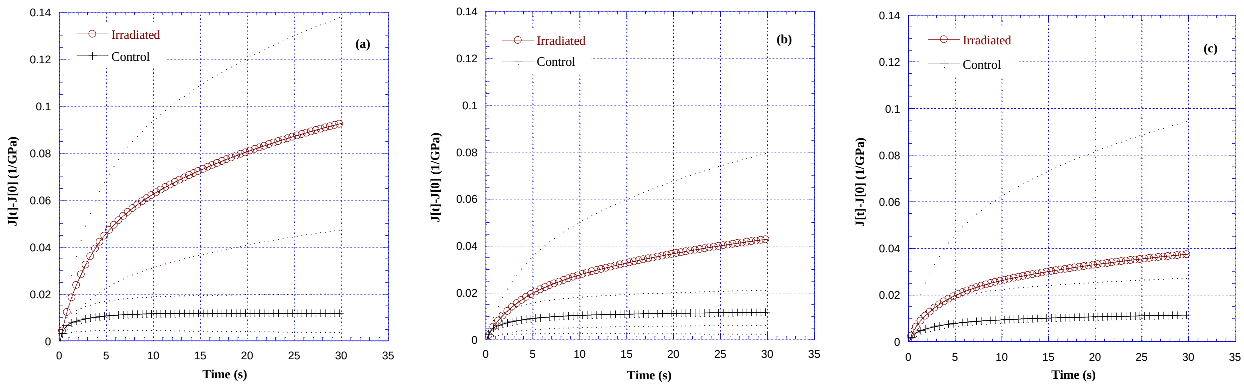

Figure 4: Normalized creep compliance of irradiated and control cement paste a) 0.35 w/c b) 0.40 w/c c) 0.50 w/c

Neutron irradiation leads to reduction in the strength of concrete (Hilsdorf et al. (1978)), since strength is dictated by the hydrated cement paste, the increase in the creep compliance of the irradiated samples under study is explainable. Concrete creep develops at any temperature and is primarily regulated by the presence and movement of moisture and the porosity of the hydrated cement paste. Another possible explanation for increase in creep compliance is increase in porosity due to irradiation with gamma radiation as documented in previously published result (Khmurovska et al. (2021)). The release of hydrogen gas due to radiolysis can create pressure inside the layered porous microstructure of C-S-H leading to the formation of microcracks, altering the creep resistance. Nuclear radiations are inclusive of ionizing and non-ionizing radiations and the influence of these radiations on the water molecules in the C-S-H may boost the porosity and leads to increase in the creep of irradiated samples.

The observed contact depth during nanoindentation creep tests for irradiated samples was considerably higher compared to the control sample (Table 3). This increase in contact depth may be due to increase in porosity. The provided justification for increased creep in irradiated samples is based on the previously published research and further investigation is necessary for the samples under study to confirm the aforementioned explanation. A consistent trend was not observed between the cement samples with different w/c.

Table 3: Mean contact depth observed for irradiated and control cement paste samples

	Mean contact depth h_c, (nm)		
Sample (w/c)	**0.35**	**0.40**	**0.50**
Irradiated	1489	645	1193
Control	219	343	330

CONCLUSIONS

The present work was focused on studying the combined effect of neutron and gamma radiation on E_r and creep of cement paste. All samples were polished prior to irradiation, which increased the sensitivity of the sample surface to damage. Nanoindentation based creep tests were performed on irradiated and control cement paste samples with 0.35 w/c, 0.40 w/c and 0.50 w/c. The results are summarised as follows:

1. Compared to the control samples, all irradiated samples showed a drop in E_r, and the degree of the percentage loss increased with increasing porosity of the sample. The data is statistically significant.
2. Reduction in reduced E_r confirms the possible mechanism of neutron-induced damage on the solid phase of cement hydrates at nanoscale. Further investigation is recommended to strengthen the result by performing chemical structural analyses.
3. Present study documented significant increase in the creep of irradiated cement paste samples irrespective of the w/c, unlike limited studies in the literature. A consistent trend was not observed with respect to the creep behaviour between the different irradiated cement paste samples.
4. The experimental data are insufficient to indicate a strong interaction between the neutron irradiation and the creep
5. Because the sample were irradiated with combined neutron and gamma radiations, the study was unable to separate the effects from neutron and gamma or the possible synergy between them. Possible mechanisms contributing to the increase in creep may be due to internal gas pressures caused by water hydrolysis under gamma irradiation and damage to the porous structure of the C-S-H microstructure.
6. For Portland cement paste, it was found that the deleterious effect of irradiation on creep and lowered modulus might occur at neutron and gamma doses that are much less than the threshold level of about 10^{19} n cm^{-2} and 200MGy cited by previous researchers.

It is important to study the porosity of irradiated cement paste microstructure using scanning electron microscope and XRD to understand its possible effect of combined neutron and gamma irradiation on the creep of cement paste.

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