Eng. Sci., 2021, **16**, 259–268

Engineered Science
DOI: https://dx.doi.org/10.30919/es8d537

In Situ Rheological Properties Monitoring of Cementitious Materials through the Piezoelectric-based Electromechanical Impedance (EMI) Approach

Guangshuai Han,[1] Yen-Fang Su,[1] Siwei Ma,[1] Tommy Nantung[5] and Na Lu[1, 2, 3, 4,*]

Abstract

More attentions have focused on the elastic properties of fresh state cement-based materials due to the emergence of additive manufacturing techniques. Without the support of the conventional formworks, the shaping of the additive manufactured concrete structure mostly depends on the rheological properties of the fresh materials. Even though the fresh state cementitious materials' rheological properties can be well characterized by using a rheometer, no previous study has investigated the effective and reliable real-time monitoring methods for in-situ rheological properties monitoring. This paper attempts to investigate the feasibility of using the piezoelectric-based electromechanical impedance (EMI) method to fill this gap. The EMI test and the rheological test were performed simultaneously. The calculated sensing index from EMI signals displays an excellent linear correlation with the results from the rheometer. The findings from this study proves that the EMI method is promising for in-situ monitoring of the fresh state cementitious materials' rheological properties.

Keywords: Piezoelectric-based electromechanical impedance; Additive manufacturing; Cementitious materials; Rheology.
Received: 29 June 2021; Accepted: 22 September 2021.
Article type: Research article.

1. Introduction

The cement paste is the (thixotropic) yield stress fluid at its fresh state,[1] in other words, it acts as a solid-like material if the applied shear stress is lower than its yield stress. It has been reported that the fresh cementitious material could exhibit the elastic modulus as short as a few seconds after the mixing. The very early age rheological paramseter and elastic modulus are very important properties for enabling many emerging technologies such as three-dimensional (3D) concrete printing technology.

3D concrete printing is an additive manufacturing technology by using concrete or mortar as ink.[2] The fresh concrete/mortar is pumped and extruded through the nozzle and deposited layer by layer without the presence of formwork. N. Roussel[3] examined the rheological requirements for printable concretes and pointed out the importance of the elastic modulus to control the layer geometry and avoid buckling failure. It is known that some viscosity modifying agents can significantly reduce the elastic modulus of fresh state cementitious materials,[4] leading to excessive deformation upon deposition, which can reach up to a few tens of centimeters for a two-meter printed wall.[3] Also, the elastic buckling failure of a freshly printed slender vertical structure has been reported in recent papers,[5-7] which illustrates that the elastic modulus has to stay higher than a critical value to avoid the loss of stability.

It remains a significant challenge to accurately measure the real-time elastic modulus for the in-situ 3D concrete printing projects. Some research groups utilized the high-end rheometer to apply small-amplitude oscillation for the measurement of the elastic modulus of fresh paste, and it increases the budget for research and industrial application. The measurements obtained from different rheometers are likely different for the same material due to errors that come from the calibration.[8] Most importantly, the laboratory

[1] *Lyles School of Civil Engineering, Sustainable Materials and Renewable Technology (SMART) Lab, Purdue University, West Lafayette, IN 47906, USA.*
[2] *Center for intelligent infrastructure, Purdue University, West Lafayette, IN 47906, USA.*
[3] *School of Materials Engineering, Purdue University, West Lafayette, IN 47906, USA.*
[4] *Birck Nanotechnology Center, Purdue University, West Lafayette, IN 47906, USA.*
[5] *Office of Research and Development, Indiana Department of Transportation, USA.*
* *E-mail:* luna@purdue.edu (N. Lu)

measurement may not reflect the "real" elastic modulus of the printed structure, especially given that the rheological properties of the cement paste vary from batch to batch, and the uncertainty can be of order 25% even the pastes have the same composition.[5] Thus, it is critical to develop an in-situ and reliable method to monitor the elastic modulus of 3D printed concrete.

The piezoelectric sensor-based electromechanical impedance (EMI) method has proven its feasibility and effectiveness for monitoring the mechanical information of structures.[9-12] It can also be used to detect the structural damage condition and monitor the in-situ mechanical properties of concrete. Owing to its high sensitivity and its direct and inverse piezometric principle, the piezoelectric sensor can act as both sensor and actuator to capture the properties changes inside the structures. Su *et al*. have researched monitoring the mechanical properties of cement mortar at its very early age (4-8 hours). The EMI results from their studies showed that the compressive strength and elastic modulus of cement mortar have high correlations with the EMI spectrum.[13-15] Tawie *et al*.[16] monitored the bonding development between steel rebar and concrete. It has been found that the conductance spectrum can be used to measure the change of the gradual adhesion between rebar and fresh concrete. Providakis *et al*.[17] used the reusable piezoelectric transducer to monitor the initial hydration states of concrete. Their results showed that the statistical indices are sensitive to concrete strength development. Narayanan *et al*.[18] found that the frequency shift and magnitude of the EMI results can well be correlated with material stiffness changes over time. Visalakshi *et al*.[19] carried out the study of using the non-dimensional parameters to track the hydration of concrete. The results indicated that the refined structural impedance parameter could eliminate the PZT (lead zirconate titanate) contribution of hydration monitoring of concrete. Thus, the EMI method has shown its reliability for monitoring the hydration of the concrete.

Most of the studies in the piezoelectric materials-based EMI method have focused on monitoring the hydration and strength gain of cementitious materials. There is a gap in exploring the potential of the EMI method for monitoring the rheological properties of cementitious materials. The merit of the EMI method, such as accurate, low-cost, and instantaneous, could provide great promises to evaluate the 3D printed concrete structure's rheological properties. To fill this knowledge gap, this study served as the pilot research to examine the feasibility of using the EMI sensing method to address real-time monitoring of the rheology of cement paste. Although most of the 3D printing projects adopted concrete or mortar as the printing ink, which includes larger, inert particles compared with the plain paste, the cement paste can be considered as the active phase in these systems regarding its elastic modulus. The time evolution of the elastic modulus due to the hydration mechanism and thixotropy rebuilding are primarily dependent on the paste phase. The EMI signals from the impedance analyzer and the elastic modulus from the rheometer are tested simultaneously in this study. A linear least square regression is utilized to determine the accuracy of the EMI sensing results. The feasibility of the piezoelectric materials-based EMI method for instantaneously monitoring the elastic modulus of the fresh state cementitious is established.

2. Rheological measurement of cementitious materials

There are more than 100 methods developed to evaluate the rheology and workability of cementitious materials.[20] According to ACI 238 report,[21] the methods can be classified as single-point tests and multi-point tests. Single point tests refer to the measurement as only standing for one point on the flow curve such as the slump test. On the other hand, the multi-point tests collected additional points for the flow curve by changing the shear rate which can constantly provide the rheological information, such as the rheometer. Another distinction of this method can be divided into a dynamic and static testing method. For dynamic methods, the instrument would transmit the energy (vibration, shear force, or jolting) into the concrete. On contrary, the static methods rely on the spontaneous gravitational force (self-weight) of concrete to flow.

To access the rheological properties of cementitious materials precisely and continuously, the rotational rheometer is typically used for the measurement. The material is sheared between two plates during the operation. The rotational speed and torque are recorded. Two types of control modes can be performed: stress-controlled and rate-controlled. The outcome of the measurement can be converted to shear rate through rotation speed and shear speed through torque if the geometry of the sample meets certain criteria. The fresh state cementitious materials have viscoelastic behavior is generally described as Bingham fluid. As such, the cementitious materials would behave their solid properties when the applied shear stress τ is lower than the yield stress τ_Y and would present their flow properties when τ is greater than τ_Y.

In the solid regime of viscoelastic materials, the elastic modulus can be measured using a rotational rheometer with a sufficiently low strain. Another alternative approach for the elastic modulus measurement is through applying the strain oscillation as Equation (1) described. The stress response $\tau(t)$ can be measured with the oscillation and be expressed as Equation (2) under the linear regime.[22]

$$\gamma(t) = \gamma_0 sin\omega t \quad (1)$$

$$\tau(t) = G'(\gamma_0 sin\omega t) + G"(\gamma_0 cos\omega t) \quad (2)$$

where γ_0 is the amplitude of shear strain, G' is defined as elastic modulus, and $G"$ is defined as the viscous modulus.

To interpret the aforementioned coefficients, we can consider the simplest model for viscoelastic solid materials, which is the Kelvin-Voigt model as Fig. 1 shown. The relationship between shear stress and strain is described in Equation (3). Hence, the modulus G' and $G"$ is further expressed as Equations (4) and (5).

$$\tau = G\gamma + \eta\dot{\gamma} \quad (3)$$
$$G'(\gamma_0, \omega) = G \quad (4)$$
$$G''(\gamma_0, \omega) = \eta\dot{\gamma} \quad (5)$$

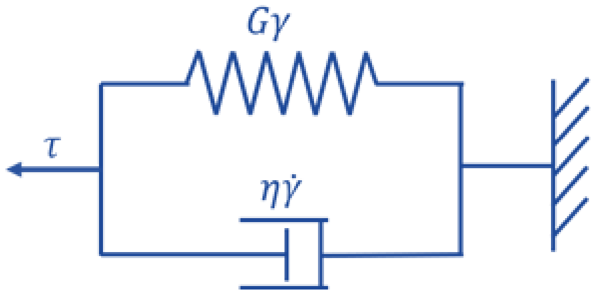


Fig. 1 Kelvin-Voigt model.

Build upon the concept of the theory in this session, the proposed EMI method would be the potential approach to identify the rheological properties of cement paste. This research compared the results of the commercially available rheometer with the EMI method, which both of these two methods are multi-point dynamic testing approaches.

3. EMI principles and signal processing

EMI method is original to be developed for identifying the structural response of the structure which is subjected to the applied force. To be more specific, the response mechanical impedance of the structural system is defined as the ratio of the applied sinusoidal driving force to the consequential velocity. Liang *et al*.[9] developed the one-dimension model to describe the mathematical relationship between the host structure and sensor, as Fig. 2 shows. A sinusoidal alternative current (AC) is applied to the sensor, which generates vibration (continuous small deformation) and is transferred to the attached structure. The response of the local area is bounced back to the sensor in the form of electrical signals. Any changes in structures can be described as the combination of mass (M), spring (K), and damper (C) as shown in the model below.

Three parts govern the dynamic interaction between sensor and host structure, including (a) piezoelectric constitutive relation of the sensor, (b) equation of motion (EOM) for host structure and piezoelectric sensor, and (c) equilibrium and compatibility conditions of the host structure. In the EMI technique, the driving force (axisymmetric vibrations) is generated by the PZT sensor, which can be expressed as piezoelectric strain-charge from constitutive relation of the sensor as Equations (6) and (7) shown.

$$S = \frac{T}{C_E} + d^T E \quad (6)$$

$$D = dT + \varepsilon_T E \quad (7)$$

where S is the strain tensor of PZT; T is the stress tensor; C_E is the elasticity matrix; d^T is the strain per unit field at constant stress; E is the applied electric field; d is the electric displacement per unit stress at the constant electric field; D is the electric displacement tensor; ε_T represents the dielectric constant. Then, the EOM for the vibrating sensor can be expressed as Equation (8).

$$\rho\frac{\partial^2 u}{\partial t^2} = Y^E\,\frac{\partial^2 u}{\partial x^2} \quad (8)$$

where u is the axial displacement at any point in the sensor patch, ρ is the material density. Y^E is Young's Modulus.

Lastly, considering the equilibrium and compatibility conditions of the host structure, the electrical admittance (Y, reciprocal of impedance) of the piezoelectric sensor can be expressed as the Equation (9) below electromechanical coupling between the host structure and the sensor.

$$Y = G + Bj = \omega j\frac{wl}{h}\left[\epsilon_{33} - \frac{2d_{31}^2 Y^E}{(1-v)} + \frac{2d_{31}^2 Y^E}{(1-v)}\left(\frac{Z_a}{Z_a+Z_s}\right)\frac{\tan kl}{kl}\right] \quad (9)$$

where the admittance is a function of conductance (G), susceptance (B) with its imaginary unit (j), angular frequency of excitation (ω), PZT sensor dimension (w, l, and h – width, length, and height), electrical permittivity (ε_{33}), piezoelectric coefficient (d_{31}), Poisson's ratio (v), and wavenumber (k). Except for Y, G, Z_a, and Z_s, all variables are material property.

This work adopted the admittance signals of the piezoelectric sensor, which is able to reflect the mechanical information of the host structure, to monitor the rheology property of the very early age cementitious materials. The cumulative Root Means Square Deviation (RMSD) matrix, which has been proved as an efficient index to monitor cementitious strength gain in practical implementation,[23] served as the EMI sensing index to estimate the rheology property of the host structure. The electromechanical admittance signal changes of the PZT sensor at adjacent ages indicated the gradational development of elastic modulus. Hence, the value between each adjacent EMI measurement can be calculated using Equation (10).

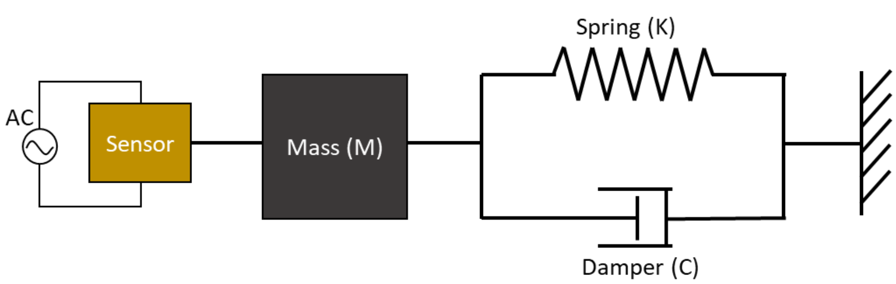


Fig. 2 One-dimension model for piezoelectric sensor-driven structural dynamic system.

$$Cumulative\ RMSD(k)(\%) = \sum_1^k \sqrt{\frac{\sum_1^N (Y_k - Y_{k-1})^2}{\sum_1^N (Y_{k-1})^2}} \quad (10)$$

where Y_k is the signal at curing age k, Y_{k-1} is the adjacent signal of Y_k, and N is the total number of collected data points of each signal. For example, the 20th minute RMSD value calculates the difference of the EMI spectrum between the 10th minute (the adjacent measurement) and 20th minutes, and so forth. Then, each value is accumulated.

4. Experimental program

4.1 Materials

Aiming to investigate the proposed rheological sensing method, two parts of experiments, namely varied mix design tests, and varied curing temperature tests were designed to evaluate the sensing performance of the fresh state cementations materials with different mix design curing temperatures. Type I cement with water was used to prepare the cement paste. To evaluate the sensing behavior on the samples with different mix designs, the water to cement ratio varied from 0.3 to 0.6. The water to cement ratio was fixed at 0.45 when the curing temperature was varied. All the samples were prepared by hand mixing for 3 minutes due to the relatively low demand for materials. The prepared samples were then used for both the EMI test and the rheology test simultaneously to minimize the variables.

4.2 Testing

4.2.1 Sensing (EMI) Test

The prepared samples were poured into a container with a size of 50 mm^3 right after the mixing. The polyester-coated sensor, fabricated by the same procedure as ref[24,25], was then deployed in the container's center. Each EMI test started 10 minutes after the water was added to the 160 minutes with 10 minutes intervals. During the EMI test, the polyester-coated sensor was excited with a 500 mV AC voltage, with a frequency range from 5 kHz to 1000 kHz. An impedance analyzer recorded the dynamic electromechanical impedance (EMI) response of the sensors. The varied mix design test was conducted in a controlled lab environment with a temperature of 23 ℃ $\pm$ 2 ℃. The samples for the varied curing temperature test were tested in a water bath with a temperature of 20 ℃, 40 ℃, and 60 ℃.

4.2.2 Rheology test

A rotational rheometer (Anton Paar MCR 502) with the parallel-plate measuring system was used to study cement pastes' elastic modulus. The diameter of the parallel plates is 25 mm, and the gap between the plates was selected as 1.1 mm. The sandpapers were applied to both plates to mitigate the wall slip effect. A round hood with a soaked sponge ring attached to its rim covered the parallel-plate measuring system to prevent the evaporation of the cement paste.

The testing program started immediately after loading fresh cement paste into the rheometer (10 min after water-cement contact). As the cement paste is a thixotropic material whose rheological properties depend on the shearing history, the pre-shearing at the shearing rate of 100 1/s was first applied for 60 s to ensure that all samples are at a reproducible state at the start of each test, followed by a rest for another 60 s. A small amplitude oscillatory shear (SAOS) measurement was then employed to monitor the evolution of the elastic modulus of cement paste. Based on our previous experiences, an angular frequency of 1 Hz and a strain value of 10^{-5} were chosen for the applied strain to measure elastic storage modulus evolution.

The cement paste can be considered as a linear elastic material when the applied strain is within the range of a few 10^{-4},[26,27] and the elastic modulus will drop dramatically, leading to a much softer material if the paste is stretched beyond the range. In the context of 3D concrete printing, we can anticipate an excessive deformation up to a few centimeters for a typical 2 m wall, which is much undesirable for the printed structure, even if the material is still at the solid regime. Thus, we focus on the elastic modulus under the strain amplitude of 10^{-5}.

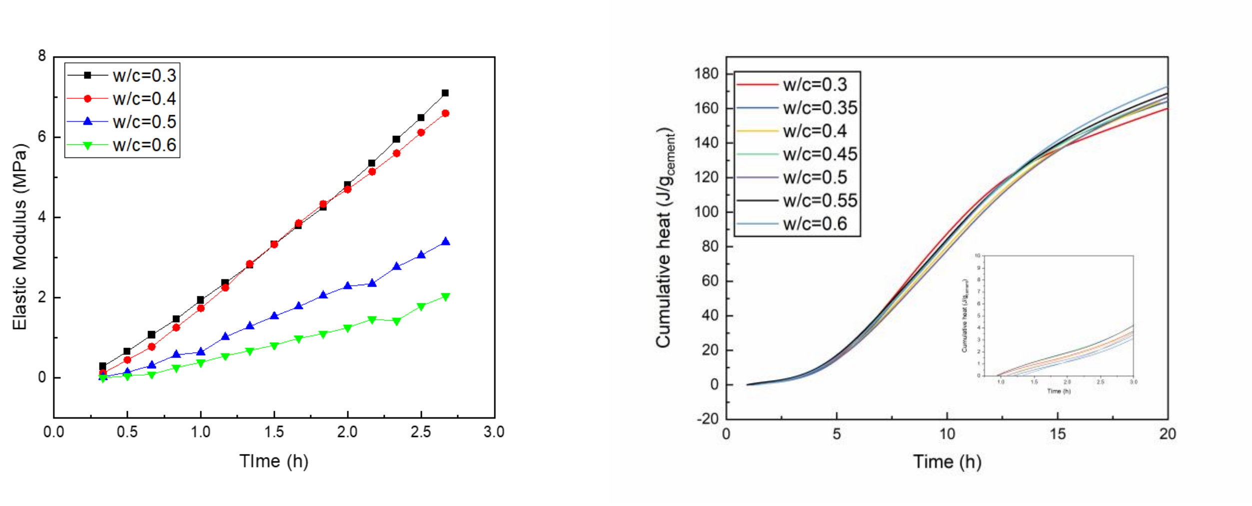
4.2.3 Calorimetry

The isothermal calorimetry was used to measure the hydration kinetics of the cement pastes. The cement paste samples were well mixed with the same mixing speed to avoid the difference in the enthalpy. Five grams of paste were added to a glass ampoule for the measurement. The ampoules were later loaded into an isothermal calorimeter (TAM Air). The rate of hydration can be obtained by monitoring the heat flow generated under constant temperature. The testing was repeated three times and averaged to ensure consistency for each sample group.

5. Result and discussion

5.1 Elastic modulus & degree of hydration

In this research, we have studied the cement pastes with different W/C ratios and curing temperatures to elucidate the feasibility of EMI as an alternative for the elastic modulus in-situ monitoring. To this end, we first measured the elastic modulus by using a rotational rheometer as a reference group. As shown in Fig. 3 (a), the elastic modulus measured by the rotational rheometer appears to increase roughly linearly during the investigated age (up to 160 min) at ambient temperature, and the increasing rate accelerates with the increase of W/C ratio. It has been suggested that the elasticity of the fresh cement originates from the formation of early hydration, which is primarily calcium silicate hydrate (C-S-H) "bridges" at the contact point between cement particles,[27] Thus, the increasing rate of elastic modulus is proportional to the quantity of the C-S-H bridge at the contact point between the flocculated cement network. A. Mostafa *et al*.[28] showed that the quantity of the formed C-S-H at the contact zones is proportional to the rate of rigidification; therefore, the elastic modulus is related to the hydration kinetics and the number of contacts between particles in a unit volume of paste. As shown



(a) (b)

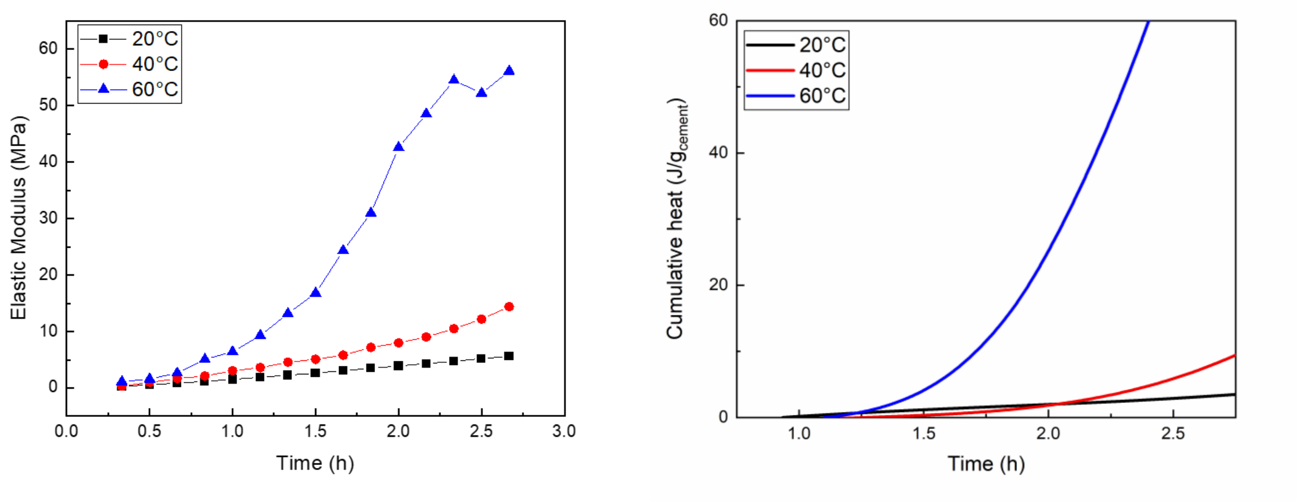
Fig. 3 Cement pastes with different W/C ratios. a) Elastic modulus results; b) Calorimetric results.

in Fig. 3 (b), the early hydration kinetics of the cement pastes are largely independent of the W/C ratio, which is in good agreement with other authors.[28-30] However, the neat cement paste with a lower W/C ratio has a higher collision frequency.[28] Therefore, the cement with a lower W/C ratio shows a higher increasing rate of elastic modulus, as seen in Fig. 3 (a).

The temperature is also a crucial factor that will influence the increasing rate of the elastic modulus since the higher temperature can increase the collision frequency of the particles.[28] Moreover, the temperature experienced by the printed materials could be much higher than 23 °C due to the hot weather and heat produced by the cement hydration process in the application of 3D concrete printing. Thus, the success of monitoring elastic module development at various temperatures is another important indicator to evaluate whether EMI can be a good alternative to rheometer. As shown in Fig. 4 (a), the temperature alters the linearity of the elastic modulus evolution and significantly accelerates its rate, especially at 60 °C; it appears that the elastic modulus evolves exponentially over time.

As shown in Fig. 4 (b), the elevated temperature increases the hydration rate during the investigated time range, which is in agreement with other works.[31,32] We also noticed that the evolution of heat flow and elastic modulus share a similar pattern, which is expected as the solid volume fraction is the same among different temperature groups; thus, the hydration kinetics could be considered as the governing factor in controlling the elastic modulus of the cement paste. Through the thermally activated process, the hydration products nucleate on the cement powder surface at a faster rate,[31] leading to a faster rate of elastic modulus increase correspondingly.

To sum up, this study measured the elastic modulus development of the cement paste with different W/C ratios and curing temperatures. The results have shown good agreement with other works, which are good to serve as evaluation criteria of the proposed EMI method.



(a) (b)

Fig. 4 Cement pastes with different curing temperatures. a) Elastic modulus results; b) Calorimetric results.

5.2 EMI sensing performance

5.2.1 Varied mix design test

The EMI signals of the designed four mixes were measured at each age of interest. Fig. 5 plots the admittance (modulus of the reciprocal of the impedance) signals of the sensors which were embedded in the fresh state cement paste. The changes in the admittance signals of the piezoelectric sensors are always considered as the mechanical properties' changes of its host sample.[33-35] The cement paste samples curing at room temperature are in the initial reaction stage and have a slow reaction before 160 minutes, mainly resulted from the cement particle wetting, dissolution, and nucleation.[36] The samples' rheological properties change due to the formation of the C-S-H bridges. These changes are reflected by the EMI spectrum of the PZT sensor change shown in Fig. 4.[37]

To fulfill the mission of rheological property in-situ sensing, the EMI sensing index, calculated by the method from section 3, was employed as an indicator to monitor the samples' rheological properties. This study adopted the frequency range from 100 kHz to 400 kHz according to the suggestions[14] from the previous work while post-processing the EMI spectrum data. Fig. 6 shows the correlation result between the EMI sensing index of the testing samples with corresponding elastic modulus. Twenty points in total were used to determine the correlation coefficient. The R-square of the sensing behavior for all mixes is above 0.93. This indicates the proposed sensing set-up with the data processing methods can be used to evaluate the rheological properties of cementitious with high accuracy.

The main reason for the high correlation between the sensing results from the piezoelectric sensor and the elastic modulus determined by the rotational rheometer is the similar strain rate during the measurement. From the discussion in section 2.2.2, we acknowledge that the applied strain from the rheometer to the cement paste sample is 10^{-4} to 10^{-5}. Similarly, according to the laser Doppler vibrometer results from the ref [25], the applied strain from the piezoelectric sensor is also in the range from 10^{-4} to 10^{-5}. The small strain is crucial to obtain an accurate elastic modulus result of the cementitious sample. Thus, the proposed piezoelectric-based method can well mimic the testing mechanism of the rheometer.

5.2.2 Varied curing temperature test

The same measurement with the same data post-processing was performed on the cement paste samples with different curing temperatures. Figs. 7 (a) to (c) shown the EMI spectrum

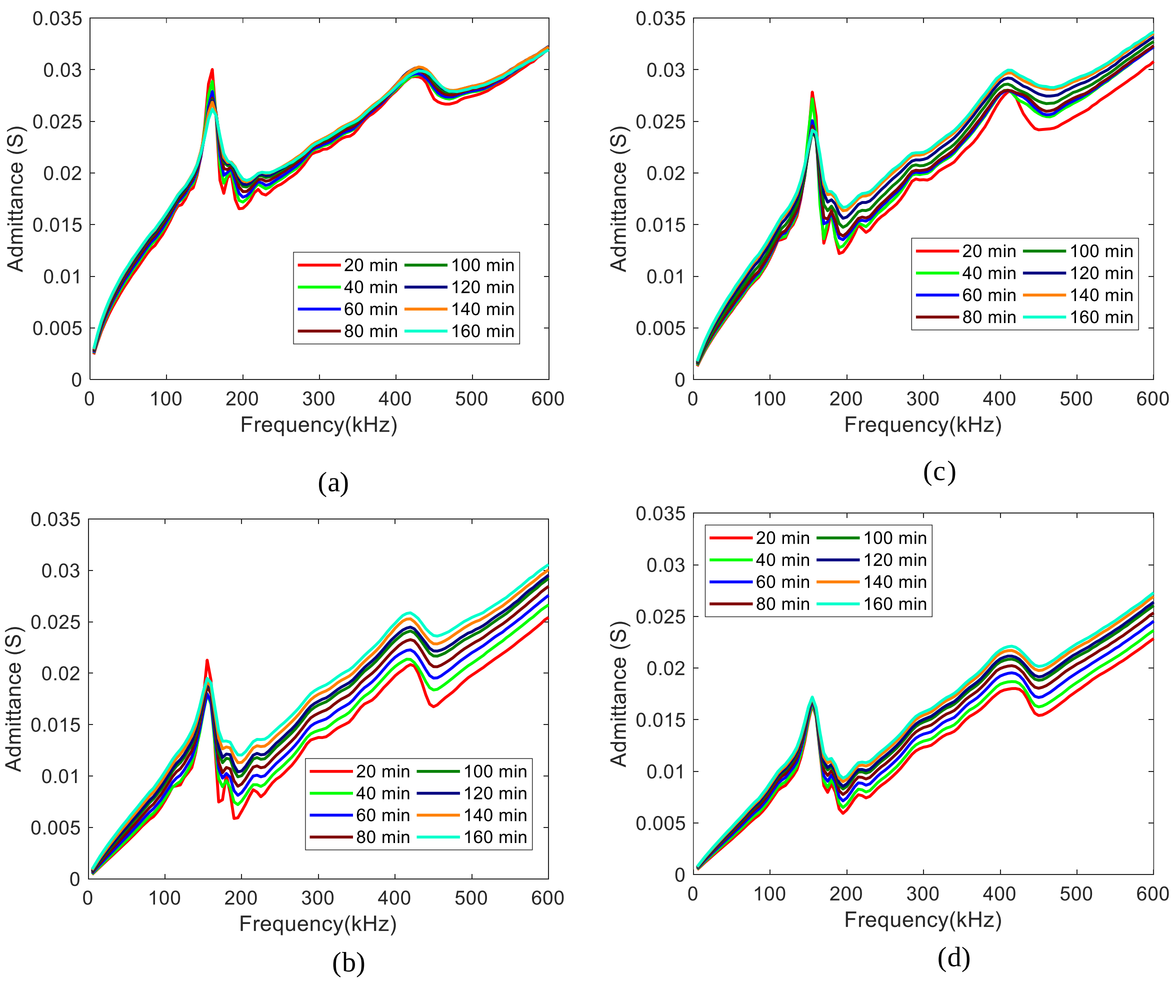


Fig. 5 EMI spectrum of the sensor embedded in the cement paste with different W/C. (a) W/C=0.3. (b) W/C = 0.4. (c) W/C = 0.5. (d) W/C = 0.6.

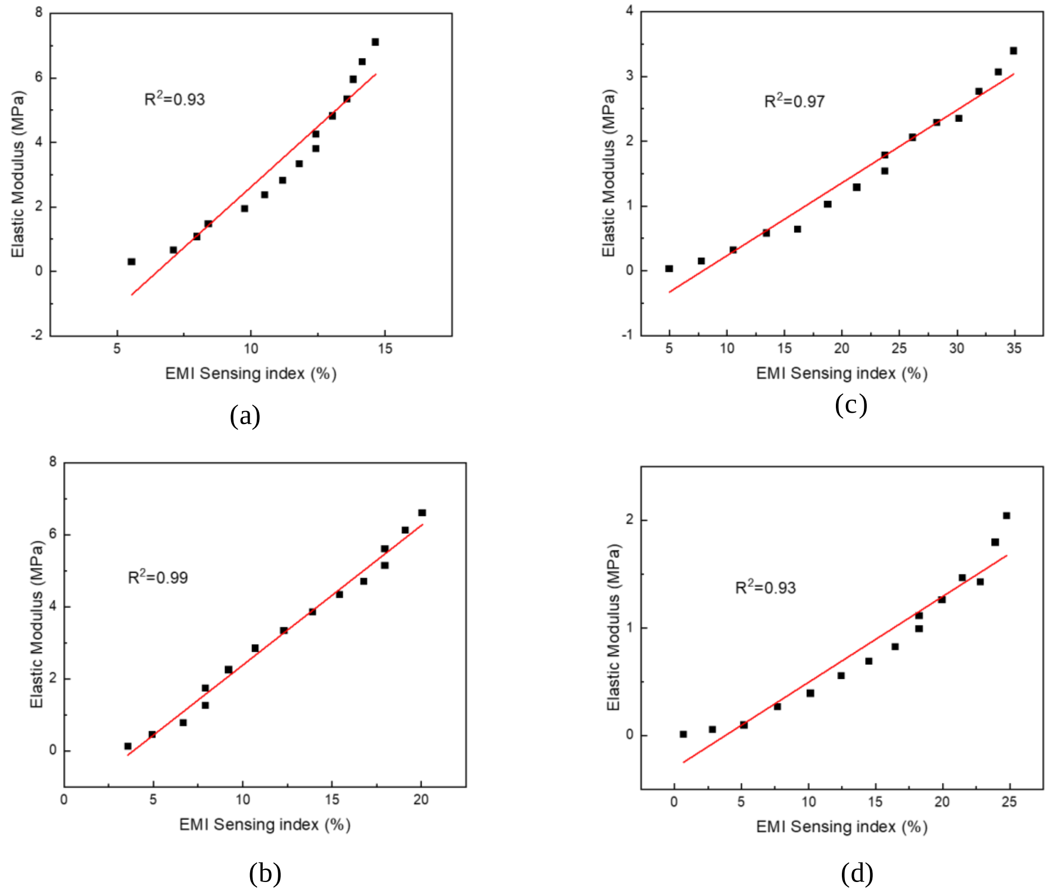


Fig. 6 Linear correlation fitting between elastic modulus with EMI-RMSD index for the cement paste with different W/C. (a) W/C=0.3. (b) W/C = 0.4. (c) W/C = 0.5. (d) W/C = 0.6.

change as the cement paste aged under different curing temperatures. The EMI spectrum of the samples curing at 20 ℃ and 40 ℃ changes in a similar way to the data shown in Fig. 6. The trend of the EMI spectrum changes in the frequency range of interest can be characterized as an upward shift without a resonant frequency shift. It is worth noting that for the EMI spectrum of the sample curing at 60 ℃, a converse downward change with the resonant frequency shift showed upstarts from 120 minutes to 160 minutes. The downward shift with a resonant frequency shift of the EMI spectrum has been reported numerous times by previous studies for using a piezoelectric sensor to monitor the hardened host structure.[38-42]

Moreover, the resonant frequency in the EMI spectrum is one of the most efficient indicators for monitoring cementitious hydration and strength gaining.[43-46] Therefore, the different changing pattern of the cement paste curing at 60 ℃ after 120 minutes, which changes to the solid phase earlier than other cement paste samples, results from the accelerated hydration speed. Differently, this study first reported the unique EMI spectrum changing pattern (Upward changing without frequency shift) when the piezoelectric sensor interacts with the cement paste in the semi-liquid phase. In this case, the resonant frequency shift is not able to monitor the elastic modulus changes at its very early age (before 120 minutes) of the cementitious material. This observation may support the hypothesis that the resonant frequency change in the EMI spectrum is the indication of the cementitious samples transit from the semi-liquid phase to the solid phase. It indicates that the cumulative RMSD is a better option when monitoring the elastic modulus monitoring at a very early age. Figs. 7 (d) to (f) demonstrates the sensing performance of the EMI sensing index calculated by the cumulative RMSD. When the cement paste subject to different curing temperatures, the speed of elastic modulus development will vary in order of magnitude due to the different hydration speeds (shown in Figs. 3 and 4). However, the high R-square and the identical developing trend between the EMI sensing index with the elastic modulus indicate that the proposed EMI method can monitor the very early age elastic modulus development.

6. Conclusion

This study has examined the feasibility of using EMI as an in-situ sensing method for monitoring the cementitious materials' elastic modulus development for additive manufacturing. Seven sets of cement paste samples with different w/c ratios and curing temperatures were tested by EMI test, calorimetry

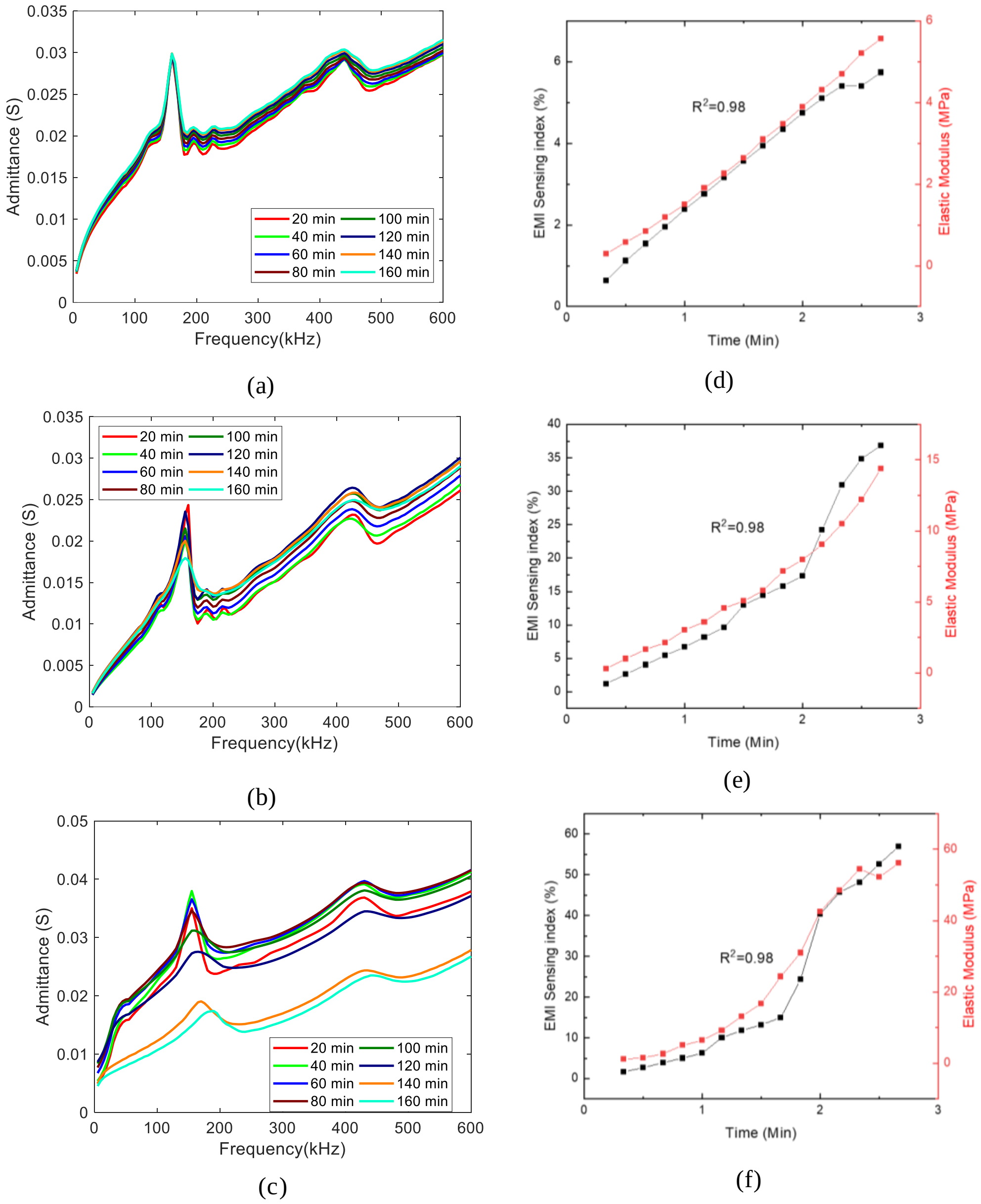


Fig. 7 Sensing performance of the sensor embedded in the cement paste with different curing temperature. EMI spectrum of cement paste curing at (a) 20 ℃, (b) 40 ℃, (c) 60 ℃. Linear correlation fitting between elastic modulus with EMI-RMSD index for the cement paste curing at (d) 20 ℃, (e) 40 ℃, (f) 60 ℃.

test, and rheological test. Based on the rheological testing results, we have identified two important factors of the elastic modulus development, the water to cement ratio and the curing temperature. These two important factors could influence the elastic modulus's development via the number of contact points and the hydration kinetic. The elastic modulus results were then correlated with the piezoelectric-based EMI test results. Since the EMI method provides a stable small oscillation to the cement paste, which can well mimic the testing mechanism of the rheometer, the measured elastic modulus can be estimated by the EMI test with satisfactory accuracy. The sensing index from the EMI test was based on extracting the physical information of the sample and further processed using the statistical approach. The correlation coefficients between the sensing index and the elastic modulus from the cement paste with different water to cement ratios and to cure temperature are all above 0.9. These results indicate that the proposed piezoelectric-based EMI method has a great potential to monitor the elastic modulus of the cementitious materials in a real-time manner.

Monitoring the quality and safety of the materials for additive manufacturing is always vitally important. This is the first report on providing an in-situ monitoring method for tracing the elastic modulus development of cementitious materials, which is one of the critical properties for additively manufactured construction. The findings reported here shed new light on the in-situ monitoring technique for burgeoning additive manufacturing. By in-situ monitoring, the elastic modulus of the cementitious materials with the proposed method, both the safety and efficiency of the additive manufacturing process can be guaranteed and strengthened. Moreover, this monitoring method can potentially be applied to similar materials systems with hardening processes during the additive manufacturing process, such as heat curing, light curing, *etc*. Since the study is limited to the rheometer requirement, this study lacks the results of the cementitious samples with the fine aggregate and the coarse aggregate. Additionally, the samples with supplementary cementitious materials (SCMs) need to be considered. Once these mentioned factors are examined, this work could significantly contribute to the monitoring of in-situ rheological properties of additive manufacturing of concrete structures.

Acknowledgments

This work is supported in part by the Joint Transportation Research Program (JTRP 4210) administered by the Indiana Department of Transportation and Purdue University, United States.

Conflict of interest

There are no conflicts to declare.

Supporting information

Applicable.

References

[1] F. Mahaut, S. Mokéddem, X. Chateau, N. Roussel, G. Ovarlez, *Cem. Concr. Res.*, 2008, **38**, 1276-1285, doi: 10.1016/j.cemconres.2008.06.001.

[2] M. S. Khan, F. Sanchez, H. Zhou, *Cem. Concr. Res.*, 2020, **133**, 106070, doi: 10.1016/j.cemconres.2020.106070.

[3] N. Roussel, *Cem. Concr. Res.*, 2018, **112**, 76-85, doi: 10.1016/j.cemconres.2018.04.005.

[4] S. Ma, Y. Qian, S. Kawashima, *Cem. Concr. Res.*, 2018, **108**, 1-9, doi: 10.1016/j.cemconres.2018.02.022.

[5] R. J. M. Wolfs, F. P. Bos, T. A. M. Salet, *Cem. Concr. Res.*, 2018, **106**, 103-116, doi: 10.1016/j.cemconres.2018.02.001.

[6] R. J. M. Wolfs, F. P. Bos, T. A. M. Salet, *Cem. Concr. Compos.*, 2019, **104**, 103344, doi: 10.1016/j.cemconcomp.2019.103344.

[7] A. S. J. Suiker, R. J. M. Wolfs, S. M. Lucas, T. A. M. Salet, *Cem. Concr. Res.*, 2020, **135**, 106016, doi: 10.1016/j.cemconres.2020.106016.

[8] C. F. Ferraris, N. S. Martys, *J. Res. Nat. Inst. Stand. Technol.*, 2003, **108**, 229-234, doi: 10.6028/jres.108.021.

[9] C. Liang, F. Sun, C. Rogers, *J. Intell. Mater. Syst. Struct.*, 1994, **8**, 323-334, doi: 10.1177/1045389X9700800405.

[10] S. Bhalla, C. Kiong Soh, *Earthq. Eng. Struct. Dyn.*, 2003, **32**, 1897-1916, doi: 10.1002/eqe.307.

[11] Z. Kong, N. Lu, *J. Aerosp. Eng.*, 2020, **33**, 04020079, doi: 10.1061/(ASCE)AS.1943-5525.0001196.

[12] R. Tawie, H. K. Lee, *Cem. Concr. Res.*, 2010, **40**, 1697-1703, doi: 10.1016/j.cemconres.2010.08.011.

[13] Y.-F. Su, G. Han, T. Nantung, N. Lu, *Constr. Build. Mater.*, 2020, **259**, 119848, doi: 10.1016/j.conbuildmat.2020.119848.

[14] Y.-F. Su, G. Han, A. Amran, T. Nantung, N. Lu, *Constr. Build. Mater.*, 2019, **225**, 340-347, doi: 10.1016/j.conbuildmat.2019.07.164.

[15] G. Han, Y.-F. Su, T. Nantung, N. Lu, *J. Intell. Mater. Syst. Struct.*, 2021, **32**, 10, doi: 10.1177/1045389X20974441.

[16] R. Tawie, H. K. Lee, *Cem. Concr. Res.*, 2010, **40**, 7, doi: 10.1016/j.cemconres.2010.08.011.

[17] C. P. Providakis, E. V. Liarakos, E. Kampianakis, *Smart Mater. Res.*, 2013, **2013**, 10, doi: 10.1155/2013/932568.

[18] A. Narayanan, A. Kocherla, K. V. L. Subramaniam, *J. Nondestr. Eval.*, 2017, **36**, doi: 10.1007/s10921-017-0442-4.

[19] T. Visalakshi, S. Bhalla, A. Gupta, *Mech. Syst. Sig. Process.*, 2018, **99**, 13, doi: 10.1016/j.ymssp.2017.05.042.

[20] N. Roussel, Understanding the rheology of concrete. Elsevier, 2011.

[21] A. Commitee, ACI Manual of Concrete Practice, Detroit, MI, USA,

[22] C. Derec, G. Ducouret, A. Ajdari, F. Lequeux, *Phys. Rev. E,* 2003, **67**, 061403, doi: 10.1103/PhysRevE.67.061403.

[23] Y.-F. Su, G. Han, C. Huang, T. Nantung, N. Lu, *ACI Mater. J.,* 2020, 118, doi: 10.14359/51726998.

[24] Y.-F. Su, G. Han, A. Adlan, G. Sean Thomas, N. Lu, Sensors and Smart Structures Technologies for Civil, Mechanical, and Aerospace Systems, 2019, **10970**, doi: 10.1117/12.2513977.

[25] Y.-F. Su, G. Han, Z. Kong, T. Nantung, N. Lu, *Eng. sci.*, 2020, **11**, 66-75, doi: 10.30919/es8d1114.

[26] S. Ma, S. Kawashima, *J. Rheol.*, 2020, **64**, 433-444, doi: 10.1122/1.5129676.

[27] N. Roussel, G. Ovarlez, S. Garrault, C. Brumaud, *Cem. Concr. Res.*, 2012, **42**, 148-157, doi: 10.1016/j.cemconres.2011.09.004.

[28] A. M. Mostafa, A. Yahia, *Cem. Concr. Res.*, 2017, **97**, 11-27, doi: 10.1016/j.cemconres.2017.03.003.

[29] P. Bentz Dale, A. Peltz Max, J. Winpigler, *J. Mater. Civ. Eng.*, 2009, **21**, 512-517, doi: 10.1061/(ASCE)0899-1561(2009)21:9(512).

[30] A. M. Ley-Hernandez, J. Lapeyre, R. Cook, A. Kumar, D. Feys, *ACS Omega*, 2018, **3**, 5092-5105, doi: 10.1021/acsomega.8b00097.

[31] J. J. Thomas, *J. Am. Ceram. Soc.*, 2007, **90**, 3282-3288, doi: 10.1111/j.1551-2916.2007.01858.x.

[32] X. Pang, W. Cuello Jimenez, B. J. Iverson, *Cem. Concr. Res.*, 2013, **54**, 69-76, doi: 10.1016/j.cemconres.2013.08.014.

[33] Y. Yang, B. S. Divsholi, C. K. Soh, *Sensors,* 2010, **10**, doi:

10.3390/s100505193.
[34] D. Wang, H. Zhu, *Constr. Build. Mater.,* 2011, **25**, 3703-3708, doi: 10.1016/j.conbuildmat.2011.04.020.
[35] D. Bahador Sabet, Y. Yaowen, *Proc. SPIE,* 2009, **7292**, 22, doi: 10.1117/12.815877.
[36] J. W. Bullard, H. M. Jennings, R. A. Livingston, A. Nonat G. W.Scherer, J. S.Schweitzer, K. L. Scrivenerg, J. J.Thomas, *Cem. Concr. Res.,* 2011, **41**, 1208-1223, doi: 10.1016/j.cemconres.2010.09.011.
[37] G. Sant, C. F. Ferraris, J. Weiss, *Cem. Concr. Res.*, 2008, **38**, 1286-1296, doi: 10.1016/j.cemconres.2008.06.008.
[38] R. Tawie, H. K. Lee, *Constr. Build. Mater.,* 2010, **24**, 1746-1753, doi: 10.1016/j.conbuildmat.2010.02.014.
[39] S. W. Shin, A. R. Qureshi, J.-Y. Lee, C. B. Yun, *Smart Mater. Struct.,* 2008, **17**, 055002, doi: 10.1088/0964-1726/17/5/055002.
[40] Y. Y. Lim, Z. S. Tang, S. T. Smith, *Smart Mater. Struct.,* 2018, **28**, 015016, doi: 10.1088/1361-665x/aaeea4.
[41] Y. Y. Lim, K. Z. Kwong, W. Y. H. Liew, C. K. Soh, *Smart Mater. Struct.,* 2016, **25**, 085021, doi: 10.1088/0964-1726/25/8/085021.
[42] C. K. Soh, S. Bhalla, *Smart Mater. Struct.,* 2005, **14**, 671-684, doi: 10.1088/0964-1726/14/4/026.
[43] D. Wang, L. Yu, H. Zhu, Proceedings of the 2010 Symposium on Piezoelectricity, 2010, 202-205, doi: 10.1109/SPAWDA.2010.5744303.
[44] X. Lu, Y. Y. Lim, C. K. Soh, *Constr. Build. Mater.,* 2018, **172**, 134-152, doi: 10.1016/j.conbuildmat.2018.03.222.
[45] P. Negi, T. Chakraborty, N. Kaur, S. Bhalla, *Constr. Build. Mater.,* 2018, **169**, 489-498, doi: 10.1016/j.conbuildmat.2018.03.006.
[46] V. Talakokula, S. Bhalla, A. Gupta, *Mech. Syst. Sig. Process.,* 2018, **99**, 129-141, doi: 10.1016/j.ymssp.2017.05.042.

Author information

Guangshuai Han *is a Ph.D. student in Civil Engineering at Purdue University. His current research interest includes flexible piezoelectric device, infrastructure sensing, and data-driven materials discovery.*

Yen-Fang Su *is a Ph.D. candidate at the Lyles School of Civil Engineering. His research interests include artificial intelligence (AI), advanced non-destructive testing (NDT) and sensing technology, and innovative multifunctional sustainable composites materials and structure. He was a structural engineer for few years, which led him equipped with strong mechanics background and practical engineering experiences. He is currently working on several exciting DOT (Department of Transportation) funded projects to solve practical engineering problems such as development of in-situ sensing technology for concrete properties evaluation and designing the self-healing cementitious materials with ultra-high ductility.*

Siwei Ma *is a Postdoc Research Associate at the Lyles School of Civil Engineering, Purdue University. His research interests include rheology, construction materials, and 3D printing cementitious materials.*

Tommy Nantung *is the Section Manager of pavement, materials, and accelerated testing in the Office of Research and Development of the Indiana Department of Transportation (INDOT). He received his BS from Parahyangan Catholic University, Bandung, Indonesia; his MSCE from the University of Michigan, Ann Arbor, MI; and his Ph.D. from Purdue University. His research interests include pavements, materials, and construction.*

Luna Lu *is ACPA Professor of the Lyles School of Civil Engineering and the founding director of Center for Intelligent Infrastructure (CII) at Purdue University. Her research program integrates civil engineering, electrical engineering, and materials science disciplines with three main topic areas: 1) thermoelectric materials for waste heat recovery, 2) piezoelectric materials for energy harvesting and sensing and 3) multi-functional infrastructure materials. She has authored over 100 peer-reviewed publications: 2 book, 6 book chapters, 1 patent and 6 provisional patents. She has won several scientific awards, including the 2014 NSF CAREER Award, 2018 Purdue Zimmerman Innovation Award, 2020 Vebleo Scientist Award and 2021 ASCE GameChanger etc.*