Analyzing the efficacy of SAF (Sulfonated Acetone Formaldehyde) as Mechano-Luminous (ML) material for detecting incipient crack

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Abstract

Certainly, the presence of cracks plays a pivotal role in assessing the structural integrity of civil infrastructure. The existence of early-age cracking in concrete presents a considerable risk to the long-term durability of structures. In the field of crack detection, numerous techniques are available, among which crack luminescence stands out as an innovative and easily accessible method for identifying incipient cracks. In this study, the luminescent properties of sulfonated actone formaldehyde (SAF) were examined in conjunction with vinyl acetate ethylene (VAE) for the purpose of detecting cracks in a substrate. The progression of cracks was systematically monitored and recorded under 365nm UV radiation. The severity of the cracks was analyzed in relation to different flexural stress and strain conditions. In this study, crack initiation was observed for the flexural strain 1.2% with a corresponding stress of 1.1 MPa, followed by mild crack propagation was observed on both sides of the specimen for strain value of 1.5% with corresponding stress of 2.2MPa. The failure occurs within a strain region of 2.1% and corresponds to a stress value of 5 MPa. Once the flexural strain surpasses 1.2%, the crack propagation started, accelerates gradually, and leading to failure with a reduced strain rate. The results highlights the utilizing SAF-VAE based mechano- luminescence as a crack detection sensor for the identification and analysis of severity of cracks in construction structures can benefit from improved safety, timely maintenance, and enhanced structural integrity. This technology offers a reliable and efficient method for detecting and monitoring cracks, ultimately contributing to the overall safety and longevity of the built environment.

Keywords: Mechano luminescent phenomina, Sulfonated acetone formaldehyde, Vinyl acetate ethylene, Non Destructive testing, Crack sensor

Introduction

Concrete stands as a highly prevalent material in civil infrastructure applications, encompassing bridges, buildings, and nuclear power plants, owing to its notable attributes of cost-effectiveness and ease of formability to achieve desired configurations. Cracks observed in concrete structures are a widely observed phenomenon that attributed to various factors, including corrosion, chemical deterioration, creep, shrinkage, and the application of unfavorable loads. The emergence of these cracks serves as a visible manifestation of structural stress, inherent weakness, and cumulative structural degradation, which potentially contribute to the risk of failure or collapse[1,2]. Certainly, cracks serve as a crucial indicator of the structural health of civil infrastructure. These cracks are deemed noteworthy when they initially exhibit an opening size of approximately 0.05-0.1mm. These cracks escalate to a significant concern when they reach a size of around 0.3-0.4mm, and they can progress further to potentially hazardous dimensions, ranging from a few millimeters to centimeters. The presence of early-age cracking in concrete poses a significant threat to the durability of structures. While cracks in brittle materials can result in instantaneous failure, crack propagation in ductile materials tends to be more gradual. It is crucial to identify and detect all forms of cracks in advance, prior to reaching a critical threshold that could lead to structural component failure or the collapse of entire structures[3].

The timely detection of all types of cracks is of paramount importance to prevent them from reaching a critical state that could lead to the failure of structural components or even the entire structures. At present, the prevailing approaches for crack detection and characterization involve periodic visual inspections and non-destructive evaluation (NDE). These methods rely on established instrumentation and traditional methodologies[4,5]. Conventional methods often exhibit drawbacks such as high costs, time-consuming procedures, and limited accuracy, particularly during the initial stages of cracking where they may fail to detect damage effectively.

For instance, experimental techniques like ultrasonic thickness meters have been employed for crack detection, but it is important to note that such inspections provide only one-dimensional measurements[6]. Due to the aforementioned limitations, multiple visualization techniques have been developed to analyze static or propagating cracks in diverse materials. These techniques encompass guided electronic speckle interferometry [7], radiography [8], wave imaging[9], thermography[10], stereography[7], and others. These methods are based on two-dimensional global measurements, allowing for enhanced crack analysis and characterization. Nevertheless, all of the aforementioned methods necessitate the utilization of sophisticated measurement devices and/or intricate post-analysis procedures. The recent integration of smart paint for the purpose of detecting and visualizing different types of cracks in both simple and complex structures exhibits significant promise and potential for substantial advancements[11,12]. In this context, there has been growing interest in the application of Mechano-Luminescent (ML) techniques to develop intelligent paint. This intelligent paint is designed to detect cracks, monitor their propagation, assess the stress distribution interpret the bridging mechanisms occurring at the crack tip. The presence of nano and micro-particles with mechano luminescent (ML) properties exhibit the capability to emit light repeatedly when subjected to minor applied stresses, such as deformation, friction, or impact. When uniformly applied as a coating into a structural element, each individual particle functions as a highly sensitive mechanical sensor. The collective emission pattern, observed in two dimensions, provides insights into the dynamic distribution of stress within the structure and conveys mechanical information pertaining to cracks and defects. The stress visualization technique, utilized for assessing the structural health of a building, presents notable advantages compared to the conventional point-by-point measurement method. It provides a comprehensive and holistic approach to analyze stress distribution, surpassing the limitations of localized measurements [13–19].

Conventional systems commonly employ strontium aluminate as mechano-luminescent particles, which have demonstrated their efficacy in various applications such as monitoring active cracks [19], detecting internal defects in pipes [20], enabling direct observation of crack tip stress fields [17,21], serving as damage sensors [22], functioning as impact sensors [23], and assessing bridging stress in different ceramics[15,18]. These applications have been realized through the development of mechano-luminescent (ML) paints or pulsed laser deposition (PLD) coatings, which serve as artificial skin to sense mechanical stress and emit visible light [13,24].

According to the existing literature, the combination of strontium aluminate and transparent resins has been employed for the formulation of mechano-luminescent paint. However, building upon this concept, a novel ML emulsion has been developed by utilizing Sulfonated Acetone

Formaldehyde (SAF) as the ML material and Vinyl Acetate Ethylene (VAE) as the binder. It is noteworthy that there is currently a lack of existing literature regarding the application of SAF in ML formulations.

Sulfonated acetone-formaldehyde (SAF), a highly efficient resin, is recognized for its exceptional superplasticizing properties within the construction industry [25,26]. Fortunately, we have observed an intriguing property of SAF, namely its pronounced mechano-luminescence (ML) phenomenon[27]. It is noteworthy to mention that these polymers exhibit several significant highlights. First and foremost, this fluorescent polymer is a novel type that originates from the CIE effect of the carbonyl groups. Secondly, it offers a novel, water-soluble fluorescent polymers with aggregation-induced emission (AEE) properties. Thirdly, the utilization of economical raw materials and a straightforward synthetic pathway for the polymer enables a convenient and cost-effective approach for the production of luminescent materials [26].

The principle underlying crack luminescence involves the phenomenon of light emission from cracks or fractures under the influence of external stimuli, such as ultraviolet radiation. When a crack is subjected to specific wavelengths of ultraviolet light, it can emit visible light in response. The emitted luminescence can be detected and analyzed to provide insights into the crack characteristics like integrity of cracks and enabling the assessment of crack growth. In this experiment luminescence was facilitated by highly excitable molecules present within the system. While a crack generates on the surface of the components, the adhesive properties of the two layers cause them to open the rip at the edges of the cracks[28]. A specific wavelength of ultraviolet (UV) rays, measuring 365nm, was utilized in the experiment. When these highly energetic rays penetrate the crack, the orbital electrons of the molecules located near the boundaries of the luminescent layer experience an energy transfer, resulting in their excitation to an excited singlet state. Subsequently, these electrons release visible radiation spanning the range of 380nm to 780nm as they return to their ground state, exhibiting a phenomenon known as fluorescence. The extent of the crack opening determines the quantity of visible light that is directed towards an observer. The presence of dark layer effectively blocks the UV rays, preventing fluorescence from occurring when the surface remains intact[29].

From this perspective, we have conducted practical investigations into the potential applications of ML sensing technique utilizing SAF and VAE copolymer as the mechano-luminescent (ML) material and binder respectively. In particular, we present several notable instances where the ML pattern has been effectively employed to visualize the presence of an active crack on a structural element, as well as the subsequent propagation of the crack, while simultaneously capturing the associated flexural stress and strain.

Experimental Study

Materials

The materials utilized in this experiment can be classified into two distinct categories: Substrate fabrication materials and luminescent formulation materials. The former category encompasses, a polypropylene fiber-reinforced cement substrate, measuring 160x40x40 mm, was obtained by introducing a 1% concentration of polypropylene fiber into the cement mortar mixture. The rationale for selecting a fiber-reinforced cement substrate is due to the enhanced flexural properties exhibited by fiber-reinforced composites in comparison to conventional cement mortar prisms. The constituent materials required for making the prisms, including cement, sand, and PP fibers, were procured from local suppliers. The latter category encompasses luminescent formulation materials, namely acetone, formaldehyde, sodium hydroxide, sodium sulfates, which were obtained from Sigma Aldrich.

Synthesis of SAF

Water soluble sulfonated acetone formaldehyde (SAF) was synthesized by introducing sulfonic groups into the structure of acetone formaldehyde using one-pot method. Fig. 1 represents the synthesis of SAF. The procedure involved by mixing formaldehyde (6.0mL, 37 wt%) and acetone (3.0 ml) in an aqueous solution with pH of 10. The mixture was then heated at 50°C for one hour, followed by the addition of 2.5 g of sodium sulfite (Na_2So_3). The polymerization process was halted following a reflux period of 5 hours at a temperature of 85°C. The remaining unreacted raw materials were eliminated using vacuum rotary evaporation. Subsequently, the pH of the resulting water-soluble product solution was adjusted to a range of 2 to 3 through the addition of hydrochloric acid. Following that, acidic products underwent sun-drying for a period of 96 hours. The resultant brown solid exhibited remarkable water solubility (solubility: exceeding 400mg/mL) and exhibited noticeable photoluminescence when dissolved in an aqueous solution[26].

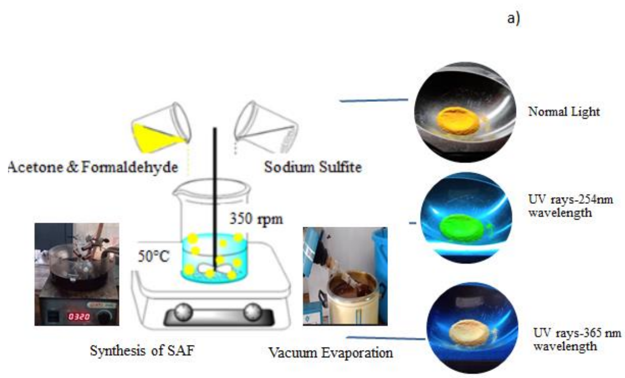


Fig. 1 Schematic diagram-Synthesis of SAF

2.1.2 Selection of luminous binder

Multiple test series were conducted to identify the appropriate binder medium for formulating the luminescent emulsion. In one of the test series, luminescent emulsion was blended with aqueous epoxy resins (Refer Fig 2 a & b). However, the low elasticity nature of epoxy resulted in brittleness upon specimen failure, causing lack of crack luminosity visibility. Subsequently, VAE, a water soluble copolymer with binding properties to the substrate, was investigated as an alternative (Fig 2c). Multiple test series were conducted to determine the optimal thickness of VAE coating. Initially VAE as such used in the formulation of luminescent emulsion. Owing to its significant density and elasticity, no cracks were observed after the failure of the specimen. In a subsequent phase, VAE was diluted with water and combined with SAF, resulting in the desired elasticity and achieving the desired thickness.

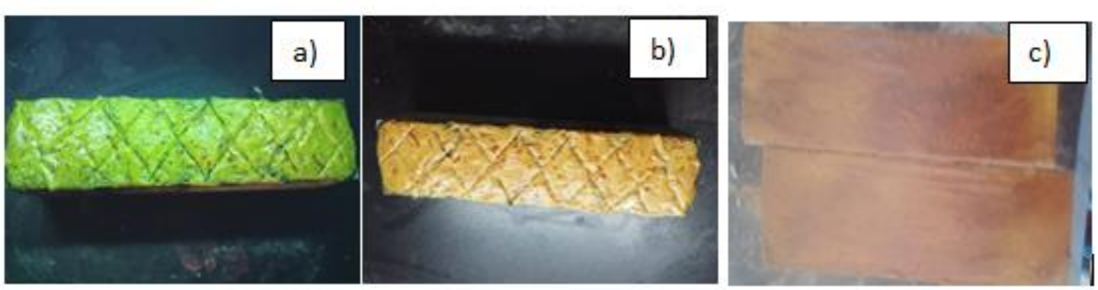


Fig 2 a, b & c –SAF with aqueous epoxy emulsion and SAF with VAE emulsion-

Luminescence under 254nm and 365 nm respectively

Formulation of Mechano luminescent (ML) emulsion

Fig. 3 represents the formulation of SAF-VAE mechano-luminescence emulsion. A mechano luminescent (ML) emulsion is composed of sulfonated acetone formaldehyde (SAF) component, which acts as the luminescent agent, vinyl acetate ethylene (VAE) copolymer emulsion, which functions as the binder for the emulsion, and water, which serves as the continuous phase of the emulsion. The luminescent emulsion was prepared by combining 15wt% of sulfonated acetone formaldehyde (SAF) with a vinyl acetate ethylene (VAE) emulsion while subjecting it to continuous stirring. The quantity of SAF used determines the desired luminescent intensity. The mixture underwent continuous stirring until the SAF was uniformly dispersed within the emulsion[30].

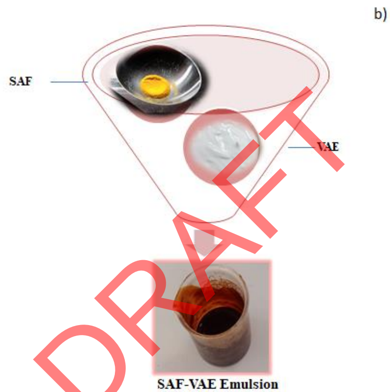


Fig. 3 Schematic diagram-Formulation of SAF-VAE emulsion

Preparation of specimen

The functional efficacy of this method necessitates a strong adhesive bond among the substrate, luminescent layer and the covering layer. The elasticity properties of the coatings play a crucial role in the detection of cracks. Coatings with higher elasticity exhibit slower crack detection or even prevent crack detection altogether. Therefore, extensive experimentation has been conducted to achieve an optimal balance between luminescence intensity and elasticity[31]. In order to achieve crack luminescence, a specialized coating with a thickness of less than 1mm was applied onto the surface of the substrate. This coating comprises of two distinct layers: a luminescent layer and a dark layer that acts as a covering for the luminescent layer[28]. The surface of the substrate underwent cleaning through sandpaper, followed by the application of the luminescent coating. The second layer known as opaque or cover layer is specifically engineered to facilitate unhindered transmission to the luminescent indicator. This layer is composed of black paint, chosen for its light-absorbing properties and ability to enhance the desired transmission characteristics. The aforementioned coatings were administered on three sides of the substrate.

Figure 4 illustrates the test specimen and provides a general overview of the procedural steps. These steps primarily involve surface cleaning, application of luminescent layer followed by drying, application of covering layer followed by drying.

Fig. 4 a, b, c Schematic representation of states of specimen preparation- Plain substrate followed by luminescent covering and finally dark layer covering.

Methods

Figure 5 illustrates the methodologies employed for crack detection in this study, which involve distinct stages, namely substrate preparation, formulation of sulfonated actone formaldehyde (SAF), formulation of the mechano-luminescent (ML) emulsion, specimen preparation and finally flexural testing. Detailed descriptions of these stages will be provided in the subsequent paragraphs.

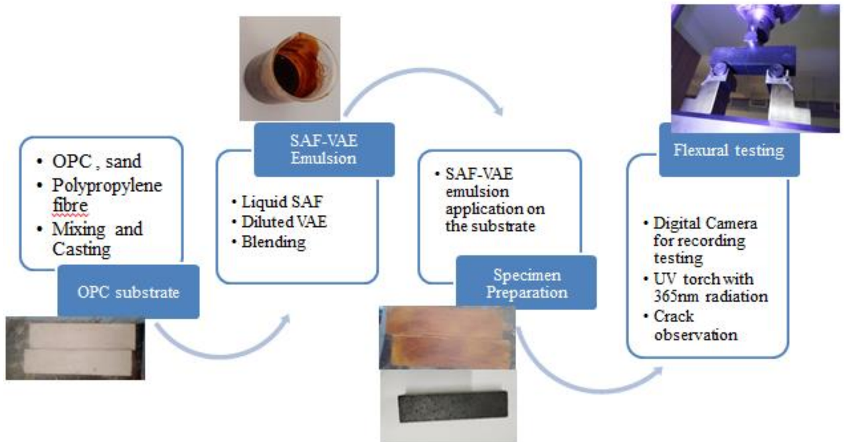


Fig. 5 Schematic diagram of experimental procedure

The flexural test, in compliance with ASTM C 348 standards, was conducted at room temperature. Samples measuring 160 mm x 40 mm x 40 mm were subjected to three-point bending. The bending test was performed using an Instron 500 testing machine, with a loading rate of 0.5mm/min. The specimens were positioned in such a way that the tensile surface was perpendicular to the lamination direction.

Figure 6 presents test setups, both involving the utilization of camera and continuous UV illumination. The incorporation of a camera system is crucial for capturing subtle changes and fluctuations in the coating's light emissions, enabling subsequent analysis and evaluation. Video analysis aims to identify the initial light emission of the coating and highlight notable stages of crack propagation by carefully examining the video frame by frame.

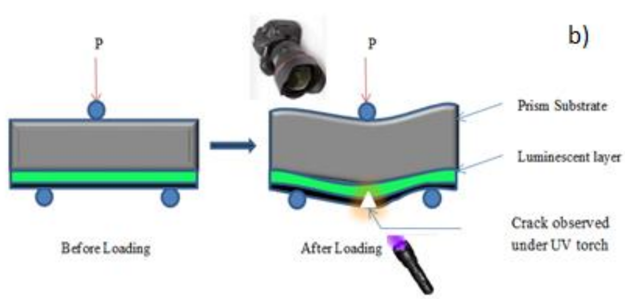


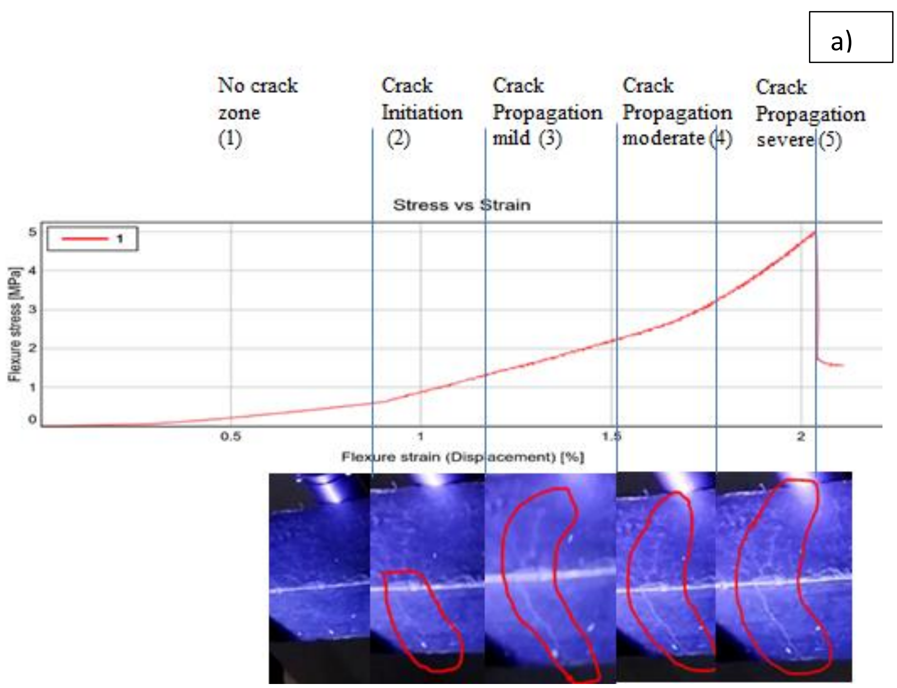
Fig. 6 Schematic diagram-Experimental setup of testing

The system synchronized the loading process, camera recording, and transmission of UV rays from the torch, ensuring simultaneous operation. The output values and captured images were analyzed and interpreted to evaluate the propagation of cracks.

Results and Discussion

Multiple test series were conducted to determine the optimal composition of the coating material, including the required mass fractions of each component. The influence of varying coating thickness was also analyzed. The findings indicated that a remarkably thin coating, ranging between 100 µm, coupled with a cover layer thickness of approximately 50µm, yielded the most sensitive detection capabilities. In order to assess the adhesive bond and delamination occurring during the deformation of the specimen, the different coatings underwent examination using a flexural test. The results of these examinations indicated that fine cracks in the coatings were observed when subjected to high strain values exceeding 1.2%.

To conduct a more comprehensive investigation, the coatings were applied to three sides of the substrate, and a flexural test was performed. Careful consideration is needed when observing the point at which the crack becomes visible for the first time. Interpolation was employed to analyze various stages of crack propagation along with corresponding flexural stress and strain values over time, as depicted in Fig 7a, b.



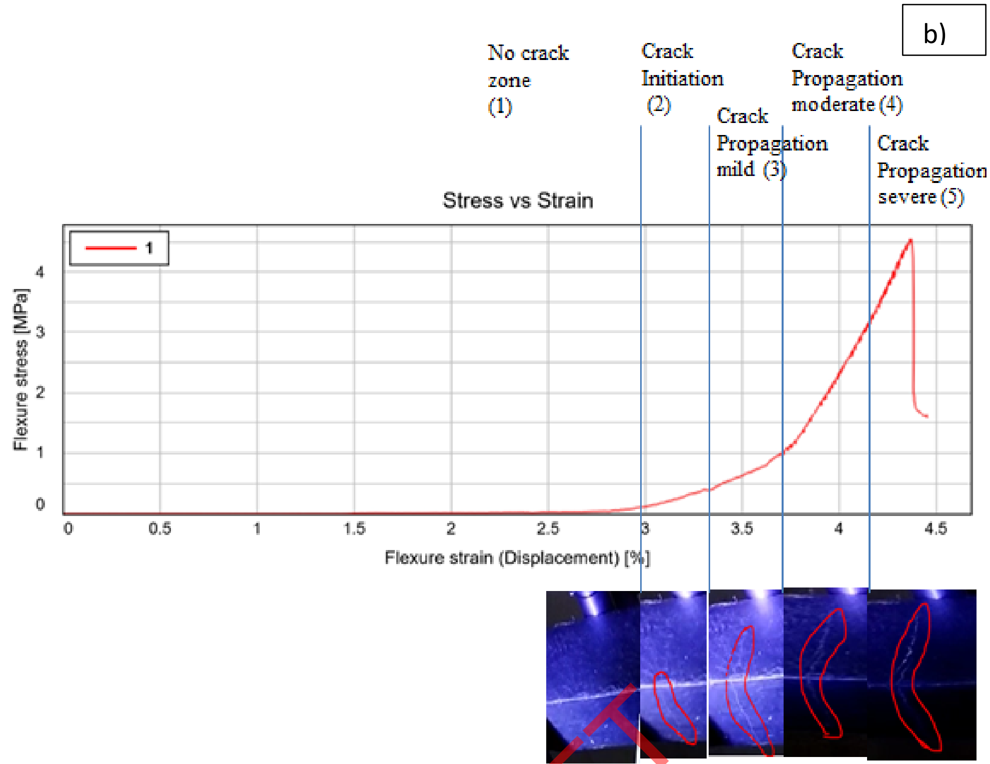


Fig. 7a, b. Stages of crack propagation corresponding to flexural stress and strain

The specimens depicted in Figures 7a and b underwent flexural testing, representing the typical substrate samples utilized in the study. From the Fig.7a it was observed that no crack formation occurred until the flexural strain reaches 0.8 %, accompanied by a corresponding stress of 0.7 MPA, Moving to next zone, crack initiation was observed for the flexural strain 1.2% with a corresponding stress of 1.1 MPa, followed by mild crack propagation was observed on both sides of the specimen for strain value of 1.5% with corresponding stress of 2.2MPa. The observed crack propagation on both sides of the specimen signifies that it has reached approximately 50% of its failure load. Advancing to the subsequent zone, the cracks exhibit a moderate appearance, but their length increases within the strain range of 1.75% and under a stress of 3.1 MPa. In the subsequent zone, the crack propagation becomes more severe compared to the previous zone and ultimately leads to failure under loading conditions. This occurs within a strain region of 2.1% and corresponds to a stress value of 5 MPa.

Fig. 7b demonstrates that no crack formation is observed until the flexural strain reaches 3%, accompanied by a corresponding stress of 0.2 MPa. Transitioning to the next zone, crack initiation is observed at a flexural strain of 3.4% with a corresponding stress of 0.5 MPa. This is followed by mild crack propagation on both sides of the specimen at a strain value of 3.6% and a corresponding stress of 1 MPa. The observed crack propagation on both sides indicates that the specimen has reached approximately 50% of its failure load.

Progressing to the subsequent zone, the cracks exhibit a moderate appearance, but their crack length increases within the strain range of 4.1% under a stress of 3.1 MPa. In the subsequent zone, the crack propagation becomes more severe compared to the previous zone, ultimately leading to failure under further loading conditions. This failure occurs within a strain region of 4.4%, corresponding to a stress value of 4.5 MPa.

The aforementioned values represent the crack growth rate of the specimen in relation to the applied flexural stress. Once the strain surpasses 0.7%, the crack propagation accelerates rapidly, ultimately leading to failure with a reduced strain rate. Evaluating the crack width and length enables the determination of crack severity. Numerous codes and standards are accessible for assessing the maximum permissible crack width and length. Typically, the allowable crack width falls within the range of 0 to 1 mm as specified by ACI 224R-01. It is crucial to note that exceeding these prescribed values has the potential to compromise the structural integrity of the concrete elements. While it is practically inevitable for concrete structures to experience some degree of cracking, restricting the occurrence and extent of cracks can significantly contribute to enhancing both structural safety and durability.

Conclusion

In this study, we conducted an investigation into the utilization of a mechano-luminousence (ML) based sensing technique for visualizing active cracks on a building and detecting crack propagation. The findings of this study led to the following insightful observations.

- The occurrence of crack initiation was noted in response to a flexural strain of 1.2%, which corresponded to a stress of 1.1 MPa. Subsequently, a gradual propagation of cracks was observed on both sides of the specimen upon reaching a strain value of 1.5%, accompanied by a corresponding stress of 2.2 MPa. This observed crack propagation on both sides of the specimen is indicative of its attainment of approximately 50% of its ultimate failure load.

- Proceeding to the next sample, the phenomenon of crack initiation becomes apparent at a flexural strain of 3.4%, coinciding with a stress of 0.5 MPa. Subsequently, a gradual propagation of cracks is observed on both sides of the specimen upon reaching a strain value of 3.6%, accompanied by a corresponding stress of 1 MPa.
- In this context, two noteworthy observations can be made. Firstly, the initiation of cracks can be effectively visualized in a repetitive manner by employing the mechano luminescence sensing technique. Furthermore, the severity of the cracks can be successfully estimated by leveraging the luminance information extracted through the ML approach.
- Based on the test results, when the crack propagation reaches approximately 60% of the specimen length, it serves as an indication that the specimen is nearing the failure zone. This observation alerts us to exercise caution and apply additional care. Even in the presence of highly subtle crack phenomena, the crack can be accurately identified and captured from the integrated PL image.
- Consequently, alterations in the mechanical equilibrium and crack propagation can be effectively visualized by examining the changes observed in the ML pattern and the displacement of the ML point, respectively.

According to usage, the ML diagnostic technique proves to be not only a highly sensitive sensor suitable for practical implementation in real-world scenarios but also user-friendly in its operation. Indeed, even individuals lacking professional expertise in the field of sensing were able to identify abnormal developments and recognize the significance of the mechano luminescence (ML) points. This heightened awareness enabled them to have a sense of impending danger and take appropriate precautions. Furthermore, one can demonstrate their ability to accurately estimate the gradient of strain and detect cracks based on the luminance information derived from the ML technique. Based on the conducted practical applications, it can be deduced that the mechano-luminescent (ML) sensing technique exhibits significant potential as a viable non-destructive evaluation (NDE) solution in domains such as aircraft runways, structures containing highly explosive pipes, railway tracks, chimneys and similar fields. Its deployment has the potential to contribute significantly to the maintenance of social safety and enhance overall structural integrity. Consequently, we anticipate that this technique will be embraced by other users with a vested interest in employing sensors for building applications.

Ethics Declarations

Conflicts of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Consent for publication

The corresponding author declares consent for publication in the journal of *Innovative infrastructure solutions.*

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