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Low electric field induction in BaTiO₃-epoxy nanocomposites

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Abstract

Epoxy is widely used material, but epoxy has limitations in terms of brittleness in failure, and thus researchers explore toughening and strengthening options such as adding a second phase or using electromagnetic fields to tailor toughness and strength, on demand and nearly instantaneously. Such approach falls into the category of active toughening but has not been extensively investigated. In this research, Si-BaTiO₃ nanoparticles were used to modify the electro-mechanical properties of a high-performance aerospace-grade epoxy so as to study its response to electric fields, specifically low field strengths. To promote uniform dispersion and distribution, the Si-BaTiO₃ nanoparticles were functionalised with silane coupling agents and mixed in the epoxy Araldite LY1564 at different content loads (1, 5, 10 wt%), which was then associated with its curing agent Aradur 3487. Real-time measurements were conducted using Raman spectroscopy while applying electric fields to the nanocomposite specimens. The Raman data showed a consistent trend of increasing intensity and peak broadening under the increasing electric field strength and Si-BaTiO₃ contents. This was attributed to the BaTiO₃ particles' dipolar displacement in the high-content nanocomposites (i.e., 5 wt% and 10 wt%). The study offers valuable insights on how electric field stimulation can actively enhance the mechanical properties in epoxy composites, specifically in relatively low fields and thin, high-aspect-ratio composite layers which would require in-situ mechanical testing equipped with electric field application, an ongoing investigation of the current research.

Keywords Barium titanate, Functional material, Dielectric polymer, Electric field polarisation, Raman, Intrinsic strain

Introduction

Fibre-reinforced polymer (FRP) composites are commonly used in high-performance sectors due to their superior specific strength and modulus [1]. FRP composites, made mainly of thermoset epoxy resins with a highly cross-linked structure, have superior properties. However, brittleness in transversal direction is a limitation to their failure, especially when exposed to impact loading or high strain rate [2]. To enhance the properties of the polymer matrix in composite fabrication, researchers have focused on adding micro- and nano-fillers, like rubber tougheners, as a second phase. As tailoring fibre properties during the process is difficult, this approach has been widely researched instead [3–7]. To improve the properties of the polymer matrix

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in fibre-reinforced composites, researchers have explored several approaches. These include adding rubber tougheners or nanoparticles, as well as incorporating silica particles, carbon nanoparticles, clay, and fibre coating into the matrix. Additionally, modifying the curing process has been studied as an alternative to modifying fibre properties during composite fabrication, which can be challenging [8, 9]. By incorporating rubber tougheners, nanoparticles, silica particles, carbon nanoparticles, clay, and fibre coating into the polymer matrix, and modifying the curing process, researchers can tailor the mechanical properties of fibre-reinforced composites. This includes enhancing stiffness and toughness, which are critical for many applications. These techniques are an alternative to modifying fibre properties during composite fabrication [10]. In order to enhance fracture toughness in composite materials, rubber tougheners like core-shell rubber or liquid rubbers are added to the polymer matrix. These tougheners help absorb energy during deformation and reduce stress concentration. Studies have demonstrated that adding rubber tougheners to epoxy resin can significantly increase the composite's fracture toughness by up to 200%. This approach is an effective way to improve the mechanical properties of composites without modifying the fibre properties during fabrication [11]. While adding rubber tougheners to polymer matrices in composite materials can enhance fracture toughness, exceeding a certain concentration threshold can result in particle clumping, leading to mechanical deterioration and reduced strength. This underscores the importance of careful consideration and optimization of the amount and type of toughener used to achieve the desired mechanical properties in the composite. Despite this challenge, incorporating rubber tougheners remains a promising approach for enhancing the mechanical performance of epoxy composites [12]. To prevent agglomeration of rubber tougheners in polymer matrices, surface modifications and multiple toughening agents can be employed. Coupling agents can improve bonding between particles and the matrix in epoxy composites, but can also lead to reduced elastic modulus, tensile strength, and glass transition temperature. Sung et al. proposed a method for toughening epoxy matrices in composites by pre-stressing via expandable hollow microspheres under heating. This approach represents a novel way to improve the mechanical properties of composites while avoiding the potential drawbacks of other toughening methods [13]. Phase-transition toughening, and transformation toughening are two techniques used to improve the toughness of polymer matrix composites. Phase-transition toughening uses expandable microspheres to induce a volume dilation effect, significantly improving the performance of composites. However, this approach may result in increased weight and potential

Table 1 BaTiO₃ characteristics

Characteristics	Value/Type
Phase	Tetragonal
Size	200 nm
Density	5.85 g/cm ³
Specific surface area (SSA)	6–7 m ² /g Spherical

compromises in mechanical properties. Transformation toughening involves causing a second phase to form under stress, creating a compressive force around the crack tip that resists crack propagation. This mechanism is effective in improving fracture toughness in a variety of materials, including ceramics and polymer matrix composites [13].

This study investigates the initial steps for development of the active toughening in Si-BaTiO₃-epoxy nanocomposites under a relatively low electric field. A real-time in-situ Raman spectroscopy was employed for the investigations to measure the response of the Si-BaTiO₃-epoxy nanocomposites under electric field stimulation. The results of this study offer valuable insights into the ongoing development by the current research for active toughening of Si-BaTiO₃-epoxy nanocomposites. The Raman data revealed a consistent trend of increasing intensity and peaks broadening under increasing electric field strength and BaTiO₃ contents. The softening effect induced by BaTiO₃'s dipolar displacement in the high-content nanocomposites dominated the stiffening trend induced by the electric fields observed in the 1 wt% specimen, which may not apply to other nanocomposites.

Materials and methods

Materials

The epoxy used in this study was Araldite LY1564, and was supplied by Huntsman, UK. The coupling agent for surface functionalisation selected in this study, 3-glycidypropyl trimethoxy silane (3-GPS), the hydrogen peroxide (H₂O₂, 30%) and the acetic acid (C₂H₄O₂, 99.9%) used as functionalisation aids were all supplied by Sigma-Aldrich, UK. The ethanol (C₂H₆O, 99.9%) used for BaTiO₃ dispersion was supplied by Fisher Scientific International, Inc., UK. BaTiO₃ was supplied by Nanostructure & Amorphous Materials Inc., US. The characteristics of the BaTiO₃ is provided in Table 1.

Pre-treatment (Hydroxylation process)

It is important to note that the method described here is for the pre-treatment and purification of BaTiO₃ nanoparticles and not for the incorporation of nanoparticles into the epoxy matrix. For the pre-treatment of the BaTiO₃ nanoparticles, 10 g of the nanoparticles were added to a 230mL solution of hydrogen peroxide (H₂O₂) in a round bottom flask. The mixture was sonicated in an ultrasonic bath for 30 min and then refluxed at the

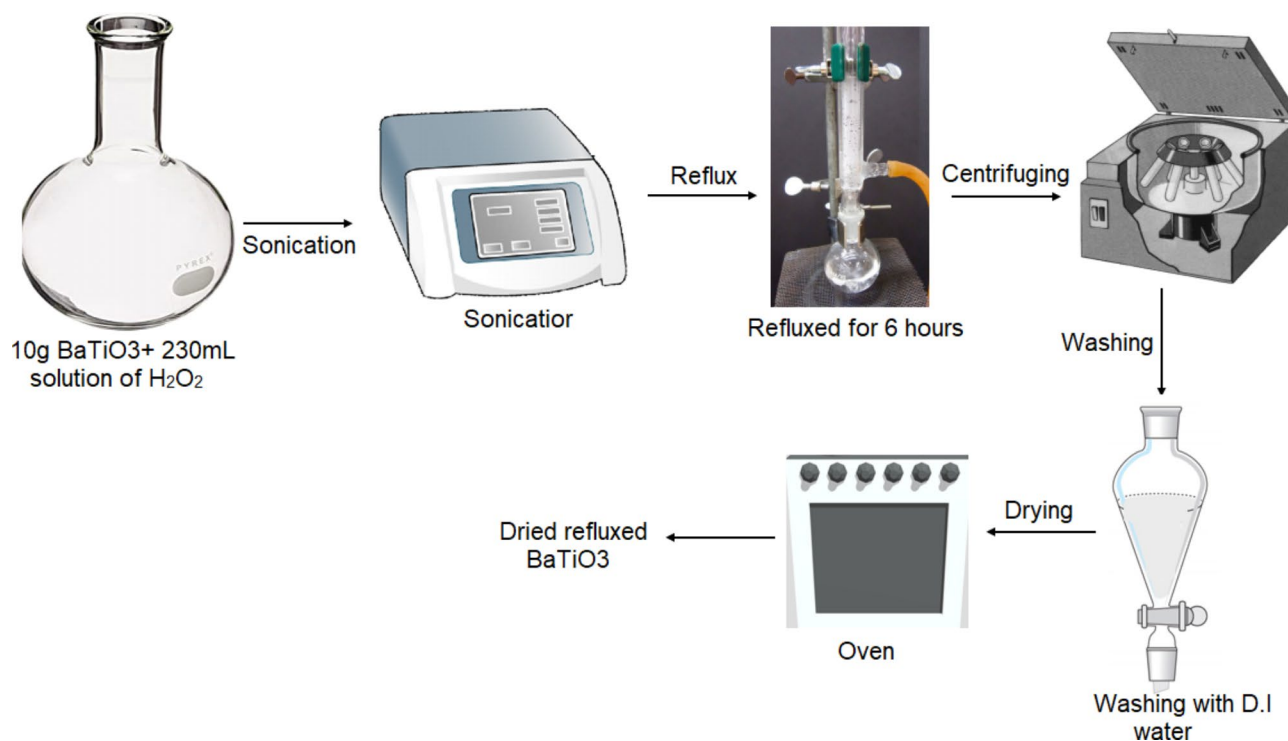


Fig. 1 Schematic diagram of the Hydroxylation Process of BaTiO₃ nanoparticles (pre-treatment for adding of hydroxyl group (-OH) to the surface of BaTiO₃)

boiling temperature of a 30% H₂O₂ solution at 108 °C for six hours with stirring at 100 rpm. The purpose of refluxing was to facilitate the process by heating without losing H₂O₂. After six hours, the resulting solution was centrifuged at 4500 rpm for 15 min to retrieve the BaTiO₃ nanoparticles, which were then washed three times with deionized water. The purified BaTiO₃ nanoparticles were dried in an oven at 80 °C for 24 h, as shown in Fig. 1.

Preparation of silane modified barium titanate (Si-BaTiO₃)

The purpose of using 3-glycidypropyl trimethoxy silane (3-GPS) was to improve the processability and dispersion of BaTiO₃ nanoparticles in the nanocomposite. To apply the silane, a solution of 1 wt% of 3-GPS with respect to BaTiO₃ reflux was prepared, and a low pH solution was made by mixing a 150mL aqueous solution of ethanol and deionized water (9:1) with acetic acid until the pH value of 3.5–4 was reached. The 0.1 g 3-GPS solution was added to the acidified solution and left in an ultrasonic bath for 30 min to form a homogeneous solution. The hydroxylated BaTiO₃ powder particles were then added to the silane solution and mixed under ultrasonic bath for 10 min to improve the wetting of the filler. The mixture was refluxed at the boiling temperature of ethanol, at 78 °C, at 100 rpm using a mechanical stirrer for six hours. After refluxing, the Si-BaTiO₃ powders were washed three times with deionized water and retrieved using centrifugation at 4500 rpm (Fig. 2). Finally, the powders

were dried at 110 °C for 24 h to avoid any condensation of silanol groups at the surface and crushed in a mortar and pestle for the nanocomposite's preparation.

FTIR analysis was performed to confirm the existence of functional groups on the surface of the BaTiO₃ nanoparticles after silane treatment (Fig. 3). The Jasco FT/IR-6200 was used in transmission mode to measure the FTIR spectra of BaTiO₃ and Si-BaTiO₃ powders in the range of 400–4000 cm⁻¹ with a resolution of 2 cm⁻¹ at room temperature. The FTIR spectra showed peaks at 1437 cm⁻¹ and 1630 cm⁻¹, indicating small traces of BaCO₃, and physically absorbed water on BaTiO₃ powders, respectively, which is a result of the combustion fabrication process. The presence of hydroxyl groups (OH) from the Si-OH group was confirmed by bands at 3200–3700 cm⁻¹. Successful silane modification of BaTiO₃ was confirmed by the appearance of spectrum bands at 850 cm⁻¹ and 1250 cm⁻¹, which indicate the Si-O, Si-O-C₂H₅, and Si-O vibrations.

Preparation of silane modified barium titanate (Si-BaTiO₃)/epoxy nanocomposites

The epoxy resin nanosuspension with 1, 5 and 10 wt% Si-BaTiO₃ nanoparticles and 10 wt% untreated BaTiO₃ nanoparticles were prepared as follows: Firstly, the weighed amount of BaTiO₃ powders was added to ethanol and sonicated with an ice water bath for 2 min with a 10s pulse to form a homogenous solution. Then a

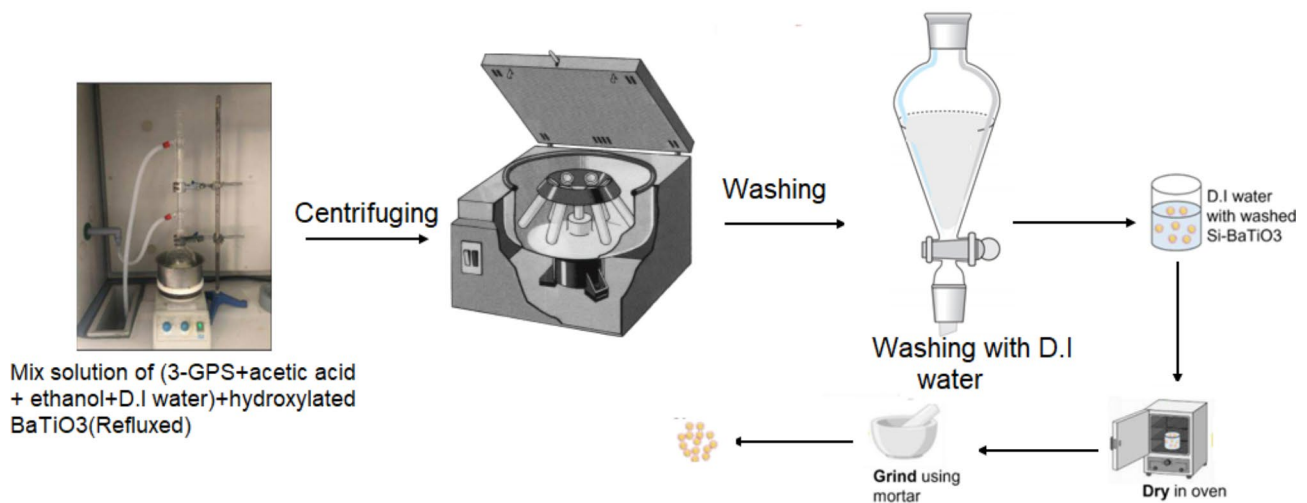


Fig. 2 Schematic diagram of the functionalisation process of BaTiO₃ with 3-GPS. The epoxy nanocomposite fabrication process including Si-BaTiO₃ functionalisation is schematically illustrated

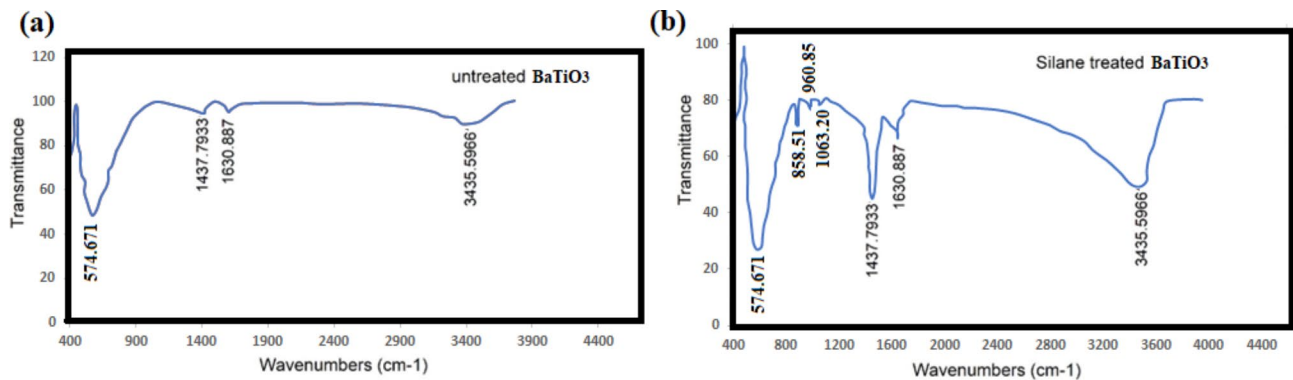


Fig. 3 FTIR spectra of unmodified BaTiO₃ and modified BaTiO₃, (a) unmodified BaTiO₃, (b) modified BaTiO₃ (Si-BaTiO₃).

weighed amount of epoxy was added to the solutions which were stirred using a mechanical stirrer at 300 rpm and 80 °C overnight under the fume hood to gradually remove the ethanol without precipitation of the particles. The epoxy mixtures were weighed before and after the evaporation of the solvent to ensure the complete removal of ethanol. The curing agent was then added to the mixture with a weight ratio recommended by the company and stirred for a further 3 min (The ratio of epoxy and curing agent was 2.94:1). Finally, the mixture was placed in the vacuum oven at 30 °C for 1 h to remove air bubbles at 29 inHg and achieve complete removal of ethanol. The whole mixture was poured into a mould made of two pieces of glass clamped with a 3 mm silicone gasket in-between, as shown in Fig. 4. This set-up assured that a uniform thickness of specimens was achieved. The specimens were then cured in the oven for 8 h at 80 °C as recommended by the manufacturer, then cooled down to room temperature. Differential scanning calorimetry has been carried out to ensure an ultimate cure degree has been achieved using the process parameters identified

above. The final specimens were of size 100×20×0.3 mm³ and cut to different sizes using a precision cut-off machine BRILLANT 220.

Characterisations

Several studies provide valuable characterisation of BaTiO₃-epoxy composites [14, 15] and introduces a novel approach to sensor placement for reliable damage detection [14]. In a different study, researchers developed a method that involves adding piezoelectric particles to a polymer matrix and extruding the mixture into a fibre [15]. By using unsintered calcined powders of PZT and BaTiO₃, the resulting ferroelectric hybrid fibres become flexible and soft. The researchers investigated the electromechanical behaviour of these fibres under varying electric fields and found that they can achieve up to 10% of the maximum polarisation observed in sintered counterparts. This approach shows promise in developing flexible and lightweight materials with piezoelectric properties [15]. This study provides exciting new possibilities for the development of memristive devices based

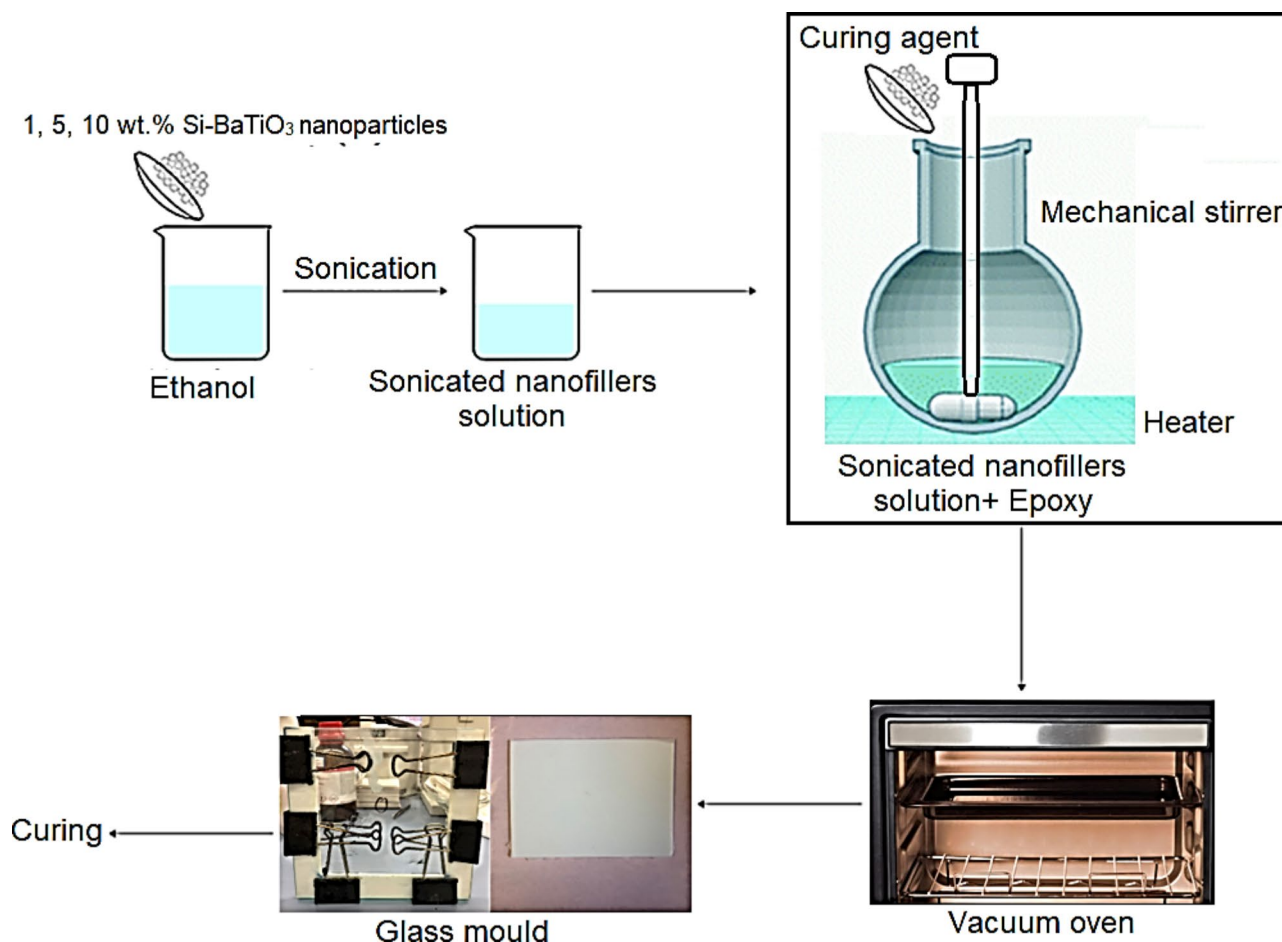


Fig. 4 Schematic diagram of the preparation of Epoxy/Si-BaTiO₃ nanocomposites

on ferroelectric films, with potential applications in information storage, computing, and neuromorphic systems. In a recent paper, “In-plane charged domain walls with memristive behaviour in a ferroelectric film” the authors investigated in-plane charged domain walls in a ferroelectric film that exhibit memristive behaviour, with a nonlinear relationship between polarisation and applied voltage and hysteresis. They used conductive atomic force microscopy (CAFM) to study the local current-voltage (I-V) characteristics of the charged DWs, with the specimens mounted on a round metal holder and connected to the internal source electrode of an atomic force microscope. Commercial silicon tips coated with Pt were used to measure the current, and bias sweeps were applied to the specimen from the internal source. The bias sweeps were performed in a positive-to-negative voltage circle (0 V to +10 V to -10 V to 0 V) with a time of 100 s (0.1 V s⁻¹), and the currents were measured with a limit of 100 μ A [16].

The research conducted usually investigated the material response to electric field with strong polarising potential (i.e. kV/cm > 10). We have designed an

in-situ electric field setup to analyse the response of the thin Si-BaTiO₃/nanocomposites at relatively low fields (<2 kV/cm), and through primary investigation via an electric field equipped Raman spectroscopy.

Microstructural analysis

Microstructure characterisation of the cryo-fractured surface of the cured epoxy nanocomposites (at different Si-BaTiO₃ wt%) was performed using a field emission scanning electron microscope TESCAN Vega 3. This technique allowed observing the microstructure of the nanocomposites, revealing the distribution of nanoparticles in the matrix and the interface between the two phases. The results of the microstructure characterisation provide information about the properties of the nanocomposites and are useful for understanding the mechanical behaviour of the material.

Electric field induced Raman spectroscopy

Raman spectroscopy is a non-destructive technique that enables the probing of the vibrational modes of a material. It is widely used for the characterization of materials

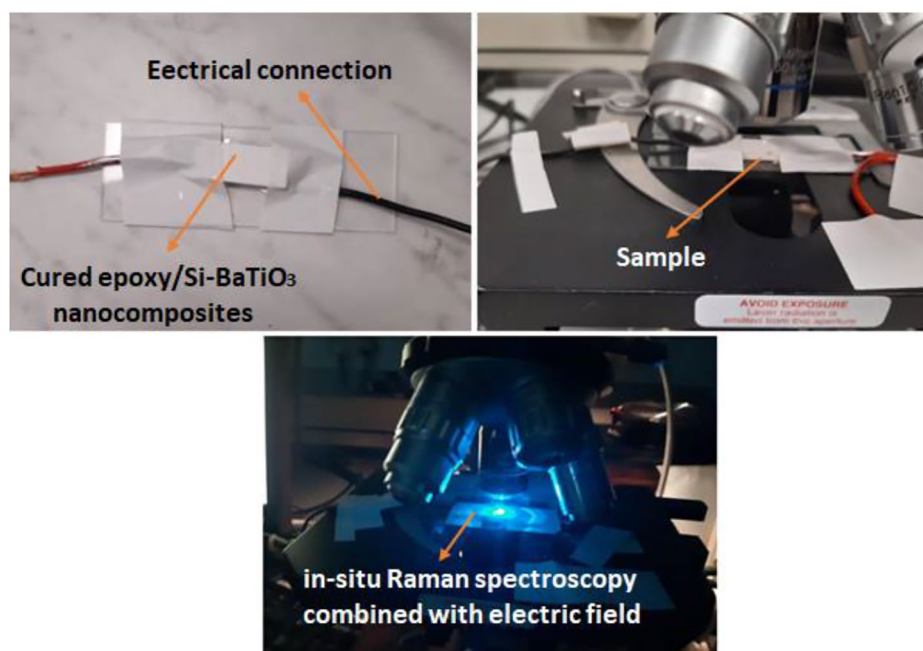


Fig. 5 In-situ electric field equipped Raman spectroscopy of BaTiO₃-epoxy nanocomposites

as it can provide information on their molecular and crystalline structure, as well as any residual strain present. For Raman spectroscopy measurements, the choice of excitation wavelength is crucial, with 514 nm being a common choice due to its balance between resolution and signal intensity. In this study, in-situ Raman spectroscopy equipped with an electric connection was employed to investigate the polarisation behaviour of the nanocomposite material in the presence of an external electric field. To this end, the voltage applied to the specimen was varied between 0 V, 6 V, 9 and 24 V. The use of in-situ Raman spectroscopy enabled the real-time monitoring of any changes in the vibrational modes of the material under the influence of the applied electric field. The setup for in-situ Raman spectroscopy typically involves the use of electrodes to apply an electric voltage to the specimen while the Raman spectra are acquired. This technique can provide valuable insights into the response of the material to external electrical stimuli, which is essential for the development of new materials with tailored electrical properties. The set-up for in-situ Raman spectroscopy equipped with an electric connection is shown in Fig. 5.

Results

To examine the internal structure of the cured nanocomposites, cryo-fracturing and field emission scanning electron microscopy (FESEM) were utilized. Cryo-fracturing using liquid nitrogen was performed to expose the cross-sectional morphology of the specimens. Before FESEM imaging, a thin layer of gold was sputter-coated onto the

specimens to enhance conductivity. The FESEM images provided insight into the distribution of BaTiO₃ within the epoxy matrix and enabled the evaluation of the modification process's efficacy. Additionally, the FESEM images provided information regarding the quantification of Si-BaTiO₃ distribution within the epoxy matrix and effect of silane functionalisation. To fully characterize the properties of the nanocomposites, Raman spectroscopy was utilized to investigate the vibrational modes of the material and provide information on its molecular and crystalline structure, as well as any residual strain present. The 514 nm excitation wavelength laser was chosen for Raman spectroscopy due to its optimal balance between resolution and signal intensity. Additionally, in-situ Raman spectroscopy equipped with an electric field was used to study the polarisation behaviour of the material in the presence of an electric field. The voltage applied to the specimen was varied between 0 V, 6 V, 9 V, and 24mV, and the results were used to evaluate any significant differences in the spectra with and without an electric field.

Scanning electron microscope (SEM)

In this study, BaTiO₃ particles were subjected to modification using the silane coupling agent 3-GPS to achieve a more finely dispersed state in an epoxy matrix. The dispersion and distribution of nanoparticles in the epoxy resin were characterized using scanning electron microscopy (SEM) at different weight percentages. With the use of the silane coupling agent, the surface of the BaTiO₃ particles was functionalized, leading to improved

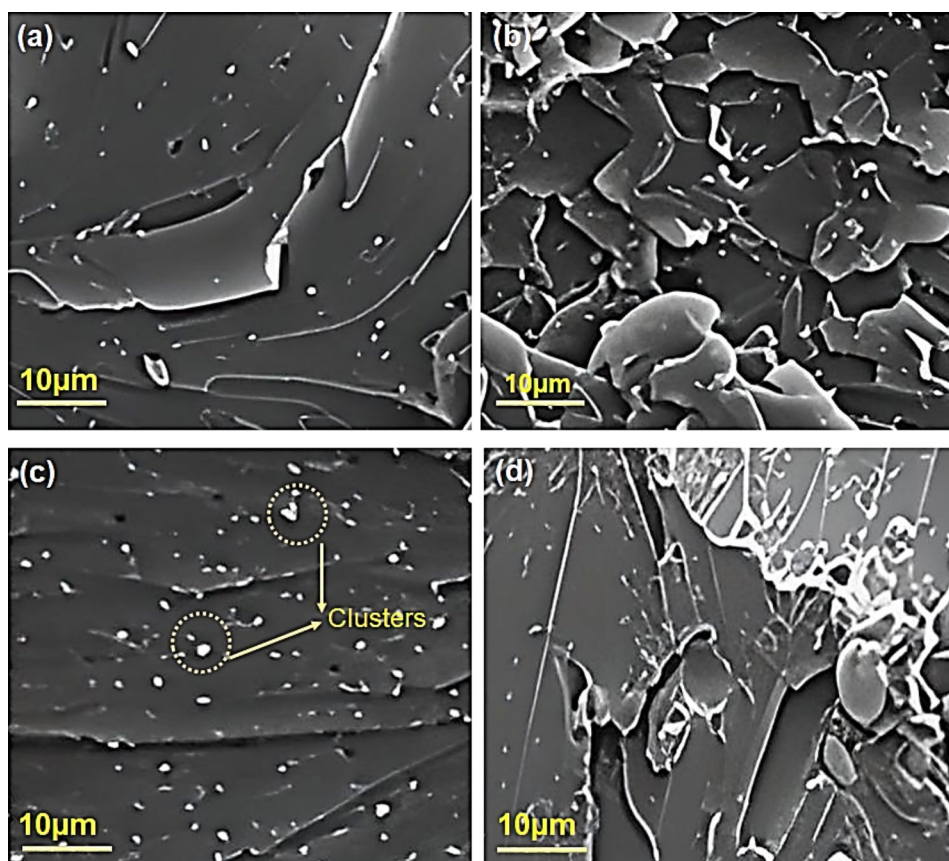


Fig. 6 SEM images from the fracture surface of epoxy nanocomposites (taken from the former research in [13]): (a)–(c) images at 1 wt%, 5 wt% and 10wt% Si- BaTiO₃, (d) untreated 10wt% BaTiO₃.

dispersion and several levels of aggregation events. SEM images of the cross-section of functionalized BaTiO₃ epoxy composites at 1%, 5%, and 10% weight fractions are presented in Fig. 6. The microstructure suggests that the functionalization of the BaTiO₃ particles with the 3-GPS silane coupling agent resulted in improved dispersion and reduced aggregation in the epoxy matrix. We have also analysed the cross-section of BaTiO₃ epoxy composites with and without a silane coupling agent at a weight% of 10% BaTiO₃ using SEM imaging. The SEM image of the non-functionalized composite in Fig. 6(d) reveals a cracked cross-section with several levels of particle aggregation, indicating poor dispersion of particles. When compared to Fig. 6(d), which depicts a non-functionalized composite, the functionalized composite in Fig. 6(c) has a more uniform particle dispersion due to the use of a coupling agent. This suggests that the coupling agent has a positive impact on achieving uniform dispersion of particles in the composite. Further analysis of the SEM images revealed that the silane-treated particles in the functionalized composite were nearly 200 nm in size, and there was no apparent aggregation of Si-BaTiO₃ particles. This indicates that the silane coupling agent was effective in achieving a finer dispersion of

particles in the epoxy resin. These findings are consistent with previous research, which highlights the effectiveness of the silane functional group in improving the dispersion of particles in polymer composites [13]. Based on the information SEM images, it can be inferred that the BaTiO₃ particles were modified using a silane coupling agent called 3-GPS to achieve a finer dispersion in the epoxy resin. The use of a silane coupling agent resulted in a more uniform dispersion of particles, which can lead to improved composite properties. Overall, the use of a silane coupling agent can be an effective way to achieve a finer dispersion of nanoparticles in polymer composites. The SEM images revealed that the use of a silane coupling agent can be an effective strategy to achieve better dispersion of nanoparticles in polymer composites and enhance their performance [17].

Electric field induced Raman spectroscopy

Under the application of an electric field (induced by applied voltage), the Raman peaks exhibit a shift in wave number, indicating the existence of residual strains induced by the applied voltage. Specifically, the Raman peak of BaTiO₃ at 515 cm⁻¹ shifts towards higher wave number with the increase in the applied voltage,

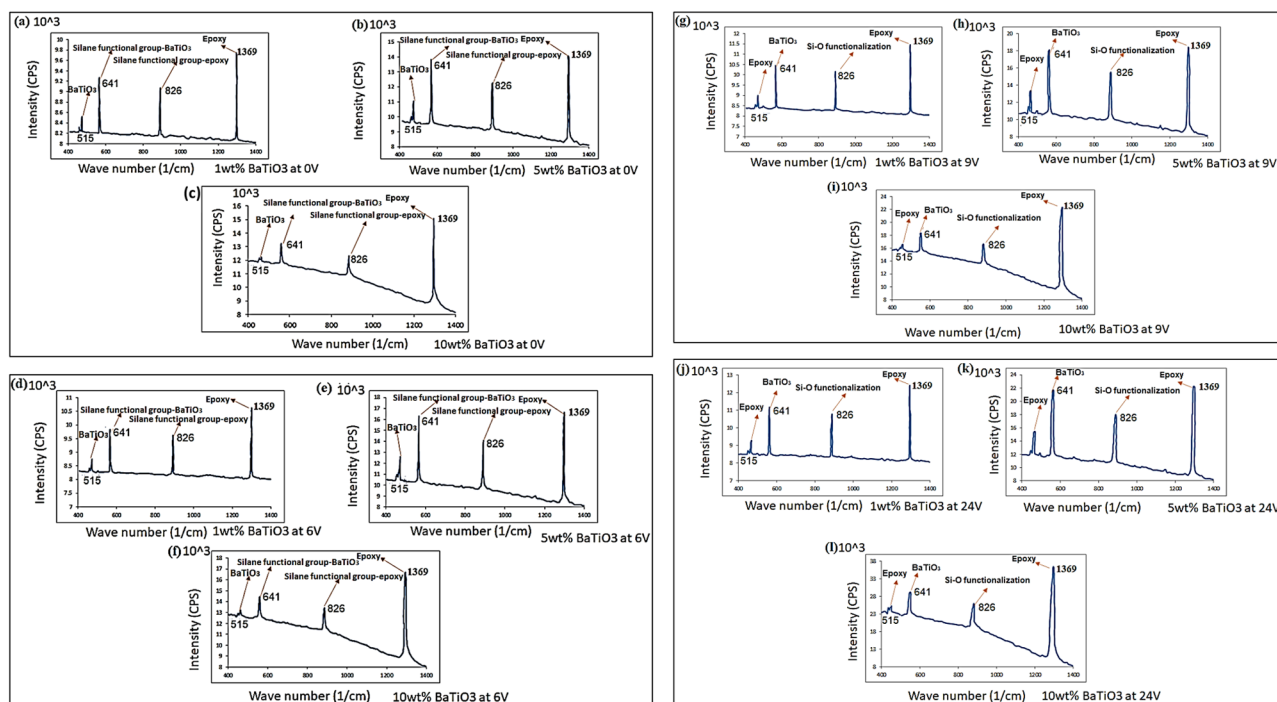


Fig. 7 Measurement of intensity and broadening of Raman peaks with respect to the wave number at different Si-BaTiO₃ content and under electric fields of (a-c) 0 V (no field), d-f) 6 V, (g-i) 12 V and j-l) 24 V

indicating a decrease in the inter-atomic distance and the presence of compressive residual strains in the nanocomposite material. The degree of shift increases with the increase in the concentration of BaTiO₃ in the nanocomposite. Similar observations are made for the Raman peaks of Si-BaTiO₃ and Si-epoxy. The Raman spectroscopy provides a powerful tool for the non-destructive characterization of the local residual strains in the nanocomposite materials, which can aid in the optimization of the nanocomposite design and performance [18]. The intensity of Raman peaks represents the polarizability of the molecule and the concentration of the active molecular groups, therefore the slight increase in intensity indicates slight increase in polarizability of Si-BaTiO₃-epoxy nanocomposites and increasing active species with the increasing content of Si-BaTiO₃. Raman spectra of Si-BaTiO₃-epoxy nanocomposites at 1, 5, 10 wt% of Si-BaTiO₃ under 6 V field (Fig. 11d-f) also shows an increase in the intensity when compared to the no-field. Such trend of increase in the intensity repeats itself in higher field strengths of 12 and 24 V (Fig. 11(g-i) and (j-l) respectively). Moreover, it is observed that the higher the voltage the broader the peaks. Such data ratifies the fact that the polarizability is not only dependent on the content of BaTiO₃ but also dependent upon the applied voltage. The broadening of peak and increasing the intensity of peak indicate the stretching of bond and structural change via field induced dipolar displacement at intrinsic

level and polarisation under such electric field (induced by applied voltage). When taking this intrinsic straining/stretching effect into account for Fig. 7 data, it can be implied that such stretch at interatomic crystalline level (under a simplified assumption of full crystalline BaTiO₃ structure) would contribute to the softening effect and thus reduction in the tensile modulus at larger extrinsic scales. This is a plausible interpretation of the data, as the broadening and intensity increase of the Raman peaks indicate a structural change induced by the electric field (induced by applied voltage). This structural change could lead to a reduction in the tensile modulus at larger scales, as observed in the tensile testing data. However, further experimentation and analysis would be required to confirm this relationship between the Raman spectroscopy results and the tensile properties of the nanocomposites (under investigation). It is important to note that the interpretation of Raman spectroscopy data can be complex and requires careful consideration of various factors, including the composition and morphology of the specimen, the laser wavelength and power, and the measurement conditions [19]. In other words, the Raman spectroscopy results suggest that the electric field not only may affect the overall mechanical properties of the nanocomposite material but also induces changes at the molecular level, particularly in the interatomic crystalline structure of the BaTiO₃ nanoparticles. The increase in polarizability and active molecular groups with

increasing Si-BaTiO₃ content and applied voltage implies that the electric field has a greater effect on the BaTiO₃ nanoparticles than on the epoxy matrix. The stretching of bonds and structural changes induced by the electric field at the molecular level can contribute to mechanically softening.

The results indicate that the application of an electric field (induced by applied voltage) introduces a shift in the Raman peaks towards higher wave numbers, which suggests the presence of compressive residual strains in the nanocomposite material. The shift in the Raman peak increases with the increase in the concentration of Si-BaTiO₃ in the nanocomposite has been observed. The increase in polarizability and active molecular groups with increasing Si-BaTiO₃ content and applied voltage implies that the electric field has a greater effect on the Si-BaTiO₃ nanoparticles than on the epoxy matrix. The broadening and intensity increase of the Raman peaks indicate that there may be a structural change induced by the applied electric field (induced by applied voltage), though seems unlikely at such low voltage, which could lead to a change in the extrinsic strains and mechanical response at larger scales. Further investigation would be required to quantitatively describe the phenomenon using an in-situ equipped mechanical testing.

Conclusions

The development of in-situ electrical field equipped dielectric nanocomposite is a state-of-the-art approach introduced to study the ferroelectric and mechanical response of BaTiO₃-epoxy composite in addition to the provision of a remote property tailoring technology. In an attempt to realise and quantify such response prior to proposing a remote tailoring innovation for engineering applications, the article investigated the alteration of mechanical properties by the dipole induced intrinsic strains in thin BaTiO₃-epoxy nanocomposites in the presence of low electric fields. A feasibility study was conducted on 1, 5 and 10 wt% of BaTiO₃ nanoparticles embedded epoxy nanocomposites. The epoxy grade was selected from high-performance grade products used in advanced and rigid structures, e.g. aerospace structures. During the fabrication of the nanocomposites, the surface functionalisation of nanoparticles was carried out to improve the interfacial particle-matrix bonding quality and their dispersion inside the epoxy. Raman spectroscopy was carried out under such electric fields. The results show that, provided achieving a high quality dispersion and particle-polymer bonding quality, the electric field application at the low strength examined has a considerable electric field-induced polarisation observed by the broadening of the peaks during the in-situ Raman measurements. These findings suggest that the intrinsic straining/stretching effect induced by the electric field

on the interatomic crystalline level may contribute to mechanical softening effects and reduction in mechanical properties, under further investigation.

Authors' contributions

R.M. developed the electric field equipped nanocomposite testing rig. D.L. fabricated the technique to develop the nanocomposite specimens. R.M. contributed to the development of the specimens. I.C., S.L. and S.G. conducted data analysis. H.Y.N. developed the initial idea, supervised the research and contributed to the analysis of the data and paper writing. All authors read and approved the final manuscript.

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Data availability

The underpinning data can be accessed publicly at DOI: <https://doi.org/10.25383/city.21651254>.

Declarations

Competing interests

I declare that the authors have no competing interests as defined by Springer, or other interests that might be perceived to influence the results and/or discussion reported in this paper.

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