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Milad Zolfipour AGHDAM [1], Naser SOLTANI [1], Hadi NOBAKHTI [1]

Application of mixed-nanoparticle coating as a novel simple method in generating speckle pattern to study small fields of view by digital image correlation

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Digital image correlation (DIC) is a powerful full-field displacement measurement technique that has been used in various studies. The first step in the DIC is to create a random speckle pattern, where the spraying method is usually employed. However, creating an optimal pattern and modification in the spraying method is not convenient. Furthermore, the size of speckles which is not so small in spraying method, limits the minimum size of the field of study. In the present research, a convenient novel technique was introduced and investigated to generate a practical kind of speckle pattern with small speckles for evaluating smaller fields of view using nanoparticles. The pattern was created by spreading a mixture of different black and white nanoparticles. To this end, the black graphene oxide particles were mixed with white nanoparticles of titanium oxide, zirconium oxide and silicon to obtain three mixtures. Displacement tests show that the mixture of graphene and titanium provides the best DIC performance. More granularly, graphene and titanium were mixed at three different ratios to find the optimal combination. Subsequently, the accuracy of the new patterning method was analyzed via tensile testing and the results were compared against those of conventional method with various subset sizes.

1. Introduction

Digital image correlation (DIC) is a powerful, non-contacting optical technique for full-field measurement of surface displacements. It has been adopted by a variety of different fields, including fracture [1–4], mechanics and bio-mechanics [5–7], thermal strain and buckling [8–10], modal analysis [11], and structure monitoring

✉ Milad Zolfipour Aghdam, e-mail: zolfipoor81@gmail.com

[1] Intelligence Based Experimental Mechanics Center, School of Mechanical Engineering, College of Engineering, University of Tehran, Tehran, Iran. ORCID (M.Z.A.): 0000-0002-0260-1673

[12], thanks to its advantageous simple setup and operation, reusability, low cost, accuracy, and pervasive compatibility. First proposed and utilized by a group of researchers at the University of South Carolina in the 80's [13–15], DIC has experienced a great deal of advancements since that first research. DIC has been and can be improved in different aspects, including the imaging instrumentation, the correlation algorithm and parameter tuning, sources of error, and the specimen surface preparation [5].

The idea behind the DIC is to take images of the specimen surface both before and after deformation and then analyze the two images to calculate displacements of the target specimen. Therefore, each point on the surface of the target specimen must be identifiable for the algorithm before it can compare the two images. In order to ensure that the points are identifiable, the specimen surface is usually coated with a random pattern (usually composed of black speckles on a white surface). Following the original DIC design, spraying a black-colored material has been the most frequent approach to creating a speckle pattern [16], because of its simplicity and cost efficiency. However, this method cannot be used in immediate cases, but rather requires time to have the coating and the speckles to be dried, not to mention that a great deal of experience is necessary for implementing this method, because it is extremely difficult to control the size and number of sprayed droplets. Accordingly, one may end up with too large or small, light or heavy speckles to be used. Furthermore, it takes considerable time to modify and regenerate a pattern through this methodology. The spraying method is also appropriate for a defined field of study, however, to create a speckle pattern in large and small scales of DIC other methods should be applied.

Many experimental studies, nowadays, are based on small displacements and small fields of study, and many attempts have been made to develop other methods to create such speckle patterns for various purposes and in small fields of study and small displacements. Loyuns [17] coated the surface of a specimen with a ceramic coating to prepare it for the DIC. Usage of this technique needs a wide range of chemical knowledge to create an appropriate ceramic to adhere to the specimen surface. This method also requires expensive instruments. Moreover, creating a coating on most specimens is not possible. The coating can also be created through air-brushing the surface with a solution of particles. Berfield et al. [18] were the first to propose this technique in 2007. They applied solution deposition of fluorescent particles to form a speckle pattern, making them able to create patterns with different speckle sizes due to the use of different particles in the solution, particularly at the micro and nano scales. Reducing the size of particles to nano scale, one could obtain very fine speckles. The minimum achievable speckle size in this technique has been as low as 10 micrometers [18–20]. The air-brushing of a solution exhibits a number of difficulties. The methods require preparation of sample-proper solutions, a great deal of experience, for example in dye spraying, and different tools for splashing. Consequently, alternative methods have been developed to spread the solution with neither spraying nor air-brushing, where nanoparticles are self-assembled into the coating. Sutton et al. [21] coated a specimen with a towner powder in a 50 × 250 micrometers field of view. In this method, similarly as in spraying and air-brushing, there is no control on powder speckles, and as a result the speckles might not be appropriate. There are also many other techniques to create speckle patterns for different applications, such as self-assembling nanoparticle [22, 23] and nano-film remodeling [24–26]. All of these are specially developed for particular research purposes rather than industrial applications. Some of them, furthermore, need complicated procedures, expensive equipment or specific knowledge to apply. In general, there are many difficulties in these methods that prove the necessity of introducing a convenient speckle pattern that can be created on a small field of interest.

The objective of the present study is to propose a novel method for generating a speckle pattern to study small fields of view. The method is based on spreading a mixture of black and white nanoparticles. In this method, a coating of mixed nanoparticles is spread on the specimen's surface to develop a pattern. In the mixture, the black nanoparticles serve as black speckles and are surrounded by a white margin established by the white nanoparticles. The proposed method facilitates the speckle generating as the speckles are already made by the black particles, and the job can be easily done, merely by spreading the mixture over the sample. The types of the black and white nanoparticles and their ratio are important factors affecting the generation of a proper speckle pattern. The two factors can be adjusted to achieve the desired speckle contrast and density and edge sharpness. Investigation of these two factors makes the process of patterning faster, easier, and more controllable. To investigate these two factors, in the first section, displacement test was selected to compare the mixture of different material, and secondly the best ratio of mixing black and white nanoparticles was determined. In the next section, to investigate application of method and subset size, the new method of pattern generation was compared with a conventional method under tensile test, and then the DIC results were qualified by the result of a strain gauge.

2. Experiment

2.1. Principle of DIC

DIC is based on capturing and analyzing two images captured from a speckle pattern before and after a deformation event. Fig. 1 shows the setup of DIC. As mentioned earlier, the DIC begins with patterning. Contrast, density and size of the speckles must be optimized to reach a high-accuracy DIC [27–30]. Once finished with patterning, two images are captured and then analyzed using a clustering algorithm that divides the area of the field of view of the pattern into subsets of a specific size. It then takes the central pixel displacement as the rigid displacement and investigates the displacement of the other pixels across the subset relative

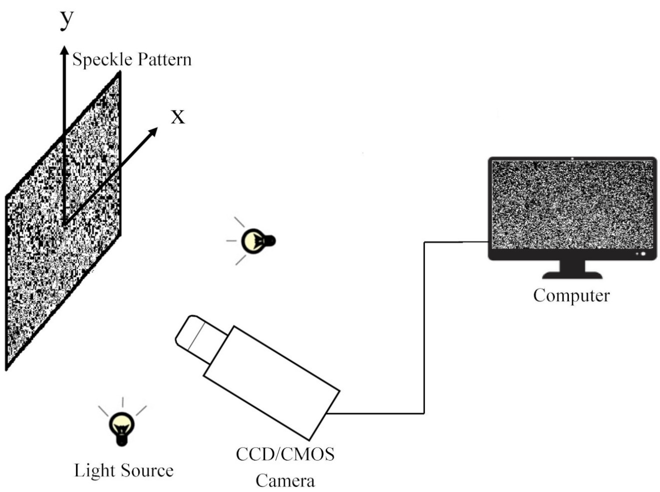


Fig. 1. Schematic of the DIC setup

to the central pixel to determine strains and rotations. Fig. 2 provides a schematic demonstration of this process. Preset by the user, the subset size affects the accuracy of DIC and must be appropriated to the required speckle pattern in the particular field of study. To achieve an accurate correlation, the subset size must be large enough to make each subset distinguishable from the other subsets, in other words,

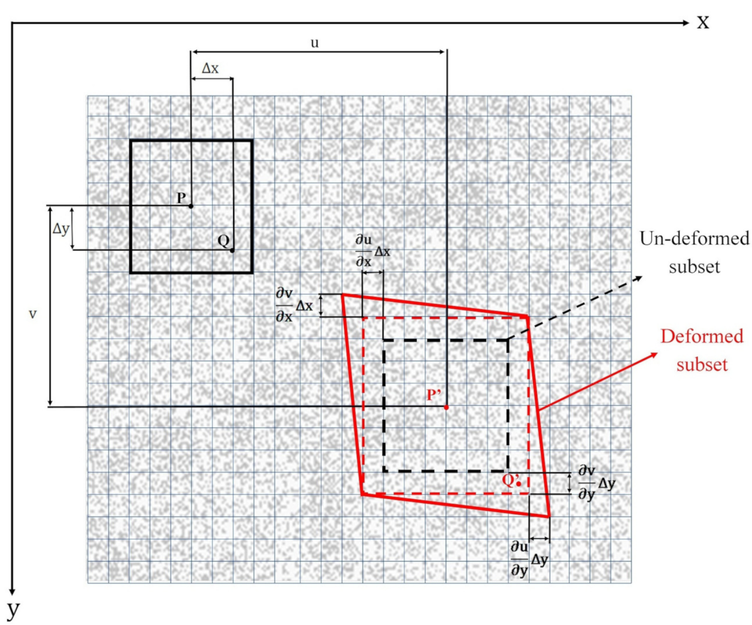


Fig. 2. The flow of the algorithm used to calculate the displacement

the subset size must not be too small for the algorithm to recognize. On the other hand, a larger subset size leads to higher levels of error in the approximation of the underlying deformation. Therefore, depending on the algorithm's capability for analyzing two images, one must select an optimal subset size to not only ensure the distinction of the subsets but also maximize the accuracy for the small deformations. A large subset size can reduce the noise in the results of the displacement and strain test [31, 32].

2.2. New patterning method

In this research, a new patterning method was developed and investigated. The method is based on spreading a mixture of black and white nanoparticles to make a speckle pattern, where the black nanoparticles serve as black speckles surrounded by colonies of white particles. For this purpose, mixtures were prepared with four different nanoparticles, namely graphene oxide (black) and titanium oxide (white), zirconium oxide (white) and silicon oxide (white). The pattern in the recent method will be simply created, modified, and removed by a small comb without any instrument and complex process. In the recent way of patterning, nanoparticles are spread on the specimen's surface, aggregate between surface reliefs and adhere with Van Der Waals forces. Due to weakness of Van Der Wall forces, reduction and movement of a nanoparticle on the specimen surface is comfortable. Thus, in comparison to other methods, there is no difficulty in this method in creation, modification and removal of the patterns. Nevertheless, despite the fact that the forces between the surface of the target specimen and nanoparticles are weak, the forces are strong enough to displace the nanoparticles due to displacement of the specimen surface.

2.3. Setup and apparatus

Once preparing the mixtures was finished, those were spread on several specimens. Next, each specimen was located on a displacement test apparatus. Fig. 3 shows the setup of the experiment. A digital microscope was used for magnification and imaging. In order to investigate and compare the patterns, different displacements in the range of 10 to 50 micrometers were applied to each specimen, and the resultant displacement was further calculated by DIC method to compare the result with actual displacement values

The new patterning method was also put on experiment through tensile testing to assess its capability and accuracy in measuring the strain. The results were compared to those of the conventional DIC, checking the accuracy of the new patterning method. For this purpose, a beam equipped with a strain gauge was used to acquire actual data and compare the results of the conventional DIC and the proposed patterning method to the actual data. Fig. 4a shows the beam. Patterns were applied on the other end of the beam; the patterns were generated using both

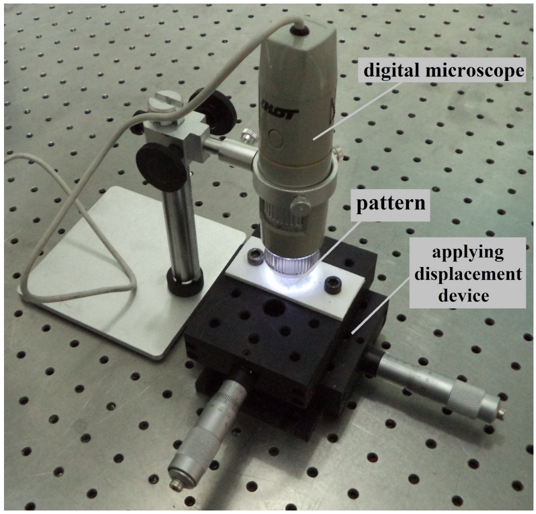
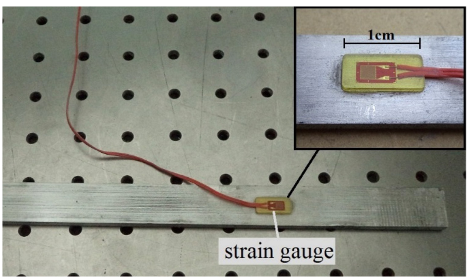
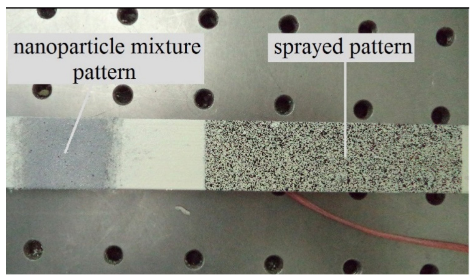


Fig. 3. Setup of displacement test



(a) The strain gauge on the beam



(b) Two patterns created on the other end of the beam

Fig. 4. Strain gauge adhered to the beam and speckle patterns created by nanoparticle spreading method and spraying method

dye spraying and coating with nanoparticle mixture. The mixture was created based on the results of the displacement test. Fig. 4b shows the two patterns created in this stage.

Fig. 5 shows the setup of the tensile testing, where the beam was extended by a tensile test machine. The digital microscope and the camera were placed on a vibration-isolated table, with the digital microscope focused on the nanoparticle mixture pattern and the camera capturing images from the sprayed pattern. Upon applying a strain to the beam, the camera and the digital microscope both captured images. Using this technique, an identical strain could be applied to the strain gauge and the two speckle patterns. Fig. 6 shows a close-up of the setup of the DIC with the digital microscope and the mixture on the beam as the speckle pattern.

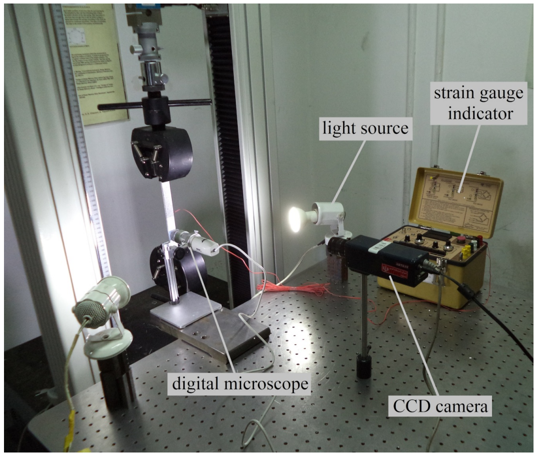


Fig. 5. Setup of tensile testing

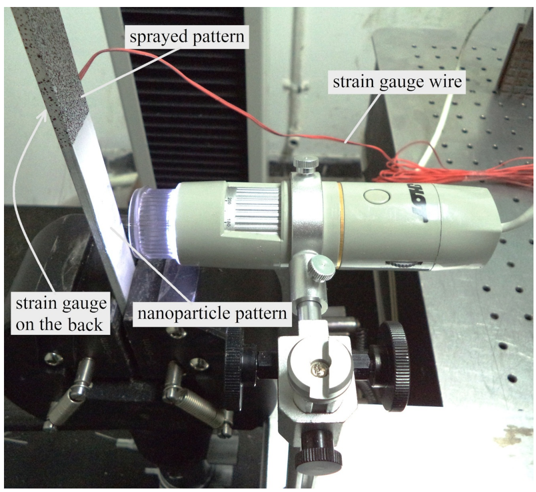


Fig. 6. DIC setup with new patterning method for tensile testing

3. Results and discussion

3.1. Investigation of different patterns through displacement test

Three mixtures of graphene with titanium, zirconium and silicon were investigated through displacement tests to determine which mixture could create better speckle pattern leading to higher accuracy. To compare the size and distribution of speckles, three samples of patterns are shown in Fig. 7. Based on these samples' size, the new method in creation of speckle pattern can be applied to study small fields of view within a range of one millimeter by one millimeter approximately.

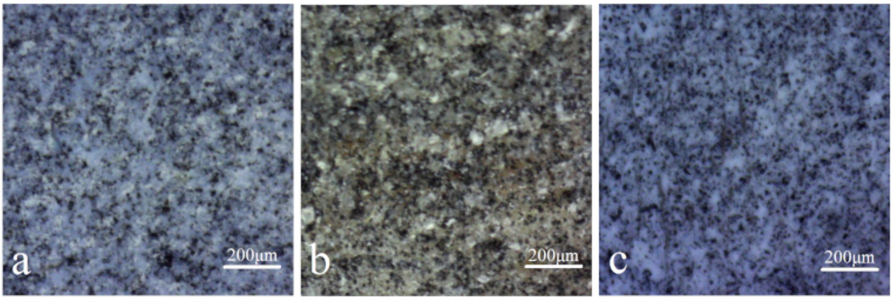


Fig. 7. Mixtures of (a) zirconium and graphene, (b) silicon and graphene, and (c) titanium and graphene

These patterns were investigated under displacement test. The displacement results for these three patterns are presented in Fig. 8, indicating the superiority of the mixture of titanium and graphene. Interestingly, this mixture was even easier to fabricate and spread.

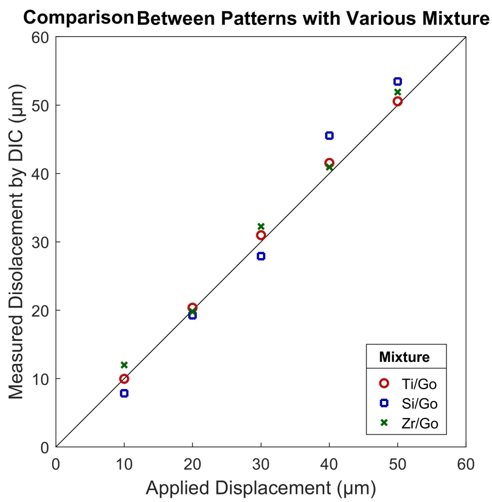


Fig. 8. Graph of experimental results vs. applied displacements

As the optimal mixture, the graphene-titanium mix was also investigated at three different weight ratios of graphene to titanium (Fig. 9). Fig. 10 shows the histogram of the patterns obtained with different ratios. Accordingly, the histogram of the mixture with graphene at 30% is wider than the two others, indicating a higher contrast in this pattern. The three patterns were further experimented through displacement tests, with the results shown in Fig. 11. The results demonstrated the superiority of the mixture containing graphene (30%) and titanium (70%). A mixture with a low percentage of black nanoparticles and a high percentage of white nanoparticles would create a pattern with black speckles in a white background.

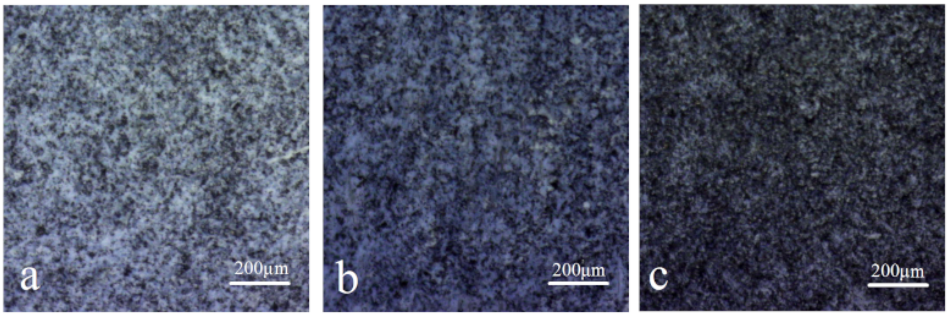
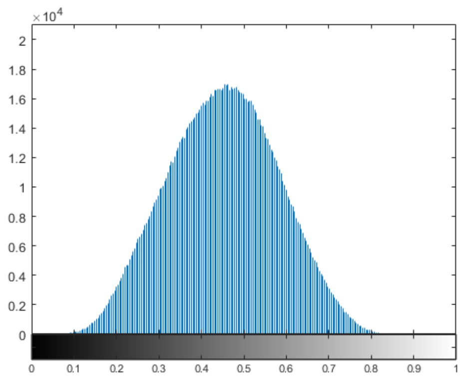
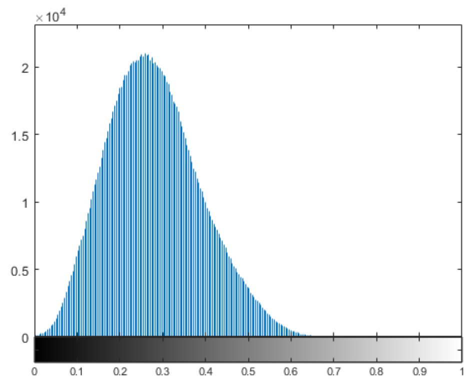


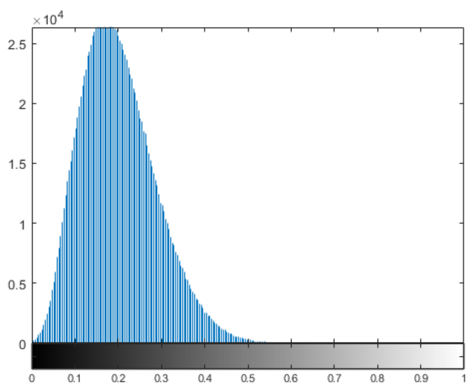
Fig. 9. Mixtures of (a) 30% graphene with 70% titanium, (b) 50% graphene with 50% titanium, and (c) 70% graphene with 30% titanium



(a) Mixture of 30% graphene and 70% titanium



(b) Mixture of 50% graphene and 50% titanium



(c) Mixture of 70% graphene and 30% titanium

Fig. 10. Histograms of patterns with various weight ratios

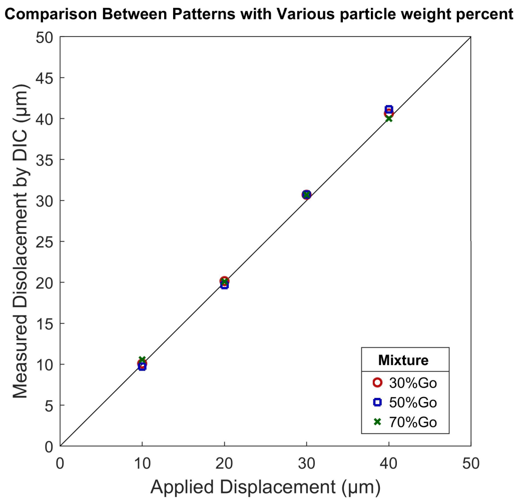


Fig. 11. Measured displacements (DIC) vs. applied displacements

Theoretically, in the displacement test, it is feasible to reach an accuracy of 0.01 pixel [33]. However, in a real test, there are some sources for error, including noise or image distortion, algorithm errors, and so on [33]. In this study, the error in the displacement test was approximately 0.7 pixels. Even though it is very high in comparison with the highest accuracies achieved, this weakness will be acceptable when we compare it with the real error. For example, in one of the studies the speckle size and error bounds were around 100 µm and 1 µm, respectively [34]. In the current study, the size of pixels is around one micrometer and the error, on average, is about 0.7 µm which is equal to 0.7 pixels, meaning that the error in this study is less than the conventional one. This error roots in devices that are used, and can be reduced by applying cutting-edge cameras and high-tech lenses. Nevertheless, the aim of this article is not to focus on errors and their reduction, but to introduce and investigate a new method of effortless creation of speckle pattern for a study on small sizes with a high gradient of displacements or a trifle displacement.

3.2. Investigation of new patterning method through tensile testing

The performance of the proposed patterning method for DIC was further investigated through tensile testing, and the results were compared to those of the conventional DIC. For this purpose, different levels of strain were applied to the specimen and the results were evaluated with different subset sizes. The subset size had no effect on the results of DIC in the displacement test, but it did affect the results in the strain test. The effect of the subset size at different levels of strain was investigated for both the conventional DIC and the proposed patterning technique. Fig. 12 and Fig. 13 show the measured strains for the conventional DIC and the

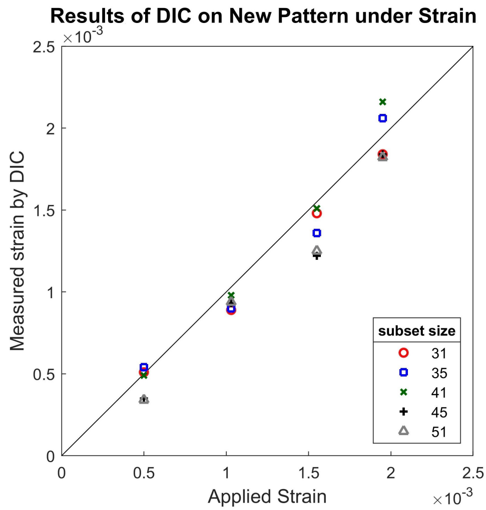


Fig. 12. Comparison of experimental results for different levels of applied strain with various subset sizes using the conventional DIC

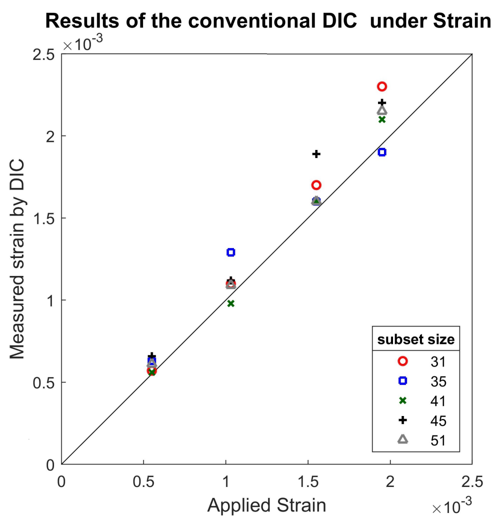


Fig. 13. Comparison of experimental results for different levels of applied strain with various subset sizes using the proposed patterning technique

proposed patterning techniques, respectively. The relative error was computed for different levels of strain (Table 1). According to the results, the highest accuracy was obtained with a subset size of 41 pixels. In comparison to conventional DIC, the proposed patterning technique provided superior accuracy. In addition, with

Table 1. Percent errors obtained at different levels of strain with various subset sizes

Subset size	Usual method	New method
31	4.2	3.5
35	7.5	4.3
41	3.5	2.8
45	7.1	6.3
51	4.3	5.6

the proposed patterning technique, the field of view could become much smaller, making the proposed technique more practical and advantageous for various fields of research.

In this study, a new method was proposed for patterning in DIC based on spreading a mixture of black and white nanoparticles on the specimen. Only graphene was used to provide black nanoparticles, while zirconium, titanium, and silicon were tested for providing the white nanoparticles. This study can be extended to check the efficiency of other particles for creating different patterns with various sizes of speckles for various fields of view in different domains of study. One may also consider solving the nanoparticles to facilitate the spreading process. In this research, the minimum accuracy of the displacement instrument was 10 micrometers, but it is anticipated that smaller displacements can also be traced by the proposed method because the pixel size was about 1 micrometer.

4. Conclusion

A new method was proposed for speckle pattern creation in DIC, where a mixture of black and white nanoparticles was spread over the specimen surface. Three mixtures were fabricated by mixing graphene (black) with either of titanium, zirconium and silicon (white). These three mixtures were investigated through displacement tests, with the results indicating the superiority of the mixture of graphene and titanium. Accordingly, the graphene and titanium were mixed at different weight ratios, and the mixture with 30% graphene 70% titanium outperformed the other mixtures. After that, the best pattern was further investigated through tensile testing, and the results were compared to those of conventional DIC method with different subset sizes. The results showed that the accuracy of the proposed method was by far acceptable. Upon the tensile tests, the most accurate results were obtained with a subset size of 41 pixels.

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