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# Full-field strain measurements at the micro-scale in fiber-reinforced composites using digital image correlation

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ABSTRACT

Few studies have been conducted to monitor inter-fiber deformations in fiber-reinforced composites. In the present work, we demonstrate full-field strain measurements in the composites at the micro-scale, using digital image correlation (DIC). The study is performed on a unidirectional glass fiber reinforced composite loaded in transverse three-point bending inside an environmental scanning electron microscope. A nano-scale random speckle pattern of high quality is created. Validity of the measured fields is assessed against results of a finite element (FE) model with boundary conditions retrieved from the experiment. A good agreement is found between the DIC-measured and FE-predicted results. The precise recognition of very small-scale strain concentrations requires enhancement of the correlation process and removal of microscopy imperfections. The investigated methodology shows promise for real-time deformation measurements in composites at the micro-scale.



## 1. Introduction

Digital image correlation (DIC) is an optical method to track changes in a series of deformed images. It is used to measure displacements and strains on the surface of a deforming material in a non-contact way. The correlation process uses the gray intensity pattern inside virtually created *subsets*. Hence, each subset should have a unique pattern, which, in the absence of an inherent surface texture, results from an artificial random speckle pattern on the surface of the material [1,2]. DIC has been widely applied to measure deformation in composites at the macro- and meso-scales [3–9]. To track deformations of individual fibers and inter-fiber deformation in the matrix, the length scale of this technique should be reduced to the micro-scale. The need for the micro-scale DIC (μDIC) is particularly important in the field of nano-engineered fiber-reinforced composites and composites with multiphase matrices where the effect of modifications is most pronounced at this scale [10–13].

A number of difficulties appear in the application of μDIC to different materials, and several studies have been conducted to progressively tackle them. Up to now, μDIC has been mostly applied to metals. It started with use of high-magnification optical microscopy in the late 90s to early 2000s, when baseline errors in displacement measurement, problems with the image contrast, refocus errors, and lens aberrations were the main difficulties [14,15]. Higher magnification DIC analyses were accomplished through engaging scanning electron microscopy (SEM) [16–18]. Challenges related to drift and spatial distortions, and SEM based noise were explored in [19–21]. Another challenge in μDIC is application of a suitable tracking pattern to the specimen surface [22]. μDIC was also applied to non-conductive materials in a few studies such as [23,24].

μDIC has been rarely applied to fiber-reinforced polymer-based composites. The promise of this technique for analysis of micro-scale deformations in these composites was first shown in [25], where Canal et al. analyzed the deformation in a unidirectional E-glass/epoxy composite under transverse compression. For a microscopic window at three different magnifications (250×, 2000×, and 6000×), they achieved fine displacement maps for the component in the loading direction. At high magnifications, the loading-direction strain maps showed lower accuracy in identifying the strain concentrations and exact location of fibers. The average composite strain in the microscopic window was derived with an error around 2.5%, but the quantitative values of strain average in each phase were not accurately obtained. The reason, pointed by the authors, was that the high deformation gradient at the fiber/matrix interface could not be captured. It was smoothed out because DIC calculates strains by taking the derivate of the displacements over a small strain window, not

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differentiating between the two phases on which it is placed. That is why it is challenging to apply μDIC to heterogeneous microstructures with a high property mismatch in the constituents.

The present study continues the line of research established in [25], applying μDIC to fiber-reinforced composites. The methodology, in line with the one proposed in [25], is exerted on an example of a unidirectional glass fiber/epoxy composite to further investigate the measurement and interpretation issues. The specimen is loaded in transverse three-point bending, and a microscopic study zone on the tensile deformation region is monitored with an environmental scanning electron microscope (ESEM). Efforts are made to produce a nano-scale random speckle pattern, with quality assessed by analysis of DIC *measurement errors*. Other difficulties related to fitting the mechanical test set-up inside the ESEM chamber, the imaging distortions and noise, charging effect, and maintaining the field of view fixed under loading are looked at. The validity of the μDIC results is evaluated against a finite element analysis (FEA). Limitations of μDIC on fiber-reinforced composites, particularly concerning high local strains, are then discussed. The preliminary studies were conducted in [26].

The authors' longer term goal is to further apply this technique to study microscopic phenomena such as damage initiation and development in composites, and to provide a tool which allows evaluation of the changes in the mechanical behavior brought by introducing nano-reinforcements in the fiber-reinforced composite [27].

## 2. μDIC methodology

The investigated μDIC approach includes such steps as preparing specimens, applying a speckle pattern, assessing the pattern, deforming the material, capturing micrographs, appraising microscopy adequacy, optimizing DIC parameters, applying DIC, filtering out random noise, and validating DIC results against FE predictions.

### 2.1. Composite material

The studied material is a unidirectional glass/epoxy composite produced by vacuum infusion, previously studied in [28]. The glass fiber is *Advantex® Glass SE 1500 2400*, with an average diameter of 17 μm, and the epoxy matrix is *EPIKOTE™Resin MGS® LR 135 LV*, with *EPIKURE™Curing Agent MGS® LH 137* as hardener. The Young's moduli of the glass fiber and epoxy matrix are 81 GPa and 3 GPa, and the Poisson's ratios are 0.22 and 0.30, respectively. The average composite thickness is 1.9 mm and the average fiber volume fraction is 61.9%. The composite plate is cut into specimens of $68.0 \times 8.0 \times 1.9\ mm^3$ dimension in such a way that fibers are aligned in the width direction. The surfaces perpendicular to the fibers direction are grinded by SiC abrasive papers (320, 800, 1200, and 4000 grit, consecutively) and polished by a diamond slurry of 1-μm particle size, followed by an oxide polishing suspension.

### 2.2. Speckle pattern enhancement and DIC parameters

#### 2.2.1. Speckle deposition

The electron microscopy image from the composite cross-section (Fig. 1a) reveals that the material does not possess a suitable texture in its natural state. The presence of large areas with similar gray intensity levels asks for an external random speckle pattern. The pattern is produced through deposition of a powder on the specimen surface. When choosing the appropriate powder, the following parameters were taken into account: the particle size, level of absorption and distribution on the surface, tendency to aggregate, and gray level contrast with composite surface. A sub-micron alumina powder (*TM-DAR* series of *TAIMICRON*) produced by *TAIMEI CHEMICALS Co., Ltd*. is selected for the study. The average size of the particles is 220 nm. To avoid particle aggregation, a dispersant for stabilizing alumina suspensions such as *DARVAN-CN* (a trademark of *R.T. Vanderbilt Holding Company, Inc.* for ammonium polymethacrylate) is used [29].

Several trials are done to achieve a suitable speckle pattern. First, a 1-wt.% suspension of alumina in distilled water is prepared, and a drop of it is applied to the polished surface of one of the specimens. Then, it is left in the ambient air to be dried. After gold sputtering, large aggregates can be observed all over the surface through electron microscopy (Fig. 1b). Aggregates could be formed before or during the deposition, causing a significant error in DIC by creating large areas with unvarying gray intensity level. Additionally, deformation of the aggregates can be different from the deformation of the underlying surface. In order to prevent particle aggregation ultrasonication along with magnetic stirring is applied to the suspension before deposition. A new polished specimen is speckled with this suspension. Fig. 1c shows that ultrasonication eliminates large aggregates. To remove small-scale aggregates and to further enhance the dispersion of nano-particles, *DARVAN-CN* is added to the suspension before ultrasonication. Fig. 1d, which is captured from the surface of a new specimen, speckled with the dispersant-including suspension, shows that the aggregation is drastically decreased.

The concentration of alumina is also optimized. In Fig. 1d, it is observed that 1 wt.% is quite high, which makes it difficult to observe the underlying surface. Thus, a 0.1-wt.% suspension is produced, taking advantage of both ultrasonication along with magnetic stirring and *DARVAN-CN*. The micrograph from the specimen speckled with the 0.1-wt.% suspension (Fig. 1e) displays that the distribution of particles is not uniform. To further enhance the distribution, the concentration is reduced to 0.05 wt.% (using ultrasonication, magnetic stirring, and *DARVAN-CN*), but the deposition process is performed twice. The second deposition is done once the first layer is dried. With this approach a well-dispersed and evenly distributed speckle pattern is achieved (Fig. 1f). The amount of *DARVAN-CN* in each suspension is 1/10 of the alumina weight. The surface of all speckled specimens in this study is coated with a thin conductive film in order to diminish the charging effect.

#### 2.2.2. Assessment of the pattern quality

To examine the quality of the optimized speckle pattern (Fig. 1f) we used a so-called *strain deviation analysis*, where DIC is applied to virtually deformed images. For this, a micrograph with the resolution of $1424 \times 968$ pixel$^2$ ($125 \times 85\ \mu m^2$) is taken from the speckled surface as the study zone (Fig. 2b). The length of the micrograph is extended by 15 pixels, using the *lanczos3* resizing method in *MATLAB*. This virtual deformation is equivalent to a value of 0.0106 for the horizontal component of the Lagrangian strain. The virtual deformation is analyzed through DIC to assess the deviations from the applied strain. The same strain deviation analysis is also applied to a poorly-speckled specimen (Fig. 1e). For DIC, *VIC-2D 2009* software (*Correlated Solutions*) is used. The sizes of *subset*, *step*, and *smoothing filter* (described in 2.2.3 and [30]) for this strain deviation analysis, are set to 21 pixels, 1 pixel, and 5 data points, respectively. All of the DIC analyses in this study are performed with a *normalized squared differences* criterion, *optimized 4-tap* interpolation, and *Gaussian* subset weights. Lagrangian strains are computed from the resulting displacements and smoothed with a 90% center-weighted decay filter [30]. All strain values given in Sections 2.2.2, 2.2.3, and 2.3 refer to the horizontal component.

It is expected to obtain homogeneous strain maps if the deformation is applied uniformly. In reality, digital resizing of an image is not totally uniform all over the image, and local deformation