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Fabrication technique of micro/nano-scale speckle patterns with focused ion beam

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The fabrication technique of micro/nano-scale speckle patterns with focused ion beam (FIB) system is studied for digital image correlation (DIC) measurement under a scanning electron microscope (SEM). The speckle patterns are fabricated by directly etching the counterpart of the specimen to the black part of a template. Mean intensity gradient is used to evaluate the quality of these SEM images of speckle patterns fabricated based on different templates to select an optimum template. The pattern size depending on the displacement measurement sensitivity is adjusted by altering the magnification of FIB according to the relation curve of the etching size versus magnification. The influencing factors including etching time and ion beam current are discussed. Rigid body translation tests and rotation tests are carried out under SEM to verify the reliability of the fabricated speckle patterns. The calculated values are in good agreement with the imposed ones.

speckle pattern, digital image correlation (DIC), micro/nano-scale, focused ion beam (FIB), scanning electron microscope (SEM)

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The increasing miniaturization of devices, especially the rapid development of Micro-Electro-Mechanical Systems (MEMS), has resulted in the demand for methods that can quantify minute loads or shapes and deformations at micro/nano-scale [1–7]. Estimation of fundamental mechanical properties requires that displacements and strains be measured at the micron and sub-micron length scale. One method applicable to micro/nano-scale measurements is digital image correlation (DIC), which is developed by a group of researchers in the 1980s [8–11]. It directly provides full-field displacements to sub-pixel accuracy and full-field strains by comparing the digital images of test object surface acquired before and after deformation. Due to its simplicity and effectiveness, DIC has been used widely in the field of experimental mechanics [12]. Combined with high resolution microscopes like scanning electron microscope (SEM), DIC can realize micro/nano-scale measurement [13]. However, SEM-DIC requires the presence of a randomly-patterned material surface and the random pattern must have a recognizable and resolvable structure on an appropriate scale for the problem of interest.

Although the fabrication of random patterns encounters a great challenge due to the tiny size of material at reduced length scale, some different patterning techniques have been developed for each length scale. Scrivens et al. [14] developed chemical vapor exposure and UV lithography to fab-

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ricate random patterns for nano-scale and micro-scale measurement respectively. Berfield et al. [15] created random speckle patterns with a fine point airbrush for micro-scale measurement and fluorescent nanoparticles for nano-scale measurement. Tanaka et al. [16] utilized electron beam lithography to make random patterns for micro/nano-scale measurement by backscattered electron imaging (BSEI) under field emission SEM. Liu et al. [17] made use of focused ion beam (FIB) milling for fabrication of periodic micro-markers on a MEMS device. Sebastiani and Korsunsky et al. [18–20] applied FIB milling to fabricate periodic holes as speckle patterns for nano-scale measurement. Sabate et al. [1,21–23] acquired speckle patterns by deposition of 20 nm pt layer with FIB for nano-scale measurement. With the use of FIB, Kregting et al. [24] first defined a rectangular grid with 250 nm spacing and then randomly positioned the markers within a square of 150 nm size around each grid point. Kammers & Daly [25] provided a survey of small- scale patterning methods for SEM-DIC and discussed their advantages and disadvantages for different applications.

Each patterning technique has its own advantages and limitations. For the micro/nano-scale measurement of micro-devices like MEMS, FIB is an ideal choice for its direct milling or depositing capability. It should be noted that due to the disadvantage of time consumption, large area pattern fabrication is impossible for FIB. However, this disadvantage is negligible for micro-device and micro-structure. Although FIB milling or deposition has been used to fabricate speckle patterns at reduced length scale, this fabrication technique itself has not been studied thoroughly. How to design an optimized template and an appropriate pattern size is not clear yet.

This paper aims at providing guidance on the fabrication of speckle patterns with FIB milling. The principle of speckle pattern fabrication with FIB milling is introduced in sect. 1. The design concept of the speckle pattern, including the design of the template and the size of the pattern region, is described in sect. 2. The influencing factors of FIB patterning technique, the etching time and the ion beam current, are analyzed in sect. 3. Verification experiments including rigid body translation tests and rotation tests are performed in sect. 4. Discussions are presented in sect. 5 followed by the conclusions in sect. 6.

1 The principle of fabricating speckle pattern with FIB milling

FIB micromachining technologies are now widely used as powerful tools in the semiconductor industry mainly for micro/nano-structuring, mask repairing, device modification, failure analysis and integrated circuit debugging [26,27]. Two broad categories of FIB micromachining are milling and deposition. This paper only involves FIB milling. FIB cuts away (mills) material from the specimen surface with dimensions typically in microns by accelerating concentrated gallium ions to a specific site, leaving a clean hole. Microstructure of various forms can be fabricated with FIB system according to different input pattern images, which is the basis to fabricate random speckle patterns with FIB milling. Using FIB milling to fabricate speckle patterns not only extends the application of FIB, but also offers a new patterning technique for DIC method.

In our experiments, FEI DB 235 dual beam system which combines FIB and SEM is used. SEM can realize precise location and real time observation of the fabrication process. The resolution of the electron beam is 2 nm and the resolution of the ion beam is 7 nm. The ion beam current ranges from 1 pA to 20 nA.

The fabrication process begins with generating a template (a binary bmp image, as seen in Figure 1) by software. Convert the template with Patterns 1.04 (software package) and input it into the pattern generator of the FIB system. Then put the specimen on the sample stage and vacuumize the FIB system. After vacuum-pumping is completed, tune focus to make the specimen seen clear and choose an area of interest. Start etching according to the template once the parameters including magnification, ion beam current and etching time are selected. Finally, speckle patterns are formed on the specimen surface. The flow chart is shown in Figure 2.

2 Design of the speckle pattern

The design of the speckle pattern involves two aspects: the morphology and the size. The former directly influences the quality of the speckle pattern and the latter determines the displacement sensitivity. At the same time, the morphology depends on the template while the size depends on the mag-

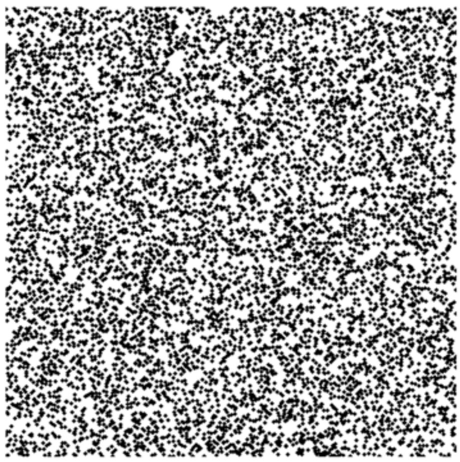

Figure 1 Schematic of a template. The size is 512 pixels×512 pixels, the number of the speckles is 9000 and the radius of a single speckle is 2 pixels. During the fabrication process, only the counterpart of the specimen to the black part is etched.

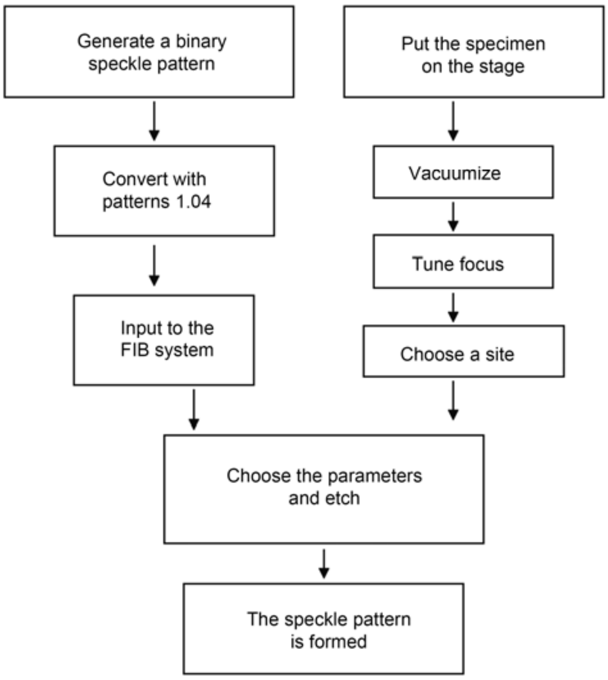

Figure 2   Flow chart of FIB fabrication process.

nification of FIB. Therefore, the design of the speckle pattern involves choosing an optimum template and a proper magnification of FIB to meet the measurement requirements.

2.1 The template

There are two parameters that influence the morphology of the template: the radius and the number of the speckle. As reported in ref. [28], the optimized radius of a single speckle is 2–3 pixels, thus only 2 pixels and 3 pixels are considered. In order to select a good template, templates with speckle amounts of 4000, 5000, 6000, 7000, 8000, 9000 and 10000 are generated respectively for radiuses of 2 pixels and 3 pixels. Corresponding speckle patterns are fabricated on silicon wafer. The etching time is 5 min, the ion beam current is 84 pA and the magnification is 5000×. Two typical SEM images of fabricated speckle patterns are shown in Figure 3, which correspond to the templates with 9000 speckles in radiuses of 2 pixels and 3 pixels.

As the quality of the speckle pattern directly influences the accuracy of the subsequent correlation calculation, it is necessary to choose a best template to get a high quality speckle pattern. Some methods or parameters have been developed to assess the quality of a speckle pattern, such as mean intensity gradient [29], mean subset fluctuation [30], image morphology method [31], subset entropy [32] and sum of squares of subset intensity gradient [33]. In the work, mean intensity gradient [29] is adopted as the evaluation criterion since it is an effective global parameter.

The expression of mean intensity gradient is

$$\delta_f = \sum_{i=1}^{W}\sum_{j=1}^{H}\left|\nabla f(\boldsymbol{x}_{ij})\right| / (W \times H), \tag{1}$$

where W and H (in unit of pixels) are image width and height respectively, $\nabla f(\boldsymbol{x}_{ij}) = \sqrt{f_x(\boldsymbol{x}_{ij})^2 + f_y(\boldsymbol{x}_{ij})^2}$ is the modulus of local intensity gradient vector with $f_x(\boldsymbol{x}_{ij})$, $f_y(\boldsymbol{x}_{ij})$ being the x- and y-directional intensity derivatives at pixel $(\boldsymbol{x}_{ij})$, which can be simply computed using a commonly used gradient operator.

The larger the mean intensity gradient is, the higher the quality of the fabricated speckle pattern is. The calculated results are shown in Figure 4. It can be found that the qualities of the speckle patterns fabricated with the templates composed of speckles of 2 pixels in radius are better than those fabricated with the templates composed of speckles of 3 pixels in radius. Mean intensity gradient keeps growing with the increase of the number of speckles within 9000. It implies that the quality of the speckle pattern is enhanced with the increase of the number of speckles until the number reaches 9000. Therefore the template with 9000 speckles of 2 pixels in radius is optimum for silicon substrate. For other materials, an optimum template can be determined in the same manner.

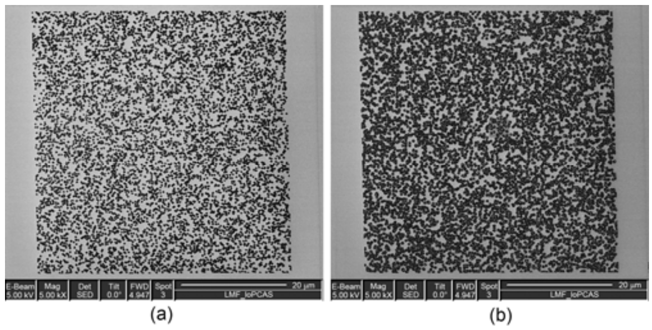

Figure 3   Fabricated speckle patterns based on two different templates. (a) Radius: 2 pixels, number: 9000; (b) radius: 3 pixels, number: 9000.

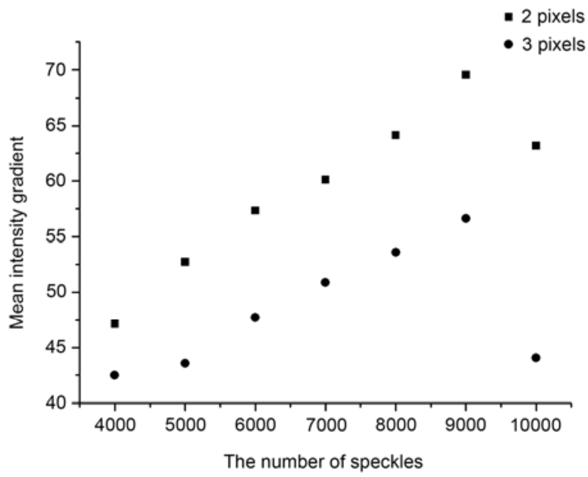

Figure 4   Mean intensity gradient of the fabricated speckle patterns.

2.2 The magnification of FIB

It is well recognized that the displacement sensitivity of DIC is 0.01 pixel. As the pixel size of the recorded SEM image is usually constant, the displacement sensitivity is directly related to the physical size of the image, which is approximate to the pattern size. Therefore the pattern size is designed according to the requirement of measurement sensitivity. On the other hand, the pattern size is adjusted by altering the magnification of FIB during the fabrication process. Once the relationship between the pattern size and the magnification is known, it is convenient to obtain a desired size by altering the magnification.

Using the same template, the corresponding etched area is inversely proportional to the magnification. To calibrate the relationship between the etched area and the magnification, the speckle patterns are fabricated on amorphous silicon carbide under the magnification of 5000×, 10000×, 12000×, 15000× and 20000× respectively with the same template. The etching time is 5 minutes and the ion beam current is 164 pA. The fabricated speckle patterns at two different magnifications are shown in Figure 5. For simplicity, the template is generated as a square so that the relationship between the etched area and the magnification is converted into the relationship between the length of the square and the magnification. It can be seen that the morphologies of the speckle patterns are identical and the size varies with the magnification of FIB. By measuring the lengths of the squares at different magnifications, the relation curve of the length versus the magnification is obtained, as shown in Figure 6. According to the data points obtained, the best fitted relation curve is derived. Therefore, we can determine the magnification of FIB according to the designed pattern size.

3 The influencing factors of FIB patterning technique

There are two major parameters that influence the quality of the speckle pattern, the etching time and the ion beam current.

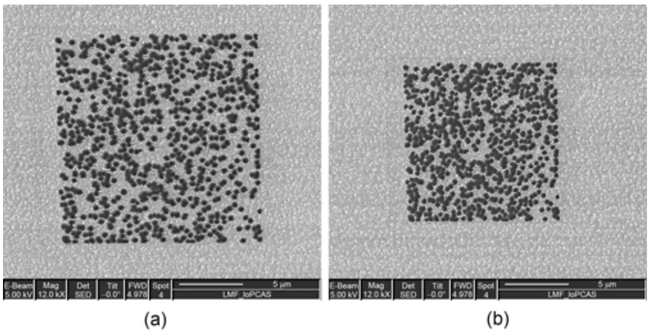

Figure 5 SEM images of speckle patterns fabricated at two different magnifications of FIB. (a) 15000×; (b) 20000×.

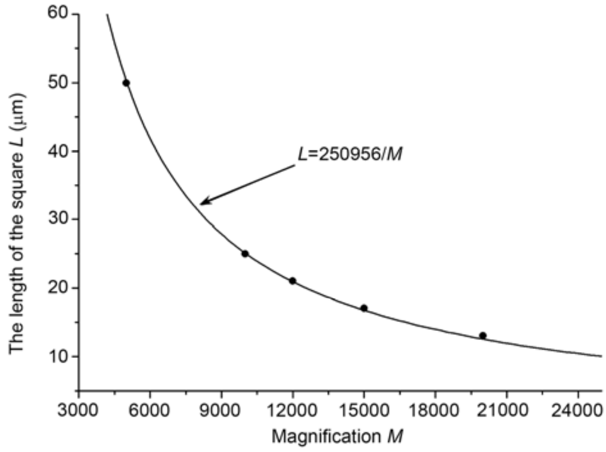

Figure 6 Relation curve of the length of the square versus magnification.

3.1 The etching time

In order to investigate the influence of the etching time to the fabricated pattern, five groups of speckle patterns are produced on silicon substrate with different etching times, as shown in Figure 7. It can be found that the contrast of the speckle image increases with the increase of etching time. This is because a longer etching time will result in a larger depth of the speckle and a higher contrast of the SEM image. However, a longer etching time will lead to a higher cost of FIB and a larger etching depth will influence the mechanical property of material. Therefore, a balanced time should be obtained by repeated experiments to ensure enough contrast and the mechanical property simultaneously. For silicon substrate, 5 minutes is optimum to fabricate 50 μm×50 μm speckle patterns.

3.2 The ion beam current

To investigate the effect of the ion beam current, the speckle patterns are fabricated on silicon wafer with different ion beam currents, including 28, 55, 222, 589 and 983 pA. The speckle patterns fabricated with three different ion beam currents are presented in Figure 8. Mean intensity gradient is still used to evaluate the quality of speckle patterns and the results are plotted in Figure 9. It suggests that 589 pA is optimum. If the ion beam current is lower, the contrast of the speckle pattern will be weak; else if it is higher, the noise level will be higher too.

4 Verification experiments

To verify the feasibility of the fabricated speckle pattern for deformation measurement, rigid body translation tests and rotation tests are carried out under FEI Siron 400 SEM utilizing the speckle pattern on a silicon wafer fabricated with the template consisting of 9000 speckles in radius of 2 pix-

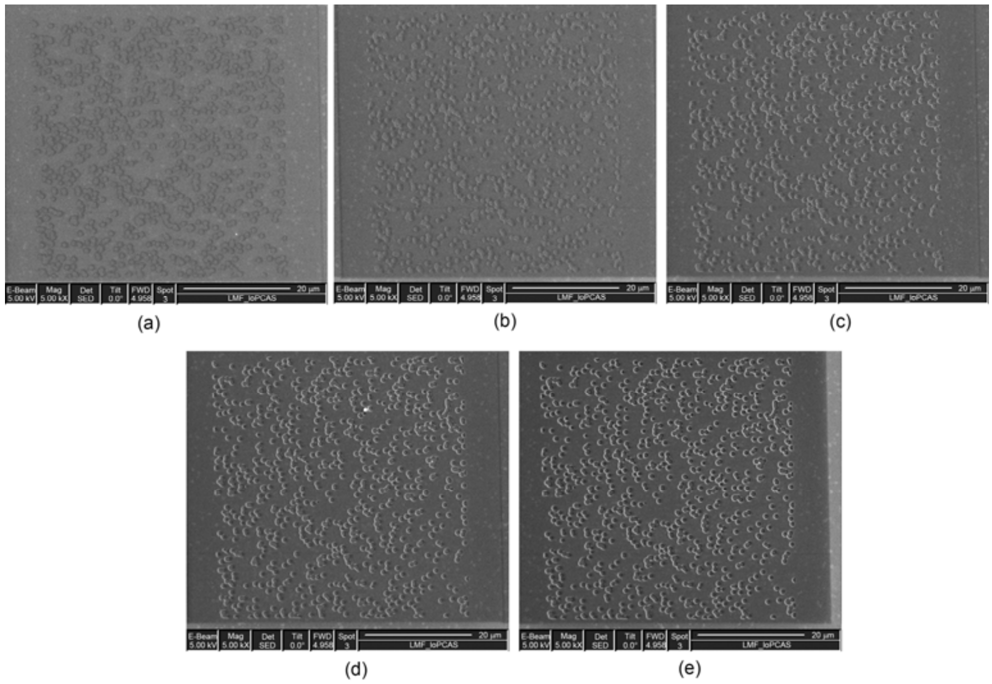

Figure 7 Speckle patterns with different etching times. The ion beam current is 986 pA and the magnification of FIB is 5000×. (a) 60 s; (b) 120 s; (c) 180 s; (d) 240 s; (e) 300 s.

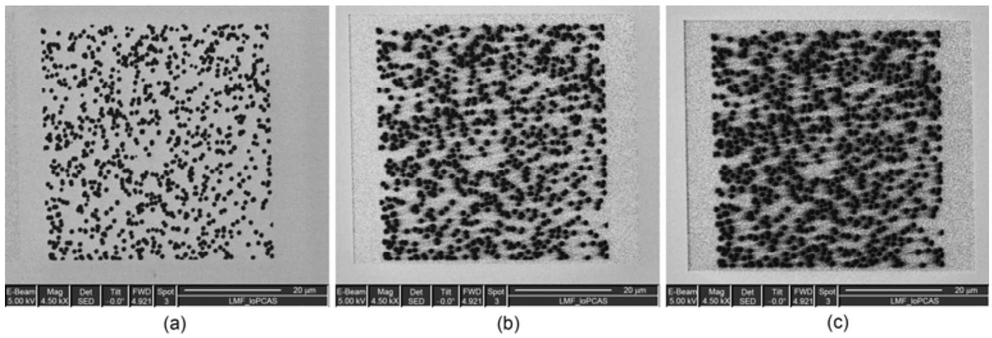

Figure 8 SEM images of speckle patterns fabricated with three different ion beam currents. The magnification is 5000× and the etching time is 5 minutes. (a) 28 pA; (b) 589 pA; (c) 983 pA.

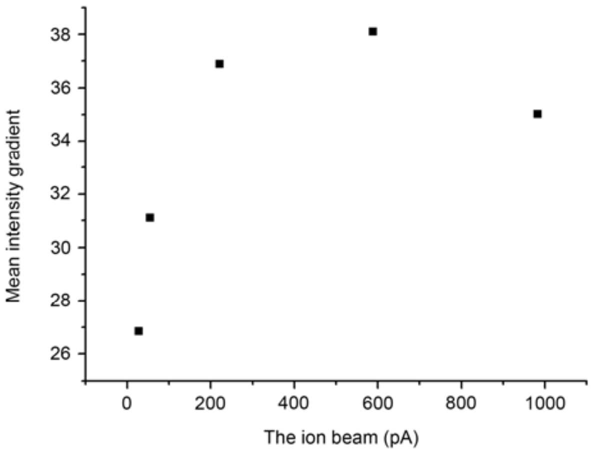

Figure 9 Mean intensity gradient corresponding to Figure 8.

els. All the displacement calculations are completed by self-developed DIC software. The subset size is 41 pixels×41 pixels and Newton-Rapshon method is used to obtain the sub-pixel displacement.

4.1 Translation tests

A piezoelectric transducer (PZT) is used to translate the specimen. This PZT is calibrated with a grating of 2000 lines/mm. By increasing the voltage from 0 to 80 V with a step of 10 V, the displacement is imposed on the specimen. The speckle patterns are recorded before and after the translation. The displacements are calculated with DIC method and the results are listed in Table 1. The calculated average displacements agree well with the imposed displacements with the maximum relative error within 5%. To

Table 1 Comparison between the calculated displacement and the imposed one

Imposed displacement (μm)	Calculated displacement (μm)	Standard error (μm)	Absolute relative error (%)
0.909	0.929	0.0027	2.2
2.000	1.973	0.0017	1.4
3.212	3.116	0.0020	3.0
4.525	4.359	0.0028	3.7
5.859	5.598	0.0026	4.4
7.212	6.851	0.0041	5.0
8.525	8.098	0.0038	5.0
9.434	9.319	0.0032	1.2

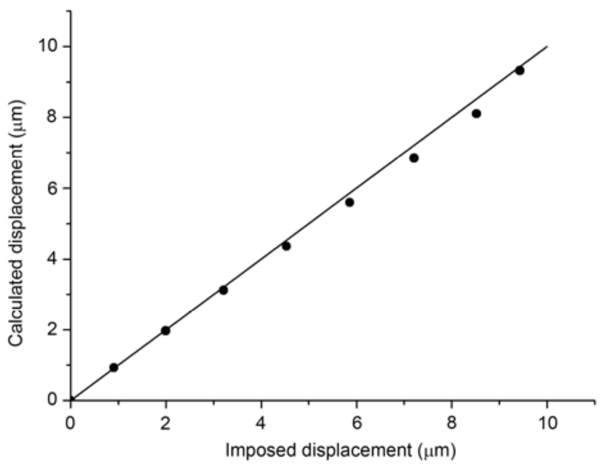

Figure 10 Calculated average displacement versus imposed displacement. The solid line is a reference line where the calculated displacement is equal to the imposed displacement and the points are the experimental data.

view the results more clearly, the results are illustrated in Figure 10. As seen from Table 1, the standard errors are relatively small compared to the average value, which implies the calculated displacement fields are very close to the average values. That's also why standard errors are not shown in the graph.

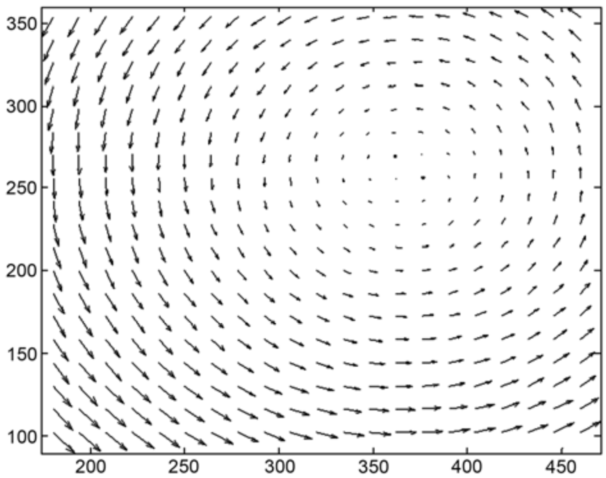

Figure 11 Displacement vector when scanning lines are rotated 3°.

Table 2 Comparison between the calculated rotation angle and the real one

Real angle (°)	Calculated angle (°)	Standard error (°)	Absolute relative error (%)
1	0.998	0.007	0.2
2	2.003	0.015	0.15
3	2.983	0.020	0.57

4.2 Rotation tests

Rotation tests are performed by rotating the scanning lines of the SEM. The speckle patterns are recorded before rotation and after rotation of 1°, 2° and 3°. The displacements are calculated with DIC method and the displacement vector when the scanning lines are rotated 3° is shown in Figure 11. The rotation angle is given by

$$\theta = \frac{1}{2}\left(\frac{\partial u}{\partial y} - \frac{\partial v}{\partial x}\right). \tag{2}$$

The results are listed in Table 2. The standard errors and relative errors are very small, which confirms the reliability of the fabricated speckle pattern.

5 Discussions

A benefit of FIB patterning is its high repeatability and the ability to generate a desired pattern. Based on the same template, the fabricated speckle patterns on the same substrate are almost identical provided that the processing parameters are constant. Thus, this technique possesses a high controllability. For the same material, only one group of trial experiments is needed to determine the optimized template and processing parameters. This will save a lot of time.

Currently, dual beam systems combining FIB and SEM are available, which have both the high-resolution imaging capability of SEM and the micromachining capability of FIB. SEM can realize precise location and real time observation of the fabrication process. Therefore FIB patterning can realize the accurate control of pattern location.

In this study, speckle patterns are successfully fabricated on the surface of silicon wafer and amorphous silicon carbide. But FIB patterning technique is not confined to these two types of materials. The characteristics of FIB also allow the micro/nano-scale speckle patterns to be produced directly on metal, glass, carbon and diamond substrates without any pattern change.

FIB offers a new tool to fabricate speckle patterns with speckle diameters down to 100 nm. However, it is a time-consuming technique and is not an efficient way to pattern a large area. That's exactly why we carefully design the size of the pattern region.

Getting high quality speckle patterns is an effective way to improve the accuracy of DIC method, which is also the ultimate goal of various patterning techniques. Although the calculated values are in good agreement with the imposed ones in verification experiments, some errors cannot be avoided as a result of the system error of the DIC method itself and the scanning distortion of SEM.

6 Conclusions

In this paper, the technique to fabricate random speckle patterns by direct milling of FIB is studied for use of SEM-DIC. It is essential to design a proper speckle pattern for micro/nano-scale deformation measurement. The design includes the selecting of a best template and a proper magnification of FIB. The template can be selected in terms of mean intensity gradient. From our experiments, the binary speckle pattern with 9000 speckles in radius of 2 pixels can serve as a good template for silicon wafer. For other materials, a best template can be determined in the same way. Since the area of interest is closely related to the displacement sensitivity, a proper size is first designed according to the measurement requirement and a proper magnification of FIB is chosen based on the calibrated relation curve between the size and the magnification. The influencing factors including etching time and ion beam current are discussed. Verification tests including rigid body translation tests and rotation tests are conducted under SEM to check out the reliability of the speckle pattern fabricated by FIB. The results demonstrate that FIB patterning technique coupled with SEM-DIC is well suited to measure the micro/nano-scale deformation of macro-specimen and micro/nano-scale device like MEMS.

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