Measurement of Strain and Stress Distributions in Structural Materials by Electron Moiré Method*

Satoshi KISHIMOTO**, Yougming XING***, Yoshihisa TANAKA**
and Yutaka KAGAWA**

**Composite Materials Group, Composite and Coating Materials Center,
National Institute for Materials Science

***Department of Applied Mechanics, Inner Mongolia University of Technology

Abstract

A method for measuring the strain and stress distributions in structural materials has been introduced. Fine model grids were fabricated by electron beam lithography, and an electron beam scan by a scanning electron microscope (SEM) was used as the master grid. Exposure of the electron beam scan onto the model grid in an SEM produced the electron beam moiré fringes of bright and dark parts caused by the different amounts of the secondary electrons per a primary electron. For demonstration, the micro-creep deformation of pure copper was observed. The creep strain distribution and the grain boundary sliding were analyzed. The residual strain and stress at the interface between a fiber and a matrix of a fiber reinforced plastic (FRP) were measured using the pushing-out test and this electron moiré method. Also, a non-uniform deformation around the boundary of 3-point bended laminated steel was observed and the strain distribution analyzed.

Key words: Electron Moiré Method, Creep Deformation, Residual Stress, Composite Materials, Strain Distribution

1. Introduction

It is very important to measure the local strain and stress distributions for understanding the mechanical properties of structural materials. Therefore, there are many techniques to measure the strain or stress distribution such as the strain gage method, optical elasticity method, optical moiré method, etc. In these methods, the optical moiré method [1] is one of the convenient methods to measure the deformation of the materials [1-3]. However, these methods are difficult to apply for deformation measurements from a microscopic aspect.

To measure the micro-deformation in a very small area, the authors have developed an electron moiré method [4, 5], and J. W. Dally and D. T. Read [6, 7] advocated it. This method keeps the main advantages of the moiré and laser moiré interferometry methods [8, 9], and has the additional ability of measuring deformation in a micro-area with a high sensitivity. For examples, non-uniform creep deformation and grain boundary sliding using parallel grid [4, 5], thermal deformation of solder joints [10], thermal residual stress in metal matrix composite [11] and the surface structure of butterfly wings [12] were measured by authors.

In order to pursue the more applications of the electron moiré method, some typical and different experiments were performed. The micro creep deformations of a pure copper specimen using cross model grids, residual stress at the interface between the fiber and the matrix in the fiber reinforced plastic and non-uniform deformations around the interface of the laminated steel were measured.

2. Principle of Electron Moiré Method

2.1 Observation of Electron Moiré Fringe

The principle of the electron moiré method is shown in Fig.1. A model grid is prepared on the surface of the specimen before deformation. An electron beam scan having spaces almost the same as that of the model grid can be used for the master-grid. The difference in the amount of the secondary electrons per a primary electron makes the moiré fringes (electron moiré fringes) that consists bright and dark parts.

Fig. 1 Principle of formation of electron moiré method

2.2 Strain Measurement

The schematic formation of moiré fringe is shown in Fig. 2. Using the moiré patterns corresponding to the u -field (x -direction) or v field (y -direction), the components of strain ϵ_x, ϵ_y in the x - y plane can be calculated by Eqs. (1).

In case of $a_x, a_y > a$,

$$\begin{aligned}\epsilon_x &= \frac{a}{d_x - a} - \frac{a'_x - a}{a} \\ \epsilon_y &= \frac{a}{d_y - a} - \frac{a'_y - a}{a}\end{aligned}\quad (1)$$

In case of $a_x, a_y < a$,

$$\begin{aligned}\epsilon_x &= -\frac{a}{d_x + a} - \frac{a'_x - a}{a} \\ \epsilon_y &= -\frac{a}{d_y + a} - \frac{a'_y - a}{a}\end{aligned}$$

where a demotes the pitch of the master grating (i.e. the spacing of electron beam scan), d_x demotes the spacing of the moiré fringe in the x -direction, d_y denotes the spacing of the moiré fringe in the y -direction. And a'_x and a'_y demote the pitch of the model grid in x -direction and y -direction before exerting the load, respectively. Usually, a'_x and a'_y are same value.

Fig. 2 Schematic formation of moiré fringe

3. Experimental Method

3.1 Grid Preparation

The procedure for producing a model is shown in Fig.3. The specimen must first be polished to a mirror-like finish (up to $0.05\mu\text{m}$ using Al_2O_3 powder), then it is covered with an electron-sensitive layer (electron beam resist, Nippon Zeon ZEP-520-22 and Toray EBR-9), spinning at a speed of 2,500 RPM for 60 seconds, and then baked in an oven for 30 minutes at 180°C for ZEP-520-22 and spinning at a speed of 2,000 RPM for 60 seconds, and then baked in an oven for 30 minutes at 195°C for EBR-9.

Fig.3. Schematic for fabrication of the model grid.

Fig.4. Parallel model grid fabricated by electron beam lithography (3 μ m pitch).

Fig.5. Electron moiré fringe fabricated by electron beam scan onto the model grid of Fig.4.

The specimen was then mounted on the specimen stage in a TOPCON SX-40A SEM for the electron beam exposure. After electron beam exposure, the specimen coated by ZEP-520-22 was developed in a solution of ZED-N50 for 60 seconds, and then immediately rinsed in ZMD-B for 30 seconds. The specimen coated by EBR-9 was developed in a solution of Type 1 for EBR-9 for 60 seconds, and then immediately rinsed in 2-propanol for 30 seconds. The specimen was coated with a very thin layer (10-20 nm) of gold by sputtering method.

The difference in the emitted amount of the secondary electrons per a primary electron between the surface and the deposited layer must be large enough to get produce a contrast in the electron moiré fringe. After removing the resist using an organic solvent, a model grid is formed on the specimen surface. Figure 4 shows an example (SEM image) of the model grid with pitches of 3 μ m fabricated by this method. Figure 5 shows a typical electron moiré fringe pattern using the model grid in Fig.4.

3.2 Creep Test

Creep specimens were machined from a pure copper bar and then annealed. On the polished surface, some model grids (4.7 μ m spacing) were prepared by the method mentioned in the Chapter 3.1 and depositing a gold-palladium alloy. After interrupt creep tests at 723K and 20MPa in argon, the electron moiré fringe and SEM images of the same area on the creep specimen's surfaces were observed.

3.3 Method for Releasing Residual Stress and Calculation of Residual Stress of Fiber Reinforced Plastics

The specimen was made of commercial boron/epoxy Textron prepreg Tape-5521 supplied by Textron Specialty Materials Inc. (Lowell, MA). It combines boron fibers with a tough and rigid epoxy resin. The diameter of the boron fiber was about 100 μ m. The boron/epoxy was primarily unidirectional with very few 90° fibers to supply lateral support. Twenty plies of the material were laid up in the manner of [0/90/0/90/0]. They were cured in an autoclave at 121°C under 0.45MPa. The physical properties of the fiber and the matrix are listed in Table1. A sample was cut from the bulk laminates perpendicular to one fiber axis using a diamond saw, and then polished into a film about 300 μ m thick.

Table 1. Physical properties of boron fiber and epoxy resin

	Epoxy resin	Boron fiber
Young's modulus (GPa)	2.41	400
Poisson's ratio	0.38	0.18
Coefficient of thermal expansion (K^{-1})	55×10^{-6}	4.5×10^{-6}

Since the epoxy matrix cannot withstand high temperature, the baking temperature was reduced to 90°C and the baking duration was increased to 16h. The specimen was coated with the gold layers both before and after fabrication of the grid. The spacing of the grid was 0.8 μ m.

To release the residual stress, the push-out test is used. A schematic illustration of this

test is shown in Fig.6. When the compressive load is applied to the top surface of the fiber, shear stresses are introduced at the interface with a maximum value occurring in the region near the top surface.

When the stress does not exceed the yield stress of the materials, the residual stress can be directly calculated by Hook's Law (Eq. (2)).

$$\sigma_{ij} = \frac{E}{(1+\nu)(1-2\nu)} [(1-\nu)\varepsilon_{ij} + \nu\varepsilon_{ji}] \quad (2)$$

where σ_{ij} and σ_{ji} denote the longitudinal and transverse stresses, respectively. E denotes Young's modulus and ν denotes Poisson's ratio. These values of this epoxy polymer are 2.41GPa and 0.38, respectively.

The equipment for pushing out was assembled in a scanning electron microscope (Topcon CL-130). The composite specimen was mounted on a 0.3 mm diameter hole, which was machined into a support block, allowing the fiber to be pushed out without resistance from the block. The specimen support block was put on a load cell holder and the holder was secured to a three axis translation table which allowed positioning of the fiber in the composite beneath the indenter. A hard steel spherical indenter with a tip radius of 100 μm was used. The push-out test was performed in vacuum condition of the SEM (Topcon CL-130) and a load was applied by moving the specimen stage upward.

Fig. 6. Schematic illustration for the push-out test.

3.4 Compressive Test

Low alloy steel sheets, WT780C, and 304 stainless steel sheets were laminated and rolled at 1473K. Thickness of each layer is about 1mm. This laminated steel was cut and machined to the specimens with 10mm width, 10mm length and 12mm high and then laminated sides were polished. On the polished surface, some model grids with a cross grid of 4.5 μm spacing were prepared by electron beam lithography. The compressive tests were performed using a universal tensile test machine (Shimadzu AUTOGRAPH AG-100kND) at room temperature. After the compressive test, the electron moiré fringe was observed.

4. Results and Discussion

4.1. Observation of Creep Deformation in Pure Copper Specimen

The change in the moiré fringe patterns in the same area of a specimen after creep deformation for 15, 90, 140 and 165h is shown in Figs. 7 (a) (b), Fig.7 (c) (d), Fig. 7 (e) (f) and Fig.7 (g) (h), respectively. These moiré fringes in Figs. 7 (a), (c), (e) and (g) were observed by scanning electron beam exposures of 4.4 μm spacing in the x-direction. Figs. 7 (b), (d), (f) and (h) were observed by scanning electron beam exposure of a 4.2 μm spacing in y-direction (loading axis). As the creep proceeds, the moiré fringe becomes more complicated and its spacing generally becomes narrower. And offsets of moiré fringe can be observed at grain boundaries. It means grain boundary sliding occurs. Figure 8 shows an SEM image of the same area as Fig.7 (g) and (h). The surface cracks can be observed at grain boundaries near the triple points.

Fig.7 Electron moiré fringe pattern in the pure copper specimen crept at 723K and 20MPa crept for 15h: (a) (b) 90h: (c) (d), 140h (e) (f) and 165h: (g) (h).

Fig.8 SEM image of pure copper specimens for 165h at 723K and 20MPa

The spacing of the moiré fringe lines was measured in a direction perpendicular (x-direction) and parallel (y-direction) to the loading axis, and the strain in the direction perpendicular and parallel to the loading axis, ϵ_x and ϵ_y , was calculated using Eq.(1). The calculated strain within and around the central grain in Fig.7 and the direction of the grain boundary sliding are shown in Figs. 9. Figures 9 (a), (c), (e) and (g) show ϵ_y and Figs. 9 (b), (d), (f) and (h) show ϵ_x . Figures 9 show that ϵ_y is tensile strain and ϵ_x is compressive strain. In Figs.9 (a) and (b), the grain boundary sliding (for example, grain boundary A-B in Figs.7.) and strain difference are shown to be slight. As the creep proceeds, a high strain area and triple point cracks are formed [see Figs. 7 (g) and (h):165h and Fig.8], the region within the central grain can be divided into two areas of high strain (central part of the grain), and low strain. From the Figs. 7, 8, and 9, the triple-point cracks are partly surrounded with low tensile strain areas. Also, grain boundary sliding is larger and larger. These results indicate that the strain concentration is strongly connected to the grain boundary sliding and the formation of triple-point cracks releases the stress or strain concentration.

Fig. 9 Strain distribution in the pure copper specimen at 723K and 20MPa crept for 15h: (a) (b) 90h: (c) (d), 140h (e) (f) and 165h: (g) (h).

4.2. Measuring Residual Stress in Fiber Reinforced Composites

The moiré fringe patterns after pushing out test are shown in Fig. 10 (y-displacement) and Fig. 11 (x-displacement). The residual strains are calculated from the moiré pattern in Figs. 10 and 11 by Eq. (1) and the residual stresses are obtained using Hooke's law (using

Eq. (2).

The calculated residual stress distribution is shown in Figure 12. This result shows that a compressive interfacial residual stress exists in the radial direction at the interface between the fiber and matrix. The level of this residual stress is about -130MPa . There has been no other experimental data available up now so that a comparison with other experimental results is not possible. The solution of the elasticity theory can be used to evaluate the experimental results. These results are shown in Figure 12. The solid curves represent the theoretical distributions of the stresses from Eq. (3). The inner pressure P has been assumed to be the stress value at the interface in the test. The triangle data points represent the residual stresses in the radial direction from the test. One can see that the profile is in quite good agreement with the theoretical one.

Fig. 10. Moiré fringe pattern represents the displacement contours in the y -direction.

Fig. 11. Moiré fringe pattern represents the displacement contours in the x -direction.

Fig. 12. Residual stress distributions along the section A-B in the boron /epoxy FRP.

The solution of the elasticity theory is used to evaluate the experimental results. Most authors use a cylinder model to analyze the residual stress. The interactive stresses (residual stresses) between the fiber and the matrix include the compressive stress in the radial direction and the shear stress along the fiber axis. If the specimen is thin enough, the effect of the shear stress in the fiber axis can be limited, and only the compressive stress needs to be considered. Hence, the analysis of the residual stress in the matrix can be simplified as a problem of a hollow cylinder under uniform pressure on the inner surface. Normally, the structure dimension is significantly greater than the fiber diameter. The stress solution using the elasticity theory [12] can be expressed by Eq. (3).

$$\sigma_r \approx -\frac{R_1^2 R_2^2 P}{(R_2^2 - R_1^2)r^2} \propto -\frac{1}{r^2} \quad (3)$$

where σ_r denotes the stress components in the radial direction; P denotes the internal pressure; and R_1 and R_2 denote the inner and the outer radii of the cylinder; respectively, and r denotes the distance to the hole center. This equation means σ_r is in inverse proportion to $-r^2$. A comparison of the theoretical and the experimental distributions of the residual stress along section AB were performed. This result is shown in Fig. 12. The solid curves represent the theoretical distributions of the stresses from Eq. (3). This inner pressure P has been assumed to be the same value as the residual stress σ_y at the interface in the test. The triangle data points represent the residual stresses in the radial direction from the test. Generally speaking, the experimental profile is in good agreement with the theoretical one.

4.3 Compressive Test of Laminated Steel

The laminated steel specimen was compressed to 1.6% and the electron moiré fringe was observed. The moiré fringe patterns are shown in Fig. 13 (b) (y -direction (loading direction)) and Fig. 14 (b) (x -direction), and the SEM images are shown in Fig. 13(a) and Fig. 14 (a). Also the boundary of the WT-720 and 304 stainless steel are shown in these figures. The spacing of the electron moiré fringes changed at the interface (Figs. 13(a)). The strain along the lines, A-A', B-B' and C-C', calculated from the electron moiré fringes in Fig. 13 (a) is shown in Fig. 15 (a) (y (loading)-direction) and the strain along the lines, A-A', B-B', C-C', D-D' and E-E' calculated from electron moiré fringes shown in Fig. 14 (b) is shown in Fig. 15 (b) (x -direction). The compressive strain in the loading direction for the 304 stainless steel and the tensile strain in the direction perpendicular to the loading direction are larger than that of WT720. Also, the compressive stain in the loading direction is largest at the interface between the 304 stainless steel and WT720.

Fig.13 SEM image of laminated steel specimen: (a) and electron moiré fringe: (b).

Fig.14 SEM image of laminated steel specimen: (a) and electron moiré fringe (x -direction): (b).

Fig.15 Strain distributions along the lines A-A', B-B' and C-C' in Fig. 13: (a) and along the lines, A-A', B-B', C-C', D-D' and E-E' in Fig. 14: (b).

5. Conclusion

The electron moiré method was applied to measure the strain distribution in the deformed specimens and measuring the residual strain (stress) around the fiber of FRP composites. A scanning electron microscope was used to fabricate very fine model grids on the specimen surfaces and to observe the electron moiré fringes. The creep strain distribution of pure copper and the strain distribution of compressed laminated steel were measured by the moiré pattern produced by the interference of the model grid and the scanning electron beam scan. The residual stress around the fibers of the FRP is released through by a push out test and the released residual stress is measured by this electron moiré method.

Acknowledgement

This work was partly supported by Research Promotion Bureau (Layer Integrated Steel and Metal Project), Ministry of Education, Culture, Sports, Science and Technology (MEXT), Japan.

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