Error Analysis and Thermal Expansion Measurement with Electron-beam Moiré

by J. R. Berger, E. S. Drexler and D. T. Read

ABSTRACT—In this paper, the authors study errors incurred when using the experimental technique of electron-beam moiré. There are two sources of error: error manifested as an apparent magnification drift and error due to fringe tracing. The error due to fringe tracing is nearly negligible in comparison to the error due to magnification drift. By investigating the thermal expansion of commercially pure copper, the authors demonstrate the usefulness of the error estimate. The average result for the coefficient of thermal expansion is within 1.8 percent of handbook values for this material, with a possible error due to apparent magnification drift of 9 percent.

Introduction

The experimental technique of electron-beam (e-beam) moiré is a relatively new member to the family of moiré methods. Based on the initial work of Kishimoto *et al.*,[1] Dally and Read[2,3] developed the method to its present, mature state. The method relies extensively on the use of a scanning electron microscope (SEM) for both production of the specimen gratings and formation of a reference grating. The specimen gratings are produced using e-beam lithography. Details of the production of the specimen gratings are given in Ref. 2, where the importance of strict control over the process variables is emphasized.

The reference grating for e-beam moiré is produced by the raster scanning motion of the electron beam in the vacuum chamber of the SEM. As such, it is necessary to perform all tests within the SEM. The raster scanning of the electron beam is very similar to the scanning video moiré technique developed by Morimoto and Hayashi[4] for low-frequency specimen gratings. Unlike the traditional geometric[5] or interferometric[6] moiré methods, e-beam moiré allows one to easily vary the effective pitch of the reference grating by varying the beam controls on a typical SEM. Read and Dally[3] developed a model to interpret fringe fields obtained as the e-beam diameter, the pitch of the raster scan and the angle between the scan lines and grating lines were varied.

In this paper, we are concerned with a determination of factors which may influence a measurement made with e-beam moiré at "fixed" settings on the SEM. This investigation was motivated by an attempt at benchmarking the method through a determination of the coefficient of thermal expansion in a bulk copper specimen. Initial measurements indicated that the fringe pattern appeared to change over time. This led us to a parametric study of the influence of instrument drift resulting in an apparent change in magnification and fringe-tracing errors on the quantitative measurement of displacements.

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Original manuscript submitted: September 1996.
Final manuscript received: February 2, 1998.

Magnification Errors

Before studying possible error sources with e-beam moiré, it is instructive to identify the key parameters in the formation of the reference grating. We are considering a scenario in which the specimen grating has been prepared and the specimen placed within the vacuum chamber of the SEM. We view the moiré fringe pattern with no thermal or mechanical load on the specimen. As noted in previous studies,[2,3] it is usually not possible to obtain an ideal null field with e-beam moiré because the magnification is adjusted in discrete increments. Under these conditions, variations in the observed fringe field must be due to variations in those parameters which contribute to the formation of the reference grating. We first consider those factors which affect the pitch of the reference grating. As described in Refs. 2 and 3, the pitch of the reference grating for e-beam moiré can be calculated as

$$p_{\mathrm{ref}} = \frac{S}{MR}, \tag{1}$$

where S is the nominal image size, M is the magnification of the SEM and R is the number of raster scan lines. The nominal image size S and the number of raster scan lines R are fixed for a given SEM during a given experiment. Therefore, according to eq (1), only a change in magnification over time can contribute to an apparent change in the pitch of the reference grating. The apparent change in magnification may be due to a number of instrument-related issues (accelerating voltage stability, stability of the electronic magnification unit, etc.). For this paper, we will treat all of these sources as an apparent change in magnification. Note that small changes in the magnification can contribute strongly to the apparent pitch of the reference grating since they are inversely proportional to each other. Read and Dally[3] noted that the value of M must be precisely known for proper interpretation of the moiré fringe fields. They further noted that the apparent magnification from the SEM character display differed from actual measured magnifications by approximately 5 percent for the SEM used in their investigations. No time variation of M was considered by the authors.

If some variation occurs in the magnification of the SEM, we can write the pitch of the reference grating as

$$p_{\mathrm{ref}} = \frac{S}{(M + \Delta M)R}, \tag{2}$$

where ΔM is the variation in the magnification. We consider the case of uniform axial strain on a specimen and calculate the error in the strain measurement due to the magnification variation. Relative to a null condition, Read and Dally[3] calculated the tensile strain in terms of reference and the moiré fringe frequencies as

$$\varepsilon = -\frac{f_i}{(f_{\mathrm{ref}} + f_i)}. \tag{3}$$

Including a variation in magnification from eq (2) which affects both f_i and f_{ref}, eq (3), rewritten in terms of grating pitches, is

$$\varepsilon = \frac{p_g MR}{S} - 1 + \frac{p_g R \Delta M}{S}, \tag{4}$$

where the last term represents the error term. It is helpful to consider the total strain as an apparent strain,

$$\varepsilon_{\mathrm{app}} = \varepsilon_{\mathrm{true}} + \varepsilon_{\Delta M} \tag{5}$$

where

$$\varepsilon_{\mathrm{true}} = \frac{p_g RM}{S} - 1 \tag{6}$$

$$\varepsilon_{\Delta M} = \frac{p_g R \Delta M}{S}. \tag{7}$$

For purposes of this analysis, we can further simplify eq (7) by noting that, for small strains,

$$\frac{p_g R}{S} \approx \frac{1}{M}. \tag{8}$$

Equation (7) can then be written as

$$\varepsilon_{\Delta M} \cong \frac{\Delta M}{M} \tag{9}$$

For a specific example, consider a specimen grating with $p_g = 180$ nm observed in the SEM at a nominal magnification of 1100X. From eq (1), we obtain a nominal reference grating pitch of $p_{\mathrm{ref}} = 170.5$ nm for our SEM, where $S = 90$ mm and $R = 480$ lines. Assume that we subject the specimen to a uniform strain of 1200 $\mu\varepsilon$. From eq (3), assuming no change in the magnification, our specimen grating then has a pitch of $p_g = 170.7$ nm. At a value of $\Delta M = 0.20$X (with a nominal magnification of 1100X), the additional strain obtained from eq (9) is 182 $\mu\varepsilon$. The error in the strain measurement would then be 15 percent, a significant error. To maintain less than 5-percent error in the strain measurement would require control on the magnification such that $\Delta M < 0.07$X.

The above analysis highlights the necessity of strict control on magnification for quantitative measurements with e-beam moiré. As noted by Dally and Reed,[2] the nominal magnification must be accurately known for proper interpretation of the moiré field. It is now clear that not only must the nominal magnification be well known but the control on factors influencing the magnification must be exceptionally stable. We have treated all time dependent errors as magnification errors for the reasons outlined at the beginning of this section. It is important to note that this error is manifested as an apparent drift in the magnification. Any drift in the instrument such as working distance, accelerating voltage, electronic magnification control or other instrument-related issues can produce an apparent drift in the magnification. Our goal is to study the influence of these apparent magnification drifts and not to isolate the precise cause for a given test on our particular instrument.

Studies of the Temporal Variation of the Fringe Fields

A series of experiments were performed in order to identify and quantify factors which varied with time in a typical e-beam moiré experiment. For each of the experiments, a single, homogeneous, polycrystalline copper specimen (99.999 percent pure copper) was used. The specimen was disk shaped with a diameter of 5 mm and a thickness of 2 mm. The specimen was instrumented with a line grating located near the center of the specimen with $p_g = 180$ nm. The specimen grating was etched into the surface of the copper. The specimen was placed in the SEM, and all tests were performed at a magnification setting of 1100X. This is the magnification setting associated with the near null field for the specimen-grating frequency used in this experiment. The temperature of the testing stage was monitored during the acquisition of images. The maximum temperature variation observed in all of our tests was ± 0.6°C.

For our initial test, moiré fringe fields were obtained at fixed settings on the SEM over a period of 240 min at 30-min intervals. For the particular test analyzed in this paper, we had an initial field of approximately four fringes. We have also observed that the number of fringes in the initial field may vary for a given specimen grating due to slight changes in either working distance or focus as the SEM is set up for a particular experiment.

Each acquired image was analyzed to obtain the average number of moiré fringes across the image. The analysis was based on a fringe analysis program developed at the National Institute of Standards and Technology. The program requires the user to specify points located along a fringe center. The software then performs a spline fit to the data. Obviously, some statistical variation can occur due to errors in locating the fringe center or from the use of different subsets of data from the same moiré field. Similar errors were noted by Barker *et al.*[8] in analyzing moiré data collected near crack tips. We will address the variation in the average number of fringes due to these errors.

The average number of fringes across each acquired image as a function of time is shown in Fig. 1. A clear variation in the average number of fringes occurs over time. Note that the first data point shows the greatest variation from the mean value. This was typical of all the experiments we performed. For the data shown in the figure, we obtain a mean value of 4.34 fringes with a standard deviation of 0.14 fringes. Our field of observation at 1100X is 81.8 μm, which yields a mean moiré fringe frequency of 0.0530 fringes/μm with a standard deviation of 0.0017 fringes/μm. Assuming this variation in the fringe field is strictly due to factors influenc-

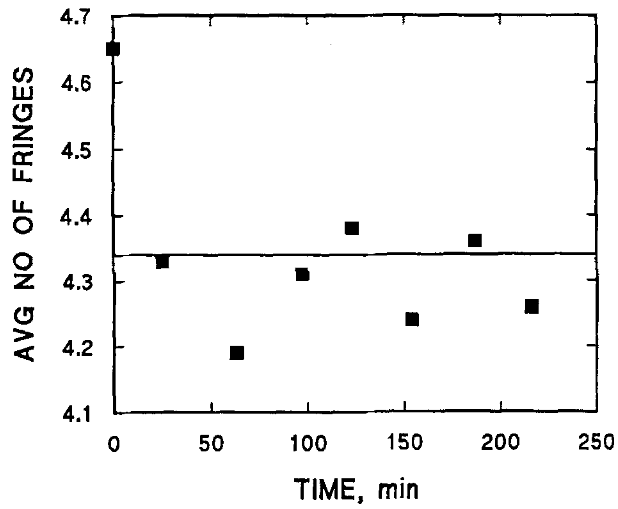

Fig. 1—Average number of fringes for the copper specimen as a function of time

ing the magnification of the SEM, we can calculate from the apparent change in magnification required to produce one standard deviation. We first calculate from eqs (1) and (2) the nominal magnification required to produce the observed moiré field frequency. We obtain a nominal magnification of $M = 1056X$. The magnification was set on the SEM at 1100X, our calculated value is within the usual 5-percent error allowed in the magnification calibration. To produce one standard deviation in the fringe field, we predict a value of $\Delta M = 0.32X$ for the data shown in Fig. 1.

As noted above, we usually observed the greatest variation in the fringe field with the first data point. This suggests that the instrument has not yet stabilized and experiments should only be performed after waiting a period of time. If we consider only the data points obtained after 30 min have elapsed, we obtain a mean value of 4.30 fringes with a standard deviation of 0.07. The mean value is comparable to that obtained previously with all data points. Therefore, our nominal magnification is approximately the same ($M = 1056X$). We note that the standard deviation has decreased from 0.14 to 0.07. We obtain a value of $\Delta M = 0.16$ required to produce one standard deviation in the fringe field. This is perhaps a more realistic estimate for expected variations in the apparent magnification.

The probe current in the SEM was initially suspected of producing apparent variations in the fringe field. This was based on recordings of probe current over time as each experiment was performed. To investigate the likelihood of probe current causing variations in the fringe field, we performed a series of tests in which the probe current was varied and images acquired. The average number of fringes plotted as a function of probe current is shown in Fig. 2. For this particular data set, the mean number of fringes is 4.95 with a standard deviation of 0.10. In comparison to Fig. 1, it is clear that the variation we observed over time is approximately the same as the variation observed over the time we varied the probe current. Note in Fig. 2 that the probe current was varied from approximately 0 pA to −100 pA. The observed variation in probe current during tests similar to that which provided the data in Fig. 1 was only ±0.6 pA. We therefore concluded that the probe current was not responsible for the observed temporal variation in the fringe field.

Finally, we considered the variation in the average number of fringes due to the fringe-tracing procedure. To investigate this, we acquired an image and performed six independent tracings of the fringe centers with the fringe analysis software used in our study. Of concern here was variation in the location of the center of the fringes and the effect of the number of points along each fringe center in the spline fit to the data. The results are shown in Fig. 3 for the average number of fringes across the entire image for each of our tracings. As shown in the figure, a variation of approximately ±0.05 fringes occurs with a standard deviation of 0.02 fringes. This is far below the observed variation over time shown in Fig. 1. We conclude that fringe tracing is not responsible for the observed variation in the fringe field.

Coefficient of Thermal Expansion for Copper

To provide a benchmark for the e-beam moiré method in thermal stress studies, we performed two experiments to de-

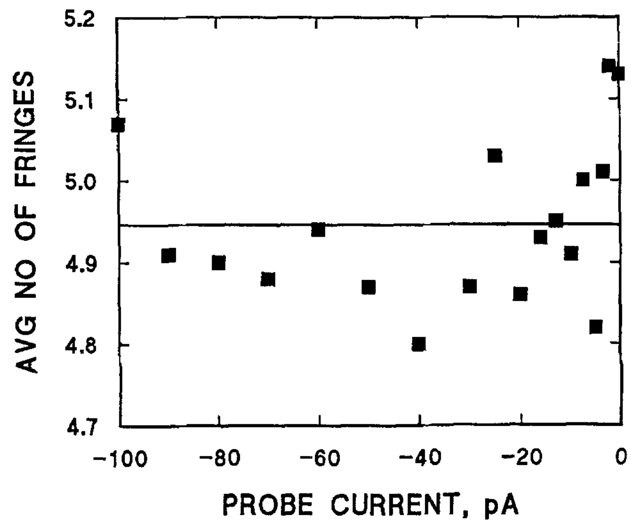

Fig. 2—Average number of fringes as the probe current is varied

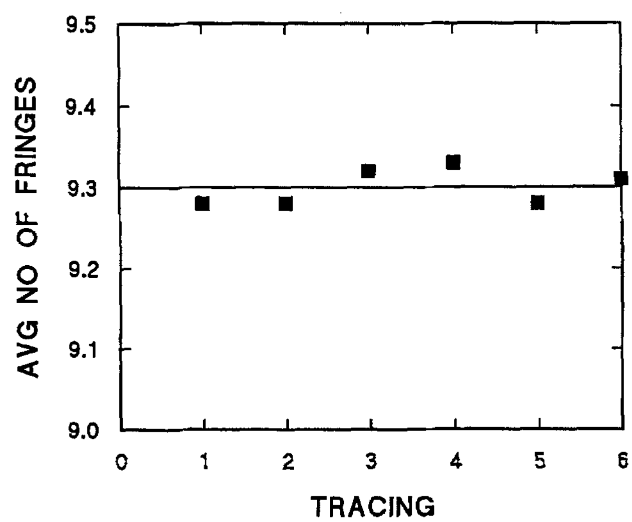

Fig. 3—Average number of fringes from different tracings of the fringe pattern with the fringe pattern analysis software used in this study

termine the coefficient of thermal expansion, α, of copper. The first experiment used a copper specimen with a geometry identical to that of the specimen previously described but with a line grating of pitch 900 nm. The second experiment was performed using the same specimen as that in the studies of the temporal variation of the fringe field. Both specimens had line gratings which were etched into the surface to avoid high-temperature deterioration of the PMMA coating normally used for producing gratings with e-beam lithography. Both specimens were tested on a thermal stage in the SEM. The heating/cooling stage fits securely on the shuttle base within the SEM chamber. The resistive heating unit has a temperature range to 400°C. The stage is cooled by circulating liquid nitrogen through the stage. The stage can be cooled down to −185°C. A platinum resistance thermocouple is located just below the specimen, and the desired stage temperature is achieved by balancing the heater output and the chilled gas. The entire stage is electrically grounded, and the 10^{-5}-torr vacuum of the SEM chamber minimizes heat transfer.

The procedure for both experiments was the same. The specimen was placed in the vacuum chamber of the SEM and thermally cycled twice between −50°C and +150°C. The temperature was then set at the starting temperature for the test of −50°C. Images of the moiré field were acquired at intervals of approximately 50°C. After each increment in temperature, the specimen was allowed to equilibrate before acquisition of the moiré field. For assessing equilibrium, we waited until the fringe field changed less than 0.25 fringes per minute across the field of view. This required waiting a maximum of 30 min. Typical fringe fields are shown in Fig. 4 at temperatures of −49.6°C and 150.6°C.

The average number of fringes across the field as a function of temperature is plotted in Figs. 5(a) and 5(b) for the two tests we performed. The first test, shown in Fig. 5(a), was conducted before we were aware of possible errors due to temporal variations in the fringe field. Only three data points were obtained in that test. After observing the temporal variations in the fringe fields detailed in the first part of this paper, we repeated the experiment but collected data during both heating and cooling of the specimen. Shown in Fig. 5(b) are the mean values of the data at each temperature with bars indicating the spread in the data.

We calculate the coefficient of thermal expansion from the fringe field following Bowles *et al.*[9] We are only interested in the change of fringe order with temperature over the uniform displacement field, so we calculate the slope of the average number of fringes with respect to temperature, s_f. The coefficient of thermal expansion can then be directly calculated as

$$\alpha = \frac{s_f}{R}. \tag{10}$$

From eq (10), we can estimate α by dividing the slope of a best-fit line through the data in Figs. 5(a) and 5(b) by R (recall that $R = 480$ for our SEM). Summarized in Table 1 are the results of this calculation.

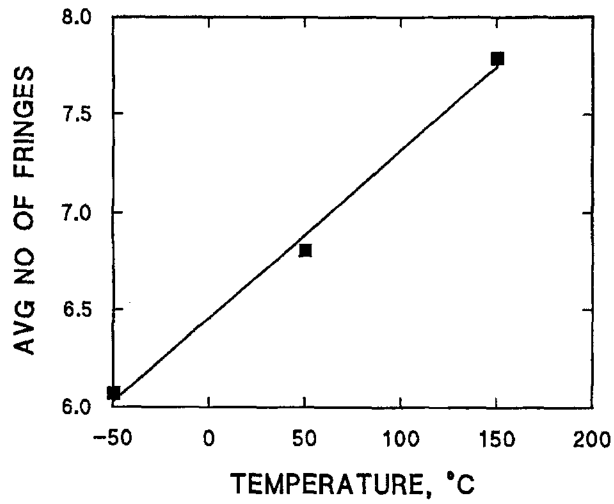

Fig. 5(a)—First test for the average number of fringes as a function of temperature change for the copper specimen

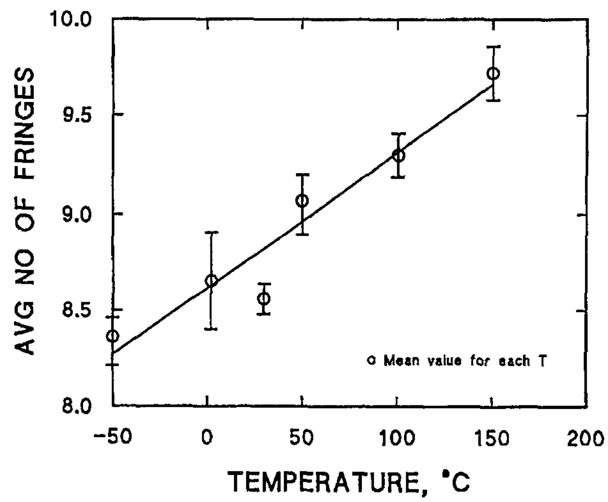

Fig. 5(b)—Second test for the average number of fringes as a function of temperature change for the copper specimen. Shown are the mean values obtained at each temperature increment. The error bars indicate the variation in the experimental data

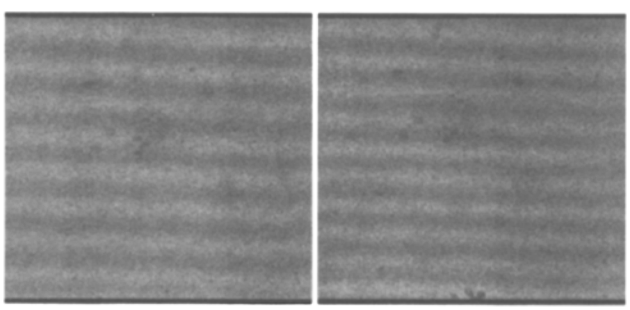

Fig. 4—Typical e-beam moiré fields acquired at temperatures of −49.6°C and 150.6°C

TABLE 1—COEFFICIENT OF THERMAL EXPANSION FROM THE E-BEAM MOIRÉ DATA

Test	Slope, s_f	Correlation Coefficient, r	α (/°C)
1 (Fig. 9)	8.61×10^{-3}	0.997	17.6×10^{-6}
2 (Fig. 10)	6.96×10^{-3}	0.966	14.8×10^{-6}
		Average α =	16.2×10^{-6}

The average coefficient of thermal expansion from the moiré data can be compared to a handbook value[10] of $\alpha = 16.5 \times 10^{-6}/°C$ for pure copper. Our average result is apparently within 1.8 percent of the handbook value. However, we must inspect our result in light of the parametric studies performed on the apparent magnification drift.

Estimate of the Error

Based on the parametric studies detailed in the previous section, we concluded that variation in the apparent magnification in the SEM is the principal source of error. This variation is not due to probe current drift. Magnification drift in the SEM can occur through slight variations in the high accelerating voltage, through drift of the magnification control unit or other instrument-related factors. Clearly, we are expecting a level of performance and stability from the SEM which it was not intended to meet. The drift of ±0.32X at 1100X which produced one standard deviation in the fringe field for the experiment of Fig. 1 represents a variation of only ±0.03 percent in the magnification.

We now consider a variation in apparent magnification during a thermal expansion test. The apparent coefficient of thermal expansion can be calculated from the apparent strains as

$$\alpha_{\text{app}} = \frac{\Delta\varepsilon_{\text{app}}}{\Delta T}, \tag{11}$$

provided the thermal strains are linear over the temperature increment ΔT. Writing the apparent strains as shown in eq (5), we have

$$\alpha_{\text{app}} = \frac{\Delta\varepsilon_{\text{true}}}{\Delta T} + \frac{\Delta\varepsilon_{\text{app}}}{\Delta T}. \tag{12}$$

We can put this equation in a more useful form as

$$\alpha_{\text{app}} = \alpha_{\text{true}}\left(1 + \frac{\Delta M}{\alpha_{\text{true}} M \Delta T}\right), \tag{13}$$

where

$$\alpha_{\text{true}} = \frac{\Delta\varepsilon_{\text{true}}}{\Delta T}. \tag{14}$$

Based on our investigations concerning the apparent variation in magnification, we are concerned with apparent variations in magnification of approximately 0.32X at 1100X. From eq (13), we obtain

$$\frac{\alpha_{\text{app}}}{\alpha_{\text{true}}} = 1.088. \tag{15}$$

We can therefore anticipate obtaining an estimate of the coefficient of thermal expansion to within approximately 9 percent. If we consider the average value of the coefficient of thermal expansion given in Table 1 and the values obtained from the individual experiments of Figs. 5(a) and 5(b), we obtain $\alpha = 16.2 \times 10^{-6}/°C$ ±8.5 percent. This is consistent with the anticipated error in the measurement predicted from eq (15).

Summary

We have presented a study concerning the magnitude of errors from a variety of sources when using e-beam moiré. Based on parametric studies of the temporal variation of the fringe patterns, we identified the major source of error as being an apparent magnification drift. It was demonstrated that the magnification error causes a change in the frequency of the observed moiré field. For the simple case of uniform strain, the error was nonnegligible for typical values of magnification drift. This demonstrates the need for strict monitoring and control of the magnification when performing measurements with e-beam moiré. Finally, we calculated the coefficient of thermal expansion for copper in light of these potential errors. We obtained an average value for the coefficient of thermal expansion within 1.8 percent of handbook values. This result was fortuitous as our expected error in the measurement due to the apparent drift in magnification was approximately 9 percent. Future work will focus on the issue of magnification control and stability in the SEM.

Acknowledgments

The first author acknowledges the support of the National Science Foundation and the National Institute of Standards and Technology under the joint NSF-NIST Program CMS-9522147. Additional support was provided by the Materials Reliability Division of the National Institute of Standards and Technology.

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