

Investigation of Deformation at the Grain Scale in Polycrystalline Materials by Coupling Digital Image Correlation and Digital Microscopy

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Abstract

The purpose of this paper is to present an approach for the investigation of in-plane strain distribution at the grain scale in polycrystalline materials. The technique is developed by coupling digital image correlation and digital microscopy. By performing and analyzing a series of validation tests, performances and limits of this approach are quantified. An example of its application is presented for a Ni based alloy specimen. It is realized that this approach can obtain accurate displacement data. This technique can thus be used to investigate micromechanical behaviors of polycrystalline materials.

Introduction

Polycrystalline materials are now widely used in industries, and studies of various materials' mechanical behaviors have thus become a crucial issue in mechanical engineering and materials science. In the past, most studies on materials' mechanical behaviors have been focused on the macroscopic level. However, in many research areas deformations need to be determined on a scale comparable to that of the microstructure. In fact, such deformations lead to significant changes in the microstructure of the materials, which could ultimately cause substantial changes in the material properties at the macroscopic level ultimately. Therefore, investigation of materials' mechanical properties on the grain scale is very important.

However, measurements on microscopic scales remain a serious challenge.¹ Especially for metals and metal alloys, displacements on the scale of grain sizes are usually within the submicron scale. It is difficult to use most traditional mechanical experimental methods to investigate microscopic deformation due to their limitations of spatial resolution. A non-contact, full-field optical deformation measurement technique named Digital Image Correlation (DIC) was developed in the1980s.² The main advantage of the technique is that it has "adjustable" measurement sensitivity, the accuracy of this method is weighed by the pixel. Therefore, the success of this method is only dependent on the quality of the images processed, and there is no spatial resolution limitation. Meanwhile, DIC is easy to set up and use; the technique only needs a digital camera to record the random speckle patterns even for long-term inspections. Digital image correlation is an ideal tool for material inspections from the macro-scale to reduced length scales.

In recent years, by coupling DIC and highmagnification devices, such as the optical microscope and the scanning electron microscope (SEM), research on polycrystalline materials' micromechanical properties has been fruitful.^{3–6} However, as to the error and precision analysis, little work has been done. In fact, common optical microscopy is not suitable for the studies on the grain scale. Due to the wavelength of light, common optical imaging systems are limited to a maximum resolution that corresponds to a magnification of about $1000 \times$. Furthermore, high magnification imaging systems are subject to a small depth of field and errors caused by lens aberration.^{7,8} Thus, most of the previous work has been done under electron microscopy (such as SEM and TEM) with high magnification and a large depth of field. However, the imaging systems based on electron microscopy also have their own inherent disadvantages. For example, SEM systems have both spatial distortion and time-varying distortion.⁹ Although some calibration procedures have been introduced to remove the errors caused by aberration and distortion,^{7–9} they are complex and may be perceived as an obstacle to those wishing to adopt the use of a microscope for micro-DIC.

The technique proposed in this study is based on the use of the DIC method coupled with a new type of microscope—the Digital Microscope. The precision is analyzed experimentally. As an illustration, simple tensile properties and low-cycle fatigue damage of a Ni-based alloy specimen are studied at the grain scale.

Digital Image Correlation

Digital image correlation is a full-field optical measurement technique, which was developed in the 1980s. The basic principle of this method is to obtain deformation data of the object surface by correlating random speckle patterns captured before and after deformation. The pattern captured before deformation is regarded as the "reference image" and the other is regarded as the "deformed image." The two patterns are both divided into several small subsets of $N \times N$ pixels. The discrete matrix of the values of pixel gray level in each subset is unique and can be used to calculate the correlation of the two subsets in the reference image and deformed image, respectively. The correlation between subsets in the two images can determine the in-plane displacements of subset centers of the reference image.

The mathematical criterion for determining correlation of the two subsets is commonly given by using a discrete cross-correlation coefficient as

$$C = \frac{\sum \sum [f(x, y) - f] \cdot [g(x^*, y^*) - \overline{g}]}{\left[\sum \sum [f(x, y) - \overline{f}]^2 \cdot \sum \sum [g(x^*, y^*) - \overline{g}]^2 \right]}$$

where f(x, y) and $g(x^*, y^*)$ are pixel gray values in the reference image and deformed image, respectively; \overline{f} and \overline{g} are the average gray values of each subset in the reference image and deformed image, respectively. The correlation coefficient *C* represents how close the two subsets are, with C = 1 corresponding to perfect correlation. Owing to systematic error,

random error, and the distortion of images, the correlation coefficient C generally cannot equal 1 in practice. Thus, the maximum value of C is considered as a measure of the coincidence of the assumed deformation with the actual one. Therefore, the experimental measurement becomes a process of mathematical optimization, which seeks the maximum value of C.

Therefore, the most important theoretical research in DIC is the study of numerical optimization algorithms. In the past few decades, many improvements on the DIC algorithms, especially on the subpixel algorithms, that provide higher precision and higher processing speeds for the DIC have been reported.^{10–16} Through a comprehensive consideration and analysis of accuracy and efficiency, a fast algorithm named the gradient-based subpixel registration algorithm^{15,16} was chosen to process images in this article. The nominal displacement measurement precision of this algorithm is reported as 0.01 pixels.

For strain measurements, an important problem is how to obtain the strain distributions from the displacement fields which have slight deviation or noise. In this article, a strain estimation technique based on least-square fitting of a local displacement field¹⁷ is used to reduce noise and extract optimized strain distributions.

Digital Microscope

The digital microscope is a new member in the family of microscopes. Early digital microscopes were actually optical microscopes equipped with imaging devices such as charge coupled device (CCD) video cameras, the captured images were transferred to computer for real-time preview and post processing. However, the qualities of images were severely affected by the limited magnification and shallow depth of field of the optical microscope available at the time. In the past decade, the digital microscope has developed rapidly. The technology has now matured to the extent that the digital microscope is a viable micro-observation and image-processing device.

In this article, the employed device was the VHX-100 digital microscope produced by KEYENCE, which is shown in Fig. 1. The microscope consists of a set of lenses which can provide a superior depth of field and various modes of observation and a powerful image-processing software system which can make the course of observation and image processing much easier for the observer. For example, multi-angle observation can be obtained by using this microscope, compared to the fixed observation



Figure 1 Digital microscope

obtained by early digital microscopes. There is also an inner lamp house in this microscope by which the diverse observation modes can be obtained and the effects of the outside environment can be minimized. In short, it is an ideal microscopic research tool. In this article, chromatic images with high magnification and excellent resolution have been obtained by using the digital microscope.

Precision Analysis

A series of experiments have been conducted to measure the errors and limits of the measurement system. The studied specimen shown in Fig. 2 is made from a Ni-based alloy called GH4169 with fine grains (less than 20 μ m). In order to fulfill the requirement of DIC, the specimens were first polished and then etched in $1H_2O_2 + 1HNO_3 + 1HCL$ for several seconds to provide distinct micrographs with enough contrast to be viewed under the digital microscope. Figure 3 is a micrograph of the specimen surface.

Baseline errors of displacements

A baseline experiment was implemented to determine the inherent minimum errors of the system. Two images of the same position on the specimen were captured at different times, without deforming or translating the specimen. The magnification, brightness, etc., were also fixed during capture of the two images. The specimen was then moved and another image pair was captured at another position. This process was repeated 10 times and these image pairs were called image pairs 1-10, respectively. Finally, these image pairs were analyzed by DIC



Figure 2 Shape and dimension of specimens (units: mm)



Figure 3 Micrograph of specimen surface

software. The experiment was conducted at $500 \times$ and $3000 \times$ magnification, respectively.

Although there is no actual deformation on the specimen, the calculated results would not be zero due to the systematic error and random error. These errors reflect the precision of the measuring system. According to the theory of mathematical statistics, the mean of all the calculated displacement values in each image pair is a statistical variable which can indicate how serious the systematic error of each measurement is and the standard deviation (SD) can reflect how serious the random error is. Therefore, the above two statistical variables can be used to weigh the baseline errors of the system. The results are shown in Figs. 4 and 5, respectively. There were 9801 calculated points in a 1200×800 pixels area of each image pair and the subset size was 61×61 pixels.

Generally, the systematic errors here come from algorithm calculation errors. It can be seen from Fig. 4 that the displacement means at $500 \times$ have the same order of magnitude as the means at $3000 \times$, and the maximum value of means is below 0.06 pixels. These results indicate that the systematic error is not significant. From Fig. 5, it can be seen



Figure 4 Analysis of systematic errors

that the SDs of displacement values at $3000 \times$ are several times larger than those at $500 \times$. This is because the effects of instability of inner lamp-house and inner noise on the quality of images captured become increasingly significant following the increase in magnification. Additionally, the maximum value of the SDs is less than 0.034 pixels. According to the theory of mathematical statistics, because the number of calculated points of each image pair is less than 10,000, the limiting error of the measurement is less than four times the maximum SD, which is 0.136 pixels. As it has been reported that the maximum value of the SD is 0.3872 pixels (at $750 \times$),¹⁸ it can be seen that the baseline error of DIC under the digital microscope is much smaller than that under SEM.

Effect of refocus errors

The deformation measured by DIC is in-plane displacement. However, in material test experiments, out-of-plane motion and deformation always exist during loading. In macroscale experiments, the effect of the out-of-plane displacement can be minimized by designing the camera lens to be positioned far away from the sample surface. However, when using a microscope, the method is impractical and the out-of-plane deformation can influence the accuracy of correlation seriously.¹⁹ Additionally, the out-ofplane displacement will also cause variance in the magnification of images captured before and after deformation. In addition, the magnification variance has a great influence on the measuring accuracy, especially to the transverse and longitudinal strains. It can be deduced that if there is a magnification



Figure 5 Analysis of random errors

variance, the measured transverse and longitudinal strains will be equal to the real values of the strains plus the magnification variance. For example, if the magnifications of an undeformed image and a deformed image are $500 \times$ and $501 \times$, respectively, the magnification variance is 0.2%, there will also be an additional strain error of 0.2% in the measured values of transverse and longitudinal strains at all calculated points. The error is large, especially for metallic materials and alloys with high strength.

To eliminate the out-of-plane effects discussed above, refocusing before acquiring deformed images is necessary in microscopic DIC. The specimen surfaces should be moved back to the original positions through refocusing, and the magnifications of the original image and the deformed image should be ensured equal. Accuracy of the refocus will govern the success of experiments.

To quantify the accuracy of refocusing under the digital microscope used in this article, original image and refocused image of the same position were treated as an image pair and processed with DIC. Experiments were repeated 10 times at nominal magnifications of $500 \times$ and $3000 \times$. DIC was performed using the parameters described above. The mean values of transverse or longitudinal strains of all the calculated points in an image pair are shown in Fig. 6.

In this "refocusing" experiment, if the specimen surface is moved back to the exact original position through refocusing, it is equal to the "baseline" experiment mentioned above, and the calculated strains here should be equal to the "baseline errors of strains." The strain errors are extracted from the part of the random errors in the overall displacement



Calculated Displacement (Pixel) Image Actual Standard Magnification Pair Displacement Mean Deviation 500× 10 µm (28 pixels) 28.4 0.033 1 15 µm (41 pixels) 0.028 2 41.5 3 20 µm (55 pixels) 54.9 0.052 4 30 µm (83 pixels) 83.3 0.061 5 40 µm (110 pixels) 110.1 0.067 $3000 \times$ 0.15 6 1 µm (17 pixels) 18.4 7 2 µm (33 pixels) 35 1 0.09 0.12 8 3 µm (50 pixels) 54.5 9 5 µm (83 pixels) 88.3 0.22 10 8 µm (132 pixels) 135.3 0.36

Table 1 Result of translation experiment

Figure 6 Effect of magnification variance on strain measurement

errors, not the part of the systematic errors. Thus according to the theory of mathematical statistics, the means of the strain baseline errors approximate zero (although the result has not been listed out, it has been proved in the "baseline" experiment that the means of transverse or longitudinal strains are below 2×10^{-5}). However, the specimen cannot be moved back to the exactly original position through refocusing in reality. It is known from the above analysis that it will cause magnification variance and the effect of the magnification variance is equal to adding a certain numerical value to the overall real strain values, so the means of the strains will no longer approximate zero. Therefore, the means of transverse or longitudinal strains of all calculated points can be used to weigh the magnification variance.

It can be seen from Fig. 6 that the magnification variance is much more significant at 500×. That is because the depth of field of the microscope at 500× is much larger than that at 3000× and it is therefore more difficult to ensure the accuracy of refocus. The errors caused by magnification variance at 3000× are smaller than 300 $\mu\epsilon$, which is enough for the strain measurement on the grain scale. In fact, it is the clear chromatic images captured by the digital microscope which can make it easier to refocus by eye. Additionally, it is shown that the mean of ϵ_x is nearly equal to the mean of ϵ_y in the same image pair. This result goes some way toward validating the above statements about the effect of magnification variance on strain measurements.

However, it should be noted that the refocus procedure is a compensatory method. In most cases, the DIC should be applied in situ, without any additional adjustments during experiments. The refocus procedure could be introduced to improve accuracy only if the errors caused by the out-of-plane motion cannot be ignored.

Another problem about microscopic DIC is that the area which can be studied in one time is limited by the small size of the vision field at high magnification. That means experiments should be conducted ex situ in many cases. Images of different positions should be captured successively to study large areas in one time. The high accuracy of refocus by the digital microscope can make it possible to apply DIC ex situ.

Accuracy of displacement measurement

A series of pure rigid translation experiments were carried out to weigh the accuracy of the system in measuring small displacements. A translation table from SEM was used to translate and position the specimens. The tests were run at nominal magnifications of $500 \times$ and $3000 \times$, respectively. Two images captured before and after translation, respectively, are regarded as an image pair, and DIC was performed using the parameters described above. Displacement results from DIC are compared with the actual translation displacements in Table 1. The translation is in the *x* direction.

It can be seen from Table 1 that the calculated displacements are close to the actual values in the image pairs at $500 \times$. Under such circumstances, the maximum difference between calculated displacements and the actual translations is within 1 pixel. Although the maximum difference in the image pairs at $3000 \times$ is more than 5 pixels, it is still within the error range of the mechanical device of the translation table. Clearly, the error of the pure translation tests is acceptable.

Lens aberration is always an important concern in the microscopic DIC using light microscopes. In fact,

the displacement fields calculated from the pure translation test can also be used to check the effects of lens aberration.¹⁹ The SD of translation displacement values of all calculated points in an image can be used to weigh the seriousness of lens aberration. It can be seen from Table 1 that the lens aberration effects under $3000 \times$ are more serious than those under $500 \times$. However, even the effects under $3000 \times$ still give errors within acceptable limits. Conversely, if the translation is large, for example, more than 100 pixels, the lens aberration effects are relatively large. On the basis of the results, a measurement can be taken to reduce lens aberration effects: the specimen should be moved back to the original position or as near as possible before acquiring the second image. This measurement can also reduce the effects of intensity field variations.

Application to Uniaxial Tensile and Fatigue Experiment

Uniaxial tensile and fatigue tests were conducted on the same specimen described above in the "Precision Analysis" section to evaluate the feasibility of the system. The experiments were conducted at $3000 \times$. The loading was in the y direction and the strain shown here was parallel to the loading direction— ε_{ν} . The tensile test was in situ, and the tensile load was 2 kN. The cyclic load amplitude was 14 kN, and the loading signal was a sinusoidal waveform with a frequency of 10 Hz and a load ratio (R) of 0. Fatigue testing was periodically interrupted at a predetermined number of fatigue cycles (often per thousand cycles). Then the specimens were transferred to the digital microscope to obtain micrographs of the areas of interests. Finally, the series of images were processed by DIC software. The subset size was 61×61 pixels. The positions of the studied regions are shown in Fig. 7. Area 1 has been studied in the fatigue experiment, with a size of 88 μ m \times 60 μ m; area 2 has been studied in the uniaxial tensile experiment, with a size of $59 \,\mu\text{m} \times 54 \,\mu\text{m}$. The results are shown in Figs. 8 and 9.

From Figs. 8 and 9, the inhomogeneous strain distributions at the microscale can clearly be seen even within an individual grain. The studied areas here are so small that they can be regarded as points at the macroscale. However, the strain gradients in these micro-areas are so large that they can even be comparative to the strain gradients at the macroscale, which is totally unexpected. However, since our intention is to highlight a new technique, we will not provide detailed analysis of the result in this article. It will be investigated in depth in future work.



Figure 7 The studied regions on the specimen



Figure 8 Strain distribution under tensile loading

Additionally, it can be seen from Fig. 9 that there is a trend of accumulation for the residual strains. The trend accords with the accumulation of fatigue damage and could, therefore, be regarded as a reverse proof of the validity of this proposed technique.

Conclusion

By coupling DIC and digital microscopy, an experimental technique which can measure the deformation of the polycrystalline materials on the grain scale was proposed. The performances and limits of this technique were analyzed systematically and it was concluded that the precision of this technique is enough for the measurement on the grain scale. As an illustration, tests of uniaxial tensile and fatigue of a Ni-based alloy specimen were carried out and



Figure 9 Evolution of cumulative fatigue residual strain distribution

inhomogeneous strain distributions on the grain scale were obtained.

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