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Method for measuring nanoscale local strain in a dual phase steel using digital image correlation with nanodot patterns

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Local strain analyses in a dual phase steel were performed using a digital image correlation (DIC) method in association with Ag nanodot patterning. The optimum size of the nanodots for DIC was estimated from image error analyses. The nanodot patterning sheds light on the DIC for nanoscale local strain analysis due to its many advantages, such as high productivity, large patterning area, easy control of the dot size, and, furthermore, good adhesion.
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Understanding microstructure–property relationships is of great importance for material development and engineering applications, and hence has attracted the attention of material scientists and engineers. Especially, measurements of the localized deformation and strain are critical in understanding the role of microstructure in the deformation behavior of multiphase materials and composites. However, understanding the local heterogeneous deformation and strain partitioning behavior in multiphase or even single phase materials from conventional tensile/compressive tests, which measure total force and displacement applied to the specimens, is difficult. Therefore, many attempts have been made to comprehend the relationship between microstructure, deformation behavior, and mechanical properties using direct observation of local deformation [1–5].

Digital image correlation (DIC) is a visual method that employs (i) tracking of local marks on the surface of the specimen and (ii) image registration for accurate measurement of the strain from changes in the coordinates of the local marks during deformation. Recently use of the DIC method has been extended to study the local and global deformation and fracture behavior for in/ex situ local strain analyses [6–14]. Local quantitative analysis, which is the major advantage of the DIC method, is also useful for accurate verification of finite element method (FEM) simulations and modeling [15–18].

In order to accurately measure local coordinates in the DIC method clearly discernible marks (generally black and white patterns) on the deforming surface are essential. In addition to the clearly discernible marks, an appropriate combination of mark sizes and facets is important in DIC. A facet is a unit domain of the pattern used to calculate the strain. The facet size (with dimensions in pixels) affects the quality of the DIC results, and, hence, sufficiently small black and white patterns should exist in a facet. Since the quality of the patterns greatly influences the measured local strain values the preparation of good quality patterns is critical for accurate analysis of local strain.

For the last several years various marking methods, such as paint spraying, silicon microparticle dispersion [6], chemical etching [7–9], scratching [10], and lithography [7,11], have been proposed for pattern preparation for accurate local strain measurement of. There are several requirements for the marks: (i) they should be

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clearly discernible; (ii) the pattern size should be small in comparison with the grain size; (iii) the patterned area should be large; (iv) there should be good bonding with the matrix with no detachment during deformation; (v) there should be little damage to the specimen surface during patterning. However, marks on microscale particles have limitations for microscale strain measurements, and chemical etching can damage the surface of the specimen. In addition, chemical etching does not produce black and white patterns in the grain interior, and, hence, local deformation of each grain cannot be detected. Nano-imprint and focused ion beam lithography have benefits on the nanoscale level, however, the high cost the equipment and the time consuming processes involved are disadvantagous, and the productivity becomes very low when the pattern sizes is decreased and the patterning area increased.

Recently nanodots have been investigated for electronic and chemical devices [19–22]. Nanoscale speckle patterns can be produced over large areas by annealing a deposited film to a substrate. In particular, the size of the nanodots can be easily controlled by controlling the deposited film thickness, and the annealing temperature and time [19]. Our idea was to utilize speckle patterns in the DIC method where regular patterns are unnecessary. In this study Ag nanodot patterns were applied as position marks in local strain measurements. To assess the usefulness of DIC with nanodot patterning for nanoscale local strain measurement dual phase (DP) steels consisting of a soft ferrite matrix and islands of hard martensite as the secondary phase were quantitatively examined. The size of the nanodots was optimized from image error analyses, and accurate strain values were obtained using the best pattern. The Ag nanodot patterns produced satisfied the above mentioned requirements of superior patterns for local strain analysis.

For tensile testing dog bone-shaped specimens were prepared from the DP steel sheet. In the elongation region the dimensions of the tensile specimen were gage length 11.0 mm, width 2.0 mm, thickness 0.7 mm. The size of the martensite islands was ~1–3 μm. The surfaces of the tensile specimens were fine polished using a 0.25 μm diamond powder paste to remove surface defects and to decrease the surface roughness. To form the Ag nanodot patterns thin 25, 50, 75, and 150 Å thick Ag films were deposited on the tensile specimens. The films were grown at a base pressure of the order of 10^{-6} torr. Then the specimens were annealed using a rapid thermal annealing (RTA) system for 5 min at 300 °C under vacuum.

The sizes of the Ag nanodots and the dot spacings were measured by field emission variable pressure scanning electron microscopy (FEVP-SEM) using a Hitachi SU6600. For the image error analysis two images were taken sequentially without deformation at various magnifications. Different facet sizes and step sizes were also applied for the strain analyses, with the different sizes of the Ag nanodot patterns. Before the tensile tests the fiducial markers were constructed using a micro-Vickers hardness indenter to indicate the detection region after the tensile tests. The strain rate was 5.0×10^{-4} s^{-1} and a total of six stages were chosen for DIC during the tensile tests.

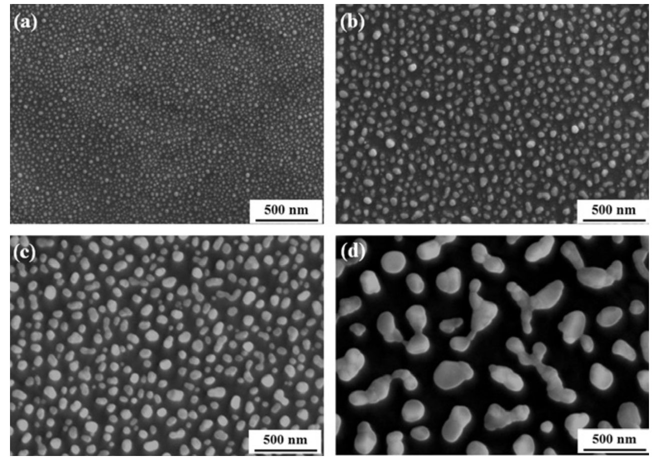

Figure 1. Ag nanodots of various patterns: (a) NP25; (b) NP50; (c) NP75; (d) NP150.

Table 1. Average dot sizes and spacings in the Ag nanodot patterns.

	NP25	NP50	NP75	NP150
Dot size (nm)	23	55	93	189
Dot spacing (nm)	20	46	70	135

Figure 1 shows various Ag nanodot patterns produced at different deposited film thicknesses. In the nanodot pattern terminology the numbers indicate the thickness of the deposited Ag films, i.e. the NP25 pattern was made from the 25 Å thick Ag film. The average dot sizes and spacings are listed in Table 1. The sizes of the Ag dots and the spacing dramatically increase as the deposition thickness increases under the same annealing conditions.

Since scanning electron microscopes produce images using an electron beam raster scan pattern, image obtained by FE-SEM can contain small errors in the pixel units. Errors in the raster scanned images should be minimized by qualitative analysis. Image errors can significantly influence local strain measurements as the DIC method calculates local strain in terms of pixel unit. The number of image errors varies with magnification, pattern size, and facet size. Thus error estimation was carried out in order to select the best pattern size for a given SEM magnification.

Sequential images without deformation were analyzed to verify the Ag nanodot DIC method. The average image errors, defined as “measured average strains in undeformed specimens”, were estimated from the major strain distributions in the NP75 specimen, as listed in Table 2. In the error analysis the step sizes were three-quarters of the facet size. The same error analysis was carried out for the other patterns (not shown here). The average image errors varied depending on the magnification and facet size, and are given in Table 2. The average image errors decreased as the facet size increased.

The DIC method was unusable at 2500 × and 20,000 × magnification for NP75 when the facet size

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