Experimental Investigation of Polycrystalline Material Deformation Based on a Grain Scale *

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We propose an experimental approach for investigation of the polycrystalline deformation behaviour at a grain scale. The technique is characterized by the joint application of micro material testing systems and the intragranular deformation analysis methods. It is attempting to map the deformation evolution at grain scale during the elastic and plastic deformations of polycrystalline specimens.

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Polycrystalline materials now are widely used in industry. Their various mechanical behaviours are determined by microstructures in the polycrystal, manufacture techniques, and external loading or environment interaction. Generally, by employing macroscopical experimental techniques, such as mechanical testing of materials, strain gauge and optical methods, only averaged mechanical behaviour can be obtained but it is difficult to predict the local deformations of the crystalline materials in a micro scale. In fact, such deformations lead to significant changes in the microstructure of the material, which causes substantial changes in the material properties vice versa. Therefore, the successful prediction of property changes is largely dependent on an accurate characterization of the deformation and the evolution of the material on the grain scale.

Although a substantial body of literature exists on the deformation measurement of polycrystalline materials, there are few reports on the characterization of individual grain surface deformation at the grain scale resulting from tensile deformation. Some previous work involving the Moiré method has been employed to investigate the mesodeformation of pure aluminium materials with the scale at several decimeters[1] and crick-tip asymptotic field[2] with millimeter fields. Another deformation or stress measuring methods were combined with a scanning electron microscope (SEM) and a grid to obtain the in-plane deformation of the polycrystalline materials, SEM and stereoimaging to measure microscopic strains within several grains that formed slip lines, macroscopical imaging and digital image correlation method to in-plane surface deformation measurement, and micro-Raman spectroscopy and x-ray to residual stress measurements.[3−9] However, to the deformation measurements, all of these studies are based on the millimeter-sized grains. Thus the macroscopical experimental method can be used to the deformation measurement of polycrystalline materials. The purpose of this article is to present the experimental approaches that combine with the micro material testing system, SEM and intragranular deformation analysis methods to investigate polycrystalline deformation behaviour at the grain scale during in-situ tensile testing.

The tested specimens were cut from a rolled sheet aluminium alloy, commercial grade LY12CZ, with thickness 1 mm and an average grain size about 40 μm. The tensile test specimens were prepared according to our testing stage's sizes and standard of ASTM. The geometries of two specimens are shown in Fig. 1(a). The one-edge notch was machined by an electrodischarge with length of $a = 0.34$ mm and radius of $r = 0.125$ mm. Both the specimens were mechanically polished to a mirror with fine diamond paste. Residual surface deformation was removed by first electrochemically anodizing the surface, followed by chemical stripping of the anodized layer. The final step for preparation of the specimens is chemical etching the polished surfaces to produce distinct grain structures. Figure 1(b) shows the grain surface image of one specimen.

Two tensile loading experiments were conducted, one on our homemade micro-vision hydraulic-electric-servo testing system (MHTS) and the other on a special loading stage for the scanning electron microscope (SEM). The MHTS can provide micro region deformation measurement with real-time and high spatial resolution (0.4 μm/pixel). A colour CCD and a digital image processing system contact with MHTS providing 8 or 12 bits truecolour image outputs. The hydraulic-electric servo system assures that the system runs stability and has a relatively large load range and high ability of force resolution. The SEM loading system

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allowed high-resolution images to be made at various stages of deformation so that the deformations could be determined subsequently using the image processing technique. The tensile specimen was placed inside the vacuum chamber of the SEM. The polished and etched surface was scanned by a focus electronic beam to obtain the microtexture information of the individual grains of the tensile specimen.

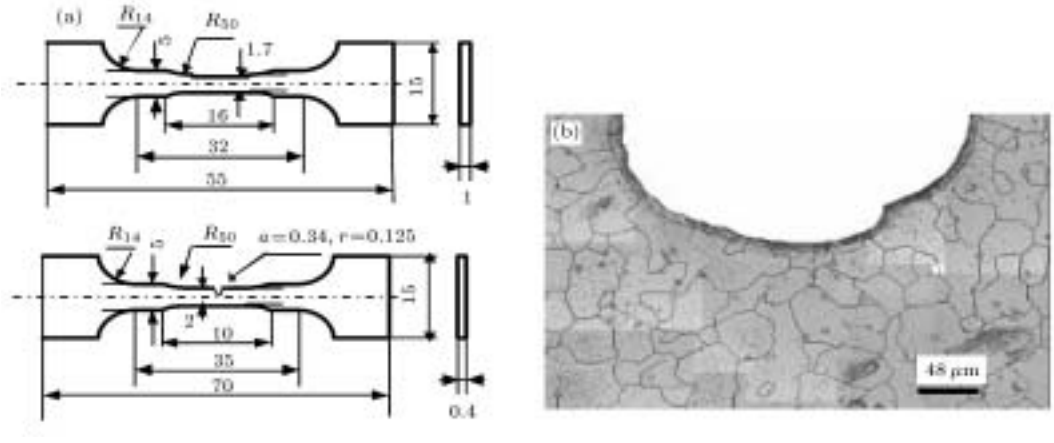


Fig. 1. (a) Specimens and (b) the grained surface in measurement.

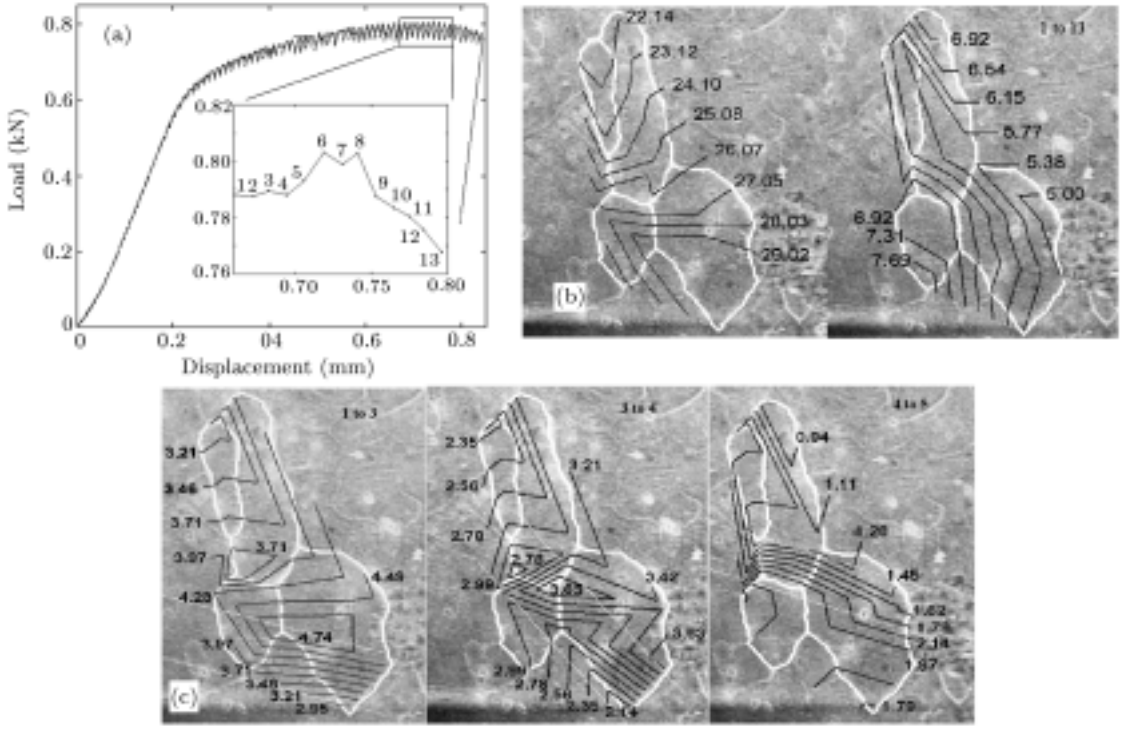


Fig. 2. Recognized grain boundaries and deformation fields under uniaxial tensile loading using SEM. (a) The load–displacement curve in the measurement and the tested load points, (b) the displacement fields in tensional and vertical directions, respectively, and (c) location deformations of the analysed grains (contour line unit: μm).

Deformations on the microscopic scale of the polycrystalline specimens were measured with sequence images taken before and after deformations. According to the properties of the sequence images captured by SEM and MHTS, different displacement procedures were adopted. To SEM images, the in-plane displacements are obtained by combining the recognition of the grain boundary and intragranular micro-nano speckle correlation. This is a digital image analysis method based on the recognition of geometrical changes of the grain boundaries and the correlation calculation of the intragranular micro-nano speckle patterns caused by chemical etching. A direct recognition of the grain boundary becomes impossible owing

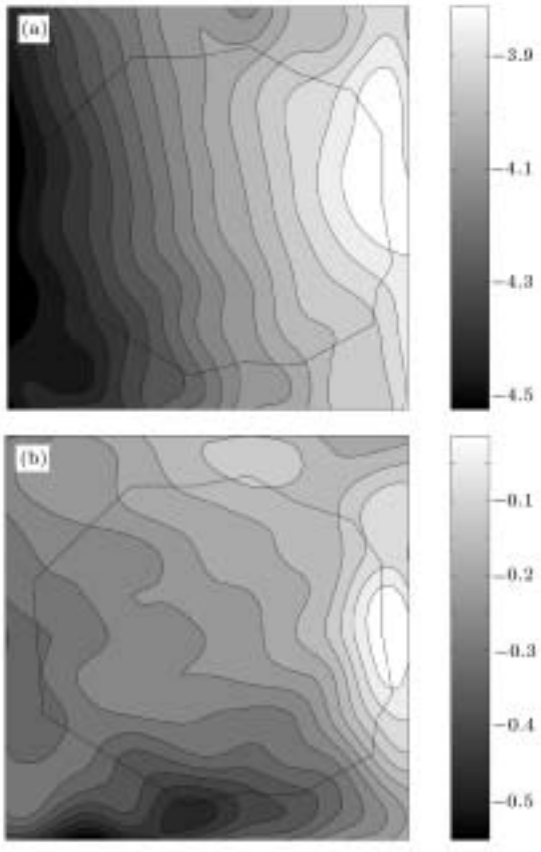


Fig. 3. Deformation evolution of an analysed grain near the crack area during tensile loading using MHTS (loads from 198 N to 201 N), (a) in tensile direction and (b) in vertical direction.

to the low contrast values for the grain boundaries in the deformed state. First, the image sharpening processing has been made to make the grain boundary clear and then the edge detection method was applied to obtain the grain boundary line. To ensure the boundary line continuum and one-pixel width, the segment, dilation and erosion operations have been employed to process the grain boundary lines subsequently. Because of the curving line form of the grain boundaries, the intragranular micro–nano speckle pattern correlation cannot provide displacement values in every position of the grain. The displacement values of these positions at any later stage are then estimated by a cubic spline interpolation using the grain boundary positions and the displacement values of the neighboring points. Thus whole-field deformations can be obtained in the analysed grains. Figure 2 shows the recognized grain boundaries and the deformation field under uniaxial tensile loading. Figure 2(a) shows the load displacement curve in the measurement and Fig. 2(b) shows the displacement contour lines. These contour values reveal that inhomogeneous deformations take place in the grain scale and the deformation localization occurs in different areas of the grains during loading. The slip regions are the sources of the plastic deformations (see Fig. 2(c)). Another result is that the deformation in tensile direction is dominant, but the magnitude of deformations in vertical direction to the tensile axis is also obvious at the grain scale.

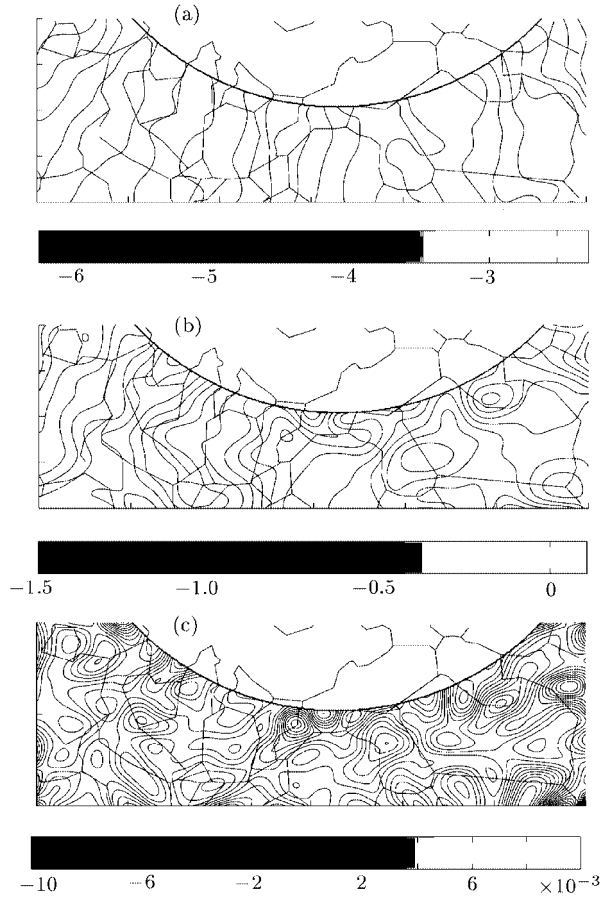


Fig. 4. Whole-field displacements in tensile (a) and vertical (b) directions and (c) the corresponding shearing strains near the crack area. The load level is from 198 N to 201 N.

The deformation measurement performed in the MHTS is relatively easy. MHTS can provide real-time imaging and good image contrast. There is not effect of unloading caused by waiting scanning imaging as in SEM testing. The grain deformations were obtained using grain boundary recognition and intragranular micro-nano speckle correlation combined with micro marker identifying method,[6] where the micro markers distributing on the surface of the grains are natural characteristics of the sample surface or the cavities caused by the chemical etching during the specimen preparation. Figure 3 presents an analysed grain deformation evolution near the crack during tensile loading. A comparatively homogeneous deformation was obtained in this grain under the loads from 198 N to 201 N. The whole-field displacements and their shearing strain distributions near the crack area are presented in Fig. 4. Besides the inhomogeneous defor-

mation caused by the crack geometry, the intragranular and intergranular deformations are nearly homogeneous in tensional direction, and inhomogeneous in the vertical direction. Two deformation bands produce at the right and left corners of the crack tip, where large numbers of slip bands occurs. The deformation localization also appears at the boundary of the grains and the tripe junction areas of the grains.

Based on the optical and scanning microscopic system, the deformation of the polycrystalline materials can be measured in-situ with real-time on the grain scale. The grain scale can be from several tens micrometers to sub-micro meters and the measured sensitivity of the grain deformation can reach to several tens of nanometres. This can provide deformation mechanism and evolution process of materials directly based on the grain scale measurement technique. Hence analysis of material failure and damage can be carried out on the level of the material microstructure.

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