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Intrinsic fracture toughness of bulk nanostructured Cu with nanoscale deformation twins

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ABSTRACT

A contactless video crack opening displacement gauging system based on digital image correlation technique was established to measure the load-line displacement of miniaturized fracture toughness samples precisely. The intrinsic fracture toughness and smoothly rising *R*-curve of bulk dynamic plastic deformation (DPD) Cu with nanotwin bundles embedded in nano-grained matrix were measured through elastic-plastic unloading compliance method using miniaturized side-grooved compact tension specimens. Nanotwin bundles exert positive bearings on enhancing the damage tolerance of the DPD Cu.

Refining grains to the nanometer scale provides attractive mechanical properties such as high strength/hardness and superior wear resistance [1,2], but it is generally conjectured that the high strength is inevitably accompanied by reduced ductility and poor damage tolerance [3–5]. This disadvantage would strongly limit the potential engineering applications of the nanostructured materials. In contrary to extensive studies on the ductility [6–8], reliable quantitative fracture mechanics evaluation of nanostructured materials is still rather limited so far [9–13]. This scarcity stems mainly from the fact that current existing techniques for preparing nanostructured materials cannot deliver sufficient specimen volume required for conventional fracture tests, thus the geometry-independent intrinsic fracture toughness is generally unavailable.

In order to evaluate the intrinsic fracture properties of nanostructured materials, the elastic-plastic fracture mechanics (EPFM) methods, which take account of crack tip plasticity, are usually applied for samples with miniaturized geometries [9,13,14]. Previous investigations have attempted to measure the elastic-plastic *J*-integral fracture toughness of bulk ultrafine-grained (UFG) metals prepared by severe plastic deformation [10–12]. The prerequisite of *J*-integral measurement is that the load-line displacement (LLD) must be precisely measured in order to compute the energy input to the tested sample. Conventionally, the LLD of standard fracture toughness specimen is easily determined by using an electron-mechanical clip-on COD gauge. However, such COD gauges are not applicable for the miniaturized nanostructured samples caused by their limited crack mouth spaces. How to accurately measure the LLD of miniaturized nanostructured materials becomes an unsolved challenge.

Dynamic plastic deformation (DPD) is an effective technique to prepare a novel bulk nanostructured Cu consisting of nano-grained matrix and embedded bundles of nanoscale deformation twins. The influence of the nanotwinned structure on the fracture behavior of the DPD Cu had been investigated in terms of single-edge notched bending tests by using miniaturized specimens [15,16]. The introduction of nanotwin bundles was found to increase the fracture toughness by promoting the formation of coarse/deep dimples [15,16]. However, because the linear elastic condition or the small-scale yielding condition was not satisfied, the intrinsic fracture toughness might be largely underrated.

In this study, the intrinsic fracture behavior of the DPD Cu is carefully evaluated by EPFM using a custom-designed contactless COD gauge with a high accuracy. Both the critical *J*-integral fracture toughness and crack growth resistance behavior are analyzed.

To accurately measure the LLD of miniaturized specimens, a contactless video crack opening displacement (VCOD) gauging system was developed based on digital image correlation (DIC) technique. The major components of the VCOD gauging include a Moritex telecentric optical lens, a high resolution CMOS camera and a computer with a custom-designed software for automatically capturing and analyzing images (Fig. 1a). The lens has a fixed magnification of 2× and a working distance of 65 mm. The resolution of the camera is 2592 × 1944 pixels, with a physical pixel size of 2.2 μm. Miniaturized

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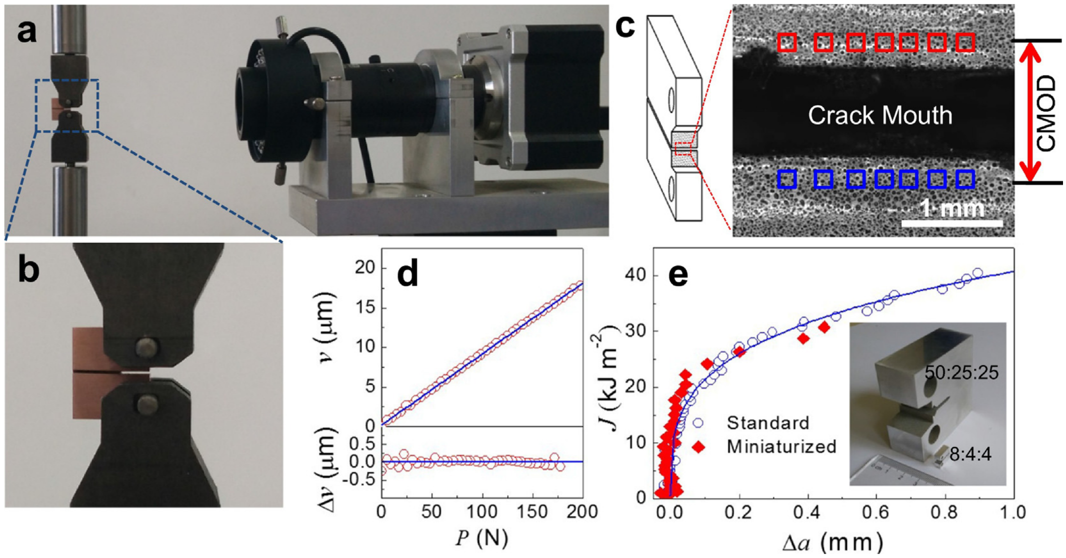

Fig. 1. (a) The VCOD gauging system based on digital image correlation (DIC) technique. (b) The compact tension (CT) specimen in (a). (c) Random speckle patterns at both sides of the CT specimen used for obtaining the load-line displacement (LLD). (d) The LLD versus force diagram measured by the VCOD. (e) The *J*-integral resistance (*J-R*) curves for miniaturized and standard specimens (inset image) tested by the VCOD and commercial clip-on COD gauge, respectively.

compact tension (CT) fracture toughness specimen with a width W of ~8.0 mm and a thickness B of ~4.0 mm was used, as shown in Fig. 1b. To mimic the measuring of conventional COD gauge, a random speckle pattern was firstly prepared by spraying black ink onto white paint background at the load plane, namely the common plane of the two pin hole centers (Fig. 1c). The original image before testing was captured as a reference image and several subsets (21 × 21 pixels) above and below the crack mouth were manually selected as tracing markers (Fig. 1c). Real-time images continuously captured by the digital camera during testing were automatically analyzed by the computer program to obtain relative displacements of the selected subsets with respect to the reference image. Since the pattern coincides with the load plane, the relative displacement representing the instantaneous crack mouth opening displacement (CMOD) can be regarded as the LLD without geometrical modification.

Fig. 1d shows a good linear relationship between the measured LLD and the imposed force during the elastic loading stage of a CT specimen. The slope representing the compliance of the specimen is 0.447 μm/N, which is quite close to the theoretical value (0.439 μm/N) calculated by the compliance equation of the CT specimen [14]. The LLD-force relationship not only demonstrates the accuracy (with a maximum deviation ≤0.3 μm) of the LLD measurement, but also proves that the measured compliance is sufficiently accurate to calculate the instantaneous crack length, which is also essential for EPFM evaluation. Therefore, the conventional single specimen technique can be employed with the stable crack advance being determined through measuring the instantaneous compliance by partially unloading the sample. Additionally, it has also been demonstrated that the contactless VCOD is sufficiently accurate to monitor crack extension of brittle materials which exhibit only limited crack tip opening displacement prior to complete fracture.

In order to verify the accuracy of the VCOD and the reliability of determining the intrinsic fracture resistance curve using a miniaturized specimen, comparison tests are performed on a commercial 7075-T651 aluminum alloy (with an average grain size of 24 ± 3 μm and a yield strength of 451 ± 4 MPa). Miniaturized specimens with a side-groove of 10%B (required for obtained plane-strain toughness, as discussed below) based on the VCOD and standard specimens ($W =$ ~50.0 mm, $B =$ ~25.0 mm, $a_0 =$ ~25 mm, non-side-grooved) with a commercial clip-on COD gauge, as shown in the inset of Fig. 1e, were tested following the procedure in accordance with ASTM E1820 [14]. In spite of the large difference in the specimen geometries, coincident curves of *J*-integral in Fig. 1e as a function of crack extension can be obtained. The comparison tests verify that the fracture toughness measured by miniaturized specimens is valid and the results are comparable to the conventional approaches.

Based on the contactless VCOD gauging system, the fracture behavior of the DPD Cu was investigated using the single specimen technique following the standard procedure of ASTM E1820 [14]. In order to obtain a sharp initial crack front, the CT specimens were first notched to a depth of ~3.2 mm by electrical discharge machining, and then fatigue pre-cracked in a tension-tension mode at a frequency of 2 Hz until an initial total crack length a_0 of ~4.0 mm was reached. The crack plane normal is in the tangential direction of the DPD Cu disk while the expected direction of crack propagation is coincident with the radial direction. The specimens were loaded under displacement control at a speed of about 0.1 mm/min to stimulate crack extension. With the recorded force, LLD measured by the VCOD and instantaneous compliance, the *J*-integral as a function of crack extension can be determined by the standard procedure in ASTM E1820 [14].

Fig. 2 shows the transverse microstructure of the DPD Cu with a total accumulated true strain of 2.0. The true strain $\varepsilon = \ln(h_i/h_f)$, where h_i and h_f are the initial and final sample thicknesses, respectively. A mixed microstructure with bundles of nanoscale deformation twins (region A) embedded in a matrix of nano-grains (region B) was obtained. Statistics show that the volume fraction of the nanotwin bundles is ~35%, and the average twin thickness is 47 ± 3 nm (Fig. 2b). Fig. 2c displays that the nano-grains are slightly elongated with a mean transverse size of 69 ± 4 nm. Uniaxial tensile tests at room temperature show that the 0.2% offset yield strength (σ_{ys}) and the ultimate tensile strength (σ_{uts}) of the DPD Cu are 595 ± 8 MPa and 629 ± 11 MPa, respectively, while the elongation to failure (δ_f) is 5.9 ± 0.5%, consistent with Ref. [16].

The representative force versus LLD curve of DPD Cu is shown in Fig. 3a. After reaching the peak value, the force decreases steadily with increasing LLD, a consequence of stable crack growth. The computed *J*-integral as a function of crack extension Δa, namely the *J*-integral resistance (*J-R*) curve, is displayed in Fig. 3b. The *J*-integral monotonically increases as crack advancing, apparently manifesting a rising *J-R* curve behavior, which is in agreement with the measurements of other miniaturized nanostructured specimens [9,13]. However, the overview micrograph (Fig. 3e) of the final crack front delineated by