A brittle-fracture methodology for three-dimensional visualization of ductile deformation micromechanisms

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An improved experimental methodology is developed and successfully evaluated to visualize deformation-induced microevents in ductile sheet metal. This easy-to-use methodology consists in a well-controlled brittle separation of samples previously deformed in a ductile manner, whereby a coupled-analysis of the two fracture surfaces permits three-dimensional features to be recombined from the ductile predeformation. The results obtained show a wealth of information regarding grain deformations, microvoid nucleation sites and growth mechanisms, and other microevents induced by ductile deformation.

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In 1900, Ewing and Rosenhain [1] stated “The microscopic study of metals was initiated by Sorby, and has been pursued by Arnold, Behrens, Charpy, Chernoff, Howe, Martens, Osmond, Roberts-Austen, Sauvreur, Stead, Wedding, Werth, and others.” About 100 years later, this list would be exponentially longer, revealing the importance of linking a metal’s microstructure to its mechanical, chemical and physical properties. A major part of the progress made can be attributed to the new and steadily improving range of microscopic techniques used to examine these microstructures. Meanwhile, the basic principles of metallographic specimen preparation, i.e. removal of the material adjacent to the layer of interest, have hardly evolved. This leaves a number of fundamental complications in the analysis of complex microphenomena in modern metals (e.g. advanced high-strength steels) unresolved, even though cutting-edge, fully automated material removal systems are nowadays available.

These complications originate from the fact that ductile damage micromechanisms (e.g. void nucleation, growth and coalescence) are masked since they are intrinsically located inside the metal (i.e. do not reveal essential details at the surface). The common approach in analyzing these micro (and increasingly nano) constituents consists in the advanced use of several experimental techniques (mechanical polishing, electropolishing, etching, etc.) for material removal.1 This can be achieved with acceptable precision with each of the techniques; however, reports in the literature show that the revealed microstructure is also modified, and in some cases deformed significantly [3–6]. While mechanical polishing causes subsurface hardening [3–5] and smearing effects [6], electropolishing and etching processes by definition round off sharp edges (edges of the specimen, microvoids, etc.), effectively altering the dimensions of the microstructural features. These adverse effects are well known in the metallography community, and hence lengthy specimen preparation protocols are generally designed to reduce surface manipulation effects to acceptable levels, and carried out in different viewing directions (i.e. in order to visualize the three-dimensional (3-D) nature of the microstructure). Such procedures, however, are impractical, expensive and not fully conclusive, as it remains experimentally difficult to assess the influence of deformation, alteration or modification due to a preparation process. Moreover,

1 Ductilely deformed microstructures can also be visualized without material removal by the use of radiography techniques (e.g. X-ray tomography [2]), which allow bulk analysis with high-energy penetrating radiation. However, limitations regarding both practical (availability, time, costs, expertise, etc.) and technical aspects (resolution, specimen size, etc.) restrict their applicability for a large class of problems.

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regardless of the type of the technique used, the material that is removed is “lost”, i.e. no data can be obtained from the removed part of the examined microstructure.

Many of the problems mentioned above have been solved with the development of the focused ion beam (FIB) technology, which allows 3-D analysis of microstructures (and crystal orientations) with almost no manipulation. However, these measurements are by no means practical, i.e. in most cases only very limited areas (or volumes) can be investigated due to time or economic constraints. In the case of ductile deformation analysis, for example, these limitations make it significantly difficult to get an overview of the multitude of damage mechanisms that are acting.

In this short paper, we propose and explore an experimental methodology that overcomes the above-mentioned difficulties, in a simple, fast and cheap but precise manner, allowing the visualization of the 3-D microstructure. As a first step, we “open-up” fractured tensile test specimens making use of a well-controlled brittle-fracture separation technique which hardly alters the large deformations and damage traces left behind by the previous ductile deformation. The advantage of using brittle fracture to open up inner surfaces has been recognized previously (for the investigation of critical void nucleation strains in a C–Mn structural steel [8]); however, this is the first time that the strength of the concept is demonstrated in all its facets. Upon separation, the two surfaces are studied by scanning electron microscopy (SEM) (or with surface profilometry tools) to reveal the original 3-D ductile deformation microstructure by correlating the observations from both surfaces.

The advantage of the brittle-fracture approach depends on two critical factors: the control of the crack path and the absence of plastic deformation upon opening. Controlling the brittle fracture in a preferential cross-section may be challenging due to the nature of brittle-crack growth. A dedicated setup is developed to trigger the brittle fracture of, for example, a previously fractured tensile test specimen (Fig. 1b) in any cross-section of interest. To keep the propagation of the crack fully brittle, specimens are opened-up under conditions that inhibit ductile deformation [10], i.e. high stress concentrations and strain rate, and low temperature.

The experimental procedure starts with a (ductile) mechanical test whereby the specimen is loaded up to complete ductile failure (Fig. 1a and b). In order to prove the principles, uniaxial tension tests are used even though the methodology can be extended for other test specimens via some minor adjustments. A scratch is made on the surface of one of the fractured parts (Fig. 1c). The location of this scratch can be chosen at any angle to the test direction, depending on the cross-section of interest. The specimen is next placed in clamps specially designed to maximize the stress intensification in the scratch, and cooled down in a container of liquid nitrogen well below the ductile-to-brittle transition temperature. The sample is then fractured into two parts (Fig. 1d) by using a conventional Charpy-impact test hammer. Microstructure and topography analyses are carried out simultaneously on both sides of the brittle-fractured specimen, to visualize the 3-D microstructural characteristics of the previously ductile deformed test sample.

To assess the applicability of the methodology to different microstructures, four different steels are studied: interstitial-free (IF), rephosphorized IF, dual-phase and low-carbon steel. Three samples from each material are analyzed to check the reproducibility. In all of the metals investigated, complete brittle fracture is achieved and fracture always occurred along the scratch. In this paper, however, the focus is on the analysis for an IF steel. IF steel is a perfect case study, since it is not trivial to achieve brittle fracture in such a ductile material. Furthermore, the use of conventional polishing methodologies for this material lead to significant void smearing effects (due to its high ductility), making the visualization of the true nature of microvoid growth in this material a challenging task.

Let us start with some general observations regarding the brittle-crack propagation. Interestingly, when the opened surfaces are examined, a smooth transition is

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2 It should be noted that significant amounts of Ga ions are embedded in the matrix of the metal being processed (e.g. [7]).

3 A recent overview over ductile-to-brittle transition and related microevents is provided in Ref. [9].

4 Additional information on the setup can be found in Ref. [11].
observed in the brittle-fracture mode along the tensile axis, i.e. as a function of the deformation history. While the brittle crack is dominantly transgranular away from the neck (i.e. at relatively low local pre-strains), the fracture is dominantly intergranular close to the ductile fracture surface of the tensile test sample (along the elongated grains in the neck (Fig. 1e)). This transition is more pronounced in more formable steels (IF, plain carbon steel), compared to less formable steels (DP600, stainless steel, IF-r).

The brittle-fracture approach provides a clear visualization of the prior (severely deformed) ductile microstructures, as shown in Figure 2 in which SEM images from the neck regions are compared for different preparation techniques. The conventional strategies, obviously, lead to unrepresentative surface finishes that reveal little microstructural information. Mechanical polishing (Fig. 2a) provides a nice representation of the fracture surface morphology (i.e. “cone”), but smearing effects limit void analyses, and observation of grains or grain boundaries is not possible. Electropolishing (Fig. 2b) removes smearing effects, whereby more voids are visible, but rounding effects change the morphology of the cross-section and these voids, as well as of the fracture surface. Grains are poorly visible, making them difficult to analyze. Etching (Fig. 2c) makes grains somewhat more visible, but voids are also affected by etching, or even completely removed. With the brittle-fracture approach (Fig. 2d), on the other hand, the specimen is separated through the grain boundaries, allowing a clear analysis of elongated grains in three dimensions, while keeping the cross-section and fracture surface morphologies undistorted, to the extent that it can be checked.

This 3-D visualization also allows for a clear analysis of void nucleation sites (e.g. the microvoids nucleated at grain boundary triple points in Fig. 3a) due to the unique property of the methodology allowing both surfaces to be available for observation (Fig. 1d). This is another important advantage of the brittle-fracture-based methodology, i.e. the separation is achieved without any noticeable plastic deformation, leading to true 3-D morphologies being obtained by assembling the micrographs of both sides of a grain (Fig. 3a), a microvoid (Fig. 3b) or other microstructural constituents, either qualitatively (e.g. by SEM observation) or quantitatively (e.g. by using confocal microscopy or atomic force microscopy). The nearly perfect complementary nature of the features in both surfaces in Figure 3 supports the hypothesis on the absence of plastic deformation in the separation of these two surfaces, enabling quantitative (three-dimensional) measurements of single voids or cracks and (area-averaged) surface fractions of damage. Note that the brittle crack tends to propagate along the internal surfaces of highest damage concentration, which are of prime importance to the failure of a deforming ductile sheet.

Furthermore, important information about ductile damage evolution and accumulation mechanisms can be gathered upon the examination of complementary surfaces. Such an observation is shown in Figure 4, which is an SEM image from the neck region of a ductile fractured tensile test specimen (fracture surface towards right) of an IF steel. Note that a portion of the SEM im-

![Figure 2](image_url)

**Figure 2.** Comparison of different specimen preparation methodologies to analyze ductile deformation. Neck regions of fractured tensile test samples examined in SEM following (a) mechanical polishing, (b) electropolishing, (c) etching and (d) brittle-fracture methodology.

![Figure 3](image_url)

**Figure 3.** Analyses on both complementary brittle-fractured surfaces allow the reconstruction of the true 3-D morphology of deformation-induced microstructures. (a) Elongated grains in the neck region of a tensile test sample, showing void nucleation at grain boundary triple points. (b) Void growth with deformation leads to the partial coalescence of three microvoids. Note that both bottom images are flipped vertically.

![Figure 4](image_url)

**Figure 4.** Void nucleation (i), growth (i–ii) and coalescence (ii–iii) observed together in one image approaching the neck of a tensile test sample. Inset images show enlarged views of these steps. The rest of the image is shadowed for visualization.
age is highlighted and the high-magnification inset images show different snapshots of the deformation process leading to fracture. The figure shows that microvoid nucleation, growth and coalescence take place at the triple junctions of the elongated grains. Nucleation starts at relatively lower local strains (i.e. further away from the fracture surface) as seen in Figure 4i. At higher local strains (i.e. closer to the fracture surface), these microvoids grow such that they begin to “touch” each other (Fig. 4ii). At even higher strains, these microvoids coalesce to form larger voids (Fig. 4iii), leading to fracture.

In summary, the proposed brittle-fracture-based methodology provides significantly more details needed for microstructure characterization, compared to traditional techniques. More data can be obtained from a given cross-section of interest (which can be chosen freely), as both separated surfaces can be examined comparatively. This data presents a significantly higher quality (in the sense that it is more representative of the true microstructure) since preparation-induced deformation is prevented. Finally, these achievements are obtained through an economic and practical methodology that is highly suited for practical and even industrial applications.

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