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Confocal laser scanning microscopy: The technique for quantitative fractographic analysis

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ABSTRACT

Brittle and ductile fracture surfaces of low-carbon steel specimens having different grain sizes were investigated by means of confocal laser scanning microscopy in conjunction with conventional scanning electron microscopy and electron backscattered diffraction. The fracture surfaces were quantitatively characterized in terms of the areal surface roughness and normalized surface area. It was demonstrated that the normalized fracture surface area rather than areal roughness can be used as a measure of fracture surface ductility. The misorientation between cleavage facets and their diameters were evaluated statistically. Good agreement was found between characteristics of fracture surface elements and the underlying microstructure.

1. Introduction

Fractography is indispensable for the study of fundamental fracture mechanisms in failure analysis. Reflecting a final crack path, the fracture topology provides a clue to understanding underlying fracture mechanisms and weakest elements in the microstructure at given loading conditions. It has long been recognized that the analysis of the fracture surface helps to improve the microstructure and its resistance to fracture [1,2]. The success of this approach relies heavily on the accuracy of acquisition and correctness of interpretation of fractographic data. Various fractography techniques have been devised to gain information from fracture surfaces [1–4]. These include many useful procedures and relationships to assess the geometric characteristics of fracture. Challenges faced by the classical quantitative fractography are reviewed in [2–4]. As a matter of fact, in view of the complexity of the fracture surfaces the vast majority of fractographic studies are performed only qualitatively [5–7] even if observations are taken on different scales. Thus, the objectivity of fracture surface examination rests on the skills and experience of an analyst. The problem is particularly acute because the terms "ductility" and "brittleness" – the key engineering mechanical properties – are vaguely defined with regard to the fracture surface topography. The latter is commonly described through the roughness, true fracture surface areas, true facet sizes, area fractions, spacing etc. (if not fully qualitatively in terms of optical reflectivity), which has yet to be related to ductility or brittleness. In other words, a reliable quantitative measure characterizing explicitly the ductility or brittleness of the fracture surface is still missing. The ductility of the fracture surface is often expressed through the extent of plastic deformation (e.g. in the dimpled relief) and the associated stored dislocation density in the underlying microstructure. A common disadvantage of all methods proposed for the measurement of the extent of plastic deformation, including those based on the X-ray diffraction [8–11],

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transmission electron microscopy (TEM) and electron back-scattered diffraction (EBSD) techniques [12] is that they are destructive, laborious and cannot be used for routine inspections.

One of the main reasons impeding the quantitative fractographic analysis is the deficit of convenient instrument capable of providing precise 3D topographic data. The conventional scanning electron microscopes (SEM), which are commonly used, in fractographic studies produce only the 2D images representing the projection of the fracture surface onto the X-Y plane. Unlike the microstructure, which can be quite comprehensively characterized by 2D metallographic, SEM or TEM images, a fracture surface is fundamentally a 3D object and three coordinates are naturally required for the adequate description of surface topology. Therefore, departing from limited information supplied by 2D trace analysis to determine crystallographic signatures of fracture, the research in fractography evolved towards the analysis of 3D surfaces. Central to this undertaking is the ability to reconstruct the 3D surface topology with high accuracy. Confocal laser scanning microscopy (CLSM) has been devised as a tool of choice for this purpose. Nowadays CLSM microscopes are the versatile high-precision 3D-measuring systems enabling digital reconstruction of an object surface topography on different scales which are commonly seen on rough, complex surfaces with marked topographic variability. Having detailed topographic data one can get a direct access to roughness, true surface area, volumes of caverns, angles between constituting elements of the surface, etc. Unique capabilities of CLSM reviewed briefly below have enabled a broad variety of applications in materials science. These include, but not limited to, examination of wear damage [15,16], topography of coatings [15], roughness characterization [17], corrosion [18,19] and fractography [17,18,20–23], etc. The present study is focused on application of CLSM to quantitative fractographic analysis with the emphasis placed on the methodological aspects of CLSM application to case studies.

Over the years, the SEM stereoimaging technique was the dominant method of quantitative 3D fractography. Despite the persistent enhancement [24,25] and some documented successful applications [26,27], no widespread usage of SEM stereoimaging is seen in routine fractography practice up to date. Among the other techniques capable of 3D reconstruction of surface topography the CLSM seems to be the most suitable for fractographic purposes. As opposes to the white light interferometry and atom force microscopy, CLSM permits 3D imaging of very rough surfaces with regions oriented at high angles to the optical axis of the microscope. Since the CLSM operates in air and there are no special requirements for specimens and their surfaces the fractographic examination of any material can be performed in a reasonably short time. Although the resolution of CLSM is lower comparing with SEM, it can serve as alternative or supplement instrument for the express 3D fracture surface analysis at a wide range of magnifications. In the present paper we endeavor to demonstrate the capabilities and discuss main methodological aspects and limitations of CLSM in qualitative and quantitative characterization of complex fracture surfaces. For this purpose two classical well-documented types of fracture surfaces – ductile dimpled and brittle cleavage – were qualitatively and quantitatively examined by CLSM. The SEM and EBSD techniques were used as the reference methods for verification of obtained results. The more specific application of certain methodological aspects described in details in this study was performed in our previous study devoted to fracture surface features of the hydrogen embrittled steel [22].

2. Experimental

2.1. Materials and testing

The commercial hot-rolled low carbon steel S235JR was used for tensile tests in the present study. The smooth flat specimens with the gage dimensions $15 \times 4 \times 1.7$ mm^3 were cut along the rolling direction by spark erosion. They were then mechanically polished and some of them were annealed in vacuum at 850 °C and at 950 °C for 30 min and furnace cooled. The uniaxial tensile tests were performed in air at 25 °C as well as in liquid nitrogen at −196 °C using the universal testing machine H50KT (Tinius Olsen).

The microstructure of the specimens before tensile testing was examined by the confocal laser scanning microscope Lext OLS4000 (Olympus) and by the EBSD technique. The EBSD patterns were obtained and processed by the EDAX/TSL facilities and software installed in the Zeiss SIGMA field emission scanning electron microscope. To investigate the specimen's microstructure just beneath the fracture surface, the microsection normal to the fracture surface was prepared and analyzed by EBSD.

2.2. Principles of confocal laser scanning microscopy

Confocal microscopy was pioneered by Marvin Minsky who patented the first confocal microscope in 1961 [28]. The main feature of that device being compared to the conventional optical microscope was the confocal optical scheme utilized for eliminating the unwanted out-of-focus scattered light and enhancing the contrast of the in-focus region of an object. This effect was enabled by a pin-hole aperture introduced between the objective lens and the light detector. With point-by-point illumination of the specimen and the movable object stage one was able to obtain a sharply focused optical image of the in-focus region of an object – the optical slice. Since the time of inception, the confocal microscopes have evolved and improved dramatically owing to and synergistic coupling of precision mechanics and advanced computing technologies [15]. The use of the short-wavelength laser as a light source allowed enhancing the brightness and lateral resolution of the confocal images. The movable stage was replaced by the lateral scanning laser beam system in order to increase the imaging