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Quantitative characterization of cleavage and hydrogen-assisted quasi-cleavage fracture surfaces with the use of confocal laser scanning microscopy

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ABSTRACT

"True" cleavage (TC) and quasi-cleavage (QC) fracture surfaces of low-carbon steel specimens tested in liquid nitrogen and after hydrogen charging respectively were investigated by quantitative confocal laser scanning microscopy (CLSM) and conventional scanning electron microscopy (SEM) with electron-backscattered diffraction (EBSD). Topological and crystallographic features of the TC fracture surface are found in good agreement with the generally accepted cleavage mechanism: TC facets diameters correspond to those of grains; the crack path strictly follows the crystallographic orientation of grains and the most of the cleavage cracks are parallel to {100} planes. On the 2D SEM images, the QC facets appeared resembling the TC ones in terms of river line patterns, shapes and sizes. However, the substantial differences between the topography of these two kinds of fracture surfaces were revealed by 3D CLSM: the average misorientation angle between QC facets and the roughness of the QC fracture surface were much lower than those measured for TC. It is demonstrated that all these features are attributed to the specific fracture mechanism operating during hydrogen-assisted cracking.

1. Introduction

"True" cleavage (TC) and quasi-cleavage (QC) represent the most common fracture modes of metals and alloys along with brittle intergranular cracking and ductile microvoid coalescence [1]. The TC is referred to as the brittle transgranular fracture mode, which is usually caused by dynamic loading or loading at low temperatures. Cleavage cracks propagate transgranularly with little or no plastic deformation in the well-defined low-index crystallographic planes. The faceted crack-path morphology with river line patterns is characteristic of cleavage [1–4]. In iron and ferritic steels, the cleavage occurs primarily along the {100} cubic planes [2,4,5], though cleavage along the {011} and other low-index planes have also been noticed [4,6]. In contrast to the TC, the term "quasi-cleavage" or "cleavage-like fracture" is less defined and is related rather to a fracture surface appearance than to a certain fracture mechanism. Therefore, many different transgranular fracture surfaces are broadly associated with QC though the underlying mechanisms of their formation can be fundamentally different. Various types of QC fracture surfaces are found in steels with ferritic [7], bainitic [8], martensitic [9] microstructures. Transgranular fracture surfaces of hydrogen embrittled steels are also often referred to as QC [10–14]. As of today the experimental database covering the nature and features of QC fracture surfaces is far from being complete. Particularly, the QC mechanism related to the so-called "fisheyes" , which are the specific round-shape defects found commonly on fracture surfaces of mild ferritic steels saturated with hydrogen, has been just scarcely studied [15–18]. Moreover, as it will be shown below, the QC facets on the fisheye surface visually resemble the TC facets in terms of size, shape and appearance of river line patterns. Nevertheless, the difference in the nature of these two fracture modes is evident. Quantitative characterization of fracture surface topography is vital for unambiguous identification of fracture mechanisms. However, the quantitative parameters such as misorientation angles between the facets, area and roughness of fisheyes QC fracture surface describing fracture surface morphology [19] are still vaguely known. *Thus, using the quantitative surface characterization techniques, the objective of this study is to establish a robust criterion capable of distinguishing the quasi-cleavage fracture surfaces of the hydrogen-induced fisheye defects from the 'true' cleavage fracture surfaces formed as a result of low-temperature embrittlement in a low carbon steel.*

With the advent of analytic microscopy and high resolution

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profilometry, quantitative fractography has become a practical methodology in materials science. The quantitative analysis of deformed or fractured microstructures based on 3d-reconstructed fractographic images allows extracting explicit information about microstructural mechanisms involved, which is not accessible otherwise. Despite a wide range of modern techniques, which are readily available nowadays for precise surface topography characterization and which include (but not limited to) confocal laser scanning microscopy (CLSM), white light interferometry, atomic force microscopy, etc., quantitative analysis of fracture surfaces is still challenging. Most fractographic studies are limited to qualitative description of the visual fracture surface features. The conclusions are therefore subjective even if observations are performed on different scales. For the purposes of quantitative fractography, the CLSM technique takes advantage of high lateral and axial resolution imaging of rough surfaces with large differences in heights (or large variance of the surface profile) [20–23]. We endeavor to demonstrate that the combination of the CLSM and the electron backscattered diffraction (EBSD) techniques in fractography paves a new avenue for quantitative characterization of complex fracture surfaces.

2. Experimental

The commercial hot-rolled low carbon steel S235JR with the chemical composition shown in Table 1 was used in this study. The smooth flat specimens for tensile tests 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, annealed in vacuum at 950 °C for 30 min and furnace cooled. Electrolytic hydrogen charging was performed in the 5% H_2SO_4 + 1.5 g/l thiourea solution during 1 h at constant current density of 100 mA/cm^2.

The uniaxial tensile tests were performed at $5 \times 10^{-3}\ s^{-1}$ initial strain rate using a screw-driven H50KT (Tinius Olsen) testing machine. To promote different fracture mechanisms of interest, three sets of specimens were tested under three different conditions:

i) not-charged specimens were tested in air at room temperature to observe an ordinary ductile fracture relief for reference;
ii) not-charged specimens were tested in liquid nitrogen at −196 °C to activate the TC fracture mode;
iii) hydrogen charged specimens were tested in air at room temperature to stimulate the QC fracture mode.

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 orientation image microscopy software installed in the Zeiss SIGMA field emission scanning electron microscope (SEM). To investigate the microstructure just beneath the fracture surface the microsection normal to the fracture surface was prepared and then analyzed by EBSD.

The fracture surfaces of the specimens were investigated using both the CLSM and SEM.

For the analysis of TC and QC facets, several 256×256 μm regions of the fracture surfaces were scanned by CLSM with the MPlanApoN50xLEXT ($\times 1000$ magnification) objective lens at 1 μm step height. More than 500 TC and 400 QC facets were analyzed. For the CLSM imaging the tensile axis of the specimen was aligned with the Z-axis of the microscope, i.e. the plane of all images is perpendicular to the normal stress direction. In order to reduce the noise and other artifacts after acquisition the images were processed and rectified with the "pre-measurement" filter built in the Lext OLS4000 software package. The same software was used for evaluation of the fracture surface roughness in terms of the 3D areal surface topography parameters Sa – arithmetical mean height of the surface and Sq – root mean square height of the surface, in accordance with ISO 25,178.

The analysis of the facet area and misorientation angles between the facets was performed with the aid of computerized procedures developed at NUST "MISIS" (Moscow, Russia). This software operates with the 2-D jpeg images of fracture surfaces and with the corresponding 3-D maps containing x, y, z coordinates for every pixel of these images. Required input data are exported from the CLSM Lext software. Using the graphical user interface, the operator distinguishes the facets on the image and outlines them with a polyline tool. Then the program finds the pixels from the outlined area in the corresponding 3-D height map, approximates this part of the surface with a plane and calculates the coefficients of the equation of the plane. This plane is considered as the facet plane. When the coefficients of the equation of the plane are known for every outlined facet, the inclination angles of the facets to the image plane as well as the misorientation angles between adjacent facets can be easily calculated.

We should notice that the term "facet" does not have a clear definition commonly accepted in the scientific community. Therefore, it is still methodologically quite difficult to formulate a non-supervised automatic procedure for determining the facet boundaries. Hence, for the purposes of this work, we determine the facets boundaries manually from visual inspection of the images. Generally, the boundary between two facets is defined as a clearly visible line separating two relatively planar regions of the fracture surface with different, but uniform contrast in the microscopy images; the shape of facets should bear resemblance with that of respective grains. The line where the river pattern changes its direction also constitutes the boundary between two facets. One can notice that the facets corresponding to two different grains are separated by a curved line reproducing the shape of the grain boundary while the facets within a single grain are separated by a straight line or a polyline. Using this method, the facets corresponding to a given single grain were labeled with identical figures. In the further analysis, the angles between these facets belonging to the same grain were not taken into account during calculations of misorientation distributions. For comparison with the grain size, the areas of these facets were summed up.

3. Results

3.1. Initial microstructure

After vacuum annealing the specimens show a ferrite-perlite microstructure, Fig. 1a, with equiaxed ferritic grain shape, Fig. 1a and b. Smaller pearlitic grains are extended along the ferritic

Table 1
Chemical composition of the steel S235JR.

Element	C	Cu	Si	Mn	P	S	Cr	Ni	Al	Fe
wt. (%)	0.129	0.067	0.02	0.42	0.019	0.015	0.05	0.007	0.028	Balance