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Assessing Fracture Surface Ductility by Confocal Laser Scanning Microscopy

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Abstract

Hitherto there is no generally accepted quantitative parameter which, on the one hand, would reliably characterize the ductility of the whole fracture surface, and, on the other hand, could be relatively quickly measured. This circumstance substantially affects the objectivity of the fractographic analysis which effectiveness is still strongly dependent on the experience and skills of an expert. Recent studies showed that the value of the normalized fracture surface area *Rs* can serve as the measure of the fracture surface ductility. This parameter can be evaluated by the quantitative confocal laser scanning microscopy (CLSM). In the present study we investigated the *Rs* value for the fracture surfaces of the low carbon steel specimens tested in the temperature range from 200 to -196 °C where the steel undergoes ductile-to-brittle transition accompanied by the alternation of the fracture mode from ductile to brittle. The temperature dependence of the *Rs* value is found to have a sigmoidal shape with the sharp drop in the range from 100 to -100 °C. It is demonstrated that the *Rs* is strongly correlated with the fracture surface appearance: the Rs decreases concurrently with increasing brittleness of the fracture surface.

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Keywords: confocal laser scanning microscopy; fracture surface; quantitative fractography; normalized surface area; low-carbon steel.

1. Introduction

A fracture surface of a material tells a lot about causes and mechanisms that lay in the basis of fracture Gilbertson and Zipp (1981), Strauss and Cullen (1978). This information can be used for improvement of the microstructure as well as mechanical and service properties of the material. Thus, the development of fractographic techniques with enhanced analytic capability and practical effectiveness is still topical and challenging.

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One of the most common routine operations in the fractographic analysis is the assessment of the fracture surface ductility of metallic parts and specimens failed during service or laboratory mechanical tests. This procedure includes visual and/or microscopic examination of the fracture surface followed by the ranking of the fracture surface ductility. However the decision regarding the extent of ductility (or brittleness) of a fracture surface is based on the number of mostly qualitative fractographic features such as presence of dimples or facets, shear lips, cross-section reduction, etc., assessed "by eyes". Thereby, the final verdict is virtually always subjective and is strongly dependent on the experience and skills of the expert.

Objectivity of the fractographic analysis can be achieved through the quantitative description of the fracture surface which is still challenging. Up to now no one generally accepted quantitative parameter can reliably reflect the ductility of the whole fracture surface on the one hand, and can be relatively quickly measured by any non-supervised technique. The conventional fractographic methods such as light or scanning electron microscopy (SEM) produce only 2D images projecting of the actual 3D fracture surface onto a plane. A fracture surface is naturally a 3D object. Therefore, its comprehensive description requires precise values of all three coordinates for every point of the surface. Advances in the confocal laser scanning microscopy (CLSM) during the last two decades provided access to the high-resolution 3D reconstruction of a surface topology even for large areas and substantial differences between peaks and valleys Claxton et al. (2006), Hovis and Heuer (2010), Tata and Raj (1998). These unique capabilities make CLSM very attractive and highly promising for the 3D qualitative and quantitative fractographic analysis Capel et al. (2006), López-Cepero et al. (2005), Merson et al., (2016a), (2016b), (2017). In particular it has been suggested recently that the fracture surface ductility of steel specimens can be assessed by the normalized fracture surface area Rs obtained from the CLSM topographic data Merson et al. (2017). It was found that Rs is sensitive enough to distinguish between the completely brittle and completely ductile extremes in the fracture surface appearance in the same steel. Nevertheless, the Rs values for the fracture surfaces exhibiting the features of both ductile and brittle fracture modes have not been investigated yet. Besides, Rs was measured on the relatively small fracture surface regions of 128x128 μm while the topographic data for the full-scale fracture surface was not available. Thus, the present study is aimed at investigation of the normalized surface area of the full-scale fracture surfaces of the low-carbon steel in the whole temperature range of ductile-to-brittle transition.

2. Experimental

2.1. Material, specimens and Mechanical Testing

Round notched cylindrical specimens with a 150 mm length and a 5 mm diameter, Fig. 1a, were mechanically machined from the hot-rolled bars of commercially available low-carbon steel grade 10 (in Russian designation) which chemical composition is presented in Table 1. The specimens were annealed in vacuum at 950 °C during 30 min. After the annealing they exhibited typical equiaxed coarse-grained ferrite-pearlite microstructure, c.f. Fig. 1b. The specimens were tensile tested using the servohydraulic universal testing machine 8872 (Instron) equipped with the environmental chamber. The tensile tests were conducted at 100 mm/min traverse velocity in the range from 200 to -196 °C. After testing all specimens were rinsed in acetone and air-dried.

Table 1. Chemical composition of the steel grade 10.

Element	C	Si	Mn	P	S	Cr	Mo	Ni	Cu	Al	Fe
Wt (%)	0,117	0,236	0,493	0,013	0,0036	0,034	0,0067	0,037	0,049	0,025	Balance

2.2. Microscopy

All fracture surfaces were scanned by the CLSM Lext OLS4000 (Olympus) using the "MPlanApoN20xLEXT" objective lens (400× magnification) at the 0.8 μm step height. A one frame obtained by this objective lens represents the 640x640 μm^2 region of specimen's surface and is composed of 1024x1024 pixels each of which contains x, y, and z coordinates as well as light intensity and color values. Such data allows reconstruction of color or gray scale 2D and 3D images and also provides opportunity to conduct any topographical and linear measurements. Panoramic images stitched from the 16 (4x4) frames with 10% overlapping, Fig. 2, have been obtained for both halves of each specimens. The size of such panoramic images was essential to contain the whole fracture surface of each specimen. Before the measurements, the images were processed by the "pre-measurement" denoising filter, Fig. 2, available in the original Olympus Lext OLS4000 software. Then, the fracture surface region was masked by the "region-of-interest" tool on every panoramic image. Finally, the true surface area *S* corresponding to the given fracture surface relief was measured and divided by the area of its projection on the plane *A* to calculate the normalized surface area value *Rs*. Additionally, all fracture surfaces were investigated by the scanning electron microscope (SEM) SIGMA (Carl Zeiss).

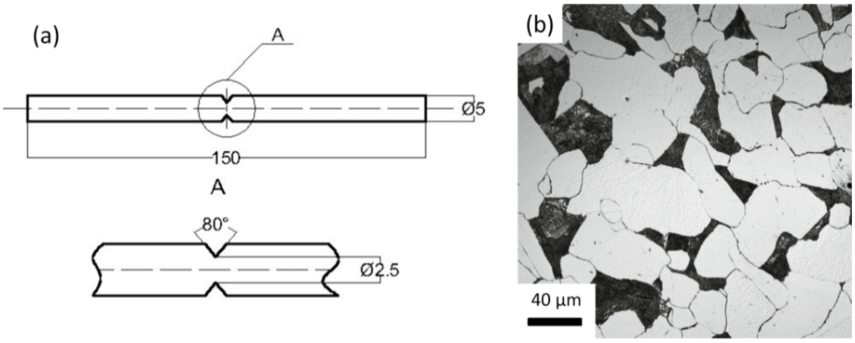

Fig. 1. Geometric characteristics (a) and microstructure (b) of the specimens.

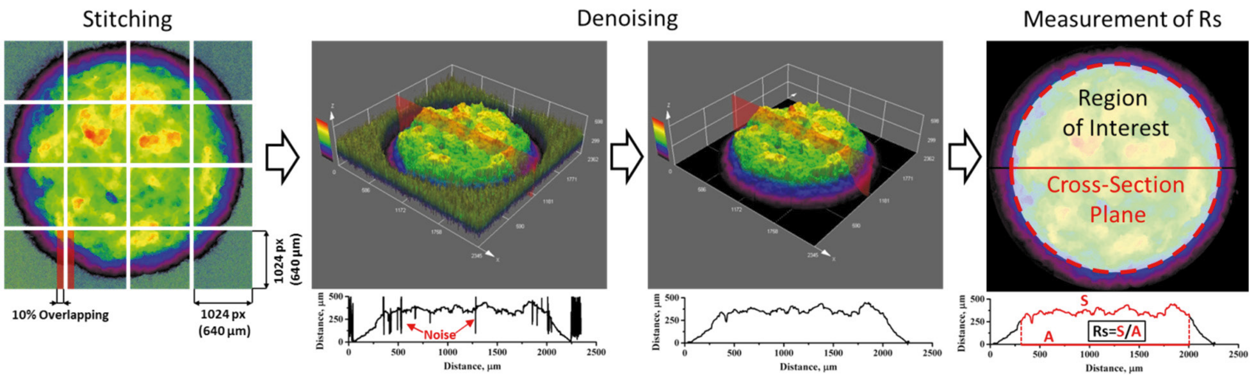

Fig. 2. Schematic illustration of the CLSM imaging and the measurement procedure including: (1) the stitching of the panoramic image, (2) denoising filtering of the images and (3) measurement of the true surface area *S* followed by the calculation of the normalized surface area *Rs*.

3. Results and Discussion

The experiments showed that shape of loading curves, Fig. 3, as well as the mechanical characteristics, Fig. 4, are quite typical for the annealed low carbon steel tested in the investigated temperature range. The elongation at break has the maximum around room temperature and drops sharply at lower temperatures, Fig. 4. Nevertheless the decrease of plasticity occurs quite monotonically from room to liquid nitrogen temperature. The ultimate tensile strength as well as the yield stress linearly increase as the temperature decreases.

The most appealing result of the present work is that the behavior of the normalized surface area of the fracture surface *Rs* as a function of temperature in the range covering the ductile-to-brittle transition is quite similar to that of the impact fracture toughness, Fig. 4. In fact, Fig. 4 reveals the typical ductile-to-brittle transition curve with the drastic decline of Rs from 100 to -100 °C. Fairly large scatter of Rs values is observed in the transition region that is also common for the fracture toughness values obtained at impact testing.

The CLSM and SEM qualitative fractographic analysis confirms the results described above. At high temperatures, the fracture surface exhibits the rough dimpled relief characteristic of ductile fracture, Fig. 5a and Fig. 6a. The decrease of testing temperature results in the appearing of cleavage facets on the fracture surface, Fig. 6c, d. The area of the faceted surface increases with decreasing temperature, Fig. 6c, d, while the fracture surface becomes more flat. Finally, at -196

°C the fracture surface is completely brittle and is fully composed of cleavage facets with no signs of dimpled morphology, Fig. 6d. As follows from CLSM topographic data, this fracture surface is more flat than the ductile one, Fig. 5b. The obtained results corroborate well the previously made suggestion that the value of the reduced surface area Rs of the fracture surface can serve as the quantitative measure of the fracture surface ductility.

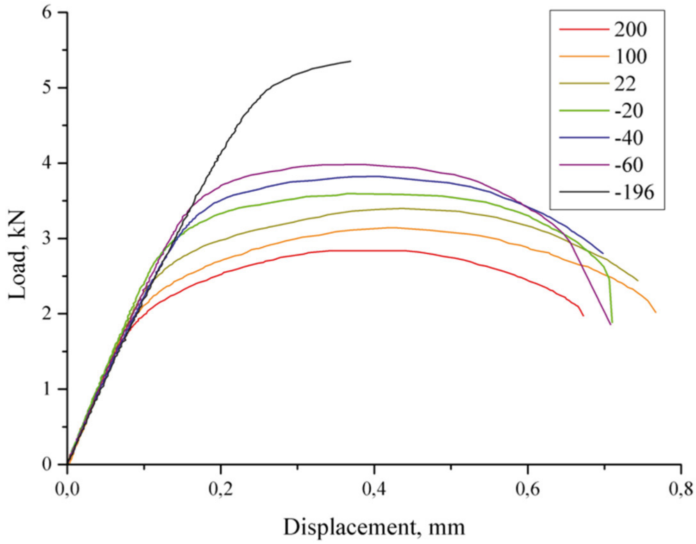

Fig. 3. Loading diagrams of the specimens tensile tested at different temperatures.

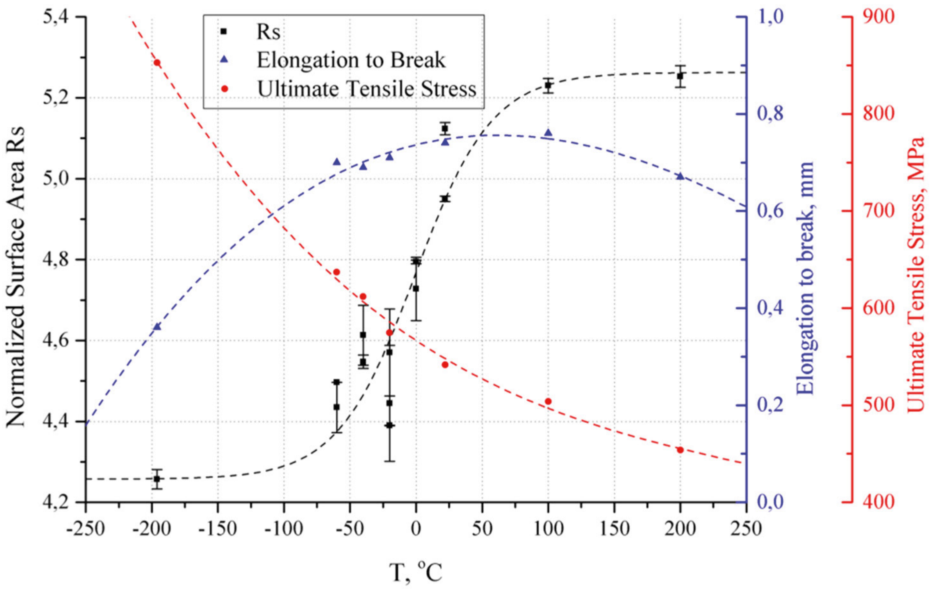

Fig. 4. Effect of the tensile testing temperature on the elongation at break, the ultimate tensile strength and the normalized fracture surface area *Rs* of the specimens.

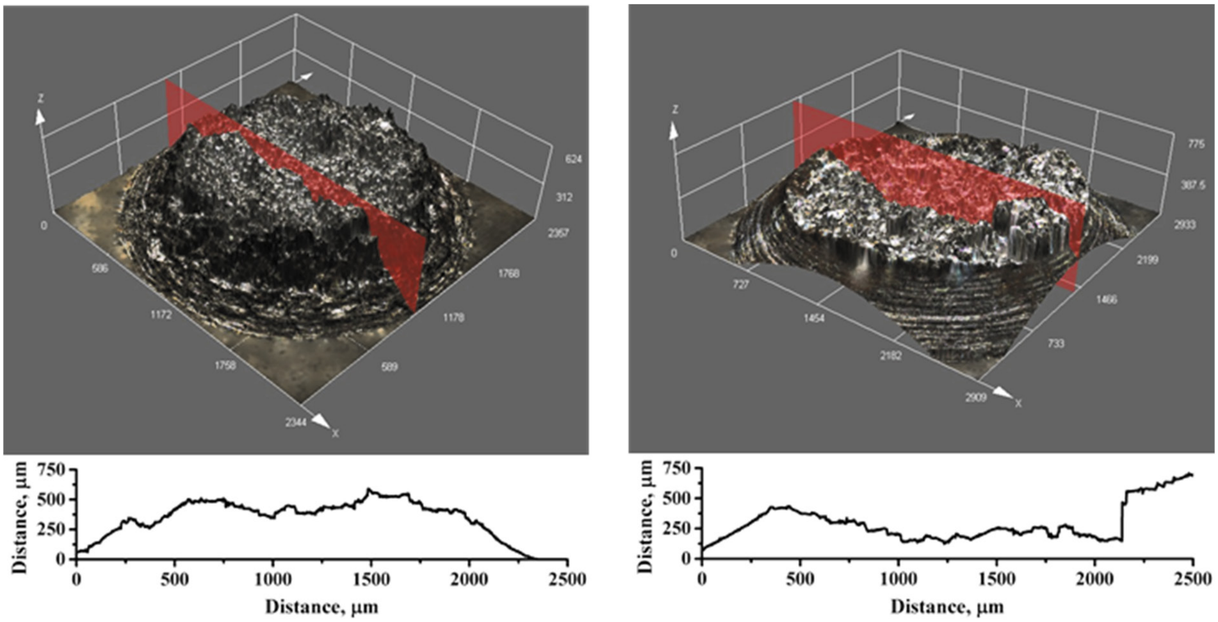

Fig. 5. CLSM 3D isometric images of the fracture surfaces with cross-section profiles for the specimens tensile tested at 200 °C (a) and -196 °C.

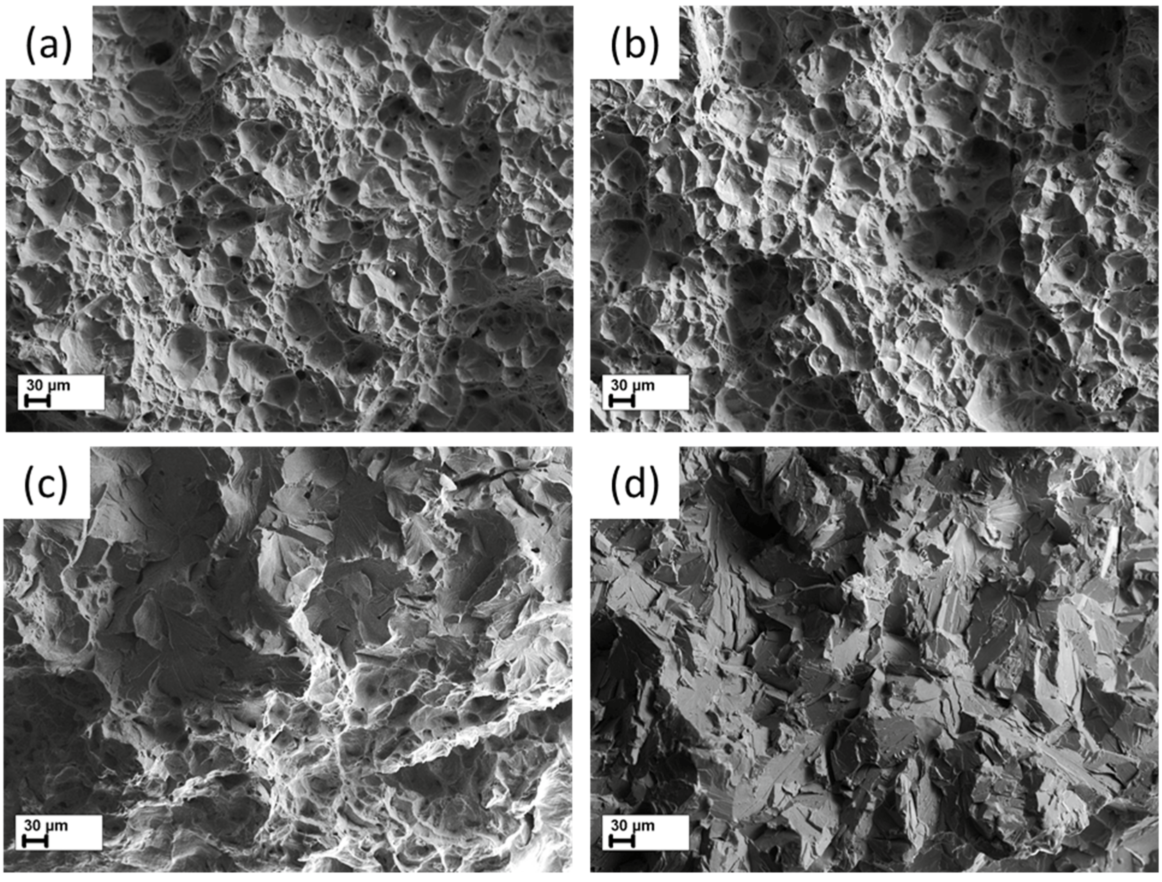

Fig. 6. SEM fracture surface images for the specimens tensile tested at 200 (a), 22 (b), -20 (c) and -196 °C (d).

4. Conclusion

We demonstrated, for the first time to our best knowledge, that the value of the normalized surface area *Rs* correlates strongly with the fracture surface appearance depending on the tensile testing temperature covering the ductile-to-brittle transition interval. Thus, in respond to the existing demand of increased objectivity of the fracture mode characterization, the Rs value can serve as a quantitative measure of the fracture surface ductility.

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