Where is the stretch zone? – 3D SEM – a powerful tool to analyze fracture

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ABSTRACT. *In this investigation the SEM MIRA3 XMU from TESCAN company is used which provides a 3D imaging technology based on a shift of the primary electron beam. The paper compares 2D with 3D imaging (as anaglyph pictures) of several crack surfaces. The focus is given to determine the stretch zone height and width. Nevertheless, other effects on the cracked specimens like local brittle fractures are shown. The 3D images give a clear view of the fracture topography which can hardly be achieved in 2D by tilting and looking from side positions. It is shown how 3D reconstruction and length measurement is possible with dual image photogrammetry software. The tool is fast, easy to use and gives also on "bad" fracture surfaces a clear view. The investigations were carried out on ductile cast iron, austenitic and ferritic steels in which multiple stretch zones are present.*

INTRODUCTION

There is always the question of assignability of static fracture mechanics test data for flaw assessment of ductile metallic materials. It is well known that physically based crack initiation values like δ_i or J_i are mostly independent on geometry. However, they are hard to measure because qualified measurements on SEM micrographs of the fracture surfaces are necessary which are not easy to perform. Therefore, often so called technical crack initiation points like $J_{0,2}$ or J_{Ic} are used. In [1] it is shown that the decission whether a physically based crack initiation point is needed can be made according to a quotient of multiaxiality in the crack section over a region of 30% of the ligament. If this quotient is below 0.4 a physically based crack initiation point must be used.
In the standard ISO 12135 J_i or δ_i is defined as the intersection between the J-Δa or δ-Δa crack resistance curve and a line parallel to the ordinate in the distance of the stretch zone width (SZW). The standard writes "A large scatter in the values of δ_i and J_i is inherent in the method due to the subjective nature of interpretation and measurement of the stretch zone width. To minimize scatter, it is suggested that only personnel with extensive experience in the interpretation of SEM fractographs be employed for this procedure. If the stretch zone cannot be distinguished from ductile crack extension, δ_i

and J_i cannot be determined." In a round robin test [2] the same SEM micrographs where given to a wide group of researchers. A large scatter in the SZW measurements was found indepently from the experience in stretch zone measurement.

The stretch zone height (SZH) gives a value for $\delta_i/2$. Using SEM, the measurement of SZH needs tilt of the specimen or conservative approximations to determine it from the SZW [3]. Other possibilities to measure the stretch zone are roughness measurements with tactile [4] or optical systems like laser or white light interferometry. However, also in these cases SEM imaging is needed in addition to avoid misinterpretations of the fracture surface profiles. It was shown in [5] that stereo-pairs of SEM micrographs which were obtained by tilting the specimen of an angle of 10° are suitable to obtain 3D reconstructions of the fracture surface by stereo photogrammetry. At that time this procedure took 3-4 hours for one profile and took a lot of effort.

EXPERIMENTAL

In the present study the fracture surfaces of several materials were analyzed using a high-resolution, field emission scanning electron microscope (HR-FE-SEM) MIRA3 XMU from TESCAN Company. The SEM is equipped with the unique real-time "In-Fligth Beam Tracing™" technique for the performance and spot optimaziation. Furthermore, the SEM provides a unique live stereoscopic imaging using an adavanced 3D beam technology. There are two different possibilities to generate 3D images of the fracture suface. In case of pronounced surface topography stereographic images were taken by beam tiliting mode. At low working distances (WD ≤ 12 mm), a set of two SEM images is taken at maximum/minium possible tilting angles of the primary electron beam. The resolution of the SEM micrographs is 1024 × 888 pixels. The other possibility is, in case of low surface roughness/topography to use the stage tilting mode, where in addition a tilting of the SEM probe stage can be applied.

Different materials ranging from commercial steels via high-alloyed TRIP steel to ductile cast iron were investigated. The goal of the selection is to show a wide range of application for the 3D SEM imaging. All materials show more or less pronounced stretch zones or multiple stretch zones due to the elastic-plastic material behaviour. All investiagted fracture surfaces are results of static fracture toughness tests using single-specimen procedure according to ISO 12135 to determine crack resistance curves. Fatigue precracking was performed until the ratio a_0/W of approximate 0.5 was reached. For the fracture toughness tests, single edge notch bend specimens (SENB) with a thickness of 10 mm and compact tension specimens (CT) with a thickness of 25 mm were used, respectively.

3D SURFACE TOPOGRAPHY

Since SEM micrographs are similar to light optical micrographs (LOM) information on the third dimension are lost in the perspectivic projection of a 3D object on a 2D image plane. In contrast to the LOM, the SEM has a solid depth of field. But nevertheless, the measurement of the topography of the specimen is impossible.

A stereoscopic reconstruction of the investigated specimen surface using SEM micrographs is the only possibility to analyze the 3D topography and to obtaine quantitative data. A set of stereographic image pairs must be generated. There are two different ways to obtaine stereographic image pairs – (1) eucentric tilt of the primary electron beam and (2) eucentric tilt of the specimen. In the first case at low working distances two SEM micrographs are taken at maximum/minium tilt angle of the primary electron beam. This procedure is sufficient for specimen surfaces with "high" topography as for the most fracture surfaces. The second procedure uses a eucentirc tilt of the specimen and is recommend for "flat" specimen surfaces. In both cases, two images of the same specimen area are taken under different perspectives.

In order to get an impression from the real topography of the investigated specimen surface, there are two different possibilities. The first – most impressive one – is the anaglyph picture. From the set of two SEM micrographs taken at different tilt angles an anaglyph picture can be calculated. Anaglyph images are obtained by the superposition of two color layers which have an offset with respect to each other to produce a depth effect. Then, the anaglyph images provide a stereographic 3D effectct, when viewed with glasses where the two lenses are of chromatically opposite colors like red and cyan. The present paper contains such anaglyph pictures. Therefore, the reader is kindly asked to use „anaglyph glasses" (red/cyan) which are commercially available (www.plano-em.de or www.3d-brillen.de). Nevertheless, the anaglyph picture provides qualtitative decription of the topography of a fracture surface, only.

In order to get quantitative data of the investiagted specimen topography, the reconstruction of a „digital object micrograph (DOM)" is performed. For this, the SEM MIRA 3 XMU is equipped with the MeX 3D software from Alicona Imaging GmbH. This software package includes an innovative algorithm which is able to recognize corresponding image points in the two stereographic images. These so-called homologous points were used to re-calculate the xyz-coordinates of the specimen surface. Therefore, for each pixel of the stereographic image the corresponding 3D point can be calculated with high precision. The MeX software provides several functions for analyzing 3D objects like 2D area measurements, 3D volume measurements as well roughness and height measurements.

As a reference for the performed measurements of the SZW and SZH a reference specimen was produced by milling of an aluminium cube containing several steps with a well defined step height of 100 µm. Figure 1 (a) shows the SEM micrograph of one single step, whereas Figure 1 (b) illustrates the 3D reconstruction of this step from two stereographic images taken at 1.5° tilt angle of primary electron beam. This sample was also analyzed by white light and laser interferometry. All three systems differ less than 2 %.

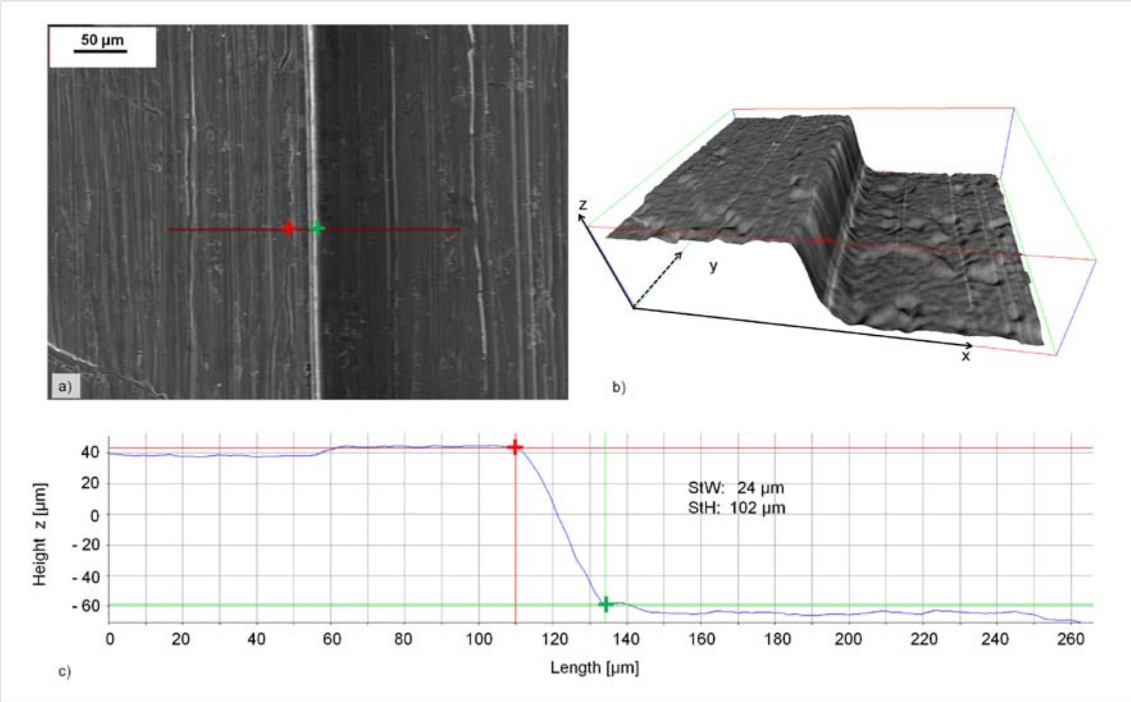

Figure 1: Reference specimen – aluminium cube with defined step height of 100 µm manufactured by milling. (a) 2D SEM micrograph. (b) 3D digital object micgraph after reconstruction. (c) Heigth profile with measurement of step width (StW) and step height (StH).

RESULTS

In particular, the height measurements are very interesting as additional information on the formation of stretch zones. During the last years, few studies using SEM for investigation of the formation of stretch zones in different kind of materials are reported in the literature [3, 6]. In all these investigations mostly the width of the stretch zones were measured with more or less accuracy. The application of the unique 3D beam technology in the SEM MIRA3 XMU in combination with the unique MeX 3D software provides the following advantages for the investigation of stretch zones:

- 3D image and reconstruction of the stretch zone,
- Clear definition of the transition from the area of fatigue crack propagation to the stretch zone and the area of stable crack propagation / instable crack initiation,
- Measurement of stretch zone width as well as stretch zone height without asumptions or tilting of the specimen,
- Calculated profiles and 3D anaglyph image or 2D image are coupled. This allows a clear definition of start and end point of the stretch zone,
- Fast calculation of the 3D reconstructions with actual standard PC hardware within seconds to few minuts.

Figure 2 shows the stretch zone area of a high strength construction steel with a yield strength of 620 MPa on a CT specimen deformed at 0°C in (a) 2D and (b) 3D anaglyph imaging which was obtained from two stereographic images taken at 2.0° tilt angle of primary electron beam. Above the fatigue crack there are multiple (up to 4) stretch zones which are terminated with small areas of stable crack growth (at all less than 0.2 mm). In the upper area of the pictures the unstable cleavage fracture is visible. A line profile from the reconstructed 3D model is shown in Figure 2c. The measuring line and points are marked in Figure 2a.

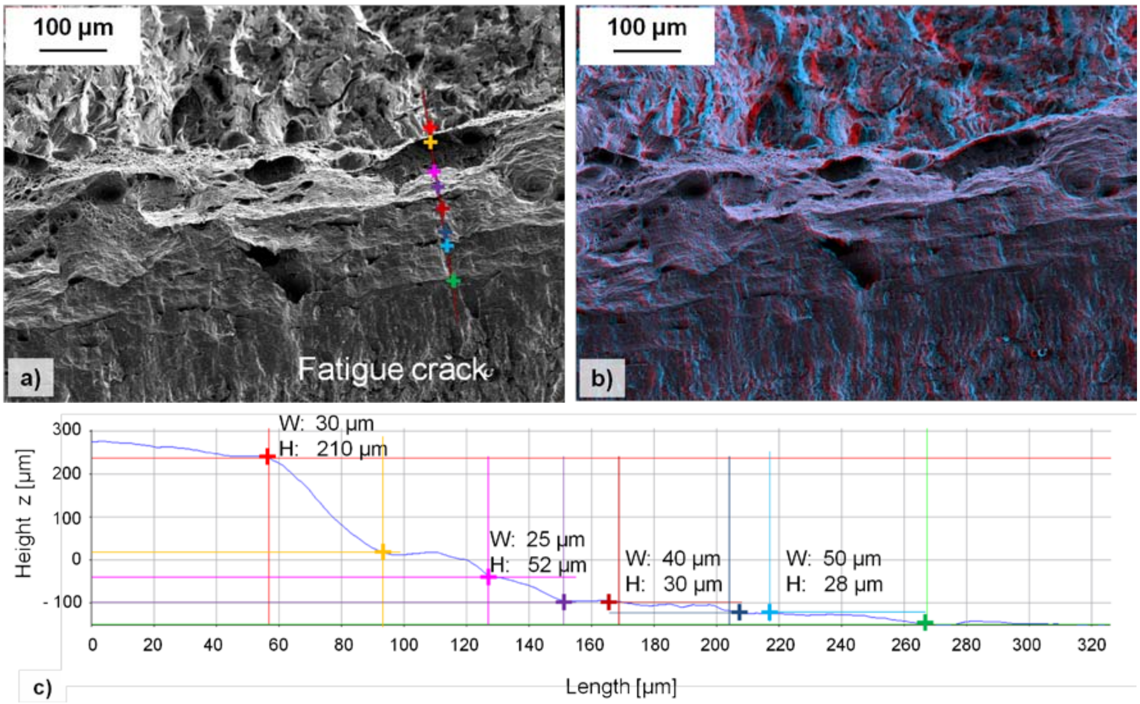

Figure 2: Multiple stretch zones in a high strength construction steel. (a) 2D SEM micrograph using SE contrast. (b) 3D anaglyph image. (c) Corresponding height profile along the measuring line indicated in (a) with determined values for SZW and SZH.

Figure 3 shows the stretch zone area of a high alloyed cast TRIP steel (16%Cr 6%Mn 6%Ni) on a SENB specimen deformed at 20°C as (a) 2D and (b) 3D anaglyph image which was obtained from two stereographic images taken at 2.0° tilt angle of primary electron beam. Blunting and stable crack growth is running simultaneously in different areas of the specimen. The obtaind surface shows multiple stretch zone areas together with stable crack growth. In this case the definition of a stretch zone width can hardly be done. For a conservative value the first stretch zone can be choosen.

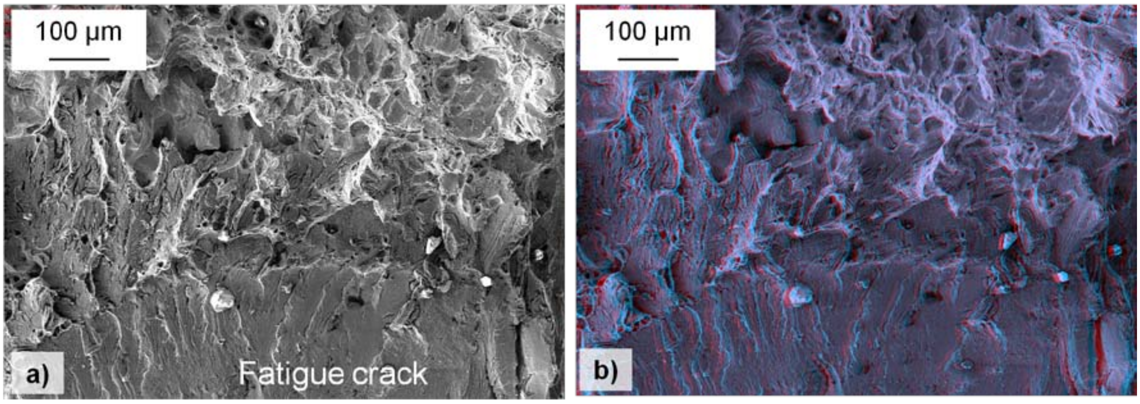

Figure 3: Multiple stretch zones in a high alloyed cast TRIP steel (16%Cr 6%Mn 6%Ni) SENB 10 mm deformed at 20°C. (a) 2D SEM micrograph using SE contrast. (b) 3D anaglyph image.

The usually expected case of a single stretch zone before stable crack growth is shown for a SENB sample of a heat-resisting steel 10CrMo9-10 (1.7380) deformed at 23°C as shown in Figure 4. The anaglyph image was obtained from two stereographic images taken at 1.0° tilt angle of the primary electron beam. Figure 5 shows the reconstructed 3D profile (a) together with the height profile (c) and the stretch zone measurement results (c) along the measuring line marked in (b).

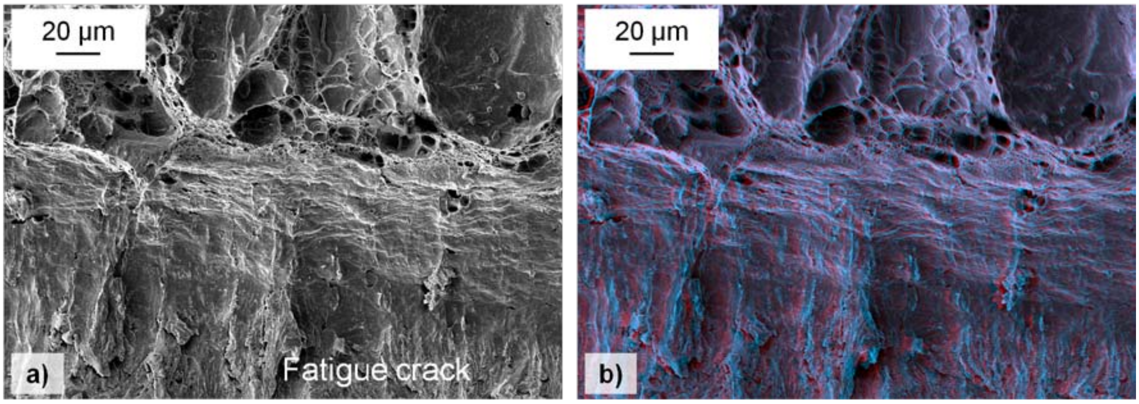

Figure 4: Stretch zone on a fracture surface of 10CrMo9-10. (SENB, tested at RT).
(a) 2D SEM micrograph using secondary electron contrast. (b) 3D anaglyph image.

Stretch zones in ferritc ductile cast iron are not easy to observe. The fracture surfaces are rough and the graphit spherolites give additional profiles. Figure 6 shows a stretch zone in cast iron of specification EN-GJS-400-18LT with a graphite sphere size of approximate 13 µm on a SENB specimen at a test temperature of -40°C. The 3D image (b) was obtained from two stereographic images taken at 1.0° tilt angle of the primary electron beam. The marked stretch zone together with the height profile is shown in Figure 6c. In the daily lab practice like requested by the fracture mechanics test standards (ISO 12135) for all cases at least 45 single measurements are done at 9 pictures with constant distance over the whole crack front to get a mean values which is representative for the specimen.

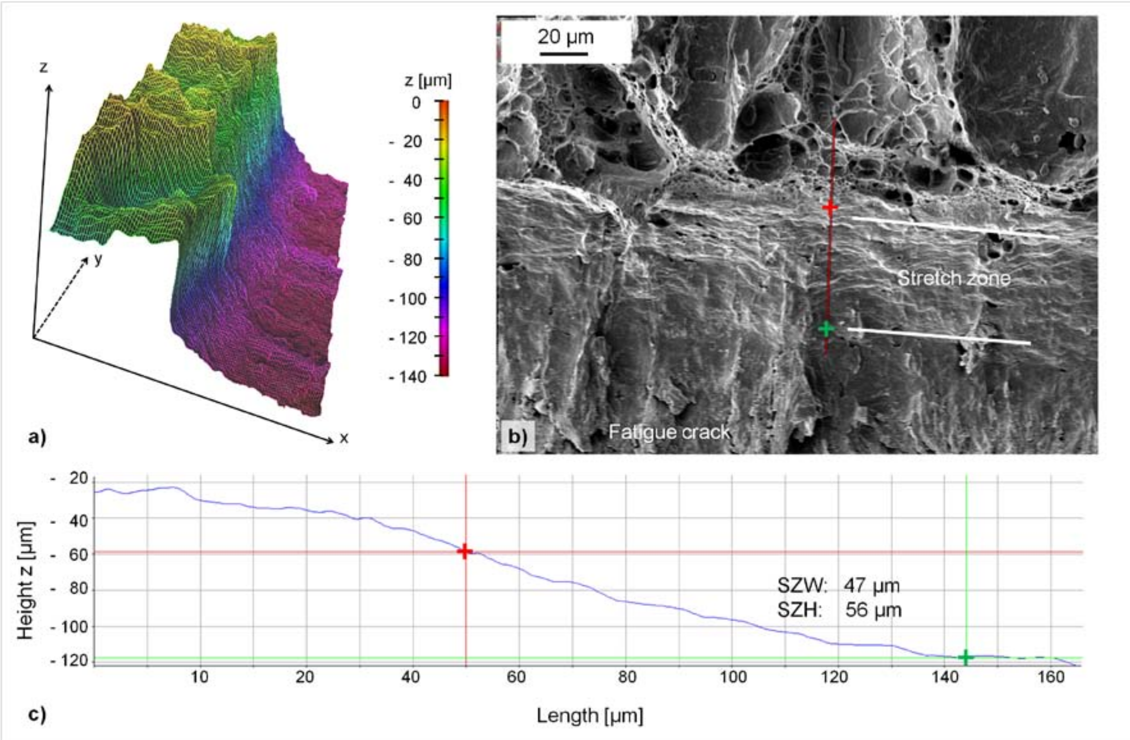

Figure 5: Measurment of the stretch zone width (SZW) and stretch zone height (SZH). (a) 3D reconstruction of the stretch zone obtained from two stereographic images taken at 2.0° tilt angle of the primary electron beam. (b) SEM micrograph with indicated measuring line and measuring points. (c) Corresponding height profile with determined values for SZW and SZH.

SUMMARY

The 3D SEM imaging together with the possibility of construction of the 3D surface profiles is a powerful tool to determine physical crack initiation parameters in materials with elastic-plastic fracture. It combines the fractographic view on SEM micrographs with the accuracy of roughness measurements in a fast and easy to handle way. The stretch zone heigth can be measured without tilting the specimen.

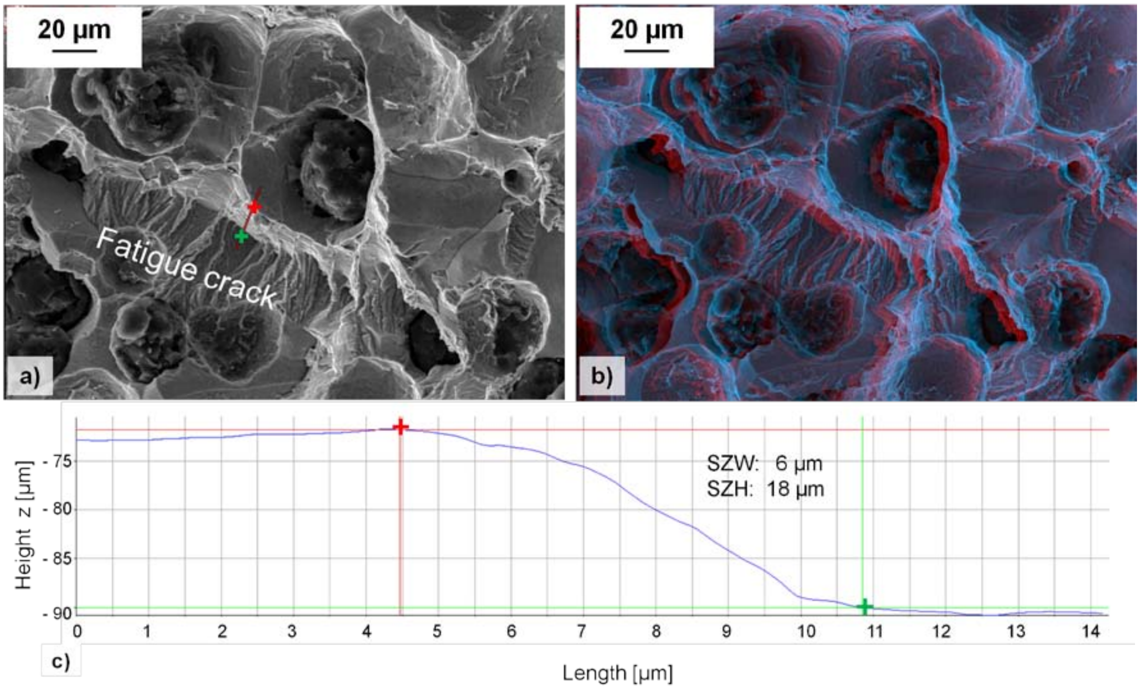

Figure 6: Stretch zone in a ductile cast iron. (a) 2D SEM micrograph taken in SE contrast. (b) 3D anaglyph image. (c) Height profile along the measuring line indicated in (a) with the measurement of the stretch zone width (SZW) and stretch zone height (SZH).

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