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Method comparison for the determination of stretch zone parameters

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Method comparison for the determination of stretch zone parameters

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Abstract. The fracture behaviour of ductile failing materials is evaluated by parameters of yielding fracture mechanics. According to ISO 12135 the determination of fracture mechanical characteristic values involves, among other things, analysing the stretch zone width (SZW) in the scanning electron microscope (SEM). This procedure is very time-consuming and requires great experience in handling SEM images of fracture surfaces. An alternative method for determining the stretch zone parameters works with 3D elevation profiles of the fracture surface in the stretch zone area taken with a digital microscope. The fracture mechanical parameters determined using the above methods were finally compared. The $J_i$ and $\delta_i$ values of scrap steels resulting from the digital determined SZW show a good agreement with analytically determined crack initiation values ($J_i/BL$, $\delta_i/BL$).

1. Introduction
Cases of failure characterized by larger plastic deformations before fracture are evaluated by means of yielding fracture mechanics, also known as elastic-plastic fracture mechanics. The J-integral and CTOD ( Crack Tip Opening Displacement) concept are the most important tools in yielding fracture mechanics. The relationship between the fracture mechanical stress parameter $J$ or $\delta$ and the stable crack propagation $\Delta a$ is shown in the crack resistance curve (R curve). The course of the J-integral- or CTOD-R curves contain the consecutive stages of blunting, crack initiation and stable crack propagation. Blunting is the rounding and bulging of a sharp fatigue crack of ductile materials under mode I loading. During the bulging process, pores are already formed in the non-cracked material in front of the crack tip. These pores are caused by breaking of inclusions or detachment of matrix material from inclusions. Newly formed and already existing pores grow. Bars of matrix material remain between the pores. The initiation of the crack is regarded as the moment when the first bars between the rounded crack tip and the pores formed in front of the crack tip tear.

Blunting creates a stretch zone in front of the crack tip, whose geometry is defined by the stretch zone width (SZW) in crack propagation direction and by the stretch zone height (SZH) perpendicular to it (Figure 1). The stretch zone is a microscopically detectable feature of crack initiation. If the SZW reaches a critical value, the crack starts. The corresponding physical crack initiation values $J_i$ and $\delta_i$ are regarded as failure criteria for ductile components, provided that they are geometry independent [1, 2].

Two main standards – ISO 12135 and ASTM E 1820 – are used to determine fracture mechanical characteristic values under quasi-static loading. In the method according to ISO 12135, the crack initiation point as well as the crack tip parameters $J_i$ and $\delta_i$ are defined as the intersection of the R curve with the blunting line. Additionally, it is necessary to determine the critical stretch zone width $\Delta a_{SZW}$ by means of analysing the fracture surface in the scanning electron microscope (SEM). The shape of the J-integral / CTOD-R curve corresponds to the function
\[ J \text{ or } \delta = \alpha + \beta \Delta a^\gamma \] [3] \hfill (1)

with the constants \( \alpha, \beta \geq 0 \) and \( 0 < \gamma < 1 \). The description of the blunting line results from

\[ J = 3.75 \times R_m \times \Delta a \] and

\[ \delta = 1.87 \times \frac{R_m}{R_{p0.2}} \times \Delta a \] [3] \hfill (2)

\hfill (3)

with \( R_m \) and \( R_{p0.2} \) as material parameters from corresponding tension tests. However, a determination of the physical crack initiation values \( J_i \) and \( \delta_i \) according to ISO 12135 is only permissible if a clear distinction between the stretch zone and the stable crack propagation on the fracture surface is possible. In this case, \( J_i \) and \( \delta_i \) also result from the orthogonal intersection of \( \Delta a_{SZW} \) with the R curve. Alternatively, the technical crack initiation value can be determined with a stable crack propagation of \( \Delta a = 0.2 \text{ mm} \).

![Figure 1. Stretch zone schematic.](image)

2. Experimental and materials

2.1. Methods for determining the stretch zone parameters

According to ISO 12135 the critical SZW measured in SEM represents the mean of nine local measurements along the entire width of the fracture surface. At the local measuring sites, at least five measurements should be carried out [3]. The detection of SZH in the SEM can't take place at the same time as SZW. Therefore, a tilting of the specimen is necessary.

The determination of the stretch zone parameters in the SEM can be subject to large scattering, thus it requires extensive experiences in the analysis of fracture surfaces according to ISO 12135 [3]. However, round robin tests of 14 participating institutions on the same set of SEM images in [4] showed approximately the same scatter of SZW regardless of the existing experience.

Alternative methods for determining the stretch zone parameters include various SEM techniques [5, 6], roughness profiles [7], optical systems such as white light interferometry [5] or the principle of confocality [8] and FEM simulation [9]. In [5], the position and dimension of the stretch zone were defined by combining a 2D SEM image with the 3D reconstruction of the fracture surface. For the 3D reconstruction, two images were taken by tilting the primary electron beam instead of tilting the specimen. Using the generated anaglyph image and the 3D profile of the fracture surface created by special software, SZW and SZH could be determined simultaneously. The authors of [6] have carried out comparative SEM investigations with the common secondary electron technique and backscatter electron contrast to determine the critical SZW. Using the backscattering technology, it should be possible to image the fracture surface even more precisely. Results from roughness measurements could only be used in combination with additional SEM images to determine the stretch zone
parameters due to the existing scatter. In order to identify the stretch zone more precisely and thus to minimize the scattering of the \( J_i \) values, the 2D SEM images in [8] were supplemented with images from a laser scanning microscope. The SZW based on 3D surface profiles displayed lower values than the SEM results. Numerical determination of the SZW is also possible. [9] presents a FEM model based on a strongly deformed stretch zone.

For our own fracture mechanical investigations on scrap steels (St38b-2, St52-3), in addition to the standardized SEM method, a procedure was applied in which topology images of the fracture surface in the area of the stretch zone were taken with a digital microscope. This approach combines the advantages of relatively simple handling with a low expenditure of time compared to scanning electron microscopy. Furthermore, it offers the possibility to evaluate both stretch zone parameters, SZW and SZH, simultaneously over a larger area of the specimen by means of 3D elevation profile per measuring site.

2.2. Standard method according to ISO 12135
In order to determine the crack initiation values of scrap steel (St38b-2, St52-3), fracture mechanics tests at -30°C were performed. After fatigue precracking (crack length 2 mm), \( J-\Delta a \) and CTOD-\( \Delta a \) curves were plotted with the partial unloading compliance method using three-point bend specimens with a thickness of 10 mm. The crack lengths achieved (fatigue precrack, stable crack propagation, fast fracture) were measured using a stereo microscope.

At first the crack initiation values were determined analytically via the intersection of the R curve with the blunting line. The \( J_i/BL \) or \( \delta_i/BL \) values are valid and as a result transferrable to components if the thickness criterion according to ISO 12135
\[
J_{i/BL} \text{ or } \delta_{i/BL} < J_{max} \text{ or } \delta_{max} [3]
\]
is complied.

The fracture surface analysis is performed with the scanning electron microscope EVO 40 from Zeiss to measure the local SZW. For this purpose, the individual SZW were marked in the SEM images and evaluated using the image processing software Adobe Photoshop CS6 Extended.

2.3. Digital microscopy
For the fracture mechanical evaluation of the scrap steels St38b-2 and St52-3 topology images of the fracture surface were analysed at nine measuring sites in the area of the stretch zone with the digital microscope VHX-900F from Keyence. In addition to the time advantage over the SEM method, this procedure offers the possibility of evaluating both stretch zone parameters, SZW and SZH, per local measuring site on the basis of a 3D elevation profile.

The nine local measuring sites along the specimen width were set by shifting the microscope stage perpendicular to the crack propagation direction by 0.5 mm each. The 3D elevation profiles were captured at 700x magnification. For this purpose, the VHX works with the D.F.D. method ("Depth from Defocus"). To calculate the 3D depth information, the blur extent of 2D images is analysed. A stepwise shift of the lens relative to the measuring object along the height axis, which takes place automatically here, enables image acquisition from all contour levels. In the top position of the lens, the highest point of the considered partial fracture surface is sharply imaged, in the lowest position the lowest point. In order to generate the 3D overall image of the surface profile, the sharp image areas for all contour levels are determined and joined together. Combined with glare removal, the D.F.D process and the Keyence image processing software can be used to precisely image metallic surfaces with complex geometries. The entire procedure, from the image acquisition to the generation of the 3D fracture surface profile per measuring site takes about 30 s. A height-colour map, which can be superimposed on the raw image, shows the surface texture and structure. The height scaling can be adjusted by means of a tilt correction.

Instead of the five individual measurements in the SEM, the Keyence software offers a measuring grid with which it is possible to average the local stretch zone parameters over a larger area.
3. Results and discussion

3.1. Standard method according to ISO 12135
For all tested specimens MP1 to MP6 ductile failure due to crack resistance behaviour was found (Figure 2). The optically measured crack lengths correspond well with the values determined during the test using the compliance method. Some specimens show very high $J_{a/BL}$ values that exceed the $J_{max}$ criterion (equation 4). Thus, there is still a geometry dependence. In this case a limitation to $J_{max}$ is recommended.

On the basis of SEM overview images, it was possible to identify the different fracture surface areas and to set the measuring sites for determining the local SZW (Figure 3a). A typical ductile fracture surface without cleavage fracture parts is shown in the area of stable crack propagation (Figure 3b). The local SZW $\Delta a_{SZW,L}$ represents the mean value of five measurements per measuring site (Figure 3c). The mean value of all $\Delta a_{SZW,L}$ defines the critical SZW per specimen whose intersection point with the J- or CTOD-R curve represents the crack initiation values $J_{i/SZW}$ and $\delta_{i/SZW}$.

The determination of the SZW in the SEM requires a great deal of time, especially if the SZH is also to be recorded for the validation of the CTOD values. Furthermore, with five individual measurements at the nine local measuring sites, only a very small area of the stretch zone is used for averaging over the entire specimen. The scattering of the measured values, which is often mentioned in literature, as well as the necessary experience to carry out this procedure additionally motivate the development of alternative methods.

3.2. Digital microscopy
Figure 4 shows the generated 3D elevation profile of a local measuring site of St38b-2. The 3D profile can be rotated in all directions so that a suitable measuring position can be easily found. For the fracture surface analyses of scrap steel, a grid of 30 measuring lines at a distance of 5 μm each was selected, which is oriented perpendicular to the crack front and corresponds to a measuring range of 145 μm. The measurement of SZW and SZH is carried out via a horizontal or vertical point to point measurement using an average profile displayed in 2D. The position of this profile section can be freely selected within the defined measuring range. The local SZW and SZH result from the mean value of the 30 individual measurements. Figure 5 shows an example measurement on the St38b-2.

With this optical method, the position and dimensions of the stretch zone can be recorded simultaneously. Another advantage over the SEM procedure is the larger local measuring range. Using
the selected measuring grid, 16 % of the net thickness of the specimen ($B_N = 8 \text{ mm}$) is used to evaluate the stretch zone parameters. Thus, individual over- or underestimates of SZW and SZH have less influence on the overall result.

Comparable 3D profiles of the fracture surface could also be recorded with the 3D laser scanning microscope VK-X200 from Keyence. Laser scanning microscopes use the principle of confocality to measure height data at the surface. In further investigations it has to be resolved whether the higher resolution up to 0.5 μm compared to the digital microscope (up to 2 μm) provides an information gain for the stretch zone analysis. According to the authors, however, there are doubts because the expected error in the determination of SZW and SZH is much greater.

**Figure 3.** Measurement of SZW in SEM for St38b-2: overview of the fracture surface specimen MP1 with the measuring sites 1.1 to 1.5 (a), fracture surface in the area of $\Delta a$ (b), local stretch zone of measuring site 1.2 with measurements M1 to M5 (c).
Figure 4. 3D elevation profile of a partial fracture surface of St38b-2 / stretch zone marked between dashed lines.

Figure 5. Determination of the stretch zone parameters with VHX at a single measuring site / St38b-2: finding measuring position (a), measuring grid (b), mean profile for measuring SZW and SZH.
3.3. Comparison of results
The stretch zone parameters and the resulting crack initiation values of the scrap steels St38b-2 and St52-3 are compared in Table 1 with the analytically determined $J_{uBL}/\delta_{uBL}$ values.

| Scrap steel | $\Delta a_{SZW}$ SEM (μm) | $\Delta a_{SZW}$ VHX (μm) | $\Delta a_{SZW}$ SZH (μm) | $J_{uBL}$ SEM (kJ/m²) | $J_{uBL}$ VHX (kJ/m²) | $\delta_{uBL}$ SEM (μm) | $\delta_{uBL}$ VHX (μm) | $\delta_{i/SZW}$ SEM (μm) | $\delta_{i/SZW}$ VHX (μm) |
|-------------|--------------------------|--------------------------|------------------------|----------------------|----------------------|------------------------|------------------------|------------------------|------------------------|
| St38b-2     | 29±1                     | 77±3                     | 25±3                   | 148±38               | 150±9                | 115±3                  | 108±5                  | 146±12                 |
| St52-3      | 34±2                     | 76±4                     | 26±4                   | 204±11               | 193±18               | 181±10                 | 157±12                 | 199±14                 |

A comparison of $J_{uBL}$ and $J_{i/SZW}$ (VHX) shows a good agreement. The $J_{i/SZW}$ values determined with the SEM method are significantly lower. This statement can be illustrated using the example of the J-R mean value curve of St38b-2 (Figure 6). The deviation is attributed to the lower SZW from the SEM. In contrast to literature, e.g. [5] and [8], the SZW was underestimated compared to the optical method. The analytically determined $\delta_{i/SZW}$ values lie between the results of $\delta_{i/SZW}$ (SEM) and $\delta_{i/SZW}$ (VHX). However, a validation of the CTOD values via the double measured SZH is currently not possible. The corresponding measured values in Table 1 were systematically underestimated. The stretch zone parameters recorded with the VHX method show a low scatter and, with the measuring range that can be used for evaluation, provide reliable values for determining $J_i$ and $\delta_i$.

![Figure 6. Average J-R curve of St38b-2 at -30°C.](image)

### 4. Conclusions
An alternative method for determining the stretch zone parameters is presented. The SZW and SZH are analysed with the digital microscope VHX-900F from Keyence using a 3D elevation profile of the fracture surface. For comparison with the standard method according to ISO 12135, in which the SZW
is determined in the SEM, the results from static fracture mechanics tests on scrap steel at -30°C were used. The crack initiation values $J_{i/SZW}$ and $\delta_{i/SZW}$ derived from the digital microscopically determined SZW show good agreement with the analytical $J_{i/BL}$ / $\delta_{i/BL}$ values.

However, a repeatable, precise determination of the stretch zone parameters with the digital microscope requires the validation of the process by comparison measurements on different materials. If a general match with $J_{i/BL}$ / $\delta_{i/BL}$ can be demonstrated, the exclusive use of the VHX method with its advantages

- low time expenditure
- user-friendly approach
- detection of position and dimensions of the stretch zone
- simultaneous determination of SZW and SZH
- averaging over a larger measuring range

instead of the standard procedure according to ISO 12135 is conceivable.

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