Experimental and numerical investigations of two material states of the material 15 NiCuMoNb5 (WB 36)

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Abstract

In this contribution, results are presented of mechanical, technological and fracture-mechanical characterization of low alloyed warm strength steel 15 NiCuMoNb5 (WB 36, material No. 1.6368) in two states of the melt E60 at a temperature of 90 °C including the determination of damage mechanical parameters for the Rousselier model.

The two states of the material are the initial state (denominated as E60A) and the aged state (denominated as E60B). The aged condition characterizes the material after 57,000 h at a service temperature of 350 °C. In service this material produces significant copper precipitates above 300 °C. These copper precipitates are the reason for strength increase as well as a shift of the transition temperature regime of the “notch” impact energy to higher temperatures. Size, shape, orientation and frequency distribution of non-metallic inclusions are found to be similar for both material states. In comparing the frequency distributions of the copper particles for both material states a significant increase of copper particles with a size between 2 and 7 nm results.

The damage mechanical results show that the Rousselier parameters for the initial state can be transferred to the aged state. The strength increase due to aging can be considered through the respective yield curves in the computer simulations. A comparison of multiaxiality relations for different specimen geometries shows that the curves of the multiaxiality coefficient $q$ in the ligament remains principally unchanged by the strength increase at identical specimen geometries. The crack resistance behaviour determined in the experiment at CT specimens correlates with the numerically determined $q$-values in the ligament for both material states E60A and E60B.

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1. Introduction

For the judgement of the integrity of pressurized components with postulated or detected defects, a quantitative prediction of initiation and growth of cracks is necessary. With the existent concepts of damage mechanics ($J$-integral) it is possible to reliably calculate the critical loading at crack initiation [1]. However, the behaviour of cracks after initiation can be predicted by damage mechanical concepts (crack resistance curves) quantitatively only to a limited extent [1]. In order to quantify the safety results, the numerical
simulation of ductile cracking behaviour is essential. With the use of damage mechanical models, the full deformation and failure procedure in the specimen and in components (starting with the plastification, followed by crack initiation and crack growth, up to instability) can be simulated [2,3]. Damage mechanical concepts do have the advantage that material characteristics, which have been determined at laboratory specimens, can be transferred to the situation in components although the loading mode, the geometry, the multiaxiality of the stress state, as well as the amount of crack extension, shows a different behaviour.

Damage mechanics does provide the possibility to quantitatively predict ductile crack behaviour in ferritic (and austenitic) components, i.e. the real crack growth, crack opening as well as load carrying behaviour modelling. Therefore, the safety reserves of components with respect to ductile cracking behaviour can be judged in a realistic manner.

In this contribution, results of the mechanical, technological as well as fracture mechanical material characterization of the low alloyed warm strength steel 15 NiCuMoNb5 (WB 36, material No. 1.6368) at a temperature of 90°C are presented. The initial state of the melt E60 (denominated as E60A) and the aged state (denominated as E60B) are analyzed. The aged state characterizes the material state after 57,000 h at a service temperature of 350 °C. This material shows significant copper precipitation above 300 °C. This copper precipitates result in a strength increase and a shift of the transition temperature of the notch impact energy to higher temperatures.

2. Scientific and technical state of the art

2.1. Description of the failure process using damage mechanics

The damage mechanical modelling of ductile failure is oriented at microstructural processes of nucleation of voids at inclusions, growth of these voids by plastic deformation, and finally coalescence which results in final failure of the material. An extensive description is given in [2,3].

In the following, only the relevant relations and parameters will be explained:

1. The extent of damage is described by the volume fraction $f$ of voids.

2. The state of damage of the material at the beginning of loading is characterized by the fraction and the frequency distribution of non-metallic inclusions (e.g. MnS and Al$_2$O$_3$). If these inclusions are only weakly bonded to the matrix, and therefore lead to void nucleation immediately after deformation initiation, the initial void volume fraction $f_0$ of the voids can be determined from the volume fraction and the shape of the inclusions. The initial void volume fraction can be determined from metallographic investigations, the chemical composition or by numerical simulation of tensile test.

3. The growth of voids is simulated with the Rousselier model [2,4] which together with the yield function describes the plastic deformation and the development of damage:

$$\Phi = \frac{\sigma_v}{1-f} + \sigma_k D f\left[\frac{\sigma_{H}}{\sigma_0}\right] - \sigma_0 = 0$$

$\sigma_v$ is the von Mises equivalent stress; $\sigma_H$, hydrostatic stress; $f$, void volume fraction; $\sigma_0$, yield limit; $\sigma_k$, material dependent constant; $D$, material independent constant. According to theoretical analyses the constant $D$ can be assumed to be equal to 2 independent of the material [7]. The second constant $\sigma_k$ can be assumed to be 445 MPa according to experiences at MPA [2,3] for different steels. The same value will be applied for the present material 15 NiCuMoNb5.

4. Failure (nucleation and propagation of a crack) of a material volume element within the structure occurs when the void volume fraction reaches a critical value $f_c$. Damage mechanical comparative studies to tensile test have shown that the value $f_c = 0.05$ describes failure best [2,3].
(5) Damage mechanical simulations of ductile failure by different authors have shown that the size of finite elements has a significant influence on the final result. From a mechanical standpoint of view this can be explained by the effect that failure propagates from void to void, therefore, the discretization has to correspond with the mean distance between voids (respective with the mean particle distance \( l_c \)).

From these explanations it can be seen that for modelling reasons the ductile failure behaviour is essentially characterized by two material parameters within the Rousselier model: initial void volume fraction \( f_0 \) and mean particle distance \( l_c \). In this contribution the determination of these two parameters is given for the material 15 NiCuMoNb5.

2.2. Material 15 NiCuMoNb5

The low alloyed warm strength steel 15 NiCuMoNb5 (WB 36, material No. 1.6368) is used in German power plants as piping and pressure containing materials. The reasons for the manifold use of this material is due to the increased yield strength at higher temperatures according to the alloying by copper and a cheap heat treatment due to the nickel and molybdenum content.

While the material in German nuclear power plants is applied in pipes at service temperatures below 300 °C and only in special cases in pressure containments up to 340 °C, its service temperatures reach 450 °C in conventional power plants. In the years 1987–1992 different damages in pipes after long time service (90,000–160,000 h) have been reported [8–11] which occurred during services.

Although different factors have played a role in these damages, in all cases strengthening and a decrease in ductility have been found. The decrease in ductility can be measured essentially by a shift of the transition temperature regime of the notch impact energy to higher temperatures. Due to the temperature dependent solubility of copper in iron, this element is used since about 100 years to increase the steel properties. Depending on the annealing conditions, different amounts of dissolved copper are present in the material which in the service conditions, above 300 °C [12], precipitates and leads to strength increase.

Therefore, it is nearly impossible to reach a stable material state in the steel WB 36 [13].

According to new electron microscopical investigations [14,15] microstructural changes in the material can be correlated with precipitation of copper in a fine disperse manner as it is shown in [16]. Accordingly, the main focus of the examinations in [17] was put on the material changes and the underlying microstructural phenomena under thermal aging conditions in the material WB 36. Other investigations in the years 1990–1995 [9,18] lead also to a shift in the transition regime of the notch impact energy between 40 and 70 K to higher temperatures as well as to an increase of strength values. The yield strength increases by 110–140 MPa and the tensile strength increases by 85–125 MPa.

The results obtained on V-notch specimens showed that the KV–T-curves [17], Fig. 1, are elevated at a temperature of 90 °C. The transition regime of the notch impact energy is shifted by 58 K to higher temperatures for the melt E60 while the notch impact energy decreases by a maximum of 15 J. The required value of 31 J respectively 41 J at 0 °C are no longer reached for the service aged state.

In Fig. 2 the crack growth resistance curves between room temperature and 350 °C for states E60A and E60B are documented from [17]. While
the crack growth resistance decreases continuously with temperature for the state E60B, the smallest crack growth resistance for the state E60A is reached at a temperature of 250 °C. As expected the $J_R$-curves of the aged state provided the lower slope as compared to the initial state, independent on temperature.

Yield stress and tensile strength are significantly increased by about 100 MPa for the state E60B, Fig. 3, while the fracture strain and cross-section reduction at fracture is reduced to smaller values. The Young’s modulus decreases continuously with increasing temperature for both material states, Fig. 4 [17]. At 90 °C Young’s modulus for state E60B is given as 204800 MPa (E60A: 201,800 MPa).

The chemical composition as determined with the emission-spectrometer (type ARL 34000 Quantovac) led to the composition as given in for the melt E60 [17].

3. Experimental investigations

The following additional investigations have been performed in order to obtain the damage mechanical parameters for the numerical analysis, [6]:

- Tensile test for state E60A and E60B at a temperature of 90 °C according to DIN50145 including fine strain measurements (T-specimen).
- Compression tests (T-specimens) for states E60A and E60B at 90 °C with compression specimens according to Siebel–Pomp.
- Notch tensile specimens with notch radii of 2 and 8 mm respectively (T-specimens) for state E60A and E60B.
- Crack resistance curves (TL-specimens) with side-notched CT25-specimens for state E60A in addition to the $J_R$-curve for the state E60B at a temperature of 90 °C.
- Metallographic cross-sections (TL, LS and TS) for state E60A and E60B to obtain size, shape and frequency distribution of non-metallic inclusions.
- TEM-investigations for state E60A and E60B.
As in [17] the tensile and compression specimens have all been obtained in T-direction at a depth of \( t/4 \) and \( 3t/4 \) respectively.

Figs. 5 and 6 show the tensile and compression yield curves of states E60A and E60B at a temperature of 90 °C. The results show only a minor scatter. The results of the tensile state for the initial state and the aged state are given in the following tables:

State E60A:

<table>
<thead>
<tr>
<th>Specimen Type</th>
<th>Temperature (°C)</th>
<th>Specimen</th>
<th>( R_{0.2} ) (MPa)</th>
<th>( R_{eH} ) (MPa)</th>
<th>( R_m ) (MPa)</th>
<th>E-modulus (MPa)</th>
<th>( A_s ) ( (10^{-2}) )</th>
<th>( Z ) ( (10^{-2}) )</th>
<th>( R_{p1.0} ) (MPa)</th>
<th>( A_g ) ( (10^{-2}) )</th>
</tr>
</thead>
<tbody>
<tr>
<td>A111</td>
<td>90</td>
<td>T</td>
<td>446</td>
<td>456</td>
<td>581</td>
<td>–</td>
<td>22.5</td>
<td>61</td>
<td>456</td>
<td>12.5</td>
</tr>
<tr>
<td>A112</td>
<td>90</td>
<td>T</td>
<td>441</td>
<td>450</td>
<td>578</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>449</td>
<td>–</td>
</tr>
<tr>
<td>A113</td>
<td>90</td>
<td>T</td>
<td>440</td>
<td>457</td>
<td>582</td>
<td>201800 (^{b})</td>
<td>23.5</td>
<td>58</td>
<td>455</td>
<td>12.5</td>
</tr>
</tbody>
</table>

\(^{a}\) Fracture in the notch.  
\(^{b}\) Mean value from six measurements.

State E60B:

<table>
<thead>
<tr>
<th>Specimen Type</th>
<th>Temperature (°C)</th>
<th>Specimen</th>
<th>( R_{0.2} ) (MPa)</th>
<th>( R_{eH} ) (MPa)</th>
<th>( R_m ) (MPa)</th>
<th>E-modulus (MPa)</th>
<th>( A_s ) ( (10^{-2}) )</th>
<th>( Z ) ( (10^{-2}) )</th>
<th>( R_{p1.0} ) (MPa)</th>
<th>( A_g ) ( (10^{-2}) )</th>
</tr>
</thead>
<tbody>
<tr>
<td>B111</td>
<td>90</td>
<td>T</td>
<td>557</td>
<td>568</td>
<td>689</td>
<td>–</td>
<td>22</td>
<td>55</td>
<td>570</td>
<td>11</td>
</tr>
<tr>
<td>B112</td>
<td>90</td>
<td>T</td>
<td>573</td>
<td>574</td>
<td>689</td>
<td>204800 (^{a})</td>
<td>19.5</td>
<td>55</td>
<td>570</td>
<td>10</td>
</tr>
<tr>
<td>B113</td>
<td>90</td>
<td>T</td>
<td>555</td>
<td>570</td>
<td>689</td>
<td>–</td>
<td>20.5</td>
<td>54</td>
<td>569</td>
<td>11</td>
</tr>
</tbody>
</table>

\(^{a}\) Mean value from six measurements.
The results of the three compression tests with the Siebel–Pomp specimen for the material state E60B are given in Fig. 6 together with the data for the initial state. The difference with respect to the 0.2% yield strength and the tensile strength in both material states amounts to about 120 MPa. The strength increase in compression is slightly higher as in tension. At a shape change of \( \varphi = 0.1 \) the difference amounts to about 40 MPa [6].

The experiment on the 20% side notch CT25 specimen was done on the basis of the \( J \)-integral with a single specimen and the method of partial unloading, according to ASTM E813–89. The result shown in Fig. 7 of the specimen complements the results documented in [17] for the melt E60. The \( J_R \)-curve at 90 °C for the initial state E60A results in a \( J \)-level of about 370 N/mm at a crack elongation of 2.5 mm. The \( J_i \)-value of the specimen amounts to 72 N/mm (stretched zone \( \Delta a_i = 0.063 \) mm). The corresponding \( J_i \)-value of the aged specimen amounts to 49 N/mm. The reduced crack resistance of the aged state is obvious.

The characteristic deformation and fracture behaviour of the initial state at a temperature of 90 °C is given by the fracture surface. In Figs. 8 and 9, fracture surfaces of the CT25-specimen are shown at different magnifications (REM). In a number of the dimples manganese sulphate particles are found which are the nuclei for the voids. Shape and orientation of the large dimples in both states, [6], reflect the shape and orientation of the respective large non-metallic inclusions.

As for the initial state, for the aged state three metallographic sections have been prepared (LS, LT and TS plane). The manganese sulphate particles in the LS-plane and LT-plane are oriented into the L-direction and elongated. Again the particles are not equidistantly distributed but are locally clustered (Fig. 10). The inspection of the metallographic cross-sections in the REM shows clearly the different shape of the MnS-particles in the different directions. It is found that especially the large particles appear in flat ellipsoidal shape, with the short axis in S-direction or thickness direction and the long axis in L-direction (rolling
direction). The same is true for the MnS-particles in the initial state. Besides the MnS-particles the metallographic cross-sections show also aluminum oxide particles with a less elongated shape (Fig. 11). These particles are also locally clustered and appear oriented in longitudinal direction.

The following table gives an overview over particle sizes which are obtained with the image analyzing system for both material states in the single cross-section planes. It has to be noted that the smallest values are practically identical with the lower resolution limit of the analyzing system at the analyzed magnification. The following values are obtained for the three cross-sectional planes at an analyzed area of 33 mm² according to a total number of 1200 measurement fields at a REM magnification of 500:

<table>
<thead>
<tr>
<th>Analyzed plane</th>
<th>Particle length $D_{\text{max}}$ ($\mu$m)</th>
<th>Particle length $D_{\text{min}}$ ($\mu$m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TS-plane</td>
<td>E60A: 1.2–56.4</td>
<td>E60A: 0.6–13.9</td>
</tr>
<tr>
<td></td>
<td>E60B: 1.2–73.6</td>
<td>E60B: 0.6–10.0</td>
</tr>
<tr>
<td>LT-plane</td>
<td>E60A: 1.2–60.5</td>
<td>E60A: 0.6–16.0</td>
</tr>
<tr>
<td></td>
<td>E60B: 1.2–78.7</td>
<td>E60B: 0.6–11.4</td>
</tr>
<tr>
<td>LS-plane</td>
<td>E60A: 1.2–130.2</td>
<td>E60A: 0.6–9.90</td>
</tr>
<tr>
<td></td>
<td>E60B: 1.2–87.5</td>
<td>E60B: 0.6–10.3</td>
</tr>
</tbody>
</table>

From this table the initial void volume fraction $f_0$ and the mean particle distance $l_c$ can be obtained, (2):

<table>
<thead>
<tr>
<th>Analyzed plane</th>
<th>Initial void volume fraction $f_0$</th>
<th>Particle distance $l_c$ (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TS-plane</td>
<td>E60A: $0.6064 \times 10^{-3}$</td>
<td>E60A: 0.049</td>
</tr>
<tr>
<td></td>
<td>E60B: $0.6486 \times 10^{-3}$</td>
<td>E60B: 0.043</td>
</tr>
<tr>
<td>LT-plane</td>
<td>E60A: $0.6849 \times 10^{-3}$</td>
<td>E60A: 0.050</td>
</tr>
<tr>
<td></td>
<td>E60B: $0.6121 \times 10^{-3}$</td>
<td>E60B: 0.044</td>
</tr>
<tr>
<td>LS-plane</td>
<td>E60A: $0.6703 \times 10^{-3}$</td>
<td>E60A: 0.053</td>
</tr>
<tr>
<td></td>
<td>E60B: $0.6439 \times 10^{-3}$</td>
<td>E60B: 0.046</td>
</tr>
</tbody>
</table>

A comparison of these results shows that the particle sizes are similar independent on the observed cross-sectional planes and the material state (Figs. 12 and 13). It should be noted that these values for the initial void volume fraction $f_0$ are practically identical to the value obtained from the chemical analysis in [3], by $0.7 \times 10^{-3}$.

For further microstructural characterizations in the nanometer regime, investigations have been performed with the analytical 200 keV transmission electron microscope (JEM–2000FX Jeol, Japan). Besides an imaging of the microstructure down to the nanometer regime, the crystal structure and orientation of phases can be principally determined. Furthermore, the microscope is equipped with a system of energy dispersive X-ray microanalysis of the company Tracer Northern, USA (TN-5500), in order to determine the chemical composition of the faces. Elements with ordering numbers larger or equal than 11 (Na) can be analyzed.

In Fig. 14, the correlation between the size distribution of copper precipitates and the strength

Fig. 10. MnS inclusions (LS-plane, M 1000:1).

Fig. 11. Alumina inclusions (LS-plane, M 500:1).
increase is schematically shown. Important is the transition from the initial to the aged state. This transition is characterized by an increase of the particle sizes, nucleation of new particles and, thus, a decrease of the particle distances. Assuming a constant volume increase of each particle results in a stronger size increase of smaller particles providing the strength increase.

Thin metal foils are used for the TEM analysis, they are prepared in the following steps:

- Initial 4 mm thick specimen.
- Polishing down to about 0.1 mm thickness with SiC polishing paper of grains 1000.
- Punching of disc of 3 mm diameter.
- Electrolytically thinning until a hole is formed with areas of a maximum thickness of 300 nm at the border of the hole.
- Reduction of the volume by punching a disc of 1 mm diameter from the central area.

The reduction of specimen volume is necessary in order to reduce disturbances from the magnetic field of the steel specimen during imaging in the TEM. The chemical composition of the available precipitates in the metal foil can be not exactly determined by EDS, because more or less matrix is measured as well, depending on the particle size. Therefore, in the frame of this work it was only qualitatively shown that the observed precipitates are really copper precipitates or copper rich precipitates, respectively.

In order to quantify the TEM observations, the TEM micrographs have been analyzed with an automatic image analysis system. As a first step, TEM micrographs (negatives) have been input into the computer of the system with a CCD-camera. After some improvement of the images the copper precipitates have been separated from the matrix with proper gray scale thresholds, because they are significantly darker as compared to the surrounding.

Particle sizes have been determined with 10 negatives for each specimen (magnification 100,000). The thickness of the specimen in the analyzed area was 200 nm. The analyzed area was 3 \( \mu m^2 \). It has to be noted that the detectability limit for the precipitates in these TEM analyses was 2 nm.

For the initial state 1321 particles have been evaluated. The size distribution of the particles...
shows that most of the particles possess a size between 2 and 15 nm. This corresponds with the large number of particles with small area (20 nm²). Most of the particles show values between 3 and 4 nm for the minimum diameter and between 4 and 5 nm for the maximum diameter, thus showing a relation between $D_{\text{max}}/D_{\text{min}}$ between 1:1 and 2:1.

The three micrographs in Fig. 15 give an impression of the present copper particles in the microstructure. Each figure gives an area of about $530 \times 680$ nm.

Fig. 15. Copper precipitates in different ferritic grains (TEM micrographs).

Fig. 16. Frequency distribution ($D_{\text{max}}$) of copper precipitates for the state E60A and E60B.

For the aged state E60B 2023 particles have been analyzed. Again, most particles are within the regime of 2–20 nm (Figs. 16 and 17). Similar as in the initial state, most particles have a width of 2–3 nm, a length of 4–5 nm and a ratio $D_{\text{max}}/D_{\text{min}}$ between 1:1 and 2:1.

The direct comparison of the results for both material states shows a significant higher number of particles with particle sizes between 3 and 7 nm for the state E60B. A comparison of lengths $D_{\text{max}}$ to $D_{\text{min}}$ shows a trend where the shape of the

Fig. 17. Frequency distribution ($D_{\text{min}}$) of copper precipitates for the state E60A and E60B.
particles is more globular in the aged state as compared to the initial state reducing the ratio $D_{\text{max}}$ to $D_{\text{min}}$ (Fig. 18). A comparison of the evaluation of particle areas shows a significant increase of particles with an area smaller than 20 nm$^2$ for the state E60B (Fig. 19).

4. Numerical investigations

Numerical analyses have been performed with the finite element program CASTEM [19] in order to determine the damage mechanical parameters for the Rousselier model for the two material states E60A and E60B. The elastic–plastic, geometrically non-linear damage mechanic finite element analyses have been performed for smooth and notched tensile specimens in axiallysymmetric manner and for different other specimen geometries (CT, TBP, SECT, CCP) two-dimensionally under the assumption of plane strain state. The following table gives an overview over the performed finite element calculations.

<table>
<thead>
<tr>
<th>Specimen shape</th>
<th>Calculation type</th>
<th>Average particle distance</th>
<th>Initial void volume fraction</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cylindrical tensile specimen</td>
<td>Axial–sym.</td>
<td>$l_{c1} = 0.05$</td>
<td>$f_{03} = 0.003^a$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$l_{c2} = 0.10$</td>
<td>$f_{04} = 0.004$ and $f_{05} = 0.005^a$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$f_{01} = 0.001–0.003^b$</td>
<td>$f_{05} = 0.003^b$</td>
</tr>
<tr>
<td>Notched cylindrical tensile specimen, $\rho = 2$ mm</td>
<td>Axial–sym.</td>
<td>$l_{c1} = 0.05$</td>
<td>$f_{01} = 0.001–0.003^b$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$l_{c2} = 0.10$</td>
<td>$f_{04} = 0.004$ and $f_{05} = 0.005^a$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$f_{01} = 0.001–0.003^b$</td>
<td>$f_{05} = 0.003^b$</td>
</tr>
<tr>
<td>Notched cylindrical tensile specimen, $\rho = 8$ mm</td>
<td>Axial–sym.</td>
<td>$l_{c1} = 0.05$</td>
<td>$f_{01} = 0.001–0.003^b$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$l_{c2} = 0.10$</td>
<td>$f_{04} = 0.004$ and $f_{05} = 0.005^a$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$f_{01} = 0.001–0.003^b$</td>
<td>$f_{05} = 0.003^b$</td>
</tr>
<tr>
<td>CT-specimen 2D/plane strain</td>
<td></td>
<td>$l_{c1} = 0.05$</td>
<td>$f_{01} = 0.001–0.003^b$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$l_{c2} = 0.10$</td>
<td>$f_{03} = 0.003^b$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$f_{01} = 0.001–0.003^b$</td>
<td>$f_{05} = 0.005^a$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$l_{c1} = 0.15$</td>
<td>$f_{03} = 0.001–0.003^b$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$l_{c2} = 0.20$</td>
<td>$f_{03} = 0.005^a$</td>
</tr>
</tbody>
</table>

Fig. 18. Ratio of length to width of the copper particles for the states E60A and E60B.

Fig. 19. Area distributions of the copper particles for the states E60A and E60B.
The number of performed loading steps amounts to about between 300 and 350, dependent on the shape of the specimen. The loading function was adapted in such a manner that after crack initiation, the crack grew by stiffness reduction by a maximum of one Gaussian point in the ligament per loading step, this corresponds to half of an element length or mean particle distance \( l_c \) respectively, otherwise convergence problems would occur [2]. The elements used for the calculation possess in both, the axial symmetric case (tensile specimen) and in the two-dimensional case (TPB, SECT and CCP specimens), eight nodal points and four integration points (reduced integration). All calculations have been performed in a displacement controlled manner. The discretization of the specimens with cracks was adjusted to the corresponding ductile crack growth. Modelling of ductile crack growth of e.g. \( \Delta a = 2.4 \) mm for \( l_c = 0.1 \) mm, requires 12 elements in the ligament, a reduction of \( l_c \) to 0.05 mm doubles the required number of elements.

In the numerical calibration of the Rousselier parameters only the parameter of the initial void volume fraction \( f_0 \) and the mean particle distance \( l_c \) are varied. The other three parameters, namely integration constant \( D = 2 \), stress \( \sigma_k = 445 \) MPa and critical void volume fraction \( f_c = 0.05 \) correspond to values from [2,3].

In Fig. 20 the chosen discretization for the CT25-specimen is shown together with the used number of nodal points and elements, making use of symmetry conditions.

The calculations for the tensile specimen with \( l_c2 = 0.1 \) mm are shown in Figs. 21 and 22. The comparison of four numerical calculations with experimental observations shows agreement to a large extent.

As expected, the technical yield curve of specimen A111 (state E60A) and specimen B111 (state E60B) can be well reproduced.

A comparison of the numerical results shows that for this specimen geometry the obtained result is practically invariant for the two parameters \( l_0 \) and \( l_c \). Also for the state E60B the calculated result is practically independent of the chosen initial void volume fraction \( f_0 \). Only before the point of failure (end of calculations) the calculated results differ to a small degree.

In calculating the notch tensile specimen the mean particle distance \( l_c \) was varied from 0.05 to 0.1 \( \mu \)m and the initial void volume fraction \( f_0 \) from 0.001 to 0.005 \( \mu \)m. The numerical results show that

---

### Specimen shape Calcula-
<table>
<thead>
<tr>
<th>Specimen shape</th>
<th>Calculation type</th>
<th>Average particle distance</th>
<th>Initial void volume fraction</th>
</tr>
</thead>
<tbody>
<tr>
<td>TPB-specimen</td>
<td>2D/plane strain</td>
<td>( l_c2 = 0.10 )</td>
<td>( f_{02} = 0.002^b )</td>
</tr>
<tr>
<td>SECT-specimen</td>
<td>2D/plane strain</td>
<td>( l_c2 = 0.10 )</td>
<td>( f_{02} = 0.002^b )</td>
</tr>
<tr>
<td>CCP-specimen</td>
<td>2D/plane strain</td>
<td>( l_c2 = 0.10 )</td>
<td>( f_{02} = 0.002^b )</td>
</tr>
</tbody>
</table>

\(^a\) Only state E60A.
\(^b\) State E60A and E60B.

---

**Fig. 20.** Finite element discretization and boundary conditions of the CT specimen with \( l_c = 0.1 \) mm.
the parameter \( l_c \) is of practically no influence on the load versus specimen cross-section reduction behaviour. Therefore, this specimen can be used to calibrate the initial void volume fraction \( f_0 \). From the comparison between the experimental and numerical fracture point it can be seen that for a notch radius of 2 mm the best agreement for \( f_0 \) lies in the regime 0.002–0.003 while for a notch radius of 8 mm smaller values of \( f_0 \) between 0.002 and 0.001 correlate better (Figs. 23–26). As a compromise between these comparisons a value of \( f_0 = 0.002 \) is used as best solution. The value \( f_0 \) of \( 0.65 \times 10^{-3} \) is obtained from metallographical examinations which results in a too late fracture point in the numerical calculations, i.e. too high values for the cross-sectional reduction \( \Delta D \).

The calculational results for the aged state show a similar behaviour as the results for the initial state. A comparison shows that for a notch radius of 2 mm the best agreement with \( f_0 \) values lies in the regime between 0.002 and 0.003, while for a notch radius of 8 mm rather smaller \( f_0 \)-values between 0.002 and 0.001 correlate better. As a compromise between these comparisons \( f_0 = 0.002 \) is seen as a best solution also for the aged state.
The numerically calibrated values for mean particle distances and initial void volume fractions are larger than the values for metallographic analyses and the $f_0$-value for the chemical composition ($f_0 = 0.009$). The numerical results seem to be more reliable as they provide the mechanical basis for the numerical damage mechanical simulations of the ductile crack growth.

For the material state E60A, 10 two-dimensional finite element calculations and the variation of the parameters $f_0$ and $l_c$ have been performed in order to analyze the CT-specimen. The analysis comprises the load versus crack opening behaviour, the crack growth resistance behaviour and the multiaxiality coefficient $q_r$ [20–23], in the ligament. The multiaxiality coefficient is defined as $q_r = (1/\sqrt{3})(\sigma_\n/\sigma_\ell)$ with $\sigma_\ell = (1/3)(\sigma_1 + \sigma_2 + \sigma_3)$. In constrast to the calculational results for the tensile specimens, the calculational results in the numerical simulation of ductile crack growth of CT-specimens influence $f_0$ as well as $l_c$. For the load versus crack opening behaviour an increase in $l_c$ means an increase in the calculated load at larger deformation values while $f_0$ influences the cracking of load decrease after the load maximum.

In general, an increase of $l_c$ results in an increase of the absolute shape level in a calculated $J_R$-curve while $f_0$ in general, is responsible for the slope of the curve [6]. With respect to the determination of the parameters $f_0$ and $l_c$ the best agreement with the experimental data from notched tensile specimens is obtained with an initial void volume fraction of $f_0 = 0.002$ and the mean particle distance of $l_c = 0.1$ mm.

In analogy to the initial state, 10 calculations have been performed for the aged state of material. The data used for the initial void volume fraction $f_0$ and the mean particle distance $l_c$ were the same as for the initial state.

A comparison of the experimental and numerical results for the load versus crack opening behaviour and the crack resistance behaviour shows that the values of $l_c = 0.1$ mm and $f_0 = 0.002$ as derived for the initial state lead also to good agreement with the experimental results for the aged state (Figs. 27 and 28). The data $l_c = 0.1$ mm and $f_0 = 0.002$ for this material state are again compromized between the results for the CT-specimen and the results for the notched cylindrical tensile specimen. Although the values $l_c = 0.1$ mm and $f_0 = 0.003$ result also in a good agreement for the CT-specimen a less favourable result is achieved for $f_0 = 0.003$ for the notched tensile cylindrical specimens with $p = 8$ mm as compared to $f_0 = 0.002$.

The comparison of the $J_R$-curves for both material states are shown in Figs. 29 and 30. As expected, the material state E60B shows a smaller crack resistance as compared to the material state E60A.
On the basis of the comparison of experimentally and numerically determined results for the initial state E60A at a temperature of 90 °C the parameters for the Rousselier model have been fixed as follows:

- Initial void volume fraction \( f_0 = 0.002 \)
- Critical void volume fraction \( f_c = 0.05 \)
- Mean particle distance \( l_c = 0.1 \) mm
- Integration constant \( D = 2 \)
- Stress \( \sigma_k = 445 \) MPa

These numerically calibrated values for the mean particle distance and the initial void volume fraction are larger than the values from the quantitative metallography (mean values from six single values are \( f_0 = 0.64 \times 10^{-3} \) and \( l_c = 0.047 \) mm) and the \( f_0 \)-value from the chemical composition \( (f_0 = 0.7 \times 10^{-3}) \). The numerically determined parameters are preferable as they provide the mechanical basis for numerical damage mechanical calculations of ductile crack growth. This discrepancy is probably due to the anisotropic shape of the inclusions and subject of present investigations.
The results for the multiaxiality coefficient \( q \) in the ligament under consideration of ductile crack growth show that \( q \) is nearly independent from the chosen pair of parameters. All chosen discretizations are fine enough (element length < 0.5 mm) in order to determine the stresses in the ligament good enough, and slightly different void volume fractions ahead of the crack tip to have no practical impact on the stress values in the ligament [6]. The analysis comprises the determination of \( q \) for five loading steps: linear elastic calculation, crack initiation load as well as three different crack growth values up to a maximum of 2.3 and 2.4 mm respectively. The area of high multiaxiality in the ligament of the CT25-specimen increases continuously with increasing load.

The results for the multiaxiality coefficient \( q \) for the aged state of the material shows a similar picture as for the initial state (Figs. 31 and 32). The multiaxiality in the ligament increases continuously with increasing load. Again the choice of the parameter pair \( f_0 \) and \( l_c \) has practically no impact on the distribution of the multiaxiality coefficient \( q \) in the ligament.

With respect to the determination of the crack growth resistance and the change of the multiaxiality coefficient \( q \) in the ligament by changing the material state, three further damage mechanical finite element calculations have been performed for three other specimen geometries with the same values for \( f_0 \) and \( l_c \). For a specimen with \( W = 50 \) mm and a crack depth relation of \( a/W = 0.5 \) (specimen thickness 20 mm) the calculations for the TPB-, the SECT- and the CCP-specimens have been performed under the assumption of plane strain state.

A comparison of calculated crack resistance curves shows the dependence on geometry (Figs. 33 and 34. The crack growth resistance increases in the sequence CT-specimen, TPB-specimen and SECT-specimen to CCP-specimen. Presently, experimental results are only available for CT-specimens.

![Fig. 31. Multiaxiality coefficient \( q \) in the ligament of the CT-specimen (state E60A).](image1)

![Fig. 32. Multiaxiality coefficient \( q \) in the ligament of the CT-specimen (state E60B).](image2)

![Fig. 33. Crack growth resistance under variation of the specimen geometry (state E60A).](image3)
The crack growth resistance behaviour is also reflected in the multiaxiality coefficient in the ligament of the specimen (Figs. 35 and 36). While the multiaxiality increases continuously for the already discussed CT-specimen under monotonically increasing load (the area with smaller $q$-values increases) the high multiaxiality decreases with increasing loading or remains restricted to a small local area for the other specimens.

In comparing the results for the new values of the CT-specimen it has to be considered that the crack depth relation of the two specimens is different (E60A: $a/W = 0.522$ and E60B: $a/W = 0.577$). Beside the material states the crack depth relation also influences the $q$-curves in the ligament. However, the changed material state does practically not change the multiaxiality in the ligament.

From these results it can also be concluded that the same $q$-curves result in the same crack resistance behaviour (CT-specimen, state E60A to be compared with TPB-specimen, state E60B). However, a similar $J_R$-behaviour does not necessarily correspond to identical $q$-values (compare the TPB-specimen in state E60A with SECT-specimen in state E60B).

5. Summary and conclusions

The aim of this contribution is to show the mechanical technological behaviour in fracture mechanical material characterization of the low alloyed warm strength material 15 NiCuMoNb5 (WB 36, material No. 1.6368). The characterization of this material at a temperature of 90 °C included the determination of the damage mechanical parameter for the Rousselier model for two material states of the melt E60. Both material states are in the ductile regime with respect to the notch impact energy at 90 °C.

The initial state (E60A) and the annealed state (E60B) of the melt E60 have been analyzed. The aged state characterizes the material state after
57,000 h of service at a temperature of 350 °C. During service, these materials show copper precipitations above 300 °C. These copper precipitations result in a strength increase as well as in a shift of the transition temperature of the notch impact energy to higher temperatures. The shift of the KV–T-curve of the melt amounts to 58 K.

The experimental analysis comprises the test of tensile specimens (smooth and notched) as well as the KV–T-curve of the melt. The experimental analysis comprises the test of tensile specimens (smooth and notched) as well as the KV–T-curve of the melt. The experimental analysis comprises the test of tensile specimens (smooth and notched) as well as the KV–T-curve of the melt.

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Two-dimensional damage mechanical finite element calculations for the two material states on the basis of the obtained Rousselier parameters and under the assumption of a plane strain state have been performed additionally for TPB-, SECT- and CCP-specimens. The $J_R$-characteristic of a specimen and its change is different for all specimens due to the dependence on the geometry. For material state E60B a reduction in crack growth resistance was obtained in all cases.

For all specimen geometries and material states the multiaxiality coefficient $q$ was evaluated for different ductile crack growth amounts in the ligament. For the CT-specimen the area of high multiaxiality increases with increasing loading while for the TPB-specimen a small, and for the SECT- and CCP-specimen a significant decrease of the multiaxiality was observed [6]. A comparison of the multiaxiality relations in the different specimens shows that for identical specimen geometry nearly no influence of the material states on the multiaxiality coefficient $q$ is observed. Similar $q$-curves in a ligament of two different specimens result in a similar $J_R$-behaviour. However, a similar $J_R$-behaviour does not automatically correspond to the same multiaxiality in the ligament of different specimens.

Finally, it should be noted that the experimentally proved hardening, as given in paragraph 3, can also be derived from dislocation theoretical calculations based on transmission electron microscopical obtained structural information of the melt [24]. Principally, it is also possible to calculate the growth of precipitates [25], and the hardening due to precipitates [26], by atomistic simulations on the nanoscale. Therefore, the damage mechanical calculations discussed in this contribution can also be embedded in a global picture of hierarchical modelling of materials.

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**References**


