THE INFLUENCE OF HIGH-TEMPERATURE CREEP ON THE ULTRASONIC VELOCITY IN ALLOY 800H

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INTRODUCTION

The occurrence of creep damage limits the lifetime of components that are exposed to stresses at temperatures higher than approximately half the melting temperature. Such conditions are generally met by a lot of structural components especially in power plants (pipes, turbines, etc.). According to conventional safety rules critical parts are usually exchanged long before any failure has to be expected. This procedure is based on statistics drawn from material tests by standardized methods rather than on the actual state of the component concerned. During the last years an increasing need can be stated to develop NDE methods for the detection of early damage stages in order to improve the reliability and safety of components. Basically, techniques are required which are sensitive to either small strains or, better, to small concentrations of micropores and microcracks, respectively. With regard to in-field applications, only replica techniques are used successfully for that purpose up to now [1,2]. These metallographic techniques are restricted to surfaces where appropriate spots have to be selected and to be prepared carefully. In this work the influence of creep damage on the ultrasonic velocity has been investigated on a representative high-temperature alloy for tube components, i.e., Alloy 800 H (X10 NiCrAlTi 32 20).

The high-temperature properties of this material are extensively studied by several laboratories within the framework of the European COST 501 action on high-temperature materials.

EXPERIMENTAL

Material

Hot-rolled rod material of X10 NiCrAlTi 32 20 (Alloy 800H) of the same cast was supplied by VEW-Austria to the laboratories concerned. The chemical composition and the solution treatment of the materials are given in Table 1 [3]. The mean grain size was 80 µm with a half-width of the distribution of about ± 50 µm [3].
Table 1. Chemical composition (wt %) and solution treatment of investigated Alloy 800H.

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>Cr</th>
<th>Ni</th>
<th>Ti</th>
<th>Al</th>
<th>N</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.070</td>
<td>20.26</td>
<td>31.11</td>
<td>0.31</td>
<td>0.34</td>
<td>0.009</td>
<td>bal.</td>
</tr>
</tbody>
</table>

1130°C/30min $\rightarrow$ < 2 min $\rightarrow$ $\sim$ 830°C $\rightarrow$ water

Specimens

Creep-tested specimens have been supplied to the author by several laboratories (see Acknowledgements). An overview on the testing conditions is given in Table 2. Except for one, all tests were performed at 800°C. Most tests were run under constant load conditions up to rupture, some tests have been interrupted after the steady state stage. Four specimens were run with a constant strain rate until steady state creep was reached.

The cylindrical creep specimens had diameters between 6 mm and 10 mm. In order to enable ultrasonic velocity measurements, plan parallel samples had to be machined out of the creep-tested specimens. Therefore, from each specimen several disks were prepared having thicknesses of about 3 mm with an accuracy of $\pm 2 \times 10^{-3}$ mm. From the reduction in diameter the local creep strain of each disk was determined. These samples were used to measure the ultrasonic velocity parallel to the load direction. In some cases samples were prepared to allow velocity measurements perpendicular to the load direction, too.

Table 2. Creep testing conditions of the investigated specimens.

<table>
<thead>
<tr>
<th>Testing procedure</th>
<th>Temp. (°C)</th>
<th>Stress (MPa)</th>
<th>Strain rate (sec$^{-1}$)</th>
<th>Number of specimens</th>
<th>Time to fracture (h) (magnitude of order)</th>
</tr>
</thead>
<tbody>
<tr>
<td>initial stress</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Constant</td>
<td>700</td>
<td>57</td>
<td>1</td>
<td>10$^4$</td>
<td></td>
</tr>
<tr>
<td>load test</td>
<td>800</td>
<td>45</td>
<td>5</td>
<td>10$^3$</td>
<td></td>
</tr>
<tr>
<td></td>
<td>60</td>
<td>6</td>
<td>5</td>
<td>10$^3$</td>
<td></td>
</tr>
<tr>
<td></td>
<td>70-80</td>
<td>6</td>
<td>6</td>
<td>10$^2$</td>
<td></td>
</tr>
<tr>
<td></td>
<td>98</td>
<td>2</td>
<td>2</td>
<td>10$^1$</td>
<td></td>
</tr>
<tr>
<td></td>
<td>130</td>
<td>2</td>
<td>2</td>
<td>10$^0$</td>
<td></td>
</tr>
<tr>
<td></td>
<td>150</td>
<td>1</td>
<td>1</td>
<td>10$^{-1}$</td>
<td></td>
</tr>
</tbody>
</table>

| saturation stress |                          |              |                         |                     |                                          |
| Constant          | 48          | $10^{-8}$    | 1                       | Test interrupt at begin of steady state creep /4/ |
| strain rate test  | 62          | $10^{-7}$    | 1                       |                                                         |
|                   | 77          | $10^{-6}$    | 1                       |                                                         |
|                   | 95          | $10^{-5}$    | 1                       |                                                         |
Velocity Measurements

Ultrasonic velocity measurements were performed by means of the pulse-echo overlap method [5]. The absolute measuring error

\[ | \Delta v/v| = |\Delta d/d| + |\Delta t/t| \]  

was approximately \(2 \cdot 10^{-3}\). Here, \(d\) is the specimen thickness and \(t\) is the time-of-flight between two backwall echoes. The frequencies used were 5 MHz for shear (T) waves and 10 MHz for longitudinal (L) waves. To minimize dispersion effects, narrow band pulses were applied by means of burst excitation. If not mentioned otherwise, the measured velocities were normalized to the velocities \(v_H\) obtained at the heads of the specimens in order to separate thermal effects from load induced effects.

RESULTS AND DISCUSSION

Aging Effects

According to the heat treatment of the material (see Table 1) most of the carbon is in solution at the as-delivered condition (Fig. 1a). The amount of carbides comes to about 0.2 wt % [6]. Aging at 800°C leads to the precipitation of \(M_{23}C_6\)-particles (Fig. 1b). As can be seen from Fig. 2a, the carbide content reaches a saturation level after approximately 15 h aging. This correlates well with an increase of both the ultrasonic velocities \(v_L\) and \(v_T\) during the first 16 hours which then remain constant within the measuring error (Fig. 2b). Concerning the creep tests referred to in what follows the specimens were usually preaged 16h/800°C before testing in order to ensure stable starting conditions.

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Fig. 1. TEM-images of Alloy 800H [7]: a) as-received material, b) after aging 16h/800°C.

Fig. 2. Amount of carbide residue [6] a) and ultrasonic velocity, b) in Alloy 800H as a function of aging time.
Figure 3 shows the creep stage map $\sigma/\dot{e}$ of Alloy 800H as proposed by Degischer et al. [3]. For stresses below 70 MPa a pronounced $\dot{e}_{\text{min}}$-range ($\dot{e}_{\text{min}} =$ minimum strain rate) is found with $\dot{e}_{\text{min}}$ being different from the steady state creep rate $\dot{e}_n$. This is explained [3] by carbide strengthening which retards the formation of subgrain structures usually encountered during primary creep. Such subgrains formed by dislocation networks have been found by TEM investigations at the $\dot{e}_{\text{min}}$-range for stresses above 40 MPa [7]. Measurements on specimens from constant strain rate tests interrupted at the beginning of steady state creep (see Table 2), i.e., end of primary creep when compared to a constant load test, did not show significant changes of ultrasonic velocity (see Fig. 4) as could eventually be expected according to the Granato-Lucke model of dislocation damping which yields

$$\Delta v/v_0 \sim \Lambda \cdot L^2$$  \hspace{1cm} (2)

for the low frequency range. Here, $v_0$ is the velocity without dislocations, $\Lambda$ is the dislocation density, and $L$ is the dislocation loop length. However, when measured during plastic deformation the effect on ultrasonic velocity due to changes in the dislocation structure is usually well below $10^{-2}$ (see [9]). Thus, the measuring accuracy was probably not sufficient in our case. In contrast, ultrasonic absorption proved to be sensitive to changes in the dislocation structure [10].

Fig. 4. Normalized ultrasonic velocities $v_L/v_L^0$ and $v_T/v_T^0$ at the beginning of steady state creep after constant strain rate tests.
Secondary and Tertiary Creep Stage

The secondary creep stage is generally described in terms of balanced strain hardening and recovery yielding a steady state creep rate $\dot{\varepsilon}_n$ with $\varepsilon \sim \sigma^n$. Towards the end of secondary creep the nucleation of cavities due to vacancy condensation and grain boundary sliding usually takes place. The growth of the vacancies leads to a loss in internal cross section, and an increasing strain rate is observed characterizing the beginning of tertiary creep. Finally, fracture will occur by plastic instability at a certain loss of cross section. For a complete description of the processes involved a lot of microstructural features have to be taken into account which are in many cases not fully understood in terms of basic principles, (see for example [11]).

Specimens originating from creep tests interrupted at the end of secondary creep as well as ruptured specimens have been investigated according to the procedure described above. The results are summarized in Fig. 5a for L-waves and in Fig. 5b for T-waves, respectively. The normalized velocities are plotted as a function of the local creep strain with initial stress $\sigma_0$ as parameter. In Fig. 6 the ratios $v_F/v_H$ ($v_F$-velocity near the fracture zone, $v_H$-velocity in the specimen head) are shown as a function of time to fracture $t_f$. Figure 7 illustrates the variation of the ultrasonic velocity from the specimen heads across the gauge length for three different stresses. From these results it can be seen that $v_L$ measured parallel to the stress direction increases with increasing plastic strain for $\sigma > 80$ MPa. Approaching the near-fracture region a maximum is reached and then $v_L$ decreases. For stresses between 45 MPa and 60 MPa a drop of $v_L$ up to 4% is measured starting at strains above 10%. Concerning T-waves the velocity decreases at strains larger than 10% for all stresses (Fig. 5b). Here the maximum effect is between 1% and 4% near the fracture zone (Fig. 6). Below strains of 10% the data are in the measuring error. Referring to the behavior of $v_L$ it is obvious that two different influences are operating. These are considered to be plastic deformation and porosity as will be shown below.
Fig. 6. Normalized ultrasonic velocities \( v_L, v_T \) near the fracture area of creep damaged specimens as a function of time to fracture \( t_f \) (Alloy 800H, \( T = 800^\circ \text{C} \)).

Fig. 7. Variation of ultrasonic velocity across the gauge length of creep damaged specimens (Alloy 800H, \( 800^\circ \text{C} \)).

Plastic Deformation

The change of ultrasonic velocity as a function of plastic deformation at room temperature (RT) is depicted in Fig. 8. Here, the measuring error \( \Delta \frac{v}{v} \) was \( \pm 10^{-3} \) because thicker samples could be used. It is found that \( v_L \) perpendicular to the stress direction is rather independent of the plastic deformation, whereas \( v_L \) parallel to the stress direction linearly increases. The transverse wave velocities \( v_T \) both parallel and perpendicular to the load direction (the latter one with
Fig. 3. Ultrasonic velocities $v_L$ and $v_T$ in Alloy 800H as a function of plastic deformation at room temperature.

Polarization in load direction ($v_L^H$) show the same linear decrease up to rupture whereas $v_T^H$ with both propagation direction and polarization perpendicular to the load levels off at plastic deformations larger than about 30%. At this strain, the onset of contraction was observed. The difference between $v_L^H$ and $v_T^H$ in the undeformed state indicates a texture due to the production process of the material. The uniaxial deformation applied here leads to a pronounced increase of the $<111>$-texture in load direction. This is a well-known effect for fcc-materials [12]. Because $v_L$ ($v_T$) in $<111>$-direction is larger (smaller) compared to the isotropic case, the behavior of $v_L$ and $v_T$ parallel to the load direction is readily explained in terms of texture. This should also hold for the propagation perpendicular to the load direction but the situation is more complex in that case.

Comparing now the results measured at RT-measurements with the results measured on the creep specimens, a rather good agreement can be stated between the plastic strain dependence of $v_L$ at RT (dashed line in Fig. 5a) and the creep strain dependence of $v_L$ at stresses above 100 MPa disregarding the near-fracture zone. Concerning shear waves (Fig. 5b) the effect of plastic deformation is larger at RT than after the creep tests if the 130 MPa-specimen is assumed to show the pure deformation effect for strains below 100%. Measurements of $v_L$ perpendicular to the stress direction did not show a dependence on the creep strain in agreement with the RT-measurements (Fig. 8). Concerning in service conditions, the applied stresses (and hence strains) are well below the stresses used here. In that case the influence of plastic deformation seems to be negligible.

Porosity

In contrast to the RT-measurements the longitudinal velocity $v_L$ drops if one approaches the fracture zone. Microstructural examinations of these zones exhibit the existence of cavities (micropores, microcracks). Some examples obtained by microradiography as well as by metallography are shown in Fig. 9. It is found that the cavity density in a given specimen is the larger the larger the strain. Additionally, for a given strain the cavity density is increasing with increasing time to fracture. It should be mentioned that the microradiographic images show the projection of the whole sample (3 mm thickness) on the film plane. According to Fig. 5a, the change of velocity at stresses below 60 MPa should be mainly correlated with the porosity whereas for higher stresses the plastic
deformation must be taken into account. The influence of porosity on the ultrasonic velocity can be calculated by ultrasonic propagation theory assuming spherical pores in an isotropic matrix [9,13,14]. The following expressions can be derived for $v_L$ and $v_T$ as a function of porosity $c_p$ (volume fraction) assuming $c_p<0.05$ and $d/\lambda \ll 1$:

$$v_L = v_L^0 (1 - k_L \cdot c_p) \quad \text{and} \quad v_T = v_T^0 (1 - k_T \cdot c_p).$$

(3)

Here, $v_L^0$, $v_T^0$ are the velocities without porosity, $d$ is the pore diameter and $\lambda$ is the ultrasonic wavelength. Using $v_L = 6 \text{ mm/\mu s}$ and $v_T = 3 \text{ mm/\mu s}$ one obtains from [14]: $k_L = 0.78$ and $k_T = 0.44$, i.e., the longitudinal wave velocity is nearly twice as sensitive with regard to porosity than the shear wave velocity. These theoretical results are in good agreement with experimental values obtained in service-exposed material as long as micropores are prevailing [15]. In the presence of microcracks the effect was even larger than expected from the values given above. In [15] the actual porosity has been determined by density measurements. If one relates the theoretical results to the velocity changes as measured on the creep specimens tested at stresses below 60 MPa, one may estimate porosities up to 5% at the fracture zone (Fig. 5). Because in that zone the damage patterns show mainly microcracks (see Fig. 9) this might be a factor 2 too large according to the results mentioned above, i.e., the theory which assumes spherical pores seems to underestimate the influence of porosity produced by microcracks.

On the other hand high precision velocity measurements should enable the detection of pore concentrations below $10^{-3}$. If one relates the creep strains $\varepsilon_{23}$ (G) at the transition from secondary to tertiary creep (see Fig. 3) to the corresponding velocity measurements, one obtains the values indicated in Fig. 5a by dots. One may recognize that the dropping of $v_L$ starts around these strains because the velocity becomes sensitive to the increasing porosity.

CONCLUSIONS

During high-temperature creep the ultrasonic velocity in Alloy 800H is mainly influenced by microstructural changes, plastic deformation and porosity. Microstructural influences (mainly precipitation processes) can be separated from damage influences by comparing material of the same microstructure, i.e., damaged and undamaged parts of a component submitted to the same temperature. The influence of plastic deformation on the ultrasonic velocity is large in the case of high stresses. Concerning service stresses, the effect is assumed to be negligible, whereas the influence of porosity becomes dominant in that case. Here, ultrasonic velocity measurements seem to be appropriate to detect small concentrations of micropores. Based on the creep properties of the considered material, it is suggested that ultrasonic velocity in Alloy 800H becomes sensitive to porosity at the transition from secondary to tertiary creep assuming reasonable measuring accuracy.

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Fig. 9. Examples of creep damage in Alloy 800H.