Creep Rupture Behaviour of Nickel Base Alloys for 700°C – Steam Turbines

C. Berger; J. Granacher; A. Thoma; Institut for Materials Technology,
Darmstadt University of Technology

Abstract
Nickel base alloys are important for future steam turbines operating at 700 °C or higher. As candidate materials special versions of the superalloys Inconel 706, Inconel 617 and Waspaloy are considered. An important aim is the determination of the creep rupture properties of these materials in a temperature range of 600 to 750 °C and partly up to 800 °C. Creep rupture tests have reached up to 15 000 h. Their results permit comparisons between the different alloys. In annealing tests performed up to 15 000 h a contraction is sometimes observed. Further, creep equations are established which support the determination of creep crack growth parameters. To develop recommendations on improved 700 °C-alloys a further testing of various derivatives of these alloys is intended.

Introduction
The mechanical long term behaviour of forged nickel base alloys is important for their use as rotor material in steam turbines at temperatures of 700 to 720 °C for service times up to 200 000 h. Different types of nickel base alloys were selected as candidate materials. These are Inconel 617 representing a solid solution hardened material, Waspaloy representing a γ'-strengthened material and Inconel 706 representing a γ''-strengthened material. Inconel 706 shall be examined in two different heat treatments (Table 1). The first version no 1A has γ and γ''-phase. The second version no 1B additionally has η-phase at the grain boundaries. This is due to a special heat treatment with a direct cooling procedure.

This paper describes the determination of the creep and creep rupture properties of the different candidate materials. They are in the focus of a joint DFG-research program combining the efforts of different partners to further develop these materials with the aim to obtain recommendations for optimum nickel base alloys for application up to 700 °C and higher.
8. Tables

<table>
<thead>
<tr>
<th>no</th>
<th>material</th>
<th>chemical composition (in mass %)</th>
<th>form of manufacture</th>
<th>heat treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Ni  Fe Cr Co Mo Nb Al Ti C other</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1A</td>
<td>Inconel 706</td>
<td>42 37 16 0.05 - 3 0.2 1.5 0.01 Mn: 0.07 Cu: 0.02 Si: 0.09 Ta: 0.01 P: 0.007</td>
<td>blocks from rotor-segment</td>
<td>980°C 2h air + 720°C 8h / 1K/min to 620°C 8h / F</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>980°C 3h / 4K/min to 820°C 10h / F + 720°C 8h / F + 720°C 8h / F</td>
</tr>
<tr>
<td>1B</td>
<td>Inconel 617</td>
<td>54 0.5 22 13 9 - 1.1 0.6 0.06 Mn: 0.03 Cu: 0.03 Si: 0.14 N: 0.015 B: 0.001</td>
<td>block</td>
<td>1180°C 2h / 4K/min to 700°C / air + 800°C 2h / air</td>
</tr>
<tr>
<td>2A</td>
<td>Inconel 617</td>
<td>54 0.5 22 13 9 - 1.1 0.6 0.06 Mn: 0.03 Cu: 0.03 Si: 0.14 N: 0.015 B: 0.001</td>
<td>block</td>
<td>1180°C 2h / 4K/min to 700°C / air + 800°C 2h / air</td>
</tr>
<tr>
<td>3A</td>
<td>Waspaloy</td>
<td>57 0.6 19 14 4.5 0.01 1.2 3 0.03 Mn: 0.05 Cu: 0.015 Si: 0.04 Zr: 0.061 P: 0.003 B: 0.005</td>
<td>rotor-segment 165 mm rd. x 70 mm thick</td>
<td>1080°C 4h / 4K/min to 700°C / air + 850°C 4h / air + 760°C 16h / air</td>
</tr>
</tbody>
</table>

Table 1. Chemical composition and heat treatment of the test materials

<table>
<thead>
<tr>
<th>material</th>
<th>microstructure</th>
<th>condition</th>
<th>no</th>
</tr>
</thead>
<tbody>
<tr>
<td>Inconel 706</td>
<td>$\gamma + \gamma''$</td>
<td>new, after heat treatment</td>
<td>1An</td>
</tr>
<tr>
<td></td>
<td>$\gamma + \gamma'' + \eta$ (matrix)</td>
<td>aged, 750°C 5000 h</td>
<td>1Aa</td>
</tr>
<tr>
<td></td>
<td>$\gamma + \gamma'' + \eta$ (grain boundaries)</td>
<td>new, after heat treatment</td>
<td>1Bn</td>
</tr>
<tr>
<td></td>
<td>$\gamma + \gamma'' + \eta$ (grain boundaries)</td>
<td>aged, 750°C 5000 h</td>
<td>1Bb</td>
</tr>
<tr>
<td>Inconel 617</td>
<td>$\gamma + \text{carbides}$</td>
<td>new, after heat treatment</td>
<td>2An</td>
</tr>
<tr>
<td></td>
<td>$\gamma + \text{carbides}$</td>
<td>aged, 750°C 5000 h</td>
<td>2Aa</td>
</tr>
<tr>
<td>Waspaloy</td>
<td>$\gamma + \gamma'$</td>
<td>new, after heat treatment</td>
<td>3An</td>
</tr>
<tr>
<td></td>
<td>$\gamma + \gamma'$</td>
<td>aged, 750°C 5000 h</td>
<td>3Aa</td>
</tr>
</tbody>
</table>
Creep Rupture Tests and their Results

On the candidate materials listed in Table 2 creep rupture tests are performed. All these materials are tested in two different conditions, in a virgin or new condition "n" after heat treatment and in an aged condition "a" after 5 000 h annealing at 750 °C. The latter condition approximates a mid-of-life-condition of 100 000 h at 700 °C following a Larson-Miller parameter $P_{LM} = T (22 + \log t)$. The creep rupture testing according to EN 10 291:2000 is mainly performed by interrupted tests and partly by uninterrupted tests. Some uninterrupted tests are continued in the interrupted mode until rupture after a certain time or plastic strain is reached. Tests on alloy Inconel 706 cover a temperature range of 600 to 750 °C, tests on alloys Inconel 617 and Waspaloy a temperature range of 600 to 800 °C.

![Graph showing density dependent strain $\varepsilon_d$ of alloys Inconel 706 and Inconel 617.](image)

Fig. 1. Density dependent strain $\varepsilon_d$ of alloys Inconel 706, no 1Bn and Inconel 617, no 2An, measured in interrupted annealing tests

In addition to the creep rupture tests interrupted annealing tests with strain measurement are performed. In most cases the density dependent strain $\varepsilon_d$ from such tests is a contraction. However, on alloys Inconel 706 (Fig. 1) and on Waspaloy no contraction is observed after longer test times. In earlier investigations Inconel 617 showed a contraction. However, on material 2An a density dependent strain $\varepsilon_d$ is observed with a saturation effect at approximately - 0.06 % after 5 000 h. Such a contraction has to be considered when a creep equation is modelled, whereas creep equations for the alloys Inconel 706 and Waspaloy can be directly modelled on the basis of the plastic strain $\varepsilon_p$.

In addition to the creep tests hot tensile tests in accordance to EN 10 002:1991 were carried out. They were performed with strain rates of 0.5 %/min up to 1% plastic strain and subsequently 5 %/min until rupture. The resulting flow curves deliver estimates of the initial plastic strain $\varepsilon_i$ of the creep tests. A comparison of the tensile strength values of the test materials shows a clearly reduced tensile strength for the aged conditions of the alloys Inconel 706 (Fig. 2), but an increase of tensile strength for alloy Inconel 617 which is caused by $\gamma'$-precipitation during ageing.
The creep rupture test results obtained until now were at first assessed by the usual graphical method which is based on plastic strain-time-diagrams and stress-time-diagrams. The values of the creep rupture strength $R_{u,T}$ and the creep strength $R_{p,T}$ (stress to produce a certain plastic strain $\varepsilon_p$ during a time $t$ at temperature $T$) were determined by crossplotting between the above mentioned diagrams. In addition the stress values were plotted against temperature $T$ and balanced curve families were established. The stress-time results are shown in Fig. 3. The values for 100 000 h could be estimated with an extrapolation time ratio $q$ of about 10. The final purpose is to reach test durations of up to 30 000 h for the candidate materials presented here as well as for newly developed materials in order to obtain secured predictions of 100 000 h-values.

Fig. 4 shows the graphically assessed values of the 0.2%-creep strength and the creep rupture strength for 10 000, 30 000 and 100 000 h against temperature $T$ for all test materials in the new condition after heat treatment ($n$). The material Waspaloy shows the best long term strength values, also the alloy Inconel 617 behaves very stable on a lower level. Inconel 706 shows relatively high values up to 650 °C in both versions. But with increasing temperature the creep rupture strength strongly decreases. If variations of the heat treatment will not decisively improve the structure of this alloy, the maximum admissible temperature will not exceed 650 °C. Further, relatively low values of rupture elongation are observed on this alloy. Especially on version 1An values as low as 3 to 4 % appear at 650 °C (Fig. 3).

**Parametric Assessment of Creep Rupture Test Results**

An alternative assessment of the creep rupture test results was performed on a time temperature parameter basis with the aid of program DESA. As a result of earlier assessments on alloys Inconel 617 and Inconel 718 the Larson-Miller-parameter with a constant of $C = 22$ can be recommended for alloys of the types considered here. This was confirmed by the new assessments carried out (Fig. 5). From the graphically determined master curves the creep rupture strength values for 10 000, 30 000 and 100 000 h can be compared in Fig. 3 to the graphically determined values. At the "main" temperature of 700 °C and similarly at 650 °C a good agreement is found for the alloys Waspaloy and Inconel 617. Some disagreement appears
Fig. 3. Creep rupture strength $R_u$ and rupture elongation $A_u$ of the test materials Inconel 706, Inconel 617 and Waspaloy in the new condition "n"
Fig. 4. Creep strength $R_{p0.2 \, \varepsilon T}$ and creep rupture strength $R_{u \, \varepsilon T}$ of the test materials for $t = 10\,000$, $30\,000$ and $100\,000$ h with extrapolation time ratio $q_e$.

Fig. 5. Time temperature parameter diagram of the creep rupture strength of the test materials in the new and aged conditions.
on the two versions of Inconel 706. For this alloy the parameter based predictions seem to be rather too optimistic as compared to the graphical predictions. If the creep rupture strength values of the materials in the new condition are compared to the correspondent values of the materials in the aged condition (Fig. 5), alloys Inconel 617 and Waspaloy show only small differences. That observation demonstrates the good stability of these alloys in the time temperature range of interest. However, the two versions of alloy Inconel 706 show a strong loss of creep rupture strength in the aged condition. This again speaks against a long term application of this alloy at temperatures exceeding 650 °C.

Creep Equation of Alloy Inconel 706, 1An

For the alloys considered, conventional creep equations are needed to support creep crack growth data evaluations which are performed by another partner of the joint research project. The modelling of such an equation for Inconel 706, 1An is described in the following. The creep equation is based on creep data up to 15 000 h at the temperatures of 650 and 700 °C and on creep data up to 1 000 h at the extrapolation temperatures 725 and 750 °C. A conventional creep equation was taken. Such equations describe primary, secondary and tertiary creep and can be modelled relatively easy. They are well appropriated to calculate the time dependent stress and strain distribution of components or test pieces which are subjected to quasistatic loading conditions. From the large number of such creep equations, the well proved modified Garofalo equation was taken, i.e.

\[ \varepsilon_p = \varepsilon_i + \varepsilon_{\ell1,max} \cdot H(t) + \varepsilon_{p,\text{min}} \cdot t + \varepsilon_{\ell3} \quad (1) \]

with initial plastic strain \( \varepsilon_i \), maximum amount of primary creep strain \( \varepsilon_{\ell1,max} \), a time function \( H(t) \) of primary creep strain, the minimum creep rate \( \varepsilon_{p,\text{min}} \) and the tertiary creep strain \( \varepsilon_{\ell3} \) (Fig. 6).

![Fig. 6. Linear creep curve and components of equation (1)](image)

![Fig. 7. Initial plastic strain determined from hot tensile tests (htt) and from creep tests (ct)](image)

The initial plastic strain \( \varepsilon_i \) was mainly taken from hot tensile tests but additional data points from uninterrupted creep tests were used (Fig. 7). Relatively small values \( \varepsilon_i \) appear. At a temperature of 700 °C and a stress of 600 MPa a value \( \varepsilon_i \) of about 0.05% is observed.
Nevertheless the initial plastic strain can not be neglected. For the modelling, the equation

$$\varepsilon_i = K_i(T) \cdot \sigma_0^n \cdot e^{c \cdot \sigma_0^d}$$

was taken. The constants $n$, $c$ and $d$ and the function $K_i(T)$ were determined by a stepwise regression analysis in a procedure which is similar to that described below for the modelling of the minimum creep rate\(^7\). Eq. (2) delivers a good interpretation of the data points up to a value of $\varepsilon_i = 0.2\%$ (Fig. 7).

With the initial plastic strain $\varepsilon_i$ from eq. (2) data points $\varepsilon_i(T, \sigma_0, t) = \varepsilon_p(T, \sigma_0, t) - \varepsilon_i(\sigma_0, T)$ could be determined to plot linear creep curves $\varepsilon_i(t)$ of the type of Fig. 6. From these curves the values of the minimum creep rate and the maximum amount of primary creep strain $\varepsilon_{\text{f,1max}}$ as well as the transition times $t_{12}$ and $t_{23}$ were graphically determined. The values of minimum creep rate and maximum primary creep strain were plotted against stress $\sigma_0$. Such plots offer the opportunity to check whether the values determined for different stresses and temperatures are well correlated.

If necessary, slightly adjusted values may improve the continuous course of the minimum creep rate $\dot{\varepsilon}_{\text{p,min}}$ and the maximum amount of primary creep $\varepsilon_{\text{f,1max}}$. Modelling of the minimum creep rate $\dot{\varepsilon}_{\text{p,min}}$ was the next step. From a plot of $\log \dot{\varepsilon}_{\text{p,min}}$ against $\log \sigma_0$ (Fig. 8) a simultaneous dependence of stress $\sigma_0$ and temperature $T$ became obvious. Such curve families can be well described by an equation of the type

$$\dot{\varepsilon}_{\text{p,min}} = K(T) \cdot \sigma_0^{n_\sigma} \cdot e^{a(T) \cdot \sigma_0^b} \big/ b$$

with constants $n_\sigma$, $T$ and temperature functions $K(T)$ and $a(T)$\(^5\). To model eq. (3) the generalized Norton-exponent

$$n_\sigma = \frac{\delta \log \dot{\varepsilon}_{\text{p,min}}}{\delta \log \sigma_0}$$

was determined from the isothermals in Fig. 8 and was plotted against stress $\sigma_0$ in Fig. 9. From this diagram the coefficient $n_0 = 1.15$ was

![Fig. 9. Stress and temperature dependence of exponent $n_{\sigma}$ of alloy Inconel 706, 1An](image)

![Fig. 10. Stress and temperature dependence of the quantity $n_{\sigma} - n_0$, Inconel 706, 1An](image)
determined and values of \( n_\sigma - n_0 \) could be plotted against stress \( \sigma_0 \) in a double logarithmic diagram (Fig. 10). From there the coefficients of an equation

\[
n_\sigma - n_0 = a(T) \cdot \sigma_0^b
\]

(5)
could be determined with a temperature dependent constant \( a(T) \) which could be modelled by the aid of a double Arrhenius equation

\[
a(T) = e^{B_3 - Q_3/T} + e^{B_4 - Q_4/T}
\]

(6)

with constants \( B_3, Q_3 \) and \( B_4, Q_4 \) determined from a plot of \( a \) against \( 1/T \). The integration of eq. (5) via eq. (4) delivers eq. (3) with an integration constant \( K(T) \). That constant was modelled in Fig. 11 with the aid of a simple Arrhenius equation\(^{(12)}\). If the minimum creep rate is recalculated with eqns. (3) to (6) the original data points are well interpreted in the whole range of temperature and stress (Fig. 8).

The primary creep strain of eq. (1) obeys to

\[
\varepsilon_{f1} = \varepsilon_{f1\text{max}} \cdot H(t)
\]

(7)

with its maximum amount \( \varepsilon_{f1\text{max}} \) and a time function \( H(t) \). The variable \( \varepsilon_{f1\text{max}} \) can be described by

\[
\varepsilon_{f1\text{max}} = a_i \cdot \sigma_0^{b_i}
\]

(8)

with coefficients \( a_i, b_i \) (Fig. 12). The transition time \( t_{12} \) can be described by

\[
t_{12} = \left( \frac{K_1}{\dot{\varepsilon}_{p\text{min}}} \right)^v
\]

(9)

with coefficients \( K_1 \) and \( v \) (Fig. 13).
The time function \( H(t) \) of primary creep\(^{7,8,12} \) can be described by
\[
H(t) = 1 - e^{-D(t/t_{12})^u}.
\] (10)

Following the principle that all components of the creep equation which are already modelled have to be used for the determination of the following components, the coefficients \( D \) and \( u \) are determined by a linear regression analysis in a plot of \( \log(-\ln(1 - (\varepsilon_f - \dot{\varepsilon}_{\text{p, min}} \cdot t) / \varepsilon_{f, \text{max}})) \) against \( \log(t/t_{12}) \)\(^{12} \).

Finally the tertiary creep term of eq. (1) has to be modelled. It can be described\(^8\)\(^{9} \) by
\[
\varepsilon_f = K_3 \cdot (t/t_{23})^f.
\] (11)

The transition time \( t_{23} \) depends on the minimum creep rate \( \dot{\varepsilon}_{\text{p, min}} \) in analogy to eq. (9). For the determination of the coefficient \( K_3 \) and the exponent \( f \), the quantity \( \log(\varepsilon_f / \varepsilon_f^1 - \varepsilon_f^2) \) is plotted against \( \log(t/t_{23}) \) with data points \( \varepsilon_f(t) \) and the quantities \( \varepsilon_f^1 \) according to eqns. (7) to (10) as well as \( \varepsilon_f^2 \) according to the expression \( \dot{\varepsilon}_{\text{p, min}} \cdot t \).

With the creep equation of Inconel 706, 1An according to eqns. (1) to (11) it was possible to recalculate all creep curves in a temperature range of 650 to 750 °C and for stresses from 100 to 600 MPa. As an essential point for practical calculations, all strain terms of these equations converge to 0 for \( \sigma_0 \rightarrow 0 \). In Fig. 14 the calculated creep curves at the main temperature of 700 °C are compared to the original results of the creep rupture tests and a good agreement is shown.

### Conclusions

In the frame of a joint DFG-research program four alloys were selected as first candidate materials for rotors of 700 to 720 °C steam turbines. These candidate materials are two versions of Inconel 706 with different heat treatment and one version of alloys Inconel 617 and Waspaloy each. The candidate materials were examined in two conditions each, a condition „new“ and a condition „aged“ which was generated by additional annealing at 750 °C for 5 000 h to approximate a mid-of-life status of 700 °C, 100 000 h. An important criterion for the long term applicability of these alloys is the creep rupture behaviour. Therefore creep rupture tests are performed on material Inconel 706 in a temperature range of 600 to 750 °C and on materials Inconel 617 and Waspaloy in a temperature range of 600 to 800 °C. Test durations of 15 000 h have been reached up to now. As preliminary result, Waspaloy has the highest 100 000 h-values of 700 °C-creep rupture strength with approximately 270 MPa. Inconel 617 has clearly smaller values with a 100 000 h-value of about 140 MPa at 700 °C. A comparison
of the creep rupture strength values of the conditions new and aged indicates a good stability of these two alloys. On the opposite, creep rupture behaviour of both versions of Inconel 706 suggests that a maximum application temperature of 650 °C should be recommended for this material. At that temperature a 100 000 h-creep rupture strength of about 230 MPa is expected.

Conventional creep equations could be established for the first candidate materials with the aim to support creep crack calculations. The modelling of such an equation of the modified Garofalo-type was demonstrated on Inconel 706. This equation is capable of interpreting the creep behaviour of the material in the full temperature, stress and time ranges of interest.

In cooperation with the other partners of the joint research work, the investigations will be continued and extended to modified alloy types.

Thanks are due to the Deutsche Forschungsgemeinschaft for the promotion of the work.

7. References
6) Preussler, T.: Numerical description of the creep behaviour of superalloys; Dr.-Ing. Thesis Darmstadt, Germany D17 (in German).