Isothermal and anisothermal creep behavior of the nickel base Alloy 602 CA

Isothermes und anisothermes Kriechverhalten der Nickelbasislegierung Alloy 602 CA

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Components in industrial furnaces and apparatus construction are usually exposed to temperatures up to 1,200 °C as well as mechanical loading and corrosive attack of the environmental atmosphere. Furthermore, they undergo numerous temperature cycles in service. To describe the characteristics of the nickel base Alloy 602 CA, which is often used in this industrial sector, the long term creep behavior of this alloy was studied at temperatures from 800 °C to 1,200 °C with and without thermal cycling. During these tests unexpected effects could be noticed. At load levels representing typical component loads and isothermal temperature control an anomalous decrease in creep rate in the third creep stage was observed. Experiments under thermal cycling showed a considerably higher creep rate in the whole range than in the isothermal case. Within the present article, these findings are outlined in detail together with a brief discussion of causes and consequences based on extensive microstructural examinations.

Keywords: Alloy 602 CA / isothermal creep / low loads / thermal cycling / microstructure / high temperature

Bauteile im industriellen Ofen- und Apparatebau sind Temperaturen bis über 1.200 °C sowie mechanischer Beanspruchung und korrosivem Angriff durch die Umgebungsatmosphäre ausgesetzt. Außerdem kommt es während des Betriebs zu zahlreichen Temperaturwechseln. Um die Eigenschaften der Nickelbasislegierung Alloy 602 CA unter diesen Bedingungen zu beschreiben, wurde das länge- lige Kriechverhalten bei Temperaturen von 800 °C bis 1.200 °C mit und ohne zyklische Temperaturwechselbeanspruchung experimentell untersucht. Bei niedrigen Spannungen und isothermer Versuchsührung wurde hierbei eine ungewöhnliche Abnahme der Dehnrate im tertiären Kriechbereich beobachtet. Unter Temperaturwechselbeanspruchung konnte dagegen eine erheblich höhere Dehnrate im Vergleich zum isothermen Fall festgestellt werden. Die Diskussion der experimentellen Ergebnisse sowie der ergründeten Ursachen auf Basis mikrostruktureller Nachuntersuchungen sind Hauptgegenstände des vorliegenden Beitrags.

Schlüsselwörter: Alloy 602 CA / isothermes Kriechen / niedrige Lasten / Temperaturwechselbeanspruchung / Gefüge / Hochtemperatur

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1 Introduction

In the industrial furnace sector and the apparatus construction components are exposed to temperatures up to 1,200 °C and low mechanical loadings mainly due to gravity. At those high temperatures, corrosion, and despite the low load levels, creep is of major concern, limiting the life time of the components. The nickel material Alloy 602 CA was especially designed for this range of application. That is why it is used for components subjected to high thermal and corrosive demands as kiln rollers, radiant tubes, furnace muffles or retort furnaces. First examinations of the material behavior showed a high creep strength up to temperatures of 1,200 °C [1, 2]. However, these investigations are restricted to short-term applications so that creep tests with component relevant operation times up to 10,000 h were still missing and thus are addressed in the following.

In addition, studying the creep behavior under temperature cycling is crucial. During operation, industrial furnaces and thermal plants are exposed to a variety of different temperature cycles. Radiant tubes for example are heated by gas-fired burners, which work in on/off-operation. Due to this, the components are exposed to continuous high frequency cycles with low temperature amplitudes [3]. Control fluctuations or batch processes can lead to temperature descents which are often followed afterwards by a temperature rise. These kinds of temperature changes are more low frequent and characterized by higher temperature differences. To address these different scenarios an extensive experimental program was conducted to investigate the relation between the high temperature loading and the resulting creep answer as well as resulting microstructural changes. Major relevant findings are described and summarized in the following sections.

2 Experimental

Within this chapter the performed creep experiments carried out at the wrought alloy VDM Alloy 602 CA (Nicrofer 6025 HT, 2.4633, NiCr25-Fe10AlY) in a temperature range from 800 °C to 1,200 °C and durations of above 10,000 h were described in detail. Prior to that, a brief summary of the alloy and his microstructural characteristics will be provided.

The alloy contains approximately 62 wt% of nickel and 25 wt% of chromium. Light microscopy images show the differences of the microstructure in the as-cast condition of different melts, Figure 1a–c. Those melts differed in the average grain-size (melt 1: 80 μm, melt 2: 33 μm and melt 3: 63 μm).

Due to the high nickel content and the primary precipitated carbides the alloy shows a high creep resistance up to temperatures of 1,100 °C. The high chromium content causes the development of oxide surface protection layers. Above 1,100 °C primary Cr3C2-carbides transform into Cr23C6-carbides. These Cr23C6-carbides do not completely dissolve up to temperatures of 1,250 °C [4]. As a result, they contribute to a high creep strength even at temper-
ature levels above 1,100 °C and prevent coarse grain formation [5]. In addition to the chromium oxide layer the examined alloy also forms an aluminum oxide layer [6]. The addition of yttrium leads to a better adhesive strength and crack resistance of the oxide layer [7]. Zirconium decreases the tendency of micro cracking at the grain boundaries under creep loading in oxygen containing environments [8].

All isothermal tests were conducted following DIN EN ISO 204 [9]. All of the specimens were manufactured out of a provided sheet material transversely to the rolling direction. For the cylindrical specimens, a gauge diameter of \( d_0 = 8.4 \text{ mm} \) has been used (melt 1 and 3), Figure 1a, c. Additionally, flat test pieces with a cross section of \( 2 \times 12.9 \text{ mm}^2 \) in the testing area were manufactured (melt 2), Figure 1b. All of the experiments were carried out in single specimen testing machines. The elongation of the specimens was measured with axial extensometers mounted at manufactured specimen ridges.

The creep tests presented in this article can be classified into two categories: isothermal and anisothermal loading both with a constant mechanical loading, Table 1. At 800 °C comparably high stresses (up to 62.5 MPa) were applied. At 1,100 °C and 1,200 °C only very small mechanical loadings were applied, representing expected loading levels in the components, which are mostly exposed to their dead weight only.

The thermal cycling experiments were conducted with two different cycle forms. For the first principal shape of cycle a heating and a cooling ramp of 0.5 h was chosen. Furthermore a hold time of 1.5 h (\( t_{\text{cycle}} = 4 \text{ h} \)) or 3.5 h (\( t_{\text{cycle}} = 8 \text{ h} \)) at the minimum and the maximum temperature of the cycle was induced, Figure 2a, Table 1. For these kind of cycles a convection furnace with three heating zones was used.

The second principle cycle shape represents a high frequency thermal cycle with a triangular form without holding times. The cycle duration here was 2 min. One test was carried out with cycles between 1,050 °C and 1,100 °C and another one between 900 °C and 1,100 °C, Figure 2b [10–12]. For the realization of these faster cycles an inductive heating device has been used.

### 3 Results

The results of the introduced creep testing campaign on Alloy 602 CA are presented and discussed in the following subchapters.

| Table 1. List of conducted creep tests. |
|-----------------------------------------|
| **Isothermal creep tests** | **Anisothermal creep tests** |
| Temperature [°C] | Stress [MPa] | \( \Delta T = 200 \text{ K} \) | \( \Delta T = 50 \text{ K} \) |
| 800 | 62.5 | 37 | t = 8 h | t = 4 h | t = 2 min | t = 2 min |
| 900 | 2 | 2.5 |
| 1,100 | 2 |
| 1,200 | 1 | 2 |

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3.1 Isothermal creep experiments

As long as not marked differently all of the subsequent results outlined in the following have been achieved by using the melt with an average grain size of 33 μm, Figure 1b. Creep tests at 800 °C and with different mechanical loadings were performed, Figure 3. For the high stress of 62.5 MPa a typical creep behavior, characterized by a decrease in creep rate in the primary creep stage down to a minimum and an increase in creep rate in the tertiary creep stage until final specimen rupture can be concluded. A pronounced secondary creep stage with a constant minimum creep strain is not present.

This well-known trend however has not been observed for the lower loading levels of 27.8 MPa and 37 MPa. Here, after the tertiary creep is visible, a subsequent significant “amid-tertiary decrease” of the creep rate has been measured. This led partially to a considerable extension of the creep rupture time as can be seen in the linear representation of the measured values, Figure 4. The two tests at 37 MPa showed the same effect even if the specimen rupture times differed noticeably. Looking at the absolute creep strain levels, the post-tertiary decrease in creep rate starts at comparably high strains in the range of 10 to 20 %, Figure 5.

This general observation of amid-tertiary decreasing creep strain rates were noticeable within the whole examined temperature range in the area of low mechanical loadings. Experiments at temperatures of 1,100 °C and 1,200 °C and low mechanical loadings showed a decrease in the strain rate already in the early stages of the tertiary creep regime at strains less than 10 %, (tests with

Figure 3. Creep curves at 800 °C (a) and plot of creep rate vs. time (b), melt 2.
Bild 3. Kriechkurven für 800 °C (a) und Verläufe der Kriechrate über der Zeit (b), Schmelze 2.
At higher mechanical loadings (5.5 MPa at 1,100 °C, 4 MPa at 1,200 °C), the decrease in creep rate started later and not before reaching roughly 10% creep strain. Further creep tests at 1,200 °C on the melt with the coarser grain structure and an average grain size of 80 μm showed relatively constant creep rates over the entire test duration without a distinctive primary creep regime, Figures 1b, 8. At this melt and temperature only one creep test at the highest mechanical loading (5 MPa) exhibited a decrease in creep rate in the late tertiary creep regime. At lower mechanical loadings the creep strain rate showed a rather serrated characteristic at longer test durations. These small increases and decreases in the creep strain rate indicate the Portevin-Le Chatelier effect which is characterized by an oscillating creep strain or strain rate [13].

3.2 Anisothermal creep experiments

To fundamentally understand the creep behavior under thermal cycling condition further anisothermal creep tests have been carried out by consistently using melt 3 with an average grain size of 63 μm in the as-cast condition, Figure 1c. Since 2 MPa have been chosen as the load level for these anisothermal tests, at first isothermal creep tests at the minimum (900 °C) and maximum (1,100 °C) cycle temperature were carried out for comparison reasons, also using melt 3 shown in Figure 1c. Again, the value of 2 MPa was chosen referring to the component “radiant tube” with stresses typically in the order of a few MPa due to dead weight loading. First for comparison isothermal creep tests were carried out, Figure 9. Here, the strain response of the first thousand hours is plotted which fits to the expected strain behavior over time.

For studying anisothermal creep, the results of two performed thermal cycling tests with cycle durations of 4 hours and 8 hours are displayed together with the isothermal creep curves, Figure 10a. The measured strain of both thermal cycling tests showed a significantly higher trend than the measured strain of the isothermal tests even for the isothermal test at 1,100 °C. A repeated test which has been conducted for the 4 h-cycle shows a nearly identical creep behavior, Figure 10b. Hence, the effect of the increased creep strain under thermal cycling condition can be reproduced.

To clarify whether this effect might be caused by a superimposed thermal induced stress, a further repeated test has been conducted but with a smaller specimen diameter. Because the thermal induced stress depends on the diameter and will be generally more pronounced for larger specimens, this direct experimental comparison is of general interest. The results of the specimen with a smaller diameter of only 5.6 mm are compared to the results of the tests performed with the standard specimen diameter.
Figure 6. Creep curves at 1,100 °C (a) and of creep rate vs. time (b), melt 2.

Bild 6. Kriechkurven für 1.100 °C (a) und Verläufe der Kriechgeschwindigkeit über der Zeit (b), Schmelze 2.

Figure 7. Creep curves at 1,200 °C (a) and creep rate vs. time (b), melt 2.

Bild 7. Kriechkurven für 1.200 °C (a) und Verläufe der Kriechgeschwindigkeit über der Zeit (b), Schmelze 2.

Figure 8. Creep curves at 1,200 °C (a) and creep rate vs. time (b), melt 1 with AGS of 33 μm.

Bild 8. Kriechkurven für 1.200 °C (a) und Verläufe der Kriechgeschwindigkeit über der Zeit (b), Schmelze 1 mit mittlerer Korngröße von 33 μm.
diameter of 8.4 mm, Figure 11. The measured strain is even slightly higher for the smaller specimen variant but generally within material scatter. Furthermore the level of a superimposed thermal induced stress gradient has been estimated by using the finite element method. For the standard specimen with the diameter of 8.4 mm a maximum temperature difference between the specimen surface and the specimen center of 0.9 K for the case of heating up from 900 °C to 1,100 °C in half an hour has been simulated. Thereof a maximum thermal stress of around 0.15 MPa is induced in this example. This stress is considered to be negligibly small in comparison to the applied stress. That concludes that thermal induced stresses are unlikely to be the reason for the higher creep strain measured in tests with cycling temperature.

Beside the comparably large cycle durations of 4 hours and 8 hours tests at higher temperature cycle frequencies with a cycle duration of 2 min but by utilizing an induction furnace have been carried out. For these tests 1,100 °C has been chosen as the maximum cycle temperature whereas different minimum temperatures of 1,050 °C and 900 °C are investigated, Figure 12. The average creep rate with

Figure 9. Isothermal creep tests at 900 °C and 1,100 °C at 2 MPa, melt 3.
Bild 9. Isotherme Vergleichs-Kriechversuche bei 900 °C und 1,100 °C bei 2 MPa, Schmelze 3.

Figure 11. Comparison of isothermal creep tests with creep tests with 4 h-cycle and different specimen diameter, melt 3.
Bild 11. Vergleich von isothermen Kriechversuchen mit Kriechversuchen mit 4 h-Zyklus mit unterschiedlichen Probendurchmessern, Schmelze 3.

Figure 10. Comparison of isothermal creep tests at minimum and maximum temperature with low cycle creep tests with 4 h- and 8 h-cycle duration (a) and repeated creep test with 4 h-cycle duration (b), melt 3.
Bild 10. Vergleich von isothermen Kriechversuchen bei Minimal- und Maximaltemperatur mit niederzyklischen Kriechversuchen mit 4 h- und 8 h-Zyklus (a) und wiederholter Kriechversuch mit 4 h-Zyklus (b), Schmelze 3.
the larger temperature range ($\Delta T = 200$ K, $T_{\text{min}} = 900$ °C) was higher than with the smaller temperature range ($\Delta T = 50$ K, $T_{\text{min}} = 1,050$ °C). This leads to the preliminary conclusion that the temperature range itself is even more dominant than the average cycle temperature.

3.3 Microstructural examination

To clarify the root cause for the two observed phenomena in the following, selected microstructural examinations of the isothermally and the anisothermally tested specimens are outlined.

3.3.1 Isothermal creep behavior

In order to investigate specific microstructural changes or transitions, examinations were carried out on some selected specimens. Here, microsections were removed from the gauge length in longitudinal direction. For all shown micrographs the loading direction is orientated horizontally.

To determine a change in the microstructure during the decrease in strain rate in the late tertiary regime two specimens were tested with the same testing parameters than the two already shown ($T = 800$ °C, $\sigma_0 = 37$ MPa) but those tests were aborted before rupture after different durations of 416 h and 715 h, Figures 4, 13. The specimen for which the test has been stopped after 416 h was just at the beginning of the tertiary regime. Here, first pores in the order of several micrometers could be found as expected. The specimen with 715 h duration was stopped right after when the amid-tertiary decrease in creep rate became obvious. The microstructural examinations show a strong increase in the amount and size of pores and separations, which were evident over the whole gauge length. The separations are macroscopically visible as cracks. Energy dispersive x-ray analyses showed that the darker phases at the crack surface are chromium and aluminum oxides. Small rod or needle-like precipitations in the matrix in the vicinity of the cracks could be identified as aluminum nitrides. Beside an increased oxide layer thickness, an analogous microstructural state can also be concluded for the specimen investigated after 1,315 h, Figure 4. Here at the crack tip the amount and size of the newly formed precipitations is increased.

A further phenomenon which could be observed can be explained by examining the microstructure of a specimen tested at 1,100 °C and 2.5 MPa. Here a pronounced coarsening of the grain structure can be observed, Figure 14. In the bulk, the grain coarsening was not as pronounced as in the near-surface region which represents the carbide depleted zone, too. That might lead to the conclusion, that in the bulk region the precipitations impeded the grain coarsening whereas the dissolution of the carbides in the surface-near area was traced back to the withdrawal of chromium of the matrix material to build an oxide layer at the surface [14]. Even though the grain coarsening in the bulk was presumably slowed down by the precipitations, the grain size still reached an average value of 90 μm in contrast to the original 33 μm average grain size. The decrease in strain rate might therefore be a result of an enhanced creep resistance of the microstructure due to the coarsening of the grain structure. However, the specimens tested at 800 °C showed no pronounced grain coarsening. In addition a further test at 1,100 °C and 3 MPa for 1,516 h but on the melt with the larger initial grain size of 80 μm developed only a small increase up to a final grain size of 105 μm observed after test-end, Figure 1a [15].
Most specimens were tested until rupture or until they reached a high strain (at least 40%) or very long test duration (> 10 kh). The micrographs of these specimens exposed strong creep damage and oxidation especially at temperatures higher than 800 °C. Cracks could be found over the whole gauge length and they were often plated with oxides and surrounded by rod shaped or polygon shaped precipitations, Figure 15. At 1,200 °C many large, round pores were formed additionally to the cracks, Figure 16. Moreover massive oxides could be observed even at the specimen core and after a rather short time of 147 h. Carbides were not found. The micrograph of a specimen tested at 1,200 °C with 4 MPa after 2,166 h showed a fully oxidized cross section, Figure 17.

### 3.3.2 Anisothermal creep behavior

The examination of the microstructure of the anisothermally tested specimens by light microscopy revealed a comparable picture. In comparison to the isothermally tested specimens no fundamentally different microstructural characteristics could be found.
Creep tests with the 8 hour cycle duration were stopped after 700 h and 5,100 h runtime, Figures 18, 19. The micrographs show an oxide layer formed at the surface. In the subjacent region inner corrosion could be detected. Similar to the isothermal specimens a carbide depletion zone developed underneath the oxide layer. With increasing running time the oxide layer grew and also the carbide depleted fringe became broader. In addition it can be very clearly seen that there is an increased creep pore formation in the near-surface area, Figure 19a. Due to the depletion of the carbides in this area, the formation of creep pores and cracks was eased. In the same region, a grain coarsening could be observed in both specimens, which might have its cause also in the depletion of precipitations. In the bulk material the grain size did not increase as much as in the surface area. The specimen with the shorter running time showed also some annealing twins, which form due to high temperature loading. In the surface region of this specimen, some creep pores could be detected even after only 700 h. Some of the earlier formed pores have agglomerated in the specimen with the longer duration, Figure 19. Furthermore, carbides moved from the inside of the grain to the grain boundaries where they formed a network. Creep pores developed at the grain boundaries or by depletion of these carbides and grew further together.

Microstructural analyses were also done for two specimens tested with the 4 h cycle. These speci-
mens were also examined after different runtimes, Figures 20, 21 (1,029 h, 7,608 h).

The specimen with the runtime of around 1,000 h showed characteristics comparable to the specimens tested with the 8 h cycle. At the specimen surface an oxide layer is visible. Below the surface, a carbide depletion zone could be discovered in which increased pore formation occurred at the grain boundaries. As mentioned before, the carbides in the core of the specimen moved from inside of the grains to the grain boundaries and formed a network.

The specimen tested for 7,600 h with the 4 h cycle showed a different microstructure, Figure 21. The pore formation as well as creep crack initiation and growth were proceeded significantly in comparison to the 1,000 h runtime specimen. To some extent the cracks were also oxidized as it could be observed in the isothermal specimen as well, Figure 15. However, in the anisothermal case the surface cracks and pores in the inner specimen part did not show as much oxidation as in the isothermal case. Rather a distinct coarsening of the carbides could be observed. It seems like some of them are dissolving leaving pores along the grain boundaries, which then turn into separations.

The micrograph of the specimen with the 2 min cycle and a temperature range of 50 K after a duration of approximately 800 h shows a comparable picture, Figure 22. In the surface region of the
specimen a carbide depletion zone with a propagated pore formation could be observed. A few grains also showed annealing twins. Moreover grain coarsening happened in the depleted area under the oxide layer. In the specimen bulk the carbides moved to the grain boundaries.

4 Discussion of results

During the creep testing of the material Alloy 602 CA unexpected effects could be noticed. In the isothermal case an unusual decrease in creep rate occurred in the tertiary creep regime whereas the creep velocity in the anisothermal case was higher than in the isothermal case at the maximum cycle temperature. Both effects will be discussed further in the following.

4.1 Results of isothermal creep testing

The observed decrease in the creep strain rate in the late tertiary creep regime might be a result of two phenomena: a changed microstructure in comparison to the initial state which leads to a higher creep strength as well as a relationship between decrease of the creep rate and the strong oxidation of the specimens and of the creep cracks.
The investigation of the chronological sequence of the microstructural changes during creep testing showed an increasing crack formation in the material and an oxidation of the creep cracks even in the deeper bulk regions of the specimens. This can be explained by means of a schematic model, Figure 23. Under creep loading condition cracks nucleate at grain boundaries in the tertiary regime. At the same time the oxide layer at the surface tears open due to the high deformation of the base material. So atmospheric oxygen and other components can penetrate into the core of the material by those cracks and the crack surfaces get oxidized. With increasing runtime and increasing creep strain new cracks form which lead to a further penetration of oxygen into the bulk material. Furthermore, needle-like nitrides form in the vicinity of cracks.

In literature, a few sources report a decrease in strain rate in the late tertiary creep region at pure nickel and nickel-chromium alloys, too [16–20]. Here the effect appears as well at low mechanical loadings and at testing temperatures above 800 °C. Creep tests, which were carried out in vacuum for comparison, did not show a decrease in the strain rate in the late third creep regime, which leads to the assumption that there must be a relationship between the oxidation of the specimen and the observed effect.

There are different mechanisms which might be responsible for the hardening of the material due to oxidation. Often thick oxide layers are formed in

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**Figure 22.** Microstructure after anisothermal creep loading with 2 min cycle duration, $\Delta T = 50$ K, 2 MPa after 817 h, a): surface area, b): specimen core, melt 3.

**Bild 22.** Gefüge nach anisothermer Kriechbeanspruchung mit 2 min Zyklus, 2 MPa und $\Delta T = 50$ K nach 817 h, a): Randbereich, b): Probeninneres, Schmelze 3.

**Figure 23.** Model of crack oxidation, schematic.

**Bild 23.** Modellvorstellung Rissoxidation.
the area of the crack tips, Figures 13, 15. They might lead to a blunting of the crack tip, whereby the crack growth is impeded and the overall deformation of the sample is slowed down. Also a strength enhancing effect of the nitrides or other precipitations, which are formed during the testing, is plausible. However, the complete oxidation of the specimen at 1,200 °C is an indication that the creep strength of the oxides themselves or rather the creep strength of the combined system of oxides and all metallic components of the material is sufficiently high and can even result in a strain hardening of the material. Tests at thin FeCrAl-foils (thickness around 50 μm) with a growing Al₂O₃-layer and an increased creep strength at low stresses and temperatures of 875 °C and 1,075 °C support this assumption [21]. It seems like all observed strengthening mechanisms (blunting of crack tip, formation of nitrides and other precipitations and a continuous oxidation network) are correlated to oxidation and appear at different states of oxidation.

On the other hand, the creep rate decrease in the early tertiary creep regime at strains in the range of 1 to 10 % and at testing temperatures of 1,100 °C and above occurs probably due to coarsening of the structure from 33 μm to up to 90 μm average grain size in the center of the specimen.

The characteristics of the different variants of the decrease of creep rate depend on temperature, stress and grain size, Figure 24. It must be taken into account that the specimens had different cross sections in the testing area and therefore both a size effect and the grain size might have an impact. A relationship between the decrease of creep rate and different specimen geometries is also reported in literature [15]. In these investigations no influence of specimen geometry could be observed for lower temperatures.

The decrease in creep rate due to oxidation occurred at specimens with a small grain size tested at 800 °C and high mechanical loadings. At 1,200 °C it could be concluded that the effect of the decreasing creep rate with decreasing specimen thickness at higher mechanical loadings occurs, while the effect of the grain size diminishes [15]. The results at 800 °C and 1,000 °C at an average grain size of 80 μm showed that the critical stress level at which the decrease in creep rate due to oxidation occurred declined with increasing temperatures. The decrease in creep rate due to grain coarsening could only be observed at specimens with an average grain size of 33 μm and at temperatures of 1,100 °C and above. There might also be a decrease in creep rate at larger average grain sizes if the specimens were tested at lower mechanical stresses and/or for a longer testing period.

Figure 24. Stress and temperature dependency of both types of strain rate reduction in the tertiary creep stage, schematic.

Bild 24. Schematische Darstellung der Spannungs- und Temperaturabhängigkeit der beiden Varianten der Dehnratenabnahme im tertiären Kriechbereich.
4.2 Results of anisothermal creep testing

The observed increased creep rate under aniso-
thermal conditions compared to the isothermal case
might be contributed by different factors with dif-
ferent intensities of dependence, differently pro-
nounced as summarized below.

One possible explanation for the increase in the
creep rate are clearly transient thermal stresses due
to temperature gradients induced in the specimens.
However this factor could be excluded by means of
both clear simulation and clear experimental re-
results.

At high temperatures (up to 1,100 °C) and low
mechanical loadings diffusional creep is the domi-
nating creep mechanism as can be seen in the so
called deformation mechanism maps (Ashby-maps)
[13]. This creep mechanism is located in the lower
right corner of the map, which indicates that this
effect is rather a “side issue” since the combina-
tion of high temperature and low mechanical stress is
not the typical loading condition in technical areas
of application. For this reason to date it is little dis-
cussed and rarely examined [22–24].

It was examined whether oxidation might con-
tribute to the increased creep rate as reported in lit-
erature [25]. Since the effect of an accelerated
creep strain development is observed already after a
few cycles and corresponding short runtimes
(10 hours to 20 hours), the influence of oxidation in
thermal cycling creep tests is rated to be not the
major influencing factor. A material damage by an
oxidative attack, which is rather a slow mechanism,
is ruled out as the main reason for the exceptional
creep rates.

The creep curve of an anisothermal creep test
with the same cycle parameters and specimen ge-
ometry but at higher mechanical loading of
4.6 MPa shows an analogous trend. This test
reached 10% strain after 550 h whereas it took
2700 h for the test with 2 MPa. An isothermal test
with 4.6 MPa at 1,100 °C reached 10% strain after
more than 4,600 h. The correlation between stress,
cycle parameters and strain development must
therefore be subject of future studies.

However the influence of the cycle parameters
by using the present state of investigation is sum-
marized and illustrated, Figure 25. The faster the
cycle is or the more thermal cycles per unit of time
are performed, the higher the corresponding meas-
ured strain will be. The right diagram shows the
creep rate of three of the discussed thermal cycling
creep tests after 1,000 h, normalized to the iso-
thermal creep test at 1,100 °C after 1,000 h. The
creep rate of the 8 h-test is for example more than
20 times higher than the creep rate of the iso-
thermal test.

The described effect under thermal cycling is re-
producible and is not only observed for the material
Alloy 602 CA, but also for the nickel cast alloys
2.4879 and Centralloy 60 HT R, as well as for the
steel 1.4841.

Figure 25. Dependence of mean creep rate on cycle duration (left), average creep rate for different cycle forms after
1,000 h, standardized on creep rate of isothermal 1,100 °C creep test after 1,000 h.

Bild 25. Abhängigkeit der mittleren Kriechgeschwindigkeit von der Zyklusdauer (links), mittlere Kriechrate nach 1,000 h für
verschiedene Zyklusformen, normiert auf die Kriechrate des isothermen 1.100 °C-Kriechversuch nach 1,000 h.
For engineering purposes a phenomenological creep description was developed within the scope of the AiF research project “Thermische Lastwechsel” 19068 N. This creep equation can describe the isothermal as well as the anisothermal material behavior [10–12]. Depending on the availability of new further findings, this description might be subject of changes with the availability of new research results and be replaced by a more mechanism based creep equation.

5 Summary and conclusion

The investigation of the creep behavior of the material Alloy 602 CA revealed new findings about its properties and behavior under isothermal and anisothermal long term creep loading and operation at temperatures up to 1,200 °C. The stress and temperature dependent results on the microstructure superimposed by oxidation and thermal cycling led to a complex conclusion.

The decrease in strain rate in the tertiary creep regime at low mechanical loadings observed in isothermal creep tests seems to be contributed by two different factors. For a fine grained structure (average grain sizes 33 μm) a decrease in creep rate could be observed for strains in the range of 1% to 10% and for high temperatures in the range of 1,100 °C. In metallographic examinations grain coarsening could be identified as a possible reason for the decrease in creep rate.

A second type of decrease in tertiary creep regime has been observed at all tested melts (80 μm and 33 μm average grain size) for all tested temperatures from 800 °C to 1,200 °C at low mechanical loadings but only after reaching 10% creep strain in the well-advanced tertiary creep regime. In those cases no grain coarsening was observed. The metallographic analyses showed, that in the tertiary creep regime creep cracks are formed, which tend to oxidize and blunt in the ongoing test. Therefore, it could be assumed that as a second factor the oxides themselves provide the supporting effect.

The stress area in which the second form of decrease in creep rate appears, is temperature as well as grain sized dependent. In general, the decrease in creep rate in the tertiary creep regime at isothermal testing condition leads to a significantly delayed rupture. The ongoing investigation of the formed oxides and their creep strength are issues for future work. For the material application the decrease in creep rate in the tertiary creep regime has minor relevance, since it appears only at relatively high strains, but the creep strength enhancing effect of the oxides can probably be used for future alloy development.

Another significant effect could be found in creep tests under cyclic temperature condition. At anisothermal temperatures and low mechanical loadings an increase in creep strain could be observed. The creep strain of those anisothermal tests achieved a higher level after only a few cycles in comparison to the creep strain of isothermal creep tests at the maximum cycle temperature. With decreasing cycle duration an increase in creep rate could be detected.

The microstructural investigations show similar phenomena as in the isothermal cases. Even if the behavior can be modelled with simple creep law extensions, the mechanism behind needs to be clarified and modeled by means of transmission electron microscopy analysis as well as with models based on dislocation hardening. Those investigations are topics of currently running follow-up research works.

Acknowledgements

The authors thank the Federal Ministry for Economic Affairs and Energy and the participating industrial partners for the financially support of the project IN-7505 of the program Innonet, project number IN-7505, support code 16IN0662. Furthermore the authors thank the AiF (Arbeitsgemeinschaft industrieller Forschungsvereinigungen e.V.) and the FOGI (Forschungsgemeinschaft Industriefenbau e.V.) for the financial support and the administrative monitoring of the AiF-project 19068N with the title “Thermische Lastwechsel von Ofenkomponenten”, which underlies this article. Open access funding enabled and organized by Projekt DEAL.
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Received in final form: January 8th 2021