Creep rupture in HP-Nb refractory steel tubes due to short-term overheating

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ABSTRACT
The premature creep rupture of cast reformer tubes made of HP-Nb steel due to short-term overheating, was investigated. Investigation was performed on specimens from ruptured, cracked, and uncracked tubes and included metallography, mechanical testing, and SEM/EDX analysis. It was found that accidental short-term overheating, after 5 years of normal operation, caused rapid tertiary creep, manifested by the formation of dense cavities and cracking, which eventually led to the premature creep rupture of several tubes of the reformer. Creep cavities formed preferentially at M$_7$C$_3$ carbides but also at M$_{23}$C$_6$ and MC carbides while crack propagation took place along the interdendritic carbide network. It appears that the radial direction of the columnar grains from the inside to outside diameter orients the grain boundaries perpendicular to the hoop stress, a situation which degrades creep rupture resistance. An analysis based on the Larson–Miller parameter confirmed the premature creep rupture of the tubes due to overheating.

1. Introduction
Creep resistance is the most fundamental requirement for processes taking place at high-temperature and high-stress conditions encountered in equipment such as furnaces, reactors, heat exchangers, and piping in power plants and oil refineries. Among the above processes, catalytic steam reforming of methane is used in oil refineries to produce hydrogen. The process takes place in a reformer furnace in tubes filled with a
nickel-based catalyst. The tubes are suspended in the furnace and are heated continuously at temperatures above 900 °C. The high temperature and the stresses due to the internal pressure in the tubes generate creep strains, thus, making creep resistance the main design requirement for the tube material. The design life is set to 100,000 h or 11.4 years according to API recommended practice (American Petroleum Institute, 1988). The tube material belongs to the class of heat-resistant steels containing Cr and Ni with additions of Nb and Ti. The state-of-the-art material is HP-Mo modified alloy (25Cr-35Ni). Increased creep resistance is due to the precipitation of highly stable NbC carbides (Barbabela, de Almeida, da Silveira, & Le May, 1991). The tubes are centrifugally cast and this generates a dendritic structure with austenitic dendrites oriented radially from the inner wall (IW) to the outer wall (OW) of the tubes. Despite the progress in tube materials, premature failures of the reformer tubing still takes place and is due to several reasons including thermal shock, excessive creep strains, overloading, and overheating. Previous research has shed light in mechanisms of creep damage either after a certain operating period of the reformer furnace (Alvino, Lega, Giacobbe, Mazzocchi, & Rinaldi, 2010; Bonaccorsi, Guglielmino, Pino, Servetto, & Sili, 2014; Gong, Tu, & Yoon, 1999; Lee, Yang, Yoo, & Cho, 2009; Ray et al., 2011), after complete creep rupture (Jian-Hua, Zeng, Zhi-Cao, & Yu-Cheng, 2018; Ray et al., 2003) or after creep testing (Alessio, Gonzalez, Pirrone, Iurman, & Moro, 2012; Attarian & Taheri, 2016; Ray, Roy, Raj, & Roy, 2016). The microstructural evolution during creep exposure has been investigated in the past (de Almeida, Ribeiro, & Le May, 2002; Kenik, 2003; Monobe & Schön, 2009). A recent study in reformer tubes indicated that various aging phenomena act as precursors to creep damage (Haidemenopoulos et al., 2019). These phenomena include carbide precipitation, coarsening and transformation. It was shown that M7C3 carbides transform to the more stable M23C6 carbides while the presence of Ti suspended the transformation of the primary NbC carbides to the Ni-Nb silicide of G-phase during the tube operating period. In addition, the room-temperature tensile elongation and the ultimate tensile strength were severely reduced (Haidemenopoulos et al., 2019).

Despite these studies, only limited work has been published on the evaluation of creep damage after overheating, either intentional, during testing (Wang, Xuan, Wang, Wang, & Liu, 2011), or accidental temperature excursions (Swaminathan, Guguloth, Gunjan, Roy, & Ghosh, 2008). The aim of this work is the study of creep rupture due to accidental short-term overheating. As stated above, most of the studies of creep damage in reformer tubes deal with tubes, which have operated for various periods under generally steady-state or isothermal conditions. There are very few reported cases of tube rupture due to accidental
overheating, lasting only few minutes, while the tubes are midway their
design creep life. The aim of the present work is to shed light in the
creep damage mechanisms that led to the premature rupture of the
reformer tubes.

2. Methodology

The reformer furnace under investigation contained a number of tubes
arranged in a radiant chamber. Heating for the reaction was provided
by burners and was transferred to the tubes by radiation. Tube opera-
tional data are given in Table 1. The tubes under investigation operated
continuously for 5 years prior to the incident at an average temperature
of 910 °C. The incident involved an accidental overheating with high
heating rate, following a first shut down, which took place after an
unexpected tripping of a combustion air blower. Following a shut-down
and restart of the reformer, the temperature increased from 450 to 1050 °C
within 15 min. A second temperature excursion took place from 700 to
1050 °C within 30 min before the final shut down of the reformer. The
temperature history is depicted in Figure 1. These temperature excursions
led to the premature rupture of several tubes as shown in Figure 2.

Table 1. Reformer tube data.

| Inside diameter | Outside diameter | Internal pressure | Operating temperature |
|----------------|------------------|-------------------|-----------------------|
| 103.3 mm       | 136 mm           | 30.8 bar          | 910 °C                |

![Figure 1. Temperature history indicating the accidental short-term overheating.](image)
As depicted in Figure 2, not all tubes of the reformer were ruptured. There were tubes with varying degrees of damage. The present analysis was based on information from three tubes, shown in Figure 3(a).

T1 was a tube from a location in the reformer where no rupture or cracking were found. T2 was a tube that did not rupture, but was in close proximity to the ruptured ones, exhibiting cracking, as depicted in Figure 3(b). Finally, T3 was a ruptured tube. The temperature distribution in the reformer is not uniform and, obviously, tube T1 was not exposed to the same maximum temperature as tubes T2 and T3. Chemical analysis of the tube material was performed on polished tube surface by optical emission spectrometry according to ASTM E1086-14 (2014) specification. The Spectrolab M8 (Spectro, Germany) spectrometer was employed. Characterization of microstructure, including carbide transformation and carbide morphology was performed by means of light optical metallography (LOM) and scanning electron microscopy equipped with an energy dispersive X-ray analysis system (EDX). Metallography was performed on through thickness specimens from all three tubes. Specimen preparation for metallography involved cutting with a Struers Accutom (Denmark) machine, grinding with SiC papers with 120, 320, 500, 800, and 1000 grit and polishing with 3 and 1 μm alumina paste. Etching was performed by swabbing with Kalling’s reagent (CuCl₂ in a mixture of hydrochloric acid and ethanol). Additional etching was performed with Marble’s reagent (CuSO₄ in a mixture of hydrochloric acid and water). Optical metallography was performed on an Olympus inverted microscope at magnifications 50-1000X. Scanning electron microscopy (SEM) was performed in etched specimens on a field emission JEOL JSM 7610 F (Japan) microscope equipped with an energy dispersive X-ray analysis (EDX) detector. Imaging was performed with secondary electron (SE) and backscattered electron (BSE) modes. Room temperature tensile testing was performed in three sections from the top, middle, and bottom of
Figure 3. (a) Schematic showing the samples location from Tubes T1, T2, and T3. (b) Tube T2 with cracking.

tube T1 according to ISO 6892:2019(B) specification (ISO. 6892-1, 2019). A Zwick SP 600 (Germany) hydraulic testing machine was employed for the tensile experiments. Stress rate was set to 20 MPa/s up to the yield point followed by a cross head separation rate of 0.1 mm/s till the end of the experiment.
3. Results and discussion

3.1. Chemical composition and tensile properties

The chemical composition of the tube material is shown in Table 2. The results indicate that it is a Fe-25Cr-35Ni HP-Nb austenitic steel with Nb and Ti additions.

Room temperature tensile testing results from three sections of T1 (top, middle, and bottom) are shown in Table 3. The results indicate a slight decrease of ultimate tensile strength (UTS) in the top and middle sections. However, there is a considerable reduction in tensile elongation, indicating that the material has aged due to continuous exposure at high temperature. Reductions in tensile ductility have also been reported in the recent study by Haidemenopoulos et al. (2019) after several operating periods as well as by Monobe & Schön (2009) after exposure for 200 h at 720–780 °C and Jian-Hua et al. (2018) after 36,000 h at 910 °C (4.12 years).

3.2. Microstructure analysis

3.2.1. Tube T1—tube with no visible damage

The microstructure of the alloy consists of austenitic matrix, forming dendrites, and a network of eutectic primary carbides. The carbides are located either in interdendritic or intradendritic regions. The dendrite length varies from 0.3 to 6.5 mm with an average length of 1.4 mm as depicted in Figure 4. The dendrites are oriented in the radial direction.

| Specimen | UTS (MPa) | Elongation (%) |
|----------|-----------|----------------|
| T1-top   |           |                |
| 1        | 429       | 4.4            |
| 2        | 452       | 5.7            |
| 3        | 424       | 5.0            |
| T1-middle|           |                |
| 1        | 428       | 6.6            |
| 2        | 430       | 6.5            |
| 3        | 386       | 5.2            |
| T1-bottom|           |                |
| 1        | 513       | 4.5            |
| 2        | 500       | 5.0            |
| 3        | 521       | 5.3            |
| Manufacturer minimum values | | 450 | 8 |
from the IW to the OW of the tube. Some dendrites are very long with a length being a significant fraction of the tube thickness. The carbides are either Cr-rich of $\text{M}_7\text{C}_3$ or $\text{M}_{23}\text{C}_6$ type and Nb-rich carbides. $\text{M}_{23}\text{C}_6$ are mostly grain boundary film-like carbides. $\text{M}_7\text{C}_3$ carbides exhibit a ‘chinese script’ morphology, either coarse-structured in interdendritic regions (Figure 5(a)) or fine-structured in intradendritic regions (Figure 5(b)). Chemical composition of carbides is provided in Table 4.

Carbide identification was based on SEM-BSE and EDX analysis, discussed below. Similar observations regarding the type and morphologies of various carbides in creep-exposed HP-Nb steels have been reported by Alessio et al. (2012) and de Almeida et al. (2002). In the as-cast condition, the austenite matrix is free of precipitates, as reported by Alvino et al. (2010). Long-term exposure resulted in aging as manifested by the secondary precipitation in the austenitic matrix and coarsening of carbides, as depicted in Figure 6, using both Kalling’s and Marble’s reagents. Such intragranular precipitation and carbide coarsening has been also observed by Attarian and Taheri (2016) in a HP-Nb steel, after aging at 982°C for 100 h.

3.2.2. Tube T2 - tube exhibiting creep cavities and cracking

Creep resistance depends, among other factors, on the thermal stability of the carbides. During operation, at high temperatures, the carbides undergo phase changes as well as morphological changes, which degrade the creep resistance of the material. Phase changes involve the transformation of primary $\text{M}_7\text{C}_3$ to the more stable, at the temperature of...
The NbC carbides transform to a Ni-Nb silicide, the G-phase, with nominal composition Ni$_{16}$Nb$_7$Si$_6$. The G-phase is hard and brittle and, therefore, prone for cavity formation when the material is subjected to temperature excursions (de Almeida Soares, de Almeida, da Silveira, & Le May, 1992; Jian-Hua et al., 2018). Morphological changes involve coarsening of carbides driven by the reduction of interfacial energy (Alvino et al., 2010; Attarian & Taheri, 2016). Finally, an aging reaction, manifested as secondary precipitation of carbides within the austenitic matrix takes place during creep exposure (Alvino et al., 2010). The microstructure of tube T2 material was investigated with

Figure 5. (a) Grain boundary film-like $M_{23}C_6$ carbides and coarse-structured chinese script $M_7C_3$ carbides, (b) fine-structured $M_7C_3$ carbides (Tube T1).
Table 4. EDX analysis (at%). Data associated with Figure 7(a,b) are taken from Haidemenopoulos et al. (2019).

|       | Cr | Ni | Fe | Nb | Ti | C  | M  | M:Cr | Carbide |
|-------|----|----|----|----|----|----|----|------|---------|
| Figure 5 |    |    |    |    |    |    |    |      |         |
| 1     | 51 | 6.5| 12.3|    |    | 30.9| 69.8| 2.25 | M7C3    |
| 2     | 29.5| 21.8| 26.2|    |    | 22.1| 77.5| 3.5  | M23C6   |
| 3     | 50.2| 7.5 | 10.5|    |    | 34.1| 68.2| 2.0  | M7C3    |
| 4     |    |    |    |    |    |    |    |      |         |
| 1     | 52.2| 1.7 | 46.1| 53.9| 1.17| MC |
| 2     | 50  | 2.7 | 47.3| 52.7| 1.11| MC |
| 3     | 51.3| 2.5 | 46.2| 53.8| 1.16| MC |
| 4     | 41.4| 5  | 53.6| 46.4| 0.86| MC |
| Figure 7(a) |    |    |    |    |    |    |    |      |         |
| 5     | 53.4| 7.8 | 16.8|    |    | 22  | 78  | 3.54 | M23C6   |
| 6     | 59  | 4.7 | 15.3|    |    | 21  | 79  | 3.76 | M23C6   |
| matrix | 25.4| 33.4| 34.7|    |    | 5.2 |      |      |         |
| matrix | 25.9| 33.2| 34.6|    |    |      |      |      |         |
| Figure 7(b) |    |    |    |    |    |    |    |      |         |
| 1     | 57.4| 1.4 | 7.6 |    |    | 33.6| 66.4| 1.97 | M7C3    |
| 2     | 28.1| 22.2| 29.2|    |    | 20.5| 79.5| 3.87 | M23C6   |
| 3     | 33.1| 19.6| 25.3|    |    | 21.3| 78  | 3.66 | M23C6   |
| 4     | 41.3| 2.7 | 56  | 44  | 0.78| MC |
| Figure 10 |    |    |    |    |    |    |    |      |         |
| 1     | 48.3| 6.7 | 15  |    |    | 30  | 70  | 2.33 | M7C3    |
| 2     | 49.2| 5.8 | 14.2|    |    | 30.8| 69.2| 2.24 | M7C3    |
| 3     | 60  | 5   | 14  |    |    | 21  | 79  | 3.76 | M23C6   |
SEM operating in the SE and BSE modes. Typical microstructures are depicted in Figure 7. MC, Nb-rich carbides, appear white, while $M_{23}C_6$, Cr-rich carbides appear dark grey in the BSE images due to the difference in atomic number between Nb and Cr. Carbide identification by SEM-BSE imaging has been used by other researchers in cast heat-resistant steels (Alvino et al., 2010; de Almeida et al., 2002; Haidemenopoulos et al., 2019). The numbers in Figure 7(a) correspond to EDX composition analysis, which is presented in Table 4. The composition analysis (EDX) of the carbides is semi-quantitative, as it depends on the spot size relative to the carbide size. The matrix contribution to the EDX spectrum was minimized by using a low voltage in the SEM. The results indicated

Figure 6. Secondary precipitation in austenite matrix and coarsening of primary carbides, (a) Kalling’s reagent, (b) Marble’s reagent (Tube T1).
that MC carbides contain Ti, which according to Swaminathan et al. (2008), stabilize the MC carbide against transformation to the G-phase. This transformation has been observed, in Ti-free alloys by Jian-Hua et al. (2018) and by de Almeida Soares et al. (1992). The transformation of the primary $M_7C_3$ carbides to $M_{23}C_6$ is depicted in Figure 7(b) with the associated EDX analysis of carbides. In addition, cavity formation can be seen around the MC carbides, while the interface of the

Figure 7. (a) SEM-BSE image depicting MC and $M_{23}C_6$ carbides, (b) Transformation of $M_7C_3$ to $M_{23}C_6$ carbide after creep exposure. Numbers indicate EDX composition analysis in Table 4 (Haidemenopoulos et al., 2019). Tube T2.
fine-structured $M_7C_3$ carbides with the matrix remains intact. Creep damage, in the form of dense cavities, has been observed in T2, as depicted in Figure 8. Higher magnifications are provided in Figure 9. Large cavities form around the primary, coarse-structured, $M_7C_3$ carbides while the fine-structured $M_7C_3$ carbides do not contribute to the formation of cavities (Figure 9(a)). Figure 9(b) depicts the very fine-structured $M_7C_3$ carbides, like the one in Figure 5(b), which transform to $M_{23}C_6$, while the coarse-structured ‘chinese script’ primary carbides do not, at least at the operation time of 5 years. It is also shown that cavities first form at the coarse-structured $M_7C_3$ and these cavities then link with microcracks along the film-like $M_{23}C_6$ carbides.

As aforementioned, Tube T2 exhibited cracking as depicted in Figure 3(b). A specimen from a ring cut through the crack is depicted in Figure 10(a), depicting numerous cracks initiating at the inner wall (IW) and propagating towards the outer wall (OW) of the tube. A collage of micrographs, in Figure 10(b) indicates the multiple through-thickness cracking. Creep crack initiation at the ID is depicted in Figure 11. Initiation at the IW is probably due to the higher hoop stress at the IW as has also been documented by Alvino et al. (2010). The cracks propagate through the wall by the linking of cavities along the interdendritic carbide network, as depicted in Figure 12. Creep cracks propagating along interdendritic carbides has also been observed by Swaminathan et al. (2008) in HP-Nb tubes, after a temperature excursion to 1150°C for 5-10 minutes. It is interesting to note that in tube T2 the cracks terminated before reaching the OW of the tube, as depicted in Figure 13(a). The reason for crack

Figure 8. Creep damage in the form of high number density of cavities in Tube T2.
arrest is probably (a) the lower hoop stress at the OW and (b) the presence of a layer of equiaxed structure, which disrupts the interdendritic carbide network for crack propagation. This layer of equiaxed grains is about 300 μm thick and, as depicted in Figure 13(b), is characterized by twinned austenite grains with intergranular and intergranular carbides.

Figure 9. (a) Cavity formation around coarse-structured M₇C₃ carbides, (b) transformation of fine-structured M₇C₃ carbides to M₂₃C₆ carbides and cavity formation in primary coarse-structured M₇C₃ carbides (Tube T2).
It is important to note that cracking of carbides, involving cracks crossing the carbides, as reported by Ribeiro, de Almeida, dos Santos, Fruchart, and Bobrovnitchi (2003) was not observed. Cracking always took place at the interface of carbides with the austenitic matrix.

3.2.3. Tube T3-ruptured tube
A specimen cut directly from the rupture area in tube T3 was used for fractography. The fracture surface in the SEM is depicted in Figure 14(a,b). The dendritic microstructure of the material is evident. The crack propagation along the interdendritic carbide network is manifested by the separation of dendrites. Regarding dendrite separation, it is worth noting that this is driven by the hoop stress since the dendrites are oriented radially from the IW to the OW of the tube. As stated above, some of these columnar dendrites are quite long, up to 6 mm in length. It appears that, as depicted in the schematic of Figure 15, the orientation of dendrite boundaries perpendicular to the hoop stress is not beneficial for creep.

Figure 10. (a) Multiple creep cracks propagating from IW to OW of the Tube T2, (b) Collage of micrographs indicating cracking in (a).
rupture resistance since the dendrite boundaries provide an easy path for crack propagation.

### 3.3. Creep analysis

A simple creep analysis was performed to investigate the effect of accidental overheating on creep strain accumulation and creep rupture life. The hoop stress variation in a thick circular cylinder subjected to internal pressure is given as follows:
where $p_i$ is the applied internal pressure in MPa, $p_0$ is the external pressure taken to be atmospheric pressure in MPa, $r_i$ is the inside radius of the tube in mm, and $r_o$ is the outside radius of the tube in mm. With the data of Table 1, the hoop stress at the IW is, according to Equation (1), $\sigma = 11.00$ MPa.

The creep strain rate is given by Norton’s law as a function of applied stress $\sigma$ and temperature $T$ as follows:

$$
\sigma = \frac{p_i r_i^2 - p_0 r_i^2}{r_o^2 - r_i^2} - \frac{r_i^2}{r_0^2 - r_i^2} \left( p_0 - p_i \right) \frac{r_i^2}{r_o^2 - r_i^2}
$$

Figure 12. (a) Crack propagation through the interdendritic carbide network, (b) crack propagation by linking of cavities (Tube T2).
The values of the creep exponent $n$ and the activation energy $Q$ were obtained from the work of Alessio et al. (2012), after creep testing of HP-Nb alloy, similar to the one investigated here. The values are $n=6$ and $Q=331$ KJ/mol. Using the creep curves from (Alessio et al., 2012), the value of the constant $A$ was obtained as $A=2.346 \times 10^{-39}$ s$^{-1}$. The creep strain rate was then computed using Equation (2) for a constant stress of $\sigma=11.00$ MPa and a constant operating temperature of 910°C.

$$\dot{\varepsilon} = A\sigma^n \exp\left(-\frac{Q}{RT}\right)$$

Figure 13. (a) Crack termination at the OW, at the equiaxed grain layer, (b) equiaxed twinned austenite grains at the OW. (Tube T2).
The strain rate resulted as $1.01 \times 10^{-11} \text{ s}^{-1}$. With this strain rate, the accumulated creep strain after 5 years of operation and just before the accidental overheating was 0.0016. Computation of the strains due to the accidental overheating was carried out by first approximating the temperature profile of Figure 1 by linear segments and then integrating Equation (2) over the time intervals. The algorithm was programmed in MATLAB computational software. The accumulated strain, during the two-peak overheating, was only $1.07 \times 10^{-6}$ and cannot explain the observed creep rupture. This is of course an expected result, since Norton’s law describes steady-state or secondary creep, where the creep

Figure 14. SEM micrographs of the rupture surface indicating separation of individual dendrites (Tube T3).
rate is constant, while creep rupture is the result of tertiary creep. Taking the above into consideration an analysis using the Larson–Miller (LM) parameter was conducted. From the Larson–Miller plot of HP-Nb alloy supplied by the manufacturer, depicted in Figure 16, the LM parameter for $\sigma = 11.00 \text{ MPa}$ is $\text{LM} = 35.2$. The LM parameter is given by

$$\text{LM} = \frac{T \left( \log t_f + C \right)}{1000}$$

with $T$ in Kelvin, fracture time $t_f$ in hours and $C = 22.4$. Using the above equation, the ratio of fracture times at 1050 °C (1323 K), which is the
peak temperature reached during overheating and 910 °C (1183 K) was calculated as:

\[ \frac{t_f,1323K}{t_f,1183K} = 0.0007 \]

i.e., the fracture time at 1050 °C is only 0.07% of the fracture time at 910 °C, which explains why the observed creep rupture took place in such a short period of time, as a result of accidental overheating to 1050 °C.

It should be noted that while for a new tube, the expected fracture time at 910 °C is 100,000 h, the life at 1050 °C would be only 70 h. However, the tubes under consideration had already been exposed for 5 years at 910 °C before the accidental overheating, so the anticipated creep rupture time at 1050 °C is much shorter than 70 h. Similar short rupture times, of the order of few minutes, have been reported by Swaminathan et al. (2008) for the case of local overheating of HP-Nb cast reformer tubes due to flame irregularities. In addition, creep testing reported by Jian-Hua et al. (2018), indicated that a temperature excursion by just 4.4% resulted in a reduction of creep rupture life by 57.9%.

4. Conclusions

An analysis of creep damage and creep rupture due to accidental short-term overheating has been conducted for HP-Nb cast reformer tubes. Based on the results presented above, the following conclusions can be drawn:

- Accidental short-term overheating, after 5 years of normal operation, caused rapid tertiary creep manifested by the formation of dense cavities and cracking, which eventually led to the premature creep rupture of several tubes of the reformer.
- Creep cavities formed preferentially at the coarse-structured ‘chinese script’ primary Cr-rich M₇C₃ carbides. Cavities also formed at film-like grain boundary Cr-rich M₁₂₃C₆ carbides and around Nb-rich MC carbides.
- Creep cracking initiated at the IW and propagated by linking of cavities along the interdendritic carbide network, separating the columnar dendritic grains. It appears that the radial direction of the columnar grains from the IW to OW orients the grain boundaries perpendicular to the hoop stress, a situation which degrades creep rupture resistance.
An analysis based on the Larson–Miller parameter confirmed the premature creep rupture of the tubes due to overheating.

The uncracked tubes experienced only advanced aging without the formation of cavities. Aging was manifested by carbide coarsening and transformation of fine-structured $M_7C_3$ to $M_{23}C_6$ carbides.

**Disclosure statement**

No potential conflict of interest was reported by the authors.

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