Analysis of transient and tertiary creep behavior of Titanium modified 14Cr-15Ni stainless steel after cold working

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Abstract

Characteristic of transient and tertiary creep behavior of ten, twenty, thirty, and forty percent cold worked indigenously developed Titanium modified 14Cr-15Ni stainless steel at 700 °C and various stress levels have been assessed. Small primary creep region followed by secondary creep and then substantial tertiary creep regions were exhibited by all steels. Creep parameters like transient strain and rate of exhaustion of transient creep in Garofalo equation and tertiary strain and rate of acceleration of tertiary creep in modified Dobes and Cadek equation have been extensively studied. The relationships among exhaustion rate of transient creep, acceleration rate of tertiary creep, and minimum creep rate revealed the prevailing theory of first order reaction rate during transient and tertiary creep deformation of the Titanium modified 14Cr-15Ni stainless steel. The exhaustion rate of transient creep decreased, whereas tertiary creep parameter decreased as the amount of cold work increased. Creep damage tolerance parameter indicated that necking, precipitate coarsening, and recovery of substructures are dominant creep damage mechanisms in the Titanium modified 14Cr-15Ni stainless steel.

1. Introduction

Creep curves of austenitic stainless steels are, generally, characterized by relatively small primary creep region followed by prolonged secondary creep region with the occurrence of minimum creep deformation rate and finally extensive tertiary creep regime [1–4]. Designing of structural components for high temperature applications involves the knowledge of creep rupture life, and variation in the creep strain leading to rupture. Thus, constitutive creep deformation equations, which describe the behavior of creep curves completely are of extreme interest to designer for safe application. Figure 1 shows the schematic representation of creep curve including instentanious strain (\(\varepsilon_0\)), transient creep strain (\(\varepsilon_T\)), minimum creep rate (\(\varepsilon_m\)), creep rupture life (\(t_r\)), strain accumulation at the point of minimum creep rate (\(\varepsilon_m t_r\)), and tertiary creep strain (\(\varepsilon_T\)). McVetty [5] and Garofalo [6] made various interpretations on transient creep, and secondary creep behavior of material. Webster \textit{et al.}[7] introduced the theory of first order kinetics process to establish the physical understanding between quantitative assesment of creep curve and mechanisms, which controls the creep rate of the material. Researchers suggested that during transient creep deformation, the dislocation rearrangements are governed by the process of dislocation climb. This process obeys the first order rate kinetics as described in equation (1).

\[
\frac{d\varepsilon}{dt} = \frac{-(\varepsilon - \varepsilon_s)}{\tau}.
\]  

where \(\varepsilon\), \(\varepsilon_s\), and \(\tau\) are creep strain, steady state creep strain, and relaxation time for dislocations to rearrange themselves into stable configuration. Garofalo [6] proposed an empirical relationship between strain (\(\varepsilon\)) and time (\(t\)) by integrating the above mentioned first-order rate equation twice, to represent the transient creep deformation of materials as described in equation (2).
where \( r' \) is the exhaustion rate of transient creep. Subsequently, Davies et al. [8] established the correlation between strain and time for entire creep curve and introduced the tertiary creep parameters, which was further modified by Dobes and Cadek [9] as described in equation (3).

\[
\varepsilon = \varepsilon_0 + \varepsilon_T [1 - \exp(-r'.t)] + \varepsilon_p.t + \varepsilon_3 . \exp [ p . (t-t_c)]
\]

where \( p \) is the acceleration rate of tertiary creep.

During constant load creep testing, cross-sectional area of creep specimen decreases during tertiary creep, leading to an increase in stress. This results in the continuous increase in creep rate of specimen. Microstructural degradation can be correlated to the change in the secondary creep deformation to tertiary creep deformation [10, 11]. Microstructural changes like grain growth, dislocation substructure recovery, recrystallization, and coarsening of precipitate particles contribute to creep deformation of materials. Internal stress decreases during creep exposure and accelerated plastic deformation rate resulted due to degradation of microstructure is reported. Moreover, nucleation of microcracks due to inadequate stress concentrations near the grain boundary region, irregularities occurred during sliding of grain boundaries, and contribution of grain growth process accelerate the creep deformation rate [12].

Plastic deformation during creep exposure and creep rupture behavior of austenitic stainless steels have been studied extensively. Austenitic stainless steels are generally preferred for structural components of fast breeder reactors (FBRs). These steels have wide range of applications as the structural and core materials in reactors [13]. Titanium modified 14Cr-15Ni stainless steel has specifically tailored chemical composition to resist void swelling during irradiation of the FBRs core. Microstructure evolution during creep exposure comprises of changes in dislocation substructure, recovery and recrystallization, precipitate coarsening of M23C6 type carbides, and fine TiC carbide precipitation during creep exposure. These microstructural changes result in variation in creep strain during different creep regimes [14].

In the current investigation, analysis of creep curves of indigenously developed Titanium modified 14Cr-15Ni stainless steel was carried out. It is an indigenously developed steel and creep testing was done on different prior cold work levels of the steel. Assessment of various transient and tertiary creep parameters was carried out to analyze the influence of cold work and stress levels on creep deformation behavior of the steel. The interdependence of creep parameters are discussed in details to understand the behavior of various stages of creep deformation.

2. Experimental

Mill-annealed Titanium modified 14Cr-15Ni austenitic stainless steel was procured from Mishra Dhatu Nigam Limited (MIDHANI), Hyderabad, India. Initial thickness of plate was 12 mm. The composition of alloying elements of the steel is shown in table 1. As received plate was cut into the bars of size 140 × 50 × 12 mm along the rolling direction of plate by EDM (Electric Discharge Machining) process. Solutionizing at 1060 °C for 30 min was carried out followed by quenching in water at ambient temperature. Cold working was done by rolling process at ambient temperature. Buhler make cold roll mill was used for cold rolling of ten (CW1),
twenty (CW2), thirty (CW3), and forty (CW4) percent. Gauge diameter and gauge length of creep specimens were kept as 5 mm, and 50 mm, respectively, as per ASTM standard E139. Uniaxial creep testing was carried out at 700 °C for 175, 200, and 225 MPa stress level by Star Testing System make creep testing machines. Test temperature was maintained within ±1 °C throughout the test. An extensometer was used to measure the elongation of specimen and data was logged continuously. The experimentally generated creep curves were analyzed by using Garofalo and modified Dobes & Cadek equation. The creep curve fitting with respect to equations (2) and (3) was done [15, 16]. After several iterations, creep curve parameters were calculated with R-square value of 0.98 and discussed in this study.

3. Results and discussion

Creep behavior of the test steel in solution annealed (CW0) condition was discussed in previous study [17]. Ti-modified 14Cr-15Ni steel is generally used in its cold worked state. Cold working generates more dislocations in the material, which can act as sinks for point defects such as vacancies and interstitials. During creep deformation process, the spacing between the barriers to the dislocation movement is an important parameter, which governs the high temperature strength of materials [14, 18]. Significant increase in creep rupture life was observed in all cold worked steels. However, the creep rupture life varied with cold work levels.

3.1. Creep behavior and rupture life dependence on stress

Figure 2 shows a typical three staged creep deformation behavior of the Titanium modified 14Cr-15Ni steel at 700 °C and 200 MPa stress level corresponding to solution annealed and different cold working levels. Creep deformation is evidently described by instantaneous strain (ε<sub>0</sub>) followed by primary, secondary, and tertiary regimes. Figure 3 shows the variations in the rate of creep deformation of different cold worked steels at 700 °C and 200 MPa stress level with normalized creep exposure time. To compare creep behaviors of all such samples during the progress of test, X-axis was normalized. Initially, creep rate was found to be decreased with time in primary stage and reached to a minimum creep rate level during secondary stage. After that, the creep rate was found to increased with the exposure time leading to creep fracture. This is a typical behavior of austenitic stainless steels during creep deformation [19]. Generally, the time (t<sub>f</sub>) at which tertiary creep deformation initiates, was delayed as the minimum creep rate (ε<sub>m</sub>) decreased during creep deformation and creep rupture life (t<sub>r</sub>) of steels varied with the variation in prior cold work levels.

Figure 4 shows the dependance of ε<sub>m</sub> with respect to σ for all creep tested steels. It was found that the variations in the ε<sub>m</sub> was increased with the increase in cold work level predominantly at higher stress levels. CW2 steel showed the lowest value of minimum creep rate among all steels. However, CW3 steel showed a prevailing decreasing trend of ε<sub>m</sub> value and at 175 MPa stress level, ε<sub>m</sub> of CW3 steel was found to be almost equivalent to

| C     | Ni    | Cr    | Mo    | Ti    | B     | N     | P     | S     | V     | Nb    | Si    | Mn    | Fe    |
|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| 0.041 | 15.52 | 14.09 | 2.21  | 0.23  | 0.005 | 0.0087| 0.023 | 0.022 | 0.022 | 0.023 | 0.74  | 2.49  | bal   |

Table 1. Alloying elements (wt%) in the investigated Titanium modified 14Cr-15Ni steel.

Figure 2. Creep curve of solution annealed, and various cold worked steels.
This variation in $\varepsilon_m$ with $\sigma$ follows the Norton’s power law relationship as described in equation (4).

\[ \varepsilon_m = A \sigma^n. \]  

where $A$ and $n$ are power law constant and stress exponent, respectively. Values of the stress exponent are an indicative of the controlling mechanism of creep deformation rate. The values of $n$ (typically $3 < n > 12$) indicate that the dislocation creep mechanism is prevailing creep deformation mechanism [20]. In current investigation, solution annealed as well as all cold worked steels exhibited the value of stress exponents ($n$) in between 5 to 9, which proves that dislocation creep was predominant deformation mechanism [21].

The minimum creep rate of CW2 and CW3 steel at 175 MPa was found to be $3.11 \times 10^{-6}$ h$^{-1}$ and $3.19 \times 10^{-6}$ h$^{-1}$, respectively, which is almost similar. As the level of cold working increases, the more dislocations generated in the steels, which leads to the availability of more dislocations for the precipitation of TiC precipitates and probability of dislocation tangling is more. This leads to more resistance for creep deformation. On the other hand, as the level of cold working increases, the stored strain energy in the steel is also increases, which leads to increase in the recovery of deformed microstructure and then leads to poor creep resistance. These two opposite phenomena are controlling the creep deformation behavior of this steel and resulted in the variation of minimum creep rate. Such behavior is influenced by temperature, applied stress (and thereby the exposure time), and prior cold work level leading to different substructure evolution and eventually the extent of recovery and recrystallization.

In Ti-stabilized stainless steels, precipitation of fine secondary titanium carbides (TiC) occurs along dislocations. These nano-sized fine secondary TiC precipitates hinder the motion of dislocations during plastic
deformation of steels. S. Latha et al [22] also discussed about the precipitation of these fine TiC in Ti-modified 14Cr–15Ni austenitic stainless steel. In CW3 steel, the precipitation of TiC was found to be more at 175 MPa stress level as compared to that in CW2 steel, which can be seen in figure 5. Pinning of dislocations were also more in CW3 steel. As the amount of cold work increased, the number of dislocations were also increased in the steel. Increase in dislocation density exerted back stress on the incoming new mobile dislocations and hindered the movement of dislocations during plastic deformation. However, excess dislocation density resulted in increased strain energy in the material, which is more inclined to recover faster at high temperature. On the other hand, fine TiCs were also more prone to precipitates near the dislocations and restrict the movement of dislocations by pinning. So, the precipitation of large number of these fine TiCs and pinning of dislocations can be a reason of similar minimum creep rate of CW2 and CW3 steel.

Figure 6 shows the dependence of \( t_r \) with respect to \( \sigma \) for different cold worked steels. Cold working affected the creep rupture behavior of the Titanium modified 14Cr-15Ni stainless steel, which were more prevalent at lower and higher stress levels. A power law relation was observed between creep rupture life and applied stress as described in equation (5).

\[
t_r = A' \sigma^{-n'}.
\]  

(5)
where $A'$ is stress coefficient and $n'$ is stress exponent. The variation of creep rupture life ($t_r$) with respect to applied stress ($\sigma$) displayed negative slope. In current investigation, solution annealed and all cold worked steels showed the value of stress exponents ($n'$) in between 5 to 8. Similar values of both $n$ and $n'$ ascertain that deformation mechanism as well as rupture processes were same for all cold worked steels.

The creep rupture life ($t_r$) with respect to minimum creep rate ($\epsilon_m$) of the Titanium modified 14Cr-15Ni steels is shown in figure 7. Generally, the relationship between $\epsilon_m$, and $t_r$ of materials represents the Monkman–Grant relationship as shown in equation (6).

$$\epsilon_m \times t_r = C_{MG}.$$  

Where $\alpha$ is a constant and $C_{MG}$ is the Monkman–Grant constant. The applicability of Monkman–Grant relationship was observed as the slope of the plots were almost equivalent to unity for all cold worked steels and it is attributed that the deformation mechanism, which is accountable for plastic deformation and creep fracture, was same for all cold worked steels [23]. The values of $C_{MG}$ were 0.1, 0.04, 0.06, 0.07, and 0.09 for solution annealed, ten, twenty, thirty, and forty percent cold worked creep tested steels, respectively. The values of the Monkman–Grant constant suggested that, during creep, accumulation of strain till the minimum creep rate level was relatively small with respect to final creep strain and all steels exhibited major fraction of strain during tertiary creep regime only [24]. Accumulation of creep strain till the time of minimum creep rate level was also increased as level of cold work increased. CW4 steel showed higher strain accumulation followed by CW3, CW2, and CW1, respectively. This may be due to different prior cold work condition and thereby, the recovery rate of deformed steels was different.

Figure 8 shows the relation between time for starting the tertiary creep stage ($t_t$) with creep rupture life ($t_r$). The $t_t$ was calculated from creep curves of cold worked steels and it is the time from which the creep rate started to accelerate. A linear relationship was observed in the form of $t_t = f \cdot t_r$, where $f$ is a constant and it was found to be around 0.6. This indicates that all the steels spent 60 percent time in their tertiary creep stage.

### 3.2. Transient creep behavior

Strain hardening and recovery occur during creep deformation. The predominance of each phenomenon varies in each stages of creep. In transient creep regime, rate of strain hardening dominates over rate of recovery. However, during tertiary creep regime, recovery rate is more dominant. In secondary creep regime strain hardening rate balances recovery rate. The Garofalo equation represents the relationship of strain ($\epsilon$) with time ($t$) during all creep regimes. Sidey and Wilshire [25] have established the direct relationship between $r'$ and minimum creep rate. In present investigation, the values of $\epsilon_T$ and $r'$ are calculated from experimentally generated creep curves of various cold worked Titanium modified 14Cr-15Ni stainless steels at various stress levels.

A power law relationship ($r' \propto \sigma^n$) was observed by exhaustion rate of transient creep ($r'$) with stress ($\sigma$), which is shown in figure 9. As the amount of cold work increased, stress index ($n$) decreased. This indicated that the exhaustion rate of transient creep ($r'$) was proportional to the amount of cold work. This further implies that steels were work hardened because of different level of prior cold work and further strain hardening events were
reduced during transient creep. Since creep loading conditions were same for all cold worked steels, the exhaustion rate of transient creep increased with increase in cold work and intended towards decrease in the minimum creep rate. Exhaustion rate of transient creep was found to be highest for CW4 steel followed by CW3, CW2, and CW1 steels, respectively, for all stress levels. It is expected that during creep exposure, the strain hardening behavior of all steels was controlled by the amount of prior cold work appreciably. Figure 10 shows the relationship between $\varepsilon_m$ and $r'$ and it was found that the dependence of $\varepsilon_m$ on the exhaustion rate of transient creep ($r'$) was decreasing with increase in the amount of prior cold work.

Figure 11 shows the relationship of transient strain ($\varepsilon_T$) with stress for various cold worked steels. The transient creep strain ($\varepsilon_T$) of cold worked steels showed the tendency to reach at constant value and subsequently decreased with increase in stress level. CW2 steel showed the minimum value of transient strain among other cold worked steel. This trend was found to be same for all stress levels. Since CW2 steel showed least minimum creep rate followed by better creep resistance among all cold worked steels for all stress level except one instance i.e. CW3 steel at 175 MPa stress level. So, it can be inferred that optimum level of cold work in CW2 steel exhibited low recovery rate leading to least transient strain.
3.3. Tertiary creep behavior

The cold worked Titanium modified 14Cr-15Ni steels showed extensive tertiary creep deformation, which can be seen in figure 3. In the current study, creep deformation behavior of cold worked steel in the tertiary stage has been analyzed by using modified Dobes and Cadek creep equation [9]. The creep curves of cold worked steels have been analyzed at various stress levels. Experimentally generated creep curves at various stress levels were used to compute creep parameters like tertiary strain ($\varepsilon_3$), and acceleration rate of tertiary creep ($p$) as described in equation (3). Figure 12 shows the relationship between $p$ and $\sigma$ for all creep tested cold worked steels. The relationship of $p$ with stress ($\sigma$) exhibited a power law relationship as described in equation (7).

$$p \propto \sigma^n.$$  

(7)

where the value of stress exponent ($n$) were found to be in the range of 6 to 8 for the various cold worked steels. It has been observed that the variation of $p$ with $\sigma$ is closely resemble with the variation of $\varepsilon_m$ with $\sigma$ as shown in figure 4. The value of $p$ varied with respect to amount of cold work as well as various stress levels. AISI 304 stainless steel also showed similar variation of $p$ with stress [11].

Acceleration rate of tertiary creep was found to be decreasing with applied stress. The value of $p$ depends on the tertiary creep deformation mechanism and recovery rate of material. In different cold worked steels, the rate of recovery was different due to different prior cold work levels, which also depends on the tertiary creep.
deformation mechanism. The acceleration rate of tertiary creep \((p)\) was found to be inversely proportional to \(t_r\) of the steels for all stress levels. CW2 and CW3 steels showed almost similar values of minimum creep rate. However, variation in recovery rate of deformed microstructure as well as extent of precipitation of fine TiC carbides led to variation in tertiary creep parameter \((p)\). This resulted in the higher values \(p\) for CW2 steel as compared to that of CW3 steel and led to higher creep life of CW3 steel at 175 MPa stress level.

The relationship between \(\varepsilon_m\) and \(p\) for all cold worked steels are described in figure 13. It can be said that, the dependence of \(p\) on \(\varepsilon_m\) was decreased with increase in amount of cold work. The relationship of acceleration rate of tertiary creep \((p)\) with \(\varepsilon_m\) and tertiary creep time were found to be near-linear, which indicated that all cold worked steels obeyed first order kinetics rate theory during tertiary creep deformation also.

Figure 14 shows the variation of tertiary creep strain \((\varepsilon_3)\) in various cold worked steels with respect to applied stress. The extent of tertiary creep strain \((\varepsilon_3)\) varied with respect to amount of cold work as well as applied stress. CW4 steel exhibited higher tertiary creep strain accumulation among all cold worked steels for all stress levels. Higher tertiary creep strain in CW4 steel indicated higher ductility on account of faster recovery and recrystallization of the steel [17]. At higher stress level the variation in tertiary creep strain with extent of cold work was found to be more as compared to that at lower stress level.
3.4. Creep ductility

The percentage elongation and area reduction of all creep tested steels are showed in figure 15. It was observed that cold working significantly affected the creep rupture elongation and area reduction. Creep ductility of cold worked steels were found to be reduced as compared to solution annealed steel (CW0). Furthermore, as the amount of cold work increased from 0 to 40 percent, the creep ductility was initially decreased till 20 percent cold work level after that increased for 30 and 40 percent cold work. During creep exposure of plastically deformed metal, the movement of dislocations are restricted. The more dislocations within a material, the more they interact and become pinned or tangled. However, due to the faster recovery and recrystallization phenomena 40 percent cold worked steel were responsible for increased creep ductility. Detailed microstructural studies were published earlier [17].

K Laha and Shinya et al [26, 27] also discussed that, in austenitic stainless steel, the beneficial effects of boron on creep ductility were more pronounced at relatively longer creep exposure times. In current steel, it can be said that the presence of boron led to the relatively higher creep ductility at 175 MPa stress level as compared to that of at 225 MPa stress level.

3.5. Creep damage

The start of tertiary creep deformation or the onset of tertiary creep regime resulted in accelerated creep rate during creep exposure. Cumulative effect with dominance of one creep deformation mechanism to other creep deformation mechanism resulted in the time spent by the steels in tertiary creep regime. Generally, tertiary creep deformation involves intergranular microcracks, necking, and degradation of microstructure such as the precipitate coarsening and dislocation substructures recovery [28]. According to 'Continuum Creep Damage Mechanisms (CDM)' [29], the initiation of tertiary creep damage process can be described by the parameter, which is called as creep damage tolerance parameter ($\lambda$) as described in equation (8).

$$\lambda = \frac{\varepsilon_f}{\varepsilon_{mt}}$$

(8)

where $\varepsilon_f$ is the strain at failure. Modeling of various creep damage processes has predicted that the value of $\lambda \sim 1$–2.5, indicated the tertiary creep is mainly caused by inter-granular cavitation. When necking dominates the tertiary creep, the value of $\lambda$ is in between 2.5 to 5. The value of $\lambda > 5$ indicates precipitate coarsening and recovery of substructures attributed to tertiary creep damage [30]. In the current study, the creep damage parameter ($\lambda$) values were found to be in between 4 to 5.5 as shown in figure 16, which suggested that necking was dominant creep damage mechanism in the Titanium modified 14Cr-15Ni stainless steels. The large values of $\lambda$ arise from the higher ductility exhibited by this steel. In earlier studies [31, 32] on 14Cr-15Ni-Titanium modified and 15Cr-15Ni-Ti modified steel, the higher values of damage tolerance parameter were also reported. The increase in ductility of the alloys, in the present study, can be attributed to the addition of Boron, around 50 ppm. Addition of Boron in ppm levels was found to improve creep ductility in 304, 321 and 347 austenitic stainless steels also [33].
Figure 15. Variation of creep rupture elongation and area reduction in various cold worked and solution annealed steels tested at 700 °C.

Figure 16. Damage tolerance parameter of various cold worked Titanium modified 14Cr-15Ni steels tested at various stress levels.
Addition of Boron in alloys for high temperature application has been discussed in several studies, in which creep rupture strength was found to be increased. It is widely believed that the addition of boron increases creep rupture life and ductility through the increase in creep cavitation resistance of the steel by grain boundary strengthening. In most instances, it is said that Boron is concentrated on grain boundaries, where it enters in the precipitates and alters the character of the grain boundary or precipitates in such a way to suppress microcavity formation [27, 34, 35]. Figure 17 (a) shows the scanning electron microstructure of the fracture surface of CW2 steel. The fracture surface of this steel is characterized by predominantly ductile dimples with little micro-cavitation indicating a ductile failure, whereas no sign of cavitation was found in the necking area of the sample as shown in figure 17(b). Similar observations were also reported by Latha et al [31]. In other studies of austenitic stainless steels, addition of boron was found to suppress cavity growth rate significantly in 347 austenitic stainless steels [33]. The significant decrease in cavity growth rate in 347 austenitic steel is believed to be associated with the segregation of Boron instead of Sulphur on cavity surface. In the present study the addition of Boron reduces creep damage and cavitation and improves ductility.

4. Conclusions

Based on the present study of the transient and tertiary creep regime of various cold worked Titanium modified 14Cr-15Ni stainless steels, following conclusions have been drawn:

1. The transient and tertiary creep deformation behavior of cold worked Titanium modified 14Cr-15Ni stainless steels obey first order reaction rate theory.
2. The increase in cold work level escalate the exhaustion rate of transient creep led to increase in recovery rate in forty percent cold worked steel.
3. Acceleration rate of tertiary creep was found to be inversely proportional to the creep rupture life of the steels for all stress levels.
4. Accumulation of creep strain at the minimum creep rate level was found to be increasing with increase in cold work level.
5. Necking was dominant creep damage mechanism in the Titanium modified 14Cr-15Ni stainless steels.
6. Creep ductility was found to be inversely proportional to applied stress level and minimum creep ductility was exhibited by twenty percent cold worked.

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