Author's Accepted Manuscript

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PII: S0921-5093(18)30067-4
DOI: <https://doi.org/10.1016/j.msea.2018.01.049>
Reference: MSA36003

To appear in: *Materials Science & Engineering A*

Received date: 9 October 2017
Revised date: 11 January 2018
Accepted date: 12 January 2018

Cite this article as: E. Hosseini, V. Kalyanasundaram, X. Li and S.R. Holdsworth, Effect of prior deformation on the subsequent creep and anelastic recovery behaviour of an advanced martensitic steel, *Materials Science & Engineering A*, <https://doi.org/10.1016/j.msea.2018.01.049>

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Effect of prior deformation on the subsequent creep and anelastic recovery behaviour of an advanced martensitic steel

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Abstract:

The creep and anelastic recovery characteristics of a 10%Cr steel have been systematically investigated at 600°C after subjecting the test material to various prior deformation histories. Constant-load forward creep tests on specimens, either with a tensile or compressive preloading history, indicated that over- and reverse-preloading respectively decreases and increases the early primary creep rate of the steel. The extent of decrease (or increase) in early primary creep rate is also found to be directly proportional to the magnitude of stress during prior loading while such a correlation is not clearly evident for material deformation in the secondary and tertiary stages. Specifically, the creep rate in the secondary and tertiary stages is lower for specimens with a compressive prior loading while the rupture time is notably shorter for tensile pre-loaded specimens. The observed effect of prior loading on the early primary creep behaviour can be explained by considering micro-backstress development (as a consequence of dislocation pile-up formation during the prior loading phase) that subsequently introduces a kinematic hardening effect to the material's viscoplastic response. The second set of experiments involve monitoring the anelastic recovery behaviour immediately after accumulation of a similar amount of time-dependent strain either under forward creep (load control mode) or stress relaxation (strain control mode) condition in completely unloaded 10%Cr steel specimens at 600°C. Experimental observations indicate that the higher the stress magnitude during the prior loading phase the greater and faster the anelastic recovery at zero stress. Further findings show the mode of prior deformation (creep or relaxation) to also not noticeably influence the subsequent anelastic recovery behaviour. The observed anelastic recovery characteristic can be mechanistically interpreted by consideration of the time-dependent material back-flow due to the relaxation of dislocation bows and pile-ups generated during the prior deformation.

Keywords:

Prior deformation; Creep; Anelastic recovery; Stress relaxation; Kinematic hardening/softening; 10%Cr steel.

1. Introduction

Fracture-critical components such as rotors in turbines and main steam pipes and headers in boilers typically operate at steam temperatures up to 625°C in ultra-supercritical (USC) power plants. Advanced high-chromium (9-12%Cr) martensitic/ferritic steels are the most preferred class of materials for such components not only because of their superior mechanical properties but also for their critical role in lowering carbon dioxide (CO₂) emissions through improved operating efficiencies [1,2]. Although the initial development of 9-12%Cr steels dates back almost a century [3], their microstructure has been progressively optimised by metallurgical alloying especially over the last few decades to further enhance long-term elevated temperature properties. These improvements, for example, have consequently led to a ~30% reduction in CO₂ and other environmentally damaging gas emissions in USC plants [4,5]. It has also been well documented that the resistance of 9-12%Cr steels to elevated temperature deformation is greatly dependent on both the distribution of various microstructural features (i.e. laths/subgrains, dislocations, precipitates etc.) and their evolution over time [6-12]. Similar to observations reported for low-alloy steels [13-17], recent experiments have indicated the creep resistance of various 9-12%Cr steels to be significantly modified as a consequence of prior monotonic/cyclic plasticity at elevated temperatures [16,18-23]. Systematic consideration of the influence of prior loading transients on creep is important for effective remnant life assessments and for improved design protocols in the future [11,23,24]. The overall objective of this work is therefore to provide detailed scientific insights on the effect of prior deformation on subsequent creep deformation behaviour of a 10%Cr martensitic steel at 600°C. The scope of study is further expanded by evaluation of the effect of prior deformation on subsequent anelastic recovery behaviour of the steel at 600°C.

Preliminary research efforts from the authors in this context showed the creep resistance of a 10%Cr steel to be modified as a consequence of prior deformation [16,19]. Test matrices included monotonic stress-relaxation experiments at strain amplitudes ranging from 0.075% to 0.50%. The available stress-relaxation data could be converted to equivalent forward creep data using different analytical methodologies [25-29] but they may not be able to satisfactorily capture the material deformation in all the three distinct creep regimes. Instead, evaluation of the constant load creep behaviour for testpieces with different prior deformation conditioning have therefore been employed in the current

study for a 10%Cr steel at 600°C. Experimental observations indicated that the effect of prior-deformation is different for the various creep regimes. A discussion on possible micromechanical mechanisms responsible for the observed behaviour has been provided, while it is acknowledged that further detailed microstructural investigation is required for definite determination of the responsible mechanism.

Anelastic recovery, first reported by Zener [30], is the retrieval of previously accumulated inelastic strain over time after stress unloading and is an important consideration in the mechanical analyses of high temperature alloys under varying stress conditions [31]. Although quite a few studies exist in the literature for other metals on the nature (and causal micromechanisms) of anelastic backflow [32], only two such studies [31,33] exist for 9-12%Cr steels to the authors' knowledge. Since any significant inelastic strain decrement due to anelastic recovery can markedly influence the material response on reloading, it is thus important to understand its effect at/near the peak operating temperature [31].

In summary, the overall objective of this work is to provide insights on the effect of prior deformation on the subsequent creep and anelastic recovery response for a 10%Cr steel at 600°C. Details of the test material and experiments conducted as part of this work are given in Section 2. Section 3 outlines the obtained experimental results, and the scientific relevance of these results is finally discussed in Section 4.

2. Experimental Details

Test Material:

The test material chosen for this study is an advanced high-chromium forged rotor steel that originated as Steel F (melt 3A) within the European COST501 programme in the early 1990s [34]. The nominal chemical composition (in weight%) of major alloying elements in the chosen material, referred hereafter as 10%Cr steel, is as follows: 9.98%Cr, 1.50%Mo, 0.60%Ni, 0.48%Mn, 0.17%V, 0.15%C, 0.10%Si, 0.05%Nb and 0.04%N. The typical maximum application temperature for this steel is 600°C implying a $\geq 50^\circ\text{C}$ temperature advantage relative to low-alloy 1%CrMoV steels [35]. Like other high-chromium steels [11], the test material has a tempered martensitic microstructure after quenching and subsequent tempering heat treatment (see Fig. 1). The excellent creep resistance offered by this steel stems from the presence of primarily Cr-rich $M_{23}C_6$ (M: metal) carbide precipitates decorated along the lath/subgrain boundaries while the high Cr content imparts enhanced oxidation resistance at elevated temperatures. Besides solid solution strengthening by Mo, carbonitride precipitates of the type MX (M: V or Nb; X: C and/or N) and M_2X (M: Cr or V) offer secondary hardening both within the subgrains and

along the boundaries. Table 1 gives the chemical composition of the investigated 10%Cr steel in this study.

Table 1. Chemical composition of the investigated 10%Cr steel (wt %)

C	Si	Mn	Ni	Cr	Mo	V	Nb	N
0.13	0.08	0.56	0.56	10.36	1.51	0.17	0.05	0.053

Test Specimen and Setup:

Cylindrical dogbone specimens with a gauge diameter and parallel length of 7mm and 20mm, respectively were used for the experimental program, refer Fig. 2. Tests were conducted in an induction heating based closed-loop servohydraulic system of 100kN capacity held under controlled atmospheric conditions (22°C, 50% relative humidity). Following the recommendation outlined in [36], an optimised split coil design with a passive coil was employed for induction heating to achieve a thermal gradient of $\pm 1^\circ\text{C}$ from the target temperature along the specimen parallel length. Three s-type thermocouples were spot-weld for monitoring temperature in the parallel length, one at the middle and one each at 9mm from either side of the middle. In particular, the middle thermocouple was digitally controlled to within $\pm 0.2^\circ\text{C}$ of the target temperature during the course of an entire test. A class 0.5 side-entry extensometer with a datum leg spacing of 15mm was used for the control and measurement of axial creep strain (see Fig. 3.). A calibrated integral load cell was used for the measurement of axial load acting on the specimen.

Test Matrix:

In accordance with the two major objectives proposed in this study, the experimental testing program comprised two sets of tests. Tests in Set A were designed to systematically evaluate the effect of prior deformation on subsequent creep behaviour. In contrast, the focus of Set B tests was to specifically investigate the anelastic recovery behaviour after different pre-loading histories. A detailed description of both sets of experimental tests is provided next.

Set A:

To systematically evaluate the influence of prior deformation on subsequent creep response, the constant-load behaviour of specimens subjected to different pre-loading histories was evaluated under otherwise similar forward creep loading. Evaluation of the subsequent creep response included constant-load creep testing at two different stress levels: a) a relatively low stress level of 200MPa for a duration of 40 hours and b) a high stress level of 300MPa for <12 hours (terminated once the specimen clearly exhibited tertiary creep deformation). It is important to point out that these stress levels were chosen after estimating the stress to be 240MPa at the limit of proportionality ($\sigma_{0.01\%}$) for

the chosen 10%Cr steel at 600°C (see Fig. 4). Moreover two baseline creep tests were also conducted on specimens with no prior deformation history, one each at 200 and 300MPa in load-control mode.

At the chosen stress of 200MPa, five forward creep tests were conducted on specimens that had either a tensile or compressive pre-loading history. For each of these tests, specimens were loaded monotonically in tension or compression to a predefined stress value and then ramped immediately to 200MPa. As evident from Fig. 4, the tensile pre-stress levels of 350 and 400MPa led to considerable plastic yielding in the test specimens before subsequent creep testing at 200MPa. Similarly, for the specimens subjected to compressive pre-loading before creep testing at 200MPa, the chosen pre-stress levels of -350 and -400MPa led to plastic yielding of the material in compression. In addition, a third test with compressive pre-loading only up to -200MPa with no significant yielding was conducted. For the chosen stress level of 300MPa, two creep tests were additionally conducted on specimens that had either a tensile or a compressive pre-loading history. The same pre-stress value of 400MPa was employed both in tension and compression that led to plastic yielding of the material before subsequent creep testing at 300MPa. The particular details for each of these tests can be found in Table 2. Fig. 5 shows the variation of stress during the tests of Set A. The loading and unloading ramp rate during the transients was kept at 100MPa/s.

Set B:

Seven experiments were specifically designed to investigate the influence of different pre-loading histories on the subsequent anelastic recovery behaviour of the considered 10%Cr steel at 600°C. Each of these tests first involved the accumulation of $\sim 0.07\%$ time-dependent strain under either constant-load (forward creep) or constant-strain (relaxation creep) conditions during the on-load phase. In the subsequent off-load phase, anelastic recovery characteristics of the material for a stipulated duration of time was monitored after full unloading i.e. at zero stress. Four of the tests involved constant-load forward creep at stress levels of 150, 200, 250 and 300MPa until accumulation of 0.066% creep strain and immediately followed by a fast unloading (at 100MPa/s). The remaining three experiments involved stress-relaxation during the on-load phase that was immediately followed by anelastic recovery measurement at zero stress. The specimens were loaded to a peak stress of 250, 275 or 300MPa, kept under constant-strain control until a stress drop of 100MPa before a quick unloading to zero stress for monitoring the subsequent anelastic recovery behaviour. The experimentally measured stiffness (E) of the 10%Cr steel at 600°C was ~ 150 GPa (see Fig 4) and

therefore a relaxation of 100MPa stress under constant-strain condition is equivalent to an accumulation of 0.066% creep strain ($\Delta\epsilon_{cr} = \Delta\sigma/E$). Therefore the amount of accumulated time-dependent strain for both the creep and stress-relaxation loading, before the anelastic recovery measurements, was similar ($\approx 0.066\%$). The particular details for each of these tests in Set B can be found in Table 2. Fig 6 shows the variation of stress and total strain during the tests of Set B. Comparison of the anelastic recovery characteristics of the different specimens clarified the effect of different loading modes and their magnitude, for a similar amount of accumulated time-dependent strain, for the considered 10%Cr steel at 600°C.

For both test campaigns, special care was taken to minimise any obvious difference in the testing protocol for the considered loading histories. As mentioned earlier, the thermal gradient along the specimen parallel length was maintained within minimal scatter bounds ($\pm 1^\circ\text{C}$) during the course of testing. Also, the ramp rate during transients for all the tests was kept nominally at 100MPa/s. This ramp rate is intentionally high and was adopted to ensure minimal creep strain accumulation during the transients. Furthermore, all the creep tests were conducted in accordance with guidelines provided in the European standard 204 for uniaxial creep testing in tension [37]. The load and strain signals were recorded at a frequency of 100Hz during the course of each conducted test.

Fig. 1. As-received microstructure of the investigated 10%Cr steel (optical microscopy image, etchant: warm picric acid)

Fig. 2. Testpiece geometry used for the proposed tests

Fig. 3. Test arrangement used for the experimental program

Fig. 4. Determination of the elastic modulus and the limit of proportionality for the considered 10%Cr steel at 600°C

Fig. 5. Variation of stress during tests of Set A

Fig. 6. Variation of stress and strain during tests of Set B

Table 2. Summary of test details for the two sets of experiments proposed in this work

Test ID	Type of test	Stress MPa	Pre-stress MPa	On-load duration h	Test ID	Type of test	Stress MPa	On-load duration h	Off-load duration h
A1	Constant-load creep test	200	+400	40	B1	Creep-recovery test	150	14.6	30
A2			+350		B2		200	0.66	
A3			-		B3		250	0.04	
A4			-200		B4		300	0.01	
A5		300	-350	<12	Test ID	Type of test	Stress-ini MPa	Stress-end MPa	
A6			-400		B5	Relaxation-recovery test	250	150	
A7			+400		B6		275	175	
A8		300	-		B7		300	200	
A9			-400						

3. Experimental Results

This section reports observations and analyses of data from experiments conducted in this work to understand the influence of prior-loading history on the subsequent creep and anelastic recovery behaviour of the considered 10%Cr steel at 600°C.

3.1. Specimen pedigree variability

It is first important to check the loading transients from all the conducted tests to ensure that comparisons presented later are not arbitrarily influenced by factors associated with specimen-to-specimen variability in mechanical response. Fig. 7 shows the initial loading transient from all the

conducted tests with the peak σ - ϵ datapoint differentiated by markers for each of the loading histories. The stress-strain curves indicate minimal variability in the early uniaxial deformation behaviour of different specimens and the repeatable accurate control of testing conditions (e.g. temperature and its gradient). This observation asserts that results presented in the subsequent sections can be assumed to be independent of specimen-to-specimen mechanical response variability. The inset in Fig. 7a shows the symmetrical σ - ϵ response of the 10%Cr steel under monotonic tensile and compressive loading.

3.2. Tests of Set A

Results from forward creep tests at 200MPa with and without prior loading history will be presented first in this subsection followed by those at 300 MPa (Figs. 8 and 9, respectively).

200MPa creep tests

A series of forward creep strain vs. time plots obtained at the starting stress level of 200MPa is shown in Fig. 8. Fig. 8(a) shows the creep strain evolution in the initial 180 seconds. Negative creep could be observed for specimens subjected to tensile pre-loading (for the first ~3-5 seconds). Furthermore, the amount of negative creep generation was found to be directly proportional to the magnitude of the applied tensile pre-loading stress. The propensity of the 10%Cr steel to exhibit negative creep after tensile pre-loading however diminishes rapidly over the next few seconds and the material starts to creep forward at a rate lower relative to pure creep. In comparison to pure creep, the test material however appears to creep relatively faster with increasing magnitudes of compressive pre-loading stress. For the test durations considered in this work (Refer Table 2 and Fig. 8(b)), compressive and tensile pre-loading respectively accelerates and decelerates the creep strain accumulation for the 10%Cr steel.

300MPa creep tests

Similar to that shown for 200MPa in Fig. 8, creep strain vs. time plots obtained for the forward creep tests at 300MPa are shown in Fig. 9. Fig. 9(a) shows the creep strain evolution in the initial 10 seconds of constant-load application. In contrast to the observations noted at 200MPa creep tests for specimens with tensile pre-loading, negative creep was not as discernible at 300MPa for the specimen with a similar history (A7). As shown in Fig. 9(b), similar to observations from the 200MPa creep tests, tensile and compressive pre-loading respectively decreases and increases the extent of creep strain accumulation during the early primary creep regime. The material response was found to gradually

change during secondary and tertiary creep stages with the A7 specimen showing relatively higher average creep rate and therefore the shortest rupture time.

Figs. 10(a) and (b) respectively present creep rates evaluated for all the forward creep tests at 200MPa and 300MPa with and without pre-loading.

3.3. Tests of Set B

Experimental observation from tests specifically designed for investigating the effect of prior-loading on subsequent anelastic recovery behaviour of the 10%Cr steel at 600°C is presented in this subsection.

Fig. 11 depicts the accumulation of creep strain and stress-relaxation behaviour of the test material during the on-load phase of tests of Set B. As expected, a time-dependent strain of 0.066% has been accumulated in all the seven specimens before the subsequent off-load phase.

After accumulation of 0.066% strain, the specimens were quickly unloaded and the anelastic recovery behaviour under zero-stress condition was monitored. Fig. 12 shows the anelastic recovery behaviour for the testpieces during the zero-stress relaxation phase. For both the constant-load forward creep and constant-strain relaxation creep conditions, anelastic recovery starts at a high rate immediately after the unloading but then slows down as a function of time. The rate of anelastic recovery after 30h appears to be negligible. As can be seen, for a given pre-accumulated time-dependent strain, the amount of anelastic recovery is higher for those specimens in which strain accumulation occurred under higher stress levels. The extent of anelastic recovery after 30h of unloading is between 0.033-0.042% which equally corresponds to 48-63% of the pre-accumulated time-dependent strain. Fig. 13 combines and compares the anelastic recovery behaviour after both forward creep and stress-relaxation testing indicating no significant effect of the mode of pre-straining (i.e. creep or stress relaxation) on the subsequent anelastic behaviour. Fig. 14 finally compares the unloading behaviour of the different specimens of Set B. Although the specimens showed very similar loading modulus, the unloading moduli are different and the higher the stress at the start of unloading the smaller the unloading modulus.

Fig. 7. Comparison of the stress-strain response during the initial loading transient as obtained from specimens employed for tests in a) Set A and b) Set B. The inset in (a), a plot of the absolute magnitude of tensile and compressive stresses vs. strains during the nine loading transients, shows the symmetrical response of the test material at 600°C

Fig. 8. Creep strain accumulation under 200MPa for specimens with different pre-loading histories. a) behaviour for the first 180s of forward creep loading and b) behaviour for the duration of testing (40h) considered in this work

Fig. 9. Creep strain accumulation under 300MPa for specimens with different pre-loading histories. a) behaviour for the first 10s of forward creep loading and b) primary creep response during the first hour of testing, with the inset showing creep deformation for the duration of testing (< 12h) considered in this work

Fig. 10. Creep rate evolution during forward creep tests at a) 200 and b) 300MPa. The shown creep rates are the derivatives of smooth splines fitted to the experimental creep strain data using the curve fitting toolbox in MatLab [38]

Fig. 11. a) creep strain accumulation and b) stress-relaxation behaviour of 10%Cr steel during the on-load phase of Set B tests

Fig. 12. Anelastic recovery behaviour at zero-stress for specimens in Set B tests after a) constant-load forward creep and b) constant-strain relaxation creep

Fig. 13. Comparison of zero-stress anelastic recovery behaviour of testpieces of Set B

Fig. 14. Stress-strain behaviour during unloading of testpieces at the end of creep and stress-relaxation tests of Set B

4. Discussion

In the following, the experimental observations presented in the previous section are further discussed.

4.1. Effect of prior-loading on subsequent creep behaviour

The experimental results indicate that reverse (i.e. compressive) prior-loading accelerates creep strain accumulation during the early primary creep regime while a tensile over-loading condition decreases the early primary creep rate. Analytical interpretation of these experimental observations based on the conventional strain- or time-hardening rule is not possible and both the hardening rules predict that any type of prior-loading decreases the primary creep rate in subsequent creep loading which is in stark contrast to an acceleration of creep rate observed in this study and in [39-46] for other materials specifically after reverse loading. A possible interpretation of the observed effect of prior-loading on creep behaviour during the early primary creep stage is provided below. Detailed electron microscopy

studies are however required to identify and support the principal microstructural mechanism(s) that govern material deformation under the tested conditions.

Inelastic deformation in metals is primarily accommodated by the generation, movement and interaction (with other microstructural features of relevance) of dislocations. Newly generated dislocations at the initiation of inelastic deformation can easily move along active slip planes but tend to pile-up when stopped by obstacles such as grain boundaries, solutes, second phase particles or sessile dislocations (immobile dislocations, e.g. jogs) [47,48]. Increasingly higher pile-up at such barriers modifies the effective stress acting on mobile dislocations as the generated localised (micro-) backstress acts against the applied external stress. The pile-up dislocations are loosely entangled, less stable and can exhibit reversible motion (and/or annihilation) to relax the backstress on reduction or reversal of the applied stress [47,49]. Further forward inelastic straining increases the density of pile-up dislocations and thereby the magnitude of the resulting backstress that consequently leads to lower effective stress acting on the mobile dislocations. Reduction of the effective stress due to an increase in backstress is thus cited as one of the major reasons for the observed decreasing deformation rate during the primary creep regime [50-54]. Ultimately and for considerably large strains, the magnitudes of dislocation density and backstress at pile-ups saturate such that the secondary (i.e. steady-state) creep regime with a constant deformation rate ensues.

The density of dislocations that exhibits reversible motion at the pile-ups increases for larger forward inelastic deformations accumulated under the higher applied stresses. It is therefore expected that the dense dislocation pile-ups already generated during a higher pre-loading routine (e.g. tensile prior-loading at 350/400MPa) reduces the deformation rate during subsequent loading at a lower stress level (e.g. forward creep at 200/300MPa). For conditions where stress level during subsequent loading is significantly lower than that during pre-loading, the significance of pile-up dislocation backflow may also be large enough to bring about 'negative-creep' strain at the new stress level (e.g. for forward creep at 200MPa after prior-loading at 350/400MPa), Fig 8a.

The dislocation pile-up consideration can also be extended to support the experimentally observed higher creep rate in specimens that were reverse pre-loaded. Development of pile-up induced backstress during reverse pre-loading acts in favour of the applied stress during subsequent forward loading and thus accelerates the forward dislocation movement (along the lines of the phenomenological description for the Bauschinger effect [55] on strain reversal) [56]. Moreover, the development of backstress during reverse prior-loading has been reported to relax during forward

loading by reverse glide and mutual annihilation mechanisms [57]. Therefore reverse pre-loading not only does not reduce the creep rate (as strain- and time-hardening rules predict), but also accelerates the creep strain accumulation during early forward creep loading.

Fig. 15 provides a simplified schematic illustration of the dislocation-barrier interaction mechanism expected after tensile prior-loading (Fig. 15a) and reverse prior-loading (Fig. 15b). A similar mechanism has been considered by Carroll et al. [44] to explain the forward creep behaviour in the CMSX-4 single crystal alloy at 950°C before and after a stress reversal. Their observations also indicated that for forward creep loading, a network of dislocations of opposite sign piled on either side of the narrow γ channels (i.e. at γ/γ' interfaces). The reversal of stress allowed opposite sign dislocations at both sides of the γ channels to move together and to result in an accelerated reverse creep.

The above directional hardening (/softening) mechanism can be analytically explained via kinematic-hardening, a concept used extensively in phenomenological cyclic-plasticity and -viscoplasticity models [58-60]. Accordingly to this concept, an effective stress can be expressed quantitatively as the difference between the external (applied) stress σ and the internal backstress X while assuming dislocation pile-ups to be the primary reason for the latter. Incorporation of the kinematic-hardening concept into the basic Norton creep equation [61] for uniaxial isothermal creep deformation gives

$$\dot{\varepsilon}_{cr} = A|\sigma - X|^{n+1}/(\sigma - X) \quad (1)$$

where $(\sigma - X)$ is the effective stress, A and n are material constants and ε_{cr} is the creep strain. As per Armstrong-Frederick [60], the equation for backstress evolution can be presented as

$$\dot{X} = C\dot{\varepsilon}_{cr} - \gamma|\dot{\varepsilon}_{cr}|X \quad (2)$$

where C and γ are hardening and softening coefficients, respectively. During a reverse (i.e. compressive, $\sigma < 0$) pre-loading routine that involves inelastic deformation, backstress tends to be negative. At the end of pre-loading and the start of subsequent forward creep loading (i.e. tensile, $\sigma > 0$), a higher creep rate can be attributed to an increase of effective stress (i.e. $(\sigma - X) > \sigma$) acting on the material as the stored negative backstress acts in favour of the applied positive stress. With continuous tensile straining, backstress however gradually increases towards a positive quantity and consequently decreases the effective stress on the material (i.e. $(\sigma - X) < \sigma$). The magnitude of backstress keeps increasing until it reaches the saturation value of C/γ wherein steady-state creep regime characterised by a minimum strain rate ensues. The kinematic-hardening formulation based

on Eqs. (1) and (2) was employed for representing the material deformation characteristics during 200MPa creep tests with and without prior-loading histories (see Fig. 16). The effectiveness of the kinematic-hardening assumption in closely simulating the experimental creep behaviour is clearly validated for all the considered prior-loading histories and that too particularly during the early primary creep stage. An ongoing study aims to further refine the existing model formulation for representing the deformation behaviour of all the presented experiments in this study.

The kinematic-hardening concept further predicts the secondary creep rate, after the early primary creep stage, to not vary significantly during forward creep for specimens with different prior-loading histories. However the obtained experimental results indicate the tensile over-loading and reverse prior-loading to increase and decrease the secondary creep rate, respectively. Although the kinematic-hardening consideration can closely interpret the observed early primary creep response after different prior-loading conditions, it fails to represent the observed experimental behaviour at the end of primary and secondary creep stages. More detailed microstructural investigations are required to better understand the underlying mechanism(s) for the observed secondary creep response after prior-loading histories.

4.2. Effect of prior-loading on subsequent anelastic recovery behaviour

Anelastic recovery has been mechanistically interpreted in the literature in relation with pile-up of dislocations and bowing of dislocation lines or sub-grain boundaries etc. [47,62-67]. For example, Hart [68] considered dislocation pile-up at barriers to be the major source of recovered anelastic strain after unloading while Ghosh [69] attributed decrease of dislocation density during unloading to backflow and annihilation of dislocations that escape from pile-ups. As explained in the previous section, decrease or removal of the external stress from a pre-deformed specimen allows dislocations in a pile-up to move backward (and annihilate) and to relax the localised stress fields therein (i.e. backstress relaxation). The backward movement of dislocations is fast at the beginning when the pile-up stress field is strong but slows down later as the stress field partially relaxes.

Although it has been observed to increase by increasing the magnitude of prior accumulated inelastic strain during the primary creep stage [42,65,70-73], the amount of recoverable strain usually saturates for deformation during the secondary creep stage [42,65,70-73]. For lower strains, most of the generated dislocations are still loosely entangled at the pile-ups and can reverse glide and annihilate on removal of the external stress and result in almost full recovery of the accumulated strain [74]. However, for higher amounts of both accumulated strain and dislocation densities, complex

interactions of dislocations with each other or with obstacles may hinder the capacity of dislocations to reverse glide and therefore decreases the ratio of recoverable to non-recoverable strains [74].

The decreasing rate in the increase of recoverable strain during creep deformation can be phenomenologically interpreted by the kinematic-hardening consideration and backstress concept described earlier. The Armstrong-Frederick [60] expression (Eq. 2) might be adopted to assume the backstress to result from an increase in the formation of dislocation pile-ups at a decreasing rate during the course of primary creep deformation and to finally saturate at the start of the secondary creep stage. Therefore it is expected that the amount of recoverable strain, that results from the relaxation of stress fields around the dislocation pile-ups, to also increase during primary creep with a decreasing rate and to saturate at the start of secondary creep stage.

Experimental observations in this study indicated that, for a constant amount of accumulated creep strain, the recovered strain is higher for those specimens pre-loaded under a higher stress level. Similar observations have been reported earlier by Es-Souni for Ti6242Si at 500°C [42]. As mentioned earlier, the dislocation density at the pile-ups is higher for larger extents of inelastic forward straining under higher stress levels. For a constant amount of accumulated time-dependent inelastic strain, faster deformation rate under higher stresses reduces the duration and therefore the extent of dislocation recovery. Therefore for a constant amount of accumulated inelastic strain, a higher stress level during the loading phase results in a denser dislocation pile up and consequently larger extent of anelastic recovery after unloading.

Furthermore, higher anelastic recovery for loading under a high stress condition can be explained by consideration of the bowing of dislocations or sub-grain boundaries under an external applied stress [62-66]. There is electron microscope evidence (e.g. [64,75]) from samples cooled down under creep loading conditions to indicate bowing of dislocation links as a result of the applied stress. On stress removal, the bowed dislocations return to their unstressed state and result in the development of a reverse strain [75]. The curvature of the bow is proportional to the magnitude of the applied stress and therefore higher applied stress leads to a greater extent of anelastic strain recovery due to the un-bowing of the dislocation lines ($k = 1/R = \tau/\alpha Gb$ where k and R are respectively curvature and radius of bow, τ is the applied stress, α , G and b are material constants [76]). In addition to dislocations, subgrain walls may also bow in response to the applied stress. Of course, when the applied stress is removed, these boundaries can un-bow as well and generate a reverse strain [75].

Experimental observations indicated the unloading modulus for experiments of Set B to be always lower than the loading modulus and also to be lower in particular for higher pre-stress levels. This can be attributed to the simultaneous occurrence of anelastic recovery during the unloading transient. The reduction of load allows some extent of anelastic recovery (negative creep) during unloading which affects the apparent unloading modulus and shows a lower value in comparison with that during the loading transient. It has already been explained that higher stresses during prior-loading increase the extent of anelastic recovery. Therefore, a higher prior-stress results in a faster anelastic recovery and a lower unloading modulus. A lower modulus during unloading and its correlation with anelastic recovery has previously been reported [47,49,67,69]. For example Pavlina et al. [77] and Cleveland [67] have considered low moduli during unloading in particular for unloading after higher amounts of deformation or from higher stresses.

Fig. 15. Simplified schematic illustration of the kinematic-hardening concept based on the consideration of dislocation pile-ups in the vicinity of dislocation barriers for (a) prior-loading to a higher magnitude in the same direction and (b) reverse prior-loading. Bold black line represents a barrier and green arrows show the extent/direction of dislocation motion

Fig. 16. Experimental data from Set A tests (only 200MPa) plotted along with predictions from kinematic-hardening rule of

$$\left(\dot{\epsilon}_{cr} = 8.1 \times 10^{-22} |\sigma - X|^{7.8} / (\sigma - X), \dot{X} = 2.3 \times 10^5 \dot{\epsilon}_{cr} - 1.7 \times 10^3 |\dot{\epsilon}_{cr}| X \right)$$

5. Concluding Remarks

Two sets of tests have been conducted to investigate the influence of prior-loading on subsequent creep and anelastic recovery for a 10%Cr steel at 600°C. The first experiment set included constant-stress creep tests conducted with 200 and 300MPa, with different tensile and compressive pre-loading histories. Experimental observations indicated that the early primary creep rate is higher for specimens with reverse prior-loading and is lower for the specimens which were over-loaded before the start of the constant-load creep testing. While neither creep strain- nor time-hardening rules could interpret the observed influence of prior-loading on the early primary creep behaviour of the alloy, the behaviour could be interpreted based on a kinematic-hardening consideration. During prior-loading, dislocation pile-ups develop in the vicinity of dislocation barriers (e.g. grain boundaries). The stress fields associated with such pile-ups deaccelerate and accelerate further deformation in the same and reverse directions, respectively. Therefore it is expected that creep loading after prior-over-loading and reverse prior-loading respectively give lower and higher primary creep rates in comparison with the condition with no prior-loading. The described mechanism cannot however interpret the influence of prior-loading on secondary and tertiary creep stages and further microstructural investigation is required to understand the governing mechanism for the observed influence.

The second set of experiments aimed to investigate the effect of prior-loading on the subsequent anelastic recovery behaviour of the alloy. The tests included accumulation of 0.066% creep strain at different stress levels under either constant-stress creep loading or constant-strain stress-relaxation, and subsequently monitored anelastic recovery behaviour. The observations indicate that although the mode of prior-loading (i.e. creep or stress-relaxation) does not affect the subsequent anelastic

recovery behaviour, the higher the level of stress during prior-loading, the greater is the amount of subsequent anelastic recovery. Such observations were interpreted based on consideration of the relaxation of dislocation bows and pile-ups upon unloading of the crept testpieces.

Acknowledgment

Financial support by the Swiss National Science Foundation (SNSF) is gratefully acknowledged.

References:

1. Klueh R. Elevated temperature ferritic and martensitic steels and their application to future nuclear reactors. *International Materials Reviews*. 2005;50(5):287-310.
2. Abe F. Research and development of heat-resistant materials for advanced USC power plants with steam temperatures of 700°C and above. *Engineering*. 2015;1(2):211-24.
3. Briggs J, Parker T. *The Super 12% Cr Steels*, Climax Molybdenum CD. Michigan; 1982.
4. Armor AF, Viswanathan, R. *Supercritical Fossil Steam Plants: Operational Issues and Design Needs for Advanced Plants in 4th International Conference in Advances in Materials Technology for Fossil Power Plants*, ASM International, Hilton Head Island: ASM International; 2005.
5. Mayer K, Bendick W, Husemann R, Kern T, Scarlin R. *New materials for improving the efficiency of fossil-fired thermal power stations*. GEC ALSTHOM Energie GmbH, Nuremberg (DE), 1998.
6. Dubey J, Chilukuru H, Chakravartty J, Schwienheer M, Scholz A, Blum W. Effects of cyclic deformation on subgrain evolution and creep in 9–12% Cr-steels. *Materials Science and Engineering: A*. 2005;406(1):152-9.
7. Fournier B, Dalle F, Sauzay M, Longour J, Salvi M, Caës C, et al. Comparison of various 9–12% Cr steels under fatigue and creep-fatigue loadings at high temperature. *Materials Science and Engineering: A*. 2011;528(22):6934-45.
8. Agamennone R, Blum W, Gupta C, Chakravartty J. Evolution of microstructure and deformation resistance in creep of tempered martensitic 9–12% Cr–2% W–5% Co steels. *Acta Materialia*. 2006;54(11):3003-14.
9. Kostka A, Tak K-G, Hellmig R, Estrin Y, Eggeler G. On the contribution of carbides and micrograin boundaries to the creep strength of tempered martensite ferritic steels. *Acta Materialia*. 2007;55(2):539-50.
10. Chilukuru H, Durst K, Wadekar S, Schwienheer M, Scholz A, Berger C, et al. Coarsening of precipitates and degradation of creep resistance in tempered martensite steels. *Materials Science and Engineering: A*. 2009;510:81-7.
11. Abe F. Coarsening behavior of lath and its effect on creep rates in tempered martensitic 9Cr–W steels. *Materials Science and Engineering: A*. 2004;387:565-9.
12. Maruyama K, Sawada K, Koike J-i. Strengthening mechanisms of creep resistant tempered martensitic steel. *ISIJ international*. 2001;41(6):641-53.
13. Binda L, Holdsworth S, Mazza E. Influence of prior cyclic deformation on creep properties of 1CrMoV. *Materials at High Temperatures*. 2010;27(1):21-7.
14. Plumbridge W, Miller K. Influence of prior fatigue on creep of a 'non-heat-treated' alloy steel. *Metals Technology*. 1975;2(1):249-52.
15. Sidey D, *Creep Fatigue Interactions in a Low Alloy Steel*. ICF4, Waterloo (Canada) 1977; 1977.
16. Kalyanasundaram V, and Holdsworth, S.R. The influence of prior plasticity on the creep resistance of two elevated temperature power plant steels. *First Structural Integrity Conference and Exhibition, SICE 2016*, Bangalore, India, 2016.
17. Ellison E, Paterson A. *Creep Fatigue Interactions in a 1CrMoV Steel*. *Proceedings of the Institution of Mechanical Engineers*. 1976;190(1):319-30.
18. Holdsworth S. Component assessment data requirements from creep-fatigue tests. *Creep-Fatigue Interactions: Test Methods and Models*: ASTM International; 2011. p. 1-14.
19. Kalyanasundaram V, Holdsworth SR. Prediction of forward creep behaviour from stress relaxation data for a 10% Cr steel at 600° C. *Transactions of the Indian Institute of Metals*. 2016;2(69):573-8.

20. Sarkar A, Vijayanand V, Parameswaran P, Shankar V, Sandhya R, Laha K, et al. Influence of prior fatigue cycling on creep behavior of reduced activation ferritic-martensitic steel. *Metallurgical and Materials Transactions*. 2014;45(7):3023.
21. Vorpahl C, Möslang A, Rieth M. Creep-fatigue interaction and related structure property correlations of EUROFER97 steel at 550° C by decoupling creep and fatigue load. *Journal of Nuclear Materials*. 2011;417(1):16-9.
22. Fournier B, Sauzay M, Caës C, Noblecourt M, Mottot M, Allais L, et al. Creep-fatigue interactions in a 9%Cr-1%Mo martensitic steel: Part I. Mechanical test results. *Metallurgical and materials transactions A*. 2009;40(2):321-9.
23. Holdsworth SR. 'Influence of prior fatigue on creep properties', 12th International Conference on Creep and Fracture of Engineering Materials and Structures, CREEP 2012, Kyoto, Japan, 2012, 27-31/5/2012.
24. Takahashi Y. Effect of cyclic loading on subsequent creep behaviour and its implications in creep-fatigue life assessment. *Materials at High Temperatures*. 2015;32(5):492-501.
25. Woodford DA. Test methods for accelerated development, design and life assessment of high-temperature materials. *Materials & Design*. 1993;14(4):231-42.
26. Rao GR, Gupta O, Pradhan B. Application of stress relaxation testing in evaluation of creep strength of a tungsten-alloyed 10% Cr cast steel. *International Journal of Pressure Vessels and Piping*. 2011;88(2):65-74.
27. Laflen J, Jaske C. Cyclic relaxation response under creep-fatigue conditions. *Stress Relaxation Testing*: ASTM International; 1979.
28. Altenbach H, Naumenko K, Gorash Y. Creep analysis for a wide stress range based on stress relaxation experiments. *International Journal of Modern Physics B*. 2008;22(31n32):5413-8.
29. Grzywnski G, Woodford D. Design data for polycarbonate from stress-relaxation tests. *Materials & Design*. 1993;14(5):279-84.
30. Zener C, Siegel S. *Elasticity and Anelasticity of Metals: The Journal of Physical and Colloid Chemistry*. 1949.
31. Bolton J. The consequences of recovery for the analysis of creep in steel structures. *International Journal of Pressure Vessels and Piping*. 2010;87(5):266-73.
32. Gibeling J, Nix W. Observations of anelastic backflow following stress reductions during creep of pure metals. *Acta Metallurgica*. 1981;29(10):1769-84.
33. Sawada K, Kimura K, Abe F. Mechanical response of 9% Cr heat-resistant martensitic steels to abrupt stress loading at high temperature. *Materials Science and Engineering: A*. 2003;358(1):52-8.
34. Mayer K, Berger C, Scarlin R, Gemeinschaften GWE. New steels and manufacturing processes for critical components in advanced steam power plants: European Commission cost physical sciences: Office for Official Publ. of the Europ. Communities; 1996.
35. Holdsworth S. Creep-fatigue in steam turbine materials. *Proceedings of the 6th International Conference on Advances in Materials Technology for Fossil Power Plants*, EPRI, Santa Fe, NM, USA2010. p. 487-503.
36. Radosavljevic M, Holdsworth S. Novel induction heating system for service cycle thermo-mechanical fatigue testing. *Materials at High Temperatures*. 2009;26(4):369-74.
37. Holdsworth SR, Mazza E. Using the results of creep crack incubation tests on CrMoV steel for predicting long time creep rupture properties. *Int J Pres Ves Pip*. 2009;86(12):838-44.
38. MathWorks I. *Curve fitting toolbox: for use with MATLAB®: user's guide*: MathWorks; 2002.
49. Andrade END, Jolliffe KH. The flow of polycrystalline metals under simple shear. *Proc Royal Soc London Ser A*. 1952 1952;213(1112):3-26.
40. Cho UW, Findley WN. Creep and creep recovery of 304 stainless steel under combined stress with a representation by a viscous-viscoelastic model. *J Appl Mech*. 1980;47(4):755-61.
41. Murakami S, Ohno N, Tagami H. Experimental evaluation of creep constitutive equations for type 304 stainless steel under non-steady multiaxial states of stress. *J Eng Mat Techn*. 1986;108(2):119-26.
42. Es-Souni M. Primary, secondary and anelastic creep of a high temperature near α -Ti alloy Ti6242Si. *Mater Charact*. 2000;45(2):153-64.
43. Penkalla HJ, Schubert F, Nickel H. Torsional creep of alloy 617 tubes at high temperature. *Proc Con Superalloys* 1988:643-52.
44. Carroll MC, Serin K, Eggeler G. Anisotropic strain hardening following load reversal during high-temperature creep testing of superalloy single crystals. *Mater Sci Eng A*. 2004;387:590-4.
45. Sonnerup L. *Stress reversals in connection with primary creep*: Scandinavian University Books; 1965.
46. Rees DWA. Deformation and rupture of stainless steel under cyclic, torsional creep. *Engineering Integrity*. 2008;25(1):15-22.

47. Kim H, Kim C, Barlat F, Pavlina E, Lee M-G. Nonlinear elastic behaviors of low and high strength steels in unloading and reloading. *Materials Science and Engineering: A*. 2013;562:161-71.
48. Reddy, L.K., *Principles of Engineering Metallurgy*. 2007, New Age International.
49. Yoshida F, Uemori T, Fujiwara K. Elastic–plastic behavior of steel sheets under in-plane cyclic tension–compression at large strain. *International Journal of Plasticity*. 2002;18(5):633-59.
50. Fressengeas C, Acharya A, Beaudoin A. Dislocation mediated continuum plasticity: Case studies on modeling scale dependence, scale-invariance, and directionality of sharp yield-point. *Computational Methods for Microstructure-Property Relationships*: Springer; 2011. p. 277-309.
51. Devincere B, Gatti R. Physically justified models for crystal plasticity developed with dislocation dynamics simulations. *AerospaceLab*. 2015 (9):p. 1-7.
52. Feaugas X. On the origin of the tensile flow stress in the stainless steel AISI 316L at 300K: back stress and effective stress. *Acta materialia*. 1999;47(13):3617-32.
53. Lorentzen T, Daymond M, Clausen B, Tomé C. Lattice strain evolution during cyclic loading of stainless steel. *Acta materialia*. 2002;50(6):1627-38.
54. Han C-S, Roters F, Raabe D. On strain gradients and size-dependent hardening descriptions in crystal plasticity frameworks. *Metals and Materials International*. 2006;12(5):407-11.
55. Bauschinger J. Über die Veränderung der Elastizitätsgrenze und der Festigkeit des Eisens und Stahls durch Strecken und Quetschen, durch Erwärmen und Abkühlen und durch oftmals wiederholte Beanspruchung. *Mitt Mech-Techn Lab K Techn Hochsch München*. 1886;13:108-12.
56. Zecevic M, Korkolis YP, Kuwabara T, Knezevic M. Dual-phase steel sheets under cyclic tension–compression to large strains: experiments and crystal plasticity modeling. *Journal of the Mechanics and Physics of Solids*. 2016;96:65-87.
57. Sriharan T, Chandel R. Phenomena in interrupted tensile tests of heat treated aluminium alloy 6061. *Acta materialia*. 1997;45(8):3155-61.
58. Chaboche JL. Constitutive equations for cyclic plasticity and cyclic viscoplasticity. *Int J Plast*. 1989;5(3):247-302.
59. Lemaitre J, Chaboche JL. *Mechanics of solid materials*: Cambridge university press; 1994.
60. Frederick CO, Armstrong PJ. A mathematical representation of the multiaxial Bauschinger effect. *Mat High Temp*. 2007;24(1):1-26.
61. Norton FH. *The creep of steel at high temperatures*: McGraw-Hill; 1929.
62. Gibbons TB, McLean D, Lupinc V. Dislocation anelasticity, or engineers creep recovery (in annealed nickel and Ni-Cr alloy). *Metal Sci*. 1975;9:437-44.
63. Lloyd GJ, McElroy RJ. On the anelastic contribution to creep. *Acta Metal*. 1974;22(3):339-48.
64. Lupinc V, Gabrielli F. Effect of grain size, particle size and γ' volume fraction on strain relaxation in Ni-Cr base alloys. *Mater Sci Eng*. 1979;37(2):143-9.
65. Morris DG. Anelasticity and creep transients in an austenitic steel. *J Mater Sci*. 1978;13(9):1849-54.
66. Pahutova M, Čadek J, Ryš P. Some stress change experiments on creep in α zirconium. *Mater Sci Eng*. 1979;39(2):169-74.
67. Cleveland R, Ghosh A. Inelastic effects on springback in metals. *International Journal of Plasticity*. 2002;18(5):769-85.
68. Hart E. A micromechanical basis for constitutive equations with internal state variables. *ASME J Eng Mater Technol*. 1984;106(4):322-5.
69. Ghosh A. A physically-based constitutive model for metal deformation. *Acta Metallurgica*. 1980;28(11):1443-65.
70. Gibeling JC, Nix WD. Observations of anelastic backflow following stress reductions during creep of pure metals. *Acta Metal*. 1981;29(10):1769-84.
71. Henderson J, Snedden JD. Creep recovery of Aluminum alloy DTD 2L42. *Appl Mater Res*. 1965;4(3):148-67.
72. Henderson J, Snedden JD. Creep recovery of commercially pure copper. *J Mech Eng Sci*. 1968;10(1):24-35.
73. Lubahn JD. The role of anelasticity in creep, tension and relaxation behavior. *Trans ASM*. 1953;45:787-838.
74. Kitayama K, Tomé C, Rauch E, Gracio J, Barlat F. A crystallographic dislocation model for describing hardening of polycrystals during strain path changes. Application to low carbon steels. *International Journal of Plasticity*. 2013;46:54-69.
75. Beere W, Crossland IG. Primary and recoverable creep in 2025 stainless steel. *Acta Metal*. 1982;30(10):1891-9.
76. Hull D, Bacon DJ. *Introduction to dislocations*: Butterworth-Heinemann; 2001.

77. Pavlina EJ, Lee M-G, Barlat F. Observations on the nonlinear unloading behavior of advanced high strength steels. *Metallurgical and Materials Transactions A*. 2015;46(1):18-22.
78. Bailey R. The utilization of creep test data in engineering design. *Proceedings of The Institution of Mechanical Engineers*. 1935;131(1):131-349.

Accepted manuscript