



# 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.

## 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 (CO<sub>2</sub>) 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 CO<sub>2</sub> 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 major objective of this work is therefore to provide detailed scientific insights on the effect of prior deformation on subsequent creep 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

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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(s).

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

### 2.1. Test material

The test material chosen for this study is an advanced high-chromium forged rotor steel that originated as Steel F (melt 3 A) 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  $\geq 50$  °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).



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

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

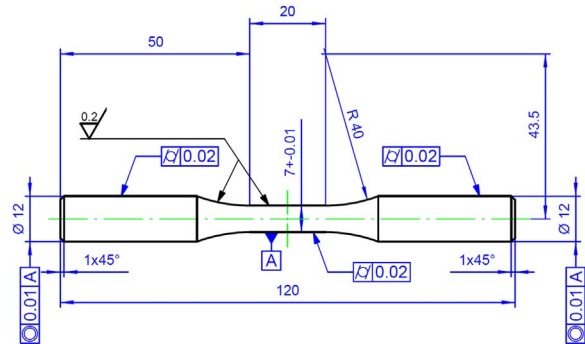


Fig. 2. Testpiece geometry used for the proposed tests.

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.

### 2.2. Test specimen and setup

Cylindrical dogbone specimens with a gauge diameter and parallel length of 7 mm and 20 mm, respectively were used for the experimental program, refer Fig. 2. Tests were conducted in an induction heating based closed-loop servohydraulic system of 100 kN 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  $\pm 1$  °C from the target temperature along the specimen parallel length. Three s-type thermocouples were spot-welded for monitoring temperature in the parallel length, one at the middle and one each at 9 mm from either side of the middle. In particular, the middle thermocouple was digitally controlled to within  $\pm 0.2$  °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 15 mm 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.

### 2.3. 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

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