Primary creep regeneration in 10%Cr martensitic steel: \textit{In-situ} and \textit{ex-situ} microstructure studies

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\textbf{HIGHLIGHTS}

- Primary creep regeneration phenomenon (PCR) has been systematically investigated for a 10\%Cr martensitic steel.
- PCR is governed by the evolution of internal stress and dislocation movement resistance of the steel during cyclic loading.
- PCR phenomenon has been explained by the formation/relaxation of dislocation pile-ups and bowing/unbowaing of dislocation-lines.
- The proposed mechanistic explanation could explain the sensitivity of PCR to various parameters of the loading profile.

\textbf{GRAPHICAL ABSTRACT}

Primary creep regeneration (PCR) is a phenomenon observed during stress-varying/cyclic creep loading conditions where a load reversal might clear the previous strain hardening memory and cause the regeneration of the primary creep regime (i.e. a period of high creep strain rate) upon reloading. In this study, \textit{in-situ} and \textit{ex-situ} microstructural examinations, including transmission electron microscopy (TEM), electron backscatter diffraction (EBSD), neutron and synchrotron X-ray diffraction were conducted to better understand the responsible mechanisms of PCR for a 10\%Cr martensitic steel at 600 °C. Our experimental evidence indicated that the PCR phenomenon is related to the change of dislocation density due to activation of dislocation generation and recovery mechanisms, formation and relaxation of dislocation pile-ups, as well as bowing/unbowaing of dislocation-lines during stress-varying creep loading conditions. These mechanisms could explain the observed creep strain accumulation in the steel during the examined stress-varying creep loading conditions reported in the current and previous studies. The presented mechanistic description of the PCR phenomenon and the reported experimental observations for the microstructural and mechanical parameters can provide a basis for the formulation of physically-based models to describe the creep behaviour of the steel under high-temperature stress-varying creep loading conditions.

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1. Introduction

Primary creep regeneration (PCR) is an important phenomenon occurring during stress-varying/cyclic creep loading conditions. For some materials, load reversal might clear the previous strain hardening memory and induce a period of high creep strain rate upon reloading, i.e. PCR. Under certain conditions, creep strain rates after the stress-transient can be even higher than that for the primary creep regime. Early observations for the PCR phenomenon were reported for lead, cadmium and aluminium in 1950–1970 [1–4]. With the requirement for more flexible operation of the thermal power plants, understanding the cyclic creep and PCR response of advanced high-temperature steels has gained attention [5–9]. Early observations for the PCR phenomenon for a 10%Cr martensitic steel during low-cycle fatigue and creep-fatigue experiments were reported in [10]. The present authors have conducted a set of stress-varying creep experiments for 316H austenitic and the 10%Cr steels to investigate the sensitivity of their PCR behaviour to the parameters of loading profile. The experimental observations revealed that PCR is more significant for smaller forward-loading levels, longer creep-testing times, higher reverse-loading magnitudes and longer reverse-loading durations. A recent study in the University of Bristol [5] employed in-situ neutron diffraction technique to evaluate the evolution of lattice strains during cyclic creep loading and provide a mechanistic interpretation of the PCR phenomenon for the 316H stainless steel. The present study focuses on the 10%Cr steel with the complex martensitic microstructure and employs a wide range of ex-situ and in-situ examination techniques to provide insights into the underlying mechanisms of PCR.

The PCR phenomenon is expected to be related to the evolution of internal stress and dislocation movement resistance in the materials during stress-varying creep loading [6–8]. The material internal stress mainly originates from dislocation pile-ups [11], strain incompatibility fields between adjacent grains [12–15] and dislocation-line bows [16,17]. X-ray diffraction (XRD), neutron diffraction (ND) and electron backscatter diffraction (EBSD) are suitable techniques for measuring the material’s internal stress [14]. On the other hand, material resistance to the dislocation movement is mainly controlled by the dislocation density [14,18], which can also be quantified by the diffraction techniques, as well as electron microscopy.

This study aims at the identification of microstructural mechanisms associated with PCR. To this end, a comprehensive microstructural analysis was performed using techniques of transmission electron microscopy (TEM), EBSD, neutron and X-ray diffractions for assessment of the evolution of dislocation density and internal lattice strain in the 10%Cr steel during stress-varying creep loading conditions at 600 °C. Interrupted stress-varying creep experiments were conducted to create representative samples for the microstructural states of the steel before, during and after stress-transients (ex-situ experiments). These samples were then investigated by TEM, EBSD and ND analysis. In addition, diffraction profiles from an in-situ synchrotron XRD experiment were analysed to determine the time-resolved evolution of dislocation density and lattice strain during stress-varying creep loading. The observations from ex-situ and in-situ experiments were combined to better understand the PCR response of the steel.

2. Experimental details

The investigated 10%Cr steel is a material routinely used for high-temperature steam turbine rotor applications. The as-received material originates from a forged block with the chemical composition of 9.8%Cr, 1.4%Mo, 0.6%Ni, 0.4%Mn, 0.2%V and 0.1%C, 0.048%N (in wt%). The heat treatment for this type of steel includes solution annealing at 1060–1080 °C followed by fast cooling and a two-stage tempering at 540–580 °C (completion of the martensite transformation) and 675–705 °C (optimization of the carbide condition). As presented in Fig. 1, such a heat treatment resulted in a martensite microstructure with a prior austenitic grain size of ~120 μm and numerous chromium carbides precipitates along the lath boundaries.

2.1. In-situ experiment

The high-temperature in-situ creep experiment was performed at beamline 1-ID of the Advanced Photon Source (APS) at Argonne National Laboratory, USA. Due to the limited duration of the allocated beamline, results of the previously conducted mechanical experiments [8] (summarised in Sec. S1) were exploited to design a dedicated short-term 43 h loading profile for the in-situ experiment to effectively investigate the sensitivity of the PCR response, dislocation structure and internal lattice strain/stress state of the steel to different parameters of the loading conditions. Fig. 2 illustrates the designed loading profile which includes different forward-loading levels, and different reverse-loading magnitudes and durations. The examined R ratios (minimum/maximum stresses) in this study are between −1.3 and 0. It should be noted that the designed loading profile for the experiment is short and simple in order to enable a systematic investigation of the underlying mechanisms of PCR. The design was also influenced by the technical

Fig. 1. EBSD maps (step size 0.8 μm) and scanning transmission electron microscopy (STEM) micrographs for the investigated 10%Cr steel: EBSD inverse pole figure, (IPF) (a), high-angle annular dark-field (HAADF) STEM (b), the Cr elemental map extracted from EDS spectrum (c). The average diameter and number density of chromium carbides are ~120 nm and ~ 5 × 1019 m−2, respectively.
limitations for conducting an in-situ experiment at APS (USA), and therefore the application of the outcomes for interpretation of industrial cases needs caution.

The testpiece had a gauge length, width and thickness of 2, 3 and 1 mm, respectively (Fig. S5). The 1 mm thickness of the specimen is chosen to assure adequate X-ray beam flux through the specimen, while including a sufficient number of grains through the thickness and therefore an acceptable statistical relevance of the generated data. The stress-varying creep experiment was carried out using an MTS servo-hydraulic test frame equipped with an infrared heating furnace controlling the specimen temperature at 600 ± 1 °C. A monochromatic 71.676 keV X-ray beam with a 150 × 150 μm² cross-section continuously passed through the specimen during the experiment. Four GE-RT41 detectors arranged as illustrated in Fig. 3 were placed at a distance of 2 m from the specimen to collect the Debye-Scherrer diffraction rings. During the stress-varying creep experiment, force and specimen temperature were recorded continuously. The setup at the APS did not allow direct macroscopic strain measurement during the in-situ experiment. To acquire the strain response of the steel under the applied stress profile, an identical test was performed using a conventional cylindrical dog-bone specimen with gauge diameter and length of 8 and 50 mm, respectively. This test is referred to as “reference mechanical test”.

2.1.1. Data analysis

The analysis of data from the in-situ experiment focused on the evaluation of diffraction patterns over an angular interval of ±2.5° (Fig. 3), which is along the loading direction and therefore corresponds to the largest imposed lattice strains. The pseudo-Voigt function was fitted to the diffraction peaks to determine their position and broadening [19]. The peak position was converted to the lattice spacing based on the Bragg’s law. The observed peak broadening included contributions from the sample and the instrument [20] and therefore a CeO2 standard sample [21] was used to determine the instrumental broadening. As explained in the following, analysis of the lattice spacing and peak broadening provides information about lattice strain and dislocation density, respectively.

2.1.2. Lattice strain and internal stress

Lattice strain from a particular family of crystallographic planes \(\{hkl\}\) can be calculated as:

\[
\varepsilon_{hkl} = \frac{d_{\text{meas}} - d_{\text{ref}}}{d_{\text{ref}}} \frac{1}{d_{\text{ref}}} \tag{1}
\]

where \(d_{\text{meas}}\) is the measured lattice spacing and \(d_{\text{ref}}\) is the material lattice spacing in a stress-free condition (i.e. M1 in Fig. 2) for the \(\{hkl\}\) plane at

Fig. 2. The designed loading profile for the in-situ XRD test. Three different sections (shown with different colours) are included to study the PCR sensitivity to reverse-loading magnitude and duration as well as forward-loading level. Readers are referred to [8] for more observations of the PCR and creep behaviour of the steel from a comprehensive testing program (e.g. under constant stress-level of 230 MPa). To facilitate referring to and discussing the experimental observations, 22 indices (M 1–8, D 1–8 and F 1–6) are introduced. M1: before start of loading; M2: 230 MPa/3 h; M3: 0 MPa/1 h; M4: 230 MPa/3 h; M5: -150 MPa/1 h; M6: 230 MPa/3 h; M7: -190 MPa/1 h; M8: 230 MPa/3 h; D1: -190 MPa/0 h; D2: 230 MPa/3 h; D3: -190 MPa/0.5 h; D4: 230 MPa/3 h; D5: -190 MPa/1 h; D6: 230 MPa/3 h; D7: -190 MPa/2 h; D8: 230 MPa/3 h; F1: -250 MPa/1 h; F2: 230 MPa/3 h; F3: 190 MPa/3 h; F4: -250 MPa/1 h; F5: 190 MPa/3 h; F6: 0 MPa/1 h.

Fig. 3. Presentation of a schematic of the test setup at the APS 1-ID-E endstation.
the test temperature. The lattice strain during the in-situ test is induced from i) external macroscopic stresses and ii) internal micro-level stresses. The contribution from the macroscopic stress can be calculated based on the lattice elastic modulus for the (hkl) crystallographic orientation (\(E_{\text{lat}}\)). Deducting the effect of macroscopic stresses from the lattice strain provides a measure of internal lattice strain [22] which can ultimately be employed for calculation of the internal lattice stress [18,23].

In principle, analysis of diffraction patterns from the angular sections of \(\pm 2.5^\circ\) and \(90 \pm 2.5^\circ\) (Fig. 3) can give the axial and one of the transverse lattice strains for consideration in stress analysis. The recorded data for the angular section of \(90 \pm 2.5^\circ\) in this study showed a low signal to noise ratio and therefore the calculated transverse lattice strains included a considerable uncertainty. Consequently, determination of the evolution of internal lattice stress during the in-situ test was not possible and, as an approximation, the internal lattice strain is considered as an indicator for the internal lattice stress.

2.2. Dislocation density

Two commonly used approaches for calculation of the dislocation density from the diffraction profiles are Convolutional Multiple Whole Profile (CMWP) [24–31] and modified Williamson-Hall (W-H) [32–37]. CMWP is a forward-modelling method which quantitatively evaluates the characteristics of the dislocation structure and other microscopic features by matching measured and theoretically modelled whole diffraction patterns. On the other hand, the modified W-H method is a simple and qualitative approach which estimates the order of magnitude for the dislocation density based on the full width at half maximum (FWHM) quantities for the diffraction peaks. The CMWP method is applied for the calculation of dislocation density in this study and is briefly introduced in the following (for a more comprehensive description, see Sec. S5).

The CMWP method assumes that the broadening of diffraction peaks is induced from two contributors: i) broadening due to the presence of sub-micron coherently-scattering-domains (size broadening), and ii) broadening because of lattice distortions induced by dislocations (strain broadening). The size broadening is considered in the CMWP analysis by assuming the theoretical effect for the presence of equiaxed coherently-scattering-domains with a log-normal size distribution (mean \(m\) and variance \(\sigma\)). Consideration of the strain broadening in the CMWP approach assumes a dislocation structure with density \(\rho\), including an edge/screw ratio described by the variable \(q\), and arranged in a manner (i.e. dipole character) defined by the dimensionless variable \(M^*\). The readers are referred to [33,38,39] and Sec. S5 for a description of these microstructural parameters.

In this study, a negligible evolution for the microstructural characteristics defining \(m, \sigma, q\) and \(M^*\) is expected during the 43 h in-situ creep experiment and the evolution of peak broadening is assumed to be mainly correlated to the evolution of dislocation density \(\rho\). The quantities of \(m, \sigma, q\) and \(M^*\) were therefore only once determined by analysing the diffraction pattern of the specimen before loading at 600 °C (index M1 in Fig. 2). Fig. 4 shows the consistency of the CMWP theoretically calculated and experimentally measured diffraction patterns for index M1, which indicates the high reliability of the analysis (uncertainty of calculated dislocation density: 0.6%). The calculated quantities of \(m, \sigma, q\) and \(M^*\) are listed in Table 1.

2.3. Ex-situ experiment

The ex-situ program included a series of interrupted stress-varying creep experiments to generate representative samples for the microstructure states of the steel before, during, and after stress-reversals. The samples were then analysed by ND, EBSD and TEM. Similar to XRD, ND measures the internal lattice strain and dislocation density by evaluation of the diffraction profiles. However, the higher penetration depth of the neutron beam allows examination over a larger gauge volume (\(4 \times 4 \times 4 \text{ mm}^3\) for ND vs. 0.15 \(\times 0.15 \times 1 \text{ mm}^3\) for XRD) and a better statistical relevance. Furthermore, TEM and EBSD provide direct images of the dislocation structure in the steel. While high-resolution EBSD provides information about the distribution of geometrically necessary dislocations (GND) by the cross-correlation analysis [40], TEM reveals individual dislocation contrast and evidence on their interactions with precipitates and other dislocations. The combination of the three microstructural examination techniques for analysis of the pre-conditioned specimens provides a more complete information about the evolution of microstructure during stress-varying creep loading conditions.

Fig. 5 shows the employed loading profile for the ex-situ study for the 10%Cr steel at 600 °C, which is designed based on the results of the previously conducted mechanical experiments [8] (summarised in Sec. S1). Seven specimens were employed for the ex-situ examination, where each was pre-conditioned to one of the states shown in Fig. 5. State 5 and state 7, i.e. immediate reloading and reloading after 1 h reverse-loading at –300 MPa, are expected to show partial/negligible and significant PCR, respectively. Uniaxial cylindrical bar specimens with a gauge length and diameter of 50 and 8 mm were used for the ex-situ study. Interrupted stress-varying creep experiments were conducted using a 100 kN MTS servohydraulic universal testing machine equipped with an induction heating system (Fig. S9). Specimens were cooled down quickly by compressed air blowers under constant load condition at the designed interruption moment to freeze the microstructures. The gauge lengths of the specimens were sectioned by electrical discharge machining (EDM) to generate microstructural representative samples for examination by ND, TEM and EBSD (Fig. 5 inset).

2.3.1. Neutron diffraction

The conducted ND measurements used the ENGINX neutron diffractometer at the ISIS facility (Oxfordshire, UK). Employed setup is shown in Fig. S10. The seven samples with dimensions of 10 \(\times 6 \times 6 \text{ mm}^3\) were...
exposed to the neutron beam for 2 h and two detectors collected the dif-
fraction data. Additionally and to determine the instrumental broaden-
ing features, an eighth measurement was conducted for a standard pure
Si sample.

As recommended in [41], the diffraction profiles from detector 1
were analysed for calculation of the dislocation density, while the informa-
tion from both detectors was used for determining the internal lat-
tice strain components and ultimately estimating the internal lattice
stress for the samples. The recorded diffraction data in terms of times
of flight (TOF) were converted to intensity vs. 2θ by using the fictive
wavelength of $\lambda = 0.0578$ nm [42]. Internal lattice strain calculations
dislocation density analysis evaluated the reflections from {110},
{200}, {211}, {220}, {310} and {222} crystallographic plane families. De-
tails of the data analysis strategy for calculation of dislocation density
and internal lattice strain were similar to that described for the in-situ
synchrotron XRD experiment. The linear elastic assumption was used
for calculation of the internal stresses where $\varepsilon_{hkl}^{11}$ and $\varepsilon_{hkl}^{22}$ ($= \varepsilon_{hkl}^{33}$) were
derived from detector 1 and 2 measurements, respectively.

2.3.2. Transmission electron microscopy
TEM and STEM studies were carried out using the Thermofischer Sci-
entific FEI Talos F200X instrument operating at 200 kV. Preparation of
the TEM samples involved their extraction from the gauge length of
the pre-conditioned specimens, mechanical grinding, polishing, dim-
pling and finally, electropolishing at $-5^\circ$ C by the A2 Struers® solution.
Bright-field (BF) imaging in TEM/STEM, as well as, low- and high-angle
annular dark-field (LAADF/HAADF) imaging in STEM were combined to
investigate the dislocation structure of the steel. For consistency and to
generate comparable micrographs for the seven states of the ex-situ
experiment, all the images were captured under the $\langle 111 \rangle$ zone axis con-
dition. The ‘line-intercept method’ was employed for quantifying the
dislocation density from the TEM images [43,44] (e.g. Fig. 6):

![Fig. 5. Designed loading profile for the ex-situ stress-varying creep experiments for the 10% Cr steel at 600 °C. Inset shows the cutting plan of the specimen gauge length for generating ND, TEM and EBSD samples.](image)

![Fig. 6. Dislocation density measurement for sample 1 of the ex-situ study based on the BF TEM micrograph.](image)

![Fig. 7. Strain evolution measured in the reference mechanical test for the stress profile of the in-situ experiment.](image)

![Fig. 8. Lattice strain development as a function of applied stress for different crystallographic planes in the axial loading direction for the 10%Cr steel at 600 °C.](image)
Table 2
Calculated elastic moduli of 6 crystallographic planes in the axial loading direction for the 10%Cr steel at 600 °C.

| (hkl) | (200) | (211) | (220) | (310) | (222) | (321) |
|-------|-------|-------|-------|-------|-------|-------|
| Elastic modulus (GPa) | 118 ± 8 | 195 ± 2 | 147 ± 8 | 138 ± 6 | 270 ± 13 | 213 ± 39 |

where \( t \) is the thickness of the TEM foil (≈200 nm, estimated from convergent beam electron diffraction under two-beam conditions); \( n \) is the number of intersections between grid lines and dislocations; \( L \) is the length and \( N \) is the number of grid lines. For each sample of the ex-situ examination, several TEM/STEM images were analysed and the average dislocation density is reported.

The higher reliability of dislocation density measurements based on neutron and X-ray diffraction analysis, relative to the TEM technique, should be acknowledged. The dislocation densities obtained from the analysis of TEM images are expected to be systematically smaller than those from the other techniques for two reasons: i) the TEM analysis was performed within the subgrains and cell interior areas where observation of individual dislocations was not handicapped by strong strain contrast modulations; ii) due to the very complex subgrain structure within a grain, the evaluations were performed for the dislocation populations visible at the given illumination conditions and were not corrected for crystallographically equivalent slip systems [45]. The TEM technique however has the advantage of providing direct observations on the dislocations and revealing their arrangement and interactions with different types of dislocation barrier.

2.3.3. Electron backscatter diffraction

EBSD measurements were performed at two areas (40 × 30 μm²) for each of the pre-conditioned specimens using a Tescan Lyra3 scanning electron microscope equipped with an Oxford Instruments Symmetry camera and AZtec data collection software. EBSD diffraction patterns were generated using a beam of 20 kV, 7.2 nA at 15 mm working distance and recorded for a step size of 100 nm with 2 × 2 binning. BLG Vantage CrossCourt v4 was used for the HR-EBSD evaluation, while standard EBSD analysis was carried out with the CHANNELS software package. For the HR-EBSD evaluation, the grain tolerance angle and minimum grain area were set to 6.7° and 50 pixels, respectively.

It is worth noting that the calculated GND density for complex steels such as the 10%Cr steel is sensitive to the parameters of EBSD measurement and cross-correlation analysis (e.g. step size, current/voltage, band contrast threshold, minimum grain size and grain tolerance angle) [46]. For example, the selected grain tolerance angle of 6.7° leads to consideration of some of the low angle grain boundaries (LAGBs) in the GND population and therefore results in relatively high GND densities. Decrease of the tolerance angle however would result in the exclusion of a significant portion of the scanned area from the analysis and therefore was not followed. As the tolerance angle and other relevant parameters were identical for the analysis of all the samples, the obtained GND values can be interpreted for comparison purposes, although the derived GND densities must be treated with caution as they might not be quantitatively representative.

3. Results

3.1. In-situ synchrotron XRD

Fig. 7 shows the strain measured in the reference mechanical test for the same stress profile applied also in the in-situ experiment. The results indicate the acceleration of creep strain rate after reverse-loadings, i.e. PCR (e.g. index F2, as shown in Fig. S6d). As expected, the PCR is more significant for larger reverse-loading magnitudes (Fig. S6a) and longer reverse-loading durations (Fig. S6c). The following presents the evolution of internal lattice strain and dislocation density based on data from the in-situ synchrotron XRD experiment.

3.1.1. Lattice strain evolution

A linear variation for the lattice strain with respect to the applied stress for loading to small stresses (or during unloading) is expected, as shown in Fig. 8. The calculated elastic moduli of 6 crystallographic planes, based on Fig. 8, are given in Table 2 and are in good agreement with [47]. Loading to higher stresses results in a nonlinear lattice strain response due to the development of internal lattice strains (stresses) induced by the systematic arrangement of dislocations and/or formation of inhomogeneous micro-strain fields within the microstructure [48]. Analysis of the lattice strain evolution therefore enables estimation of the developed internal lattice strains which are expected to influence the PCR response.

Fig. 9 shows the evolution of lattice strains during the in-situ stress-varying creep experiment for different crystallographic planes in the axial loading direction. It can be observed that the lattice strain magnitudes for (200) and (310) planes are much larger than the others, and therefore are less affected by the measurement noise/scatter and are better candidates for the analysis of internal lattice strain. To reveal
the evolution of internal lattice strain during the in-situ experiment, the contribution from the externally applied stress is subtracted from the lattice strain records by consideration of the calculated elastic moduli in Table 2. Accordingly, Fig. 10 presents the obtained internal lattice strain evolution for {200} and {310} planes. It can be observed that, during the initial creep loading (i.e., index M2), an internal lattice strain field progressively forms. A subsequent unloading or reverse-loading period decreases the internal lattice strain (e.g., indices M3) and might even lead to the formation of strain fields which contradict the one initially developed, i.e., reverse internal lattice strains (e.g., index M5). The internal lattice strain reduction is more significant for larger reverse-loading magnitudes (e.g., M5 vs. M7) and longer reverse-loading durations (e.g., D3 vs. D5). These experimental observations are further discussed in Sec. 4 to describe their correlation with the steel’s PCR behaviour.

3.1.2. Dislocation density evolution

Fig. 11 shows the calculated evolution of dislocation density based on the CMWP method for the 10%Cr steel during the in-situ experiment. As presented in Fig. S8, a similar dislocation density trend was also derived from the modified W–H method.

Fig. 11 indicates that a reverse-loading in most cases decreases the dislocation density. The decrease is more pronounced for larger and longer reverse-loading conditions. It should however be noted that reverse-loading to a significantly large stress level might increase the dislocation density (e.g., reverse-loading to ~250 MPa during F1 and F4 periods). These experimental observations are further discussed in Sec. 4 to understand the PCR mechanisms.

3.2. Ex-situ experiment results

The in-situ measurements dealt with a single specimen and the derived evolution for the dislocation density and internal lattice strain is immune to the specimen-to-specimen variability. On the other hand, the ex-situ study allows a more detailed evaluation of the steel’s microstructure by employing a wide range of examination techniques and
therefore can provide complementary information for understanding the correlation between microstructure and the PCR phenomenon. However, a possible source of uncertainty in ex-situ measurements is specimens variability. As a qualitative measure, Fig. 12 compares the deformation response of the ex-situ specimens during 15 h creep loading at 230 MPa. Such a comparison indicates a very low variability for the examined 10%Cr steel. This is an important observation which indicates that the main difference between the microstructure of ex-situ samples originates from their different loading histories and can therefore be interpreted to understand the PCR phenomenon.

3.2.1. Neutron diffraction
This section presents the outcomes of ND examination for determining the evolution of internal lattice strains/stresses and dislocation densities for the seven ex-situ samples. Figs. 13a and b present the internal lattice strains in the axial and transverse directions for different crystallographic planes of the examined samples. Similar to the in-situ synchrotron XRD experiment, it can be seen that the internal lattice strain magnitudes for the (200) and (310) planes are much larger than those for the other crystallographic planes and hence are less affected by the noise/scatter of the measurement. Fig. 13c presents the calculated evolution of internal lattice stress in the axial direction for the two crystallographic planes of (200) and (310). It can be seen that internal lattice stress progressively forms during forward-loading and drops upon reverse-loading. Immediate reloading then recovers a significant portion of the drop, while long-duration reverse-loading leads to the development of internal lattice stresses which oppose the one generated during the forward-loading (i.e. reverse internal lattice stress). This reverse internal lattice stress was partially relaxed by reloading from state 6 to 7.

The diffraction patterns from the ND experiment were also analysed by the CMWP method and the derived dislocation densities and other microstructural parameters are presented in Fig. 13d and Table S2. A similar dislocation density evolution trend was also derived from the

![Fig. 13. Evolution of internal lattice strains for different crystallographic planes in axial (a) and transverse (b) directions; calculated internal lattice stress evolution for (200) and (310) planes in axial direction, i.e. $\sigma_{11}$ (c); dislocation densities calculated from the CMWP method (d) for the 7 specimens of the ex-situ examination.](image)

![Fig. 14. STEM micrographs presenting the dislocation population morphologies of ex-situ specimens (a-b) and the derived dislocation density evolution for the ex-situ experiment (c).](image)
modified W–H method (Table S3). Fig. 13d indicates that the dislocation density increases upon forward-loading and then slightly decreases during the 15 h loading under 230 MPa at 600 °C. Reverse-loading to −300 MPa causes a quick drop in the dislocation density, which is partially restored upon immediate reloading. Longer duration reverse-loading at −300 MPa induces an increase in the dislocation density. In contrast to the immediate reloading from sample 4 to 5, reloading from sample 6 to 7 (i.e. after 1 h reverse-loading) decreases the dislocation density.

3.2.2. Transmission electron microscopy
Examples of TEM/STEM images for the ex-situ specimens are presented in Figs. 14a and b, and additional micrographs are given in Fig. S12. Fig. 14c presents the evolution of the dislocation density of the steel during the ex-situ experiment, which is comparable with the outcomes of the ND examination. The derived dislocation densities are based on analysing areas of the presented TEM/STEM micrographs in Fig. S12 where identification of individual dislocations was feasible. Consequently and also since some of the dislocations are invisible in the TEM micrographs taken along the <111> zone axis, the calculated dislocation densities are lower than the real dislocation densities.

3.2.3. Electron backscatter diffraction
Cross-correlation analysis was employed for the calculation of GND density for the ex-situ specimens and the constructed GND maps are presented in Figs. 15a and S12. Fig. 15b illustrates the evolution of GND density during the ex-situ experiment which is primarily comparable with that of the internal lattice stress (Fig. 15c, derived from ND experiment). The main inconstancy between the two trends occurs during the initial loading (from sample 1 to 2), where the GND density unexpectedly decreases. Subsequently and during the 15 h forward-loading, the GND increases while the reverse-loading to −300 MPa leads to a drop in the GND density. Afterwards, reloading to the forward-stress level or 1 h reverse-loading both increase the GND density. Finally, a drop in the GND density was observed for reloading from state 6 to 7. The EBSD evidence along with the other microstructural observations will be discussed in the next section.

4. Discussion
Inelastic deformation and associated phenomena such as PCR are mainly governed by dislocation kinetics which is controlled by i) the applied stress (i.e. the resolved shear stress), ii) material resistance against dislocation movement, and iii) internal stress state [14]. Dislocation obstacles such as MX (M: V, Nb, etc. and X: C, N) and M23C6 (M: Cr, Fe, Mo, etc.) precipitates [49,50], grain boundaries and dislocations themselves increase the resistance against dislocation movement. For negligible variation in the state of the precipitates and grain size, the evolution of the resistance against dislocation movement is governed by the GND density [14]. On the other hand, the internal stress originates from a systematic arrangement of dislocations (pile-ups and dislocation bows) and/or development of inhomogeneous micro-strain fields in the steel. The state of resistance against dislocation movement and internal stress evolves during the deformation and therefore affects the dislocation motion kinetics. As an example and as can be seen in Figs. 11 (transition from M1 to M2) and Figs. 13d and 14c (transition from 1 to 2), dislocation density grows upon initial loading of the steel due to the activation of dislocation generation mechanisms. For sufficiently high temperatures, dislocation recovery mechanisms tend to reduce the dislocation density. The driving force for dislocation recovery is proportional to the dislocation density. For materials with a high initial dislocation density (e.g. martensitic steels), a high driving force for the recovery processes exists which might cause reduction of the dislocation density during slow straining processes [51], e.g. constant load.
creep, e.g. Fig. 11 (index M2) and Figs. 13d and 14c (transition from 2 to 3).

Furthermore, inelastic deformation is accompanied by a series of mechanisms which induce internal stresses in the materials. The internal stress might result from the incompatibility of deformation response of different grains, formations of dislocation pile-ups (Fig. 16a) and bowing of dislocation-lines (Fig. 16b). During monotonic loading, the internal stress progressively increases and acts against the applied stress, hence slows down the dislocation motion kinetics. Internal lattice strain/stress and GND density are both indicative of the internal stress [14,52,53]. As can be seen in Fig. 10 (indices M1 and M2) and Fig. 13c (transition from 2 to 3), an increase in the extent of the lattice strain/stress was observed during the monotonic straining. Consistently, Fig. 15b indicates that the GND density increases during the constant-load creep loading (transition from 2 to 3). However, the decrease of the GND density during the initial loading in Fig. 15b (i.e. transition from 1 to 2) is the opposite of the expected trend and the outcomes of the ND investigations. The EBSD examinations for samples 1 and 2 had been repeated to assure the reliability of the measurements. Up to now, the decrease of GND density cannot be rationalized by the authors and might be related to the unknown prior loading history of the investigated steel.

As mentioned earlier, the dislocation movement kinetics and creep rate are governed by both evolutions of dislocation motion resistance (dislocation density) and internal stresses. For the examined 10%Cr steel during the initial constant-load creep loading, the experimental observations indicate that the increase of internal stress was dominant and slowed down the creep rate, see Fig. 7 (index M2) and Fig. 12 (transition from 2 to 3).

Unloading of a creeping specimen allows relaxation of the internal stresses induced by dislocation pile-ups, bows and incompatibility micro-strains (Fig. 10, transition from M2 to M3). Moreover, unbowing of dislocation-lines, relaxation of pile-ups and recovery processes decrease the dislocation density (Fig. 11, transition from M2 to M3). Unbowing of dislocation-lines shortens the dislocation length and therefore decreases the dislocation density. Furthermore, relaxation of the dislocation pile-ups by back-movement of dislocations might cause mutual annihilation of dislocations and a reduced dislocation density.

Unbowning of dislocation-lines and back-movement of dislocations from pile-ups during the unloading period is expected to induce some level of macroscopic deformation which opposes that previously accumulated, i.e. anelastic recovery. The anelastic recovery phenomenon was observed during the conducted in-situ experiment (index M3 in Fig. 7) as well as in the stress-varying creep records reported in [8] and reviewed in Sec. S1 (e.g. Fig. S2).

Reloading of the steel after a period of unloading results in a slightly higher creep rate than that before the unloading. This is due to the reduction of the dislocation motion resistance (dislocation density) and internal stress during the unloading and can be experimentally observed by comparing the creep rates in Fig. 7, dislocation density in Fig. 11 and internal stress in Fig. 10 (at the end of index M2 and the start of index M4). The reduction in the dislocation motion resistance and internal stress during the unloading period is time-dependent and therefore, while short-duration unloading does not induce any noticeable creep rate increase, long-duration unloading results in an obvious creep acceleration upon reloading, i.e. partial PCR (Fig. S2d).

Similarly and for a creeping material under a forward-stress level, a reverse-loading causes quick change in the state of the dislocation motion resistance and internal stress. The extent of such change and activation of PCR depends on the magnitude and duration of the reverse-loading, as well as the prior deformation history of the steel.

For short-duration small reverse-loading magnitudes (e.g. transient D1 in Fig. 2: reverse-loading to −190 MPa and immediate reloading), recovery mechanisms do not have sufficient time for significant reduction of the dislocation density and therefore the evolution of dislocation density is mainly governed by the dislocation unbowing/bowing and dislocation pile-ups relaxation/reformation. Immediately after reverse-loading, partial relaxation of dislocation pile-ups and bows start and therefore decreases the dislocation density. Subsequent reloading leads to restoration of dislocation pile-ups and bows and therefore increases the dislocation density to a level close to that before the unloading, as observed in Fig. 11 (index D2). The relaxation and reformation of dislocation bows and pile-ups (and incompatibility micro-strains) similarly affect the internal stresses in the steel. As can be seen in Fig. 10, the slight reduction in the internal lattice strain during the momentary transition to D1 was fully recovered shortly after reloading (D2). The similarity of the state of dislocation density and internal stress state of the material before and after reverse-loading indicates that the dislocation kinetics is not notably influenced by such reverse-loadings and therefore a negligible acceleration of creep rate and PCR is expected (Fig. 7, index D1).

Short-duration reverse-loading to larger stress levels (e.g. transient from sample 3 to 5 in Fig. 5: reverse-loading to −300 MPa and immediate reloading) has a more significant influence on the state of dislocation density and internal stress state of the steel. Reverse-loading to large stress levels causes significant back-movement of dislocations from the pile-ups which possibly leads to mutual annihilation of dislocations. Therefore, the dislocation density upon reloading is expected to be smaller than that before the reverse-loading. The observed lower dislocation density of the steel for the sample 5 in comparison with sample 3 for the ex-situ experiment (Figs. 13d and 14c) is consistent with the provided interpretation. It should be noted that such a reduction in the dislocation density during reverse-loading has been predicted in several modelling studies [54–56], where dislocations were divided into two categories of ‘reversible’ and ‘irreversible’, where reversible dislocations were being annihilated upon a change in their glide direction.

The variation of the internal stress during a quick stress-transient to a large reverse-stress level is expected to be similar to that for the dislocation density. Quick relaxation of dislocation bows and pile-ups causes a significant drop in the internal stress upon reverse-loading (Figs. 13c and 15b, transition from 3 to 4). Subsequent reloading increases the internal stress to a value which is smaller than that before the reverse-loading. The reduction of internal stress is due to the annihilation of some of the pile-up-dislocations during the reverse-loading and can be observed in Fig. 14c (sample 3 vs 5). On the other hand, the experimental observations from Fig. 15b indicate that the short-duration reverse-loading did not considerably affect the incompatibility micro-strains and the GND density.

As described above, short-duration stress-transient to a large reverse-stress slightly decreases the dislocation density (i.e. dislocation motion resistance) and internal stress, and therefore leads to partial activation of PCR (e.g. Fig. S2a).

Longer durations of reverse-loading to a low-stress level might introduce more significant changes to the state of dislocation motion resistance and internal stress. Simple reduction of the stress level during creep loading (e.g. from +230 MPa to +190 MPa) slows down the dislocation generation mechanisms and the microstructure recovery mechanisms might gradually decrease the dislocation density (Fig. 11, index F3). Similarly, recovery mechanisms are expected to decrease the dislocation density during reverse-loading to −190 MPa. For such reverse-loading conditions, relaxation of dislocation bows and pile-ups, and backward movement and annihilation of reversible dislocations, would also contribute to the dislocation density reduction. Therefore, the reverse-loading to −190 MPa is expected to result in a much more significant dislocation density decrease in comparison to that for stress reduction to +190 MPa (Fig. 11, index D3 vs. F3).

Such a reverse-loading condition also affects the internal stress state of the material and might even lead to the development of reverse internal stresses (Fig. 11, index D3) due to formation of new dislocation bows and pile-ups which oppose the previously formed ones. The
The evolution of dislocation density during reverse-loading to large reverse-stresses, e.g. -250 MPa vs. +230 MPa for sample 6 of the ex-situ experiment, denser dislocation pile-ups and more dislocation bows form during the reverse-loading, and therefore develop reverse internal stresses whose magnitudes are larger than those existing before the reverse-loading, e.g. Fig. 13c (sample 3 vs. 6). As mentioned earlier, the GND density is an indication of the extent of incompatibility micro-strains and therefore internal stress. Although the measured GND densities do not provide information about the direction of the internal stress, the increase of GND density during the transition from sample 4 to sample 6 in Fig. 15b can be interpreted as an indication of full relaxation of the pre-existing micro-strains and generation of new incompatibility micro-strains during the 1 h reverse-loading at -300 MPa.

The extent of dislocation density increase is higher for larger ratios of reverse to forward stress magnitudes, e.g. Fig. 11, index F1 (−250/+230 MPa) vs. F4 (−250/+190 MPa). A similar dislocation density increase was also observed during 1 h reverse-loading at −300 MPa from a forward-stress level of 230 MPa for the ex-situ experiment (Figs. 13d and 14c, transition from 4 to 6).

The described evolution of dislocation density and importantly, the change in the direction of the internal stress fields leads to a very fast dislocation movement kinetics upon reloading, in particular for the larger ratio of reverse to forward stress magnitudes. The developed reverse internal stress sums up with the applied forward-stress after the reloading and results in the observation of creep rates even higher than those for the primary creep regime, i.e. very significant PCR, see Fig. 7 (index F2), Fig. 5d and Fig. 5b.

Reloading of the steel after long-duration reverse-loading to large stresses is expected to result in relaxation of the newly generated dislocation bows, pile-ups and incompatibility micro-strains and therefore reduces the internal stress magnitude and GND density. The experimental observations presented in Fig. 10 (transitions from F1 to F2 and F4 to F5), Fig. 13c and Fig. 15b (transition from 6 to 7) consistently indicate the reduction in the magnitude of the internal stress and the density of GNDs upon reloading. The relaxation of the newly formed dislocation bows and pile-ups, and backward movement and annihilation of the newly generated reversible dislocations, also decrease the dislocation density as experimentally observed during both in-situ and ex-situ experiments; transitions from F1 to F2 and F4 to F5 in the in-situ experiment (Fig. 11) and transition from 6 to 7 for the ex-situ study (Figs. 13d and 14c).

The above discussion provides a microstructure-based description for the PCR phenomenon. Consistent with the observations from the stress-varying creep experiments reported in [8] and reviewed in Sec. S1 (Fig. S4), it can be concluded that the activation of PCR upon reloading is more significant for the reverse-loading from smaller forward-stress levels to larger reverse-stresses and for longer durations. Fig. 17 provides a schematic representation for the effect of different unloading/reverse-loading conditions on the evolution of dislocation structure (density, bows, pile-ups, reversible dislocations and GND) and consequently on the extent of PCR extent upon reloading.

5. Conclusions and outlook

In this study, different in-situ and ex-situ examination techniques were employed to investigate the microstructure of a 10% Cr steel during stress-varying creep loading conditions at 600 °C to understand the mechanisms of the primary creep regeneration (PCR) phenomenon. The experimental observations indicated that PCR is governed by the

![Fig. 17. Schematic representation for the effect of unloading/reverse-loading on the evolution of dislocation structure and PCR activation. For simplicity, only edge dislocations are presented.](image)
evolution of internal stress and dislocation movement resistance of the steel during stress-varying creep loading conditions. Different parameters of unloading/reverse-loading profile (e.g., durations and magnitudes) affect the dislocation motion resistance and internal stress state of the steel and consequently, different extents of PCR were observed for different reverse-loading conditions. The following conclusions are drawn in this study:

1. Consideration of the formation/relaxation of dislocation pile-ups (and incompatibility micro-strains), bowing/unbowing of dislocation-lines and the activity of dislocation generation and recovery mechanisms can well explain the experimentally measured evolution of dislocation density and internal stress state during in-situ and ex-situ experiments, and can be used to interpret the observed creep and PCR response of the steel under stress-varying creep loading conditions in the current and previous studies.

2. Inelastic straining results in the formation of internal stresses and an increase in the dislocation density of the material which subsequently slows down the kinetics of dislocations and reduces the deformation rate. Reverse-loading was found to relax the generated internal stresses and reduce the dislocation density of the material, hence again easing the movement of dislocations and resulting in the incidence of a period of high strain rate upon reloading, i.e. PCR.

3. Reverse-loading from smaller forward-stress levels to larger reverse-stresses and for longer durations results in a more significant reduction of the internal stress and even formation of internal stresses which oppose those developed during the forward-loading, i.e. ‘reverse internal stresses’. Upon reloading, the reverse internal stresses superimpose to the applied stress and cause creep rates even higher than those for the primary creep regime.

4. Similarly, dislocation density evolution during reverse-loading is determined by the parameters of the loading profile. Reverse-loading to small magnitudes and for short-durations decreases the dislocation density. However, reverse-loading to magnitudes and for durations greater than those for the forward-loading might increase the dislocation density.

This study presented a mechanistic description for the PCR phenomenon based on observations from a series of short-term stress-varying creep experiments for the 10Cr steel at 600 °C. Understanding the PCR behaviour and the associated microstructure evolutions for long-term cyclic loading and at different temperatures is crucial and serve the basis for the future research.

Data availability

All raw/processed data necessary for reproducing results in this study can be accessed on reasonable request.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jmatdes.2020.104905.

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