The soften mechanisms of martensitic heat resistant steels during hot deformation

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Abstract. Hot deformations for the experimental steels were carried out in the temperature range of 900-1200 °C and the strain rate range 10-3-100 s-1. In this study, the critical condition for dynamic recrystallization (DRX) was determined by a cooperation of the minimum value of −(∂θ/∂σ) and the deviation point of the linear slope of θ-σ curve. From the analysis, it was found that only one slope ratio of critical strain, εc versus critical stress, σc appeared in the 9Cr and 10Cr steels, whilst two different slopes in P92 and NS steels due to the augmentation of auxiliary softening effect of the dynamic strain-induced transformation (DSIT). The results also exhibited that ln(Z) kept an approximate positive linear relationship with ln(εc) below the value of 40, but barely changed beyond it. However, the ln(σc) value increased continuously with the rise of the ln(Z) value at a smaller growth rate after the value of ln(Z) reached 40 than that of the initial part.

1. Introduction
Thermomechanical processing (TMP) has become a crucial tool to advance the structure and enhance the mechanical properties of steels, including the heat resistant steels. During a TMP process, recrystallization plays the main role in the associated microstructure evolution [¹] which mainly determines the deformation characteristics of the steels during the hot working processes. Besides of the recrystallization referring to dynamic recrystallization (DRX), metadynamic recrystallization (MDRX), static recrystallization (SRX), other softening mechanisms may also be strongly auxiliary to have some effects on the deformation behaviour, such as dynamic recovery (DRV), static recovery (SRV) and dynamic strain induced transformation (DSIT). Which softening mechanisms execute the microstructure evolution depends on chemical compositions, prior machining procedures and the deformation parameters.

Actually, each of the softening mechanisms has to satisfy its own prerequisite in order to take place, i.e., the DRX only occurs when a critical strain is reached, and at the same time, the minimum rate of energy dissipating is received [²]. To control the microstructure during deformation, the softening mechanisms should be well analysed. Generally, whether a softening procedure occurs or not during a specific hot deformation, it could be identified through both analysing the stress-strain curves up to the peak and the microstructure observation [³].

The heat resistant steels being used in the ultra-supercritical (USC) power plants not only bear extrusion at high temperature but also experience a creep deformation under a constant stress at high temperature due to the service situation. The creep deformation could also be analysed through the constitutive equation, and the parameters calculated for the hot deformation are suitable for the creep deformation. Therefore, it is of essential importance to study the deformation characteristics and the...
microstructure evolution of the experimental steels during hot deformation. The martensitic heat resistant steels are assumed to be the most competitive candidates for the USC application at 650 °C thanks to their high heat conductivity, nice thermal fatigue resistance and high stress corrosion resistance [6]. Thus, the main objective of this study is to establish the soften mechanisms that took place during the hot deformation, to predict the creep behaviour and the failure mechanism during service.

2. Experimental
This study was conducted on four types of martensitic heat resistant steels. The chemical compositions of the experimental steels are listed in Table 1. The steels were machined into samples of 8 mm in diameter and 12 mm in gauge length.

The rob samples were homogenized in vacuum at 1200 °C for 5 min, and then cooled down to the deformation temperature of 900-1200 °C at a cooling rate of 10 °C/s. After stabilizing holding for 1 min at the deforming temperature, the compression testing samples were deformed to 60% at the strain rate range of 10⁻³-10⁰ s⁻¹. The samples were quenched to room temperature as soon as the compressions were finished.

The microstructure of deformed samples under different compression parameters was observed through an optical microscope, a scanning electron microscope (SEM).

Table 1. Chemical compositions of the experimental steels, wt%

| Steel | C  | Si  | Mn  | Cr  | Mo | W  | V  | Nb | B  | Co | N  |
|-------|----|-----|-----|-----|----|----|----|----|----|----|----|
| NS    | 0.021 | 0.09 | 1.25 | 9.37 | -  | 1.42 | 0.15 | 0.06 | -  | -  | 0.037 |
| P92   | 0.11 | 0.37 | 0.46 | 8.77 | 0.42 | 1.73 | 0.17 | 0.057 | 0.0028 | - | 0.048 |
| 9Cr   | 0.089 | 0.31 | 0.50 | 8.58 | 0.40 | 1.65 | 0.18 | 0.060 | 0.0022 | 1.64 | 0.040 |
| 10Cr  | 0.088 | 0.31 | 0.50 | 10.42 | 0.40 | 2.55 | 0.18 | 0.056 | 0.0022 | 2.19 | 0.058 |

3. Results
3.1 Stress-strain curves critical conditions for DRX
The stress-strain curves of the experimental steels obtained from four different strain rates labelled beside their corresponding curves are shown in Figure 1, in which the critical strain, $\varepsilon_c$, and the peak strain, $\varepsilon_p$, are marked on the curves. All the curves showed an initial work hardening but only a few out of them developed to a clear stress peak, indicating a DRX taking place. It was found that the distinct peaks only appeared in the curves that obtained under high temperature and low strain rate deformation situations (low Z values). With the increase of Z value in the beginning, the peak became broad and the critical strain climb up correspondingly. But when the Z value reached a higher level, the stress curve showed a “flat-top” shape with no peak. This flat-top behaviour of stress curve was traditionally thought to be an indication of no occurrence of DRX and implied that the dynamic recovery was the only softening mechanism operated during the deformation process [4].
3.2 The change of dominate soften mechanisms with different Z parameters

Generally speaking, the increase of Z value prompted the critical strain for the initiation of DRX correspondingly. The theory was confirmed by the approximate linear growth of the third polynomial order fitting curves of $\ln(\varepsilon_c)$ plots at the beginning, as shown in Figure 2 for all the experimental steels. However, the increase of $\ln(\varepsilon_c)$ value slowed down and then levelled off at Z value beyond 40. This resulting hump recorded by Stewart et al. was explained by the segregation of substitutional impurities (P in particular) to the subboundaries of newly nucleated DRX grains [3]. But Nanba acclaimed that the critical strain was expected to slightly depend on the temperature and have no apparent dependence on either the strain rate or the initial grain size for plain carbon steels [5]. That is to say, at low deformation temperature, the higher dislocation density would prompt the initiation of DRX [6] and thus slowed down the increase of the critical strain leading to a barely varying $\ln(\varepsilon_c)$ value.

Figure 1. Equivalent stress-strain curves with indication of $\varepsilon_c$ and $\varepsilon_p$ of 9Cr steel, 10Cr steel, P92 steel and NS steel in sequence.

The existing approach to the onset of DRX was based mainly on energetic considerations. The critical strain for the initiation of DRX can be deduced from either the direct microstructure observation or the analysis of the flow curve. The former is a more complicated and time consuming process.

Figure 2. Curves of $\ln(\varepsilon_p)$ and $\ln(\varepsilon_c)$ values versus $\ln(Z)$ value for the experimental steels
Unlike \( \ln(\varepsilon_c) \) values, the \( \ln(\sigma_c) \) values exhibited two different trends with the increase of \( \ln(Z) \) value, as shown in Figure 3. For the 9Cr and 10Cr steels, the trends of \( \ln(\sigma_c) \) versus \( \ln(Z) \) were similar to that of \( \ln(\varepsilon_c) \) versus \( \ln(Z) \). But as for the NS and P92 steels, the \( \ln(\sigma_c) \) values still kept linearly rising rather than barely varied when the \( \ln(Z) \) values were beyond 40. This phenomenon gave a possible explanation to the trends of \( \ln(\sigma_c) \) curves: the slope ratio of \( \sigma_c \) and \( \varepsilon_c \) got augmented at high \( Z \) value, which ensured the continuous growth of \( \ln(\sigma_c) \) after the \( \ln(Z) \) value of 40.

### 3.3 Microstructure evolution

The evolution of microstructure during hot deformation was closely related to DRV, DRX, MDRX and DSIT in the experimental steels [5]. With the change of \( Z \) value, the steels exhibited various microstructure characteristics, as demonstrated in Figure 4.

An analysis on the stress curve suggested that DRV took place before the critical strain for initiation of DRX under all investigation conditions [6,8]. At high temperature, the DRV got prompted, and the DRX started at a lower strain and proceeded faster [7]. Meanwhile, the fine and medium size particles that reduced the recovery process stabilized the substructure which would slow down the nucleation and pin the grain boundaries diminished to rare [8]. All the above factors gave explanations to the isometric martensitic grains deformed at 1200 ºC/10\(^{-3}\) s\(^{-1}\). However, the deformation temperature was high enough for the nucleated DSIT ferrite to grow at a high speed. That was why almost 50% of the grains were composed by the equiaxed ferrite.

As denoted by other studies [13-15], the decrease of temperature would promote the DSIT nucleation and consequently resulted in relatively finer ferrite grain size. The ferrite grains formed in the experimental steels tended to merge into allotriomorphic rather than equiaxial grains at the relatively low temperature and low strain rate (\( Z \) value around 40). This was because the nucleation size of the DSIT ferrite was small and the growth of DSIT ferrite was sensitive to both temperature and strain rate. But the DSIT ferrite exhibited an elongated shape which implied no DRX occurring in DSIT ferrite, while the martensitic grains showed approximately equiaxed shape indicating that DRX happened in the austenite during deformation.

The MDRX process only involved the growth of previously nucleated dynamic grains and thus enlarged the grain size in a certain extent. Therefore, the MDRX grain size depended only on the \( Z \) parameter [15,16] and was finer with the \( Z \) value increasing. This is why when the \( Z \) value rose up to 60, there was rarely distinct DSIT ferrite left, replaced by a certain volume of ferrite-martensite blend and why the martensitic microstructure of high \( Z \) value showed finer grains compared with that of low \( Z \) value.
Figure 4. Microstructure evolution and precipitation behavior of NS steel during hot deformation.

4. Conclusion

Beneath the diversity of microstructures and hot deformation processes, there are some fundamental characteristics of four types of martensitic heat resistant steels as follows.

- The value of $\ln(\varepsilon_c)$ of all the experimental steels rose linearly with $\ln(Z)$ increasing at the beginning but this acceleration slowed down and levelled off at high $Z$ conditions. The $\ln(\sigma_c)$ curve changed with $\ln(Z)$ in a similar manner to the trend of $\ln(\varepsilon_c)$ versus $\ln(Z)$ for 9Cr and 10Cr steels while it still kept a smaller linear growth when $\ln(Z)$ value beyond 40 for NS and P92 steels.
- The high temperature provoked the starts of DRX at lower strain and prompted its proceeding, leading to equiaxial grains. DSIT ferrite was fond to grow along the prior austenite grain boundaries, which formed the stripe structure. The MDRX and DSIT processes were responsible for the ferrite-martensite blend structure.

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