

## The effect of processing parameters on the dynamic recrystallisation behaviour of API-X70 pipeline steel

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### ABSTRACT

The effect of deformation temperature and strain rate on the dynamic recrystallisation (DRX) behaviour of X70 pipeline steel was investigated. DRX parameters such as the critical and peak stresses and strains as well as the deformation activation energy were determined in the temperature range between 925 °C and 1125 °C for strain rates of 0.1, 1 and 5 s<sup>-1</sup>. The relationship between the peak stresses and strains with the Zener–Hollomon parameter was determined. The dynamically recrystallised volume fraction was computed as a function of the different temperatures and strain rates. The APRGE software was applied for the first time on electron back-scattering diffraction data of dynamically recrystallised microstructures in order to reconstruct the prior austenite from the as-quenched martensite phase. The dynamically recrystallised flow stress curves and microstructure were also predicted using cellular automata modelling. The results show an earlier onset of DRX with a decrease in strain rate or an increase in deformation temperature. The dynamically recrystallised grain size is also found to decrease with an increase in strain rate and a lowering of deformation temperature.

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### 1. Introduction

The increased demand for oil and gas transportation has encouraged the development of specialised and cost-effective pipeline steels with improved mechanical strength, toughness and corrosion resistance and increased ease of weldability. In this regard, the American Petroleum Institute (API) X70 grade is one of the more commonly used pipeline steels. Research into this particular steel has largely focused on improving its mechanical properties by optimising the thermo-mechanical processing (TMP) history and/or chemical composition. Thus, while some papers characterised the various microstructural phenomena that evolve during TMP [1–4], other studies have concentrated on improving the chemical composition of API-X70 which helps in reducing the deleterious effects of precipitate segregation during continuous casting [5–7].

TMP simulations are typically undertaken in order to achieve a better understanding and control of the different microstructure–mechanical property phenomena; for example—the complex interplay between dynamic softening (recovery and/or recrystallisation)

and work (or strain) hardening. While dynamic recovery involves processes that reduce the overall stored energy of a material through the localised rearrangement of dislocations, dynamic recrystallisation (DRX) is the creation of low dislocation density nuclei at the originally deformed grain boundaries. The subsequent growth of these nuclei results in a final microstructure with a homogenous grain size when equilibrium conditions are reached [8]. DRX is crucial in dictating the final microstructure and mechanical properties of API-X70 as its prevalence during TMP causes significant grain refinement [9]; which in turn, leads to an increase in the strength and toughness values of the steel. The latter two mechanical properties are essential requirements for pipeline applications [10].

Consequently, a full understanding of the DRX processes that occur during TMP is essential when designing the hot deformation processing schedule for pipeline steels. DRX can be studied in two ways: (i) by microstructure investigation or, (ii) by flow stress analysis. As opposed to microstructure-based investigations which require a significant time investment in order to verify the occurrence of DRX [11], the flow curve analysis method is easier and faster. Here, DRX is generally initiated at a critical strain ( $\epsilon_c$ ) value just before the peak stress ( $\sigma_p$ ) is reached [11,12] and extends into the steady state region of the flow curve. In order to extract the critical and peak ( $\epsilon_p$ ) strains, different empirical equations have been developed over the years that take advantage of the relationship between the deformation

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temperature ( $T$ ), the strain rate ( $\dot{\epsilon}$ ) and the Zener–Hollomon parameter ( $Z$ ) as follows [9,13]:

$$Z = \dot{\epsilon} \exp\left(\frac{Q}{RT}\right) \quad (1)$$

where,  $Q$  is the deformation activation energy and  $R$  is the gas constant ( $=8.314 \text{ JK}^{-1} \text{ mol}^{-1}$ ). The critical drawbacks of the above mathematical constructs of DRX is that: (i) they are essentially fitting functions that require experimental data points as input and, (ii) they are unable to predict the morphology (and phase constituents) of the final microstructure.

In this regard, newer analytical and computational modelling methods have been developed that: (i) predict the deformation behaviour of different steels during TMP and, (ii) return the final microstructure upon completion of the TMP cycle. To this end, while the vertex [14], Potts-type Monte Carlo [15] and phase field [16] models are successful in predicting microstructure evolution, the cellular automata (CA) model is more readily calibrated to real-world time and length scales. The CA method inherently returns a time and space dependent description of recrystallisation and provides a visible evolution of the virtual microstructure throughout the TMP process [17]. Since static recrystallisation is less complicated to model compared to DRX, a greater number of CA studies have been devoted to modelling static recrystallisation [18–20] instead of DRX [21].

With respect to the above outlook, the present study investigates the effect of the various deformation parameters on the DRX behaviour of API-X70 steel. The parameters for DRX were extracted using the flow stress analysis technique. Lastly, a CA model was developed to simulate the final DRX microstructure and flow curves for a range of temperatures and strain rates.

## 2. Experimental procedure

A commercial API-X70 pipeline steel with the nominal chemical composition Fe-0.081C-1.2Mn-0.27Si-0.10Mo-0.04Al-0.06Nb-0.003V-0.021Ti wt% was obtained from BlueScope Steel Ltd. in the form of an as-cast slab. The as-cast slab was cut into  $20 \times 15 \times 10 \text{ mm}^3$  specimens along the original casting, transverse and thickness directions, respectively.

Using a Gleeble 3500 thermo-mechanical simulator, the non-recrystallisation (or the temperature below which recrystallisation does not occur,  $T_{nr}$ ) and the austenite-to-ferrite transformation ( $A_{F3}$ ) temperatures were estimated. This was accomplished by an initial multi-pass plane strain compression test between 1100 and 675 °C wherein a strain of 0.1 was imparted at each 25 °C temperature interval<sup>2</sup>.

As illustrated in Fig. 1, the DRX behaviour was also studied via a set of plane strain compression tests. Here the samples were: (i) reheated at  $5 \text{ }^\circ\text{C s}^{-1}$  to a reheating (RH) temperature ( $T_{RH}$ ) of 1250 °C, (ii) isothermally held for 300 s, (iii) cooled at  $1 \text{ }^\circ\text{C s}^{-1}$  to 1100 °C at which a first roughing (R) strain of 0.35 was imparted and then, (iv) further cooled at  $1 \text{ }^\circ\text{C s}^{-1}$  to finishing (F) temperatures of 925, 975, 1025, 1075 and 1125 °C at which a second strain of 1.2 was imparted at strain rates of 0.1, 1 and  $5 \text{ s}^{-1}$  followed by, (v) immediate water quenching. The resultant flow stress curves were analysed to evaluate the DRX behaviour of the API-X70 pipeline steel.

The RD-ND cross-sections were ground up to 1  $\mu\text{m}$  diamond, fine polished using colloidal silica and then lightly etched at 68 °C using Modified Winstead's reagent (80 ml saturated picric

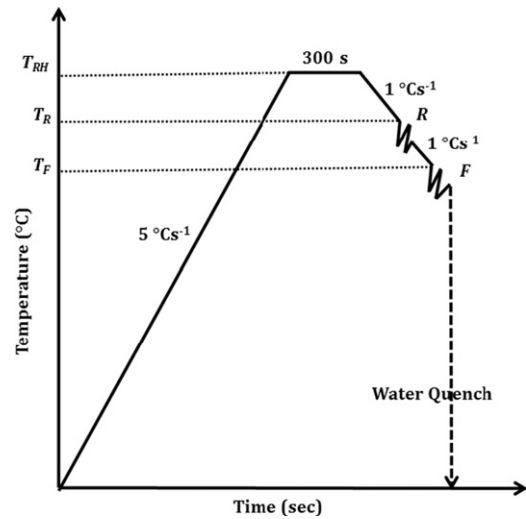


Fig. 1. A schematic of the thermo-mechanical processing schedule.  $T_{RH}$ ,  $T_R$  and  $T_F$  are the reheating, roughing and finishing temperatures, respectively.

acid+8 drops hydrochloric acid+8 drops detergent). Thereafter, the as-quenched martensitic microstructure of selected samples was characterised by Electron Back-Scattering Diffraction (EBSD). The maps were obtained from the centre of the specimen cross-section using a JEOL JSM-7001F field emission gun-scanning electron microscope fitted with a Nordlys-II(S) detector and the Oxford Instruments AZtec software suite operating at 15 kV and 2.8 nA. Depending on the structure size of the martensite matrix, the step size for EBSD mapping was varied between 0.1 to 0.25  $\mu\text{m}$  such that at least 6 pixels constitute the smallest relevant feature.

Initial post-processing of data was carried out using the Oxford Instruments Channel-5 software package. Low-angle boundaries (LAGBs) comprise misorientations between 2–15° while high-angle boundaries (HAGBs) extend from 15° onwards. In many samples, the standard metallographic etching procedure for prior austenite determination from an as-quenched martensite microstructure was unsuccessful due to the magnitude of the imparted strain. Consequently, the prior austenite grains were reconstructed from the EBSD maps via the ARPGE software package [22]. The ARPGE software algorithm is based on the general crystallographic theory of austenite to martensite transformation [23] and uses the orientation relationship between austenite and martensite given in Ref. [24] wherein  $(111)_{fcc}$  is nearly parallel to  $(011)_{bcc}$  within an angular deviation of  $\sim \pm 0.5^\circ$  while  $[\bar{1}01]_{fcc}$  deviates by  $\sim 2.5^\circ \pm 1^\circ$  from  $[\bar{1}\bar{1}1]_{bcc}$ .

## 3. Results and discussion

### 3.1. Determination of the non-recrystallisation ( $T_{nr}$ ) and transformation ( $A_{F3}$ ) temperatures

In order to estimate the non-recrystallisation ( $T_{nr}$ ) and transformation ( $A_{F3}$ ) temperatures, the mean flow stress (MFS) was calculated from the initial multi-pass plane strain compression test as the area under the stress–strain curve for each deformation pass [25,26].

As shown in Fig. 2, if the MFS is plotted against the inverse of the deformation temperature (in K), the resultant graph can be sub-divided into four different regions. Region I extends between 1100 and 975 °C and is the area in which the temperature is mostly above  $T_{nr}$  ( $=975 \text{ }^\circ\text{C}$ ). Within this region, austenite is

<sup>2</sup> The original casting, transverse and normal directions of the as-cast slab correspond to the plane strain (or rolling (RD)), transverse (TD) and normal (ND) directions of the specimen during plane strain compression.

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