Microstructure characterisation of an X70 grade pipeline steel and its dislocation development during Bauschinger testing

T S Pereira, Y L Chiu and I P Jones
School of Metallurgy and Materials, University of Birmingham, Edgbaston, B15 2TT, UK.
E-mail: TXS890@bham.ac.uk

Abstract. The microstructure of an X70 alloy and its dislocation development during one cycle of compression/tension strain has been analysed. The microstructure of the as received material and the dislocation arrangements before, during and after the Bauschinger test have been characterised. Results for this particular alloy show no evidence of grain boundary dislocation pile-ups or of precipitates pinning dislocations. A comparison between the dislocation arrangement in the as received, compressed and compressed-tensioned samples shows the formation of small dislocation cells, implying that the conception of the Bauschinger effect comes from dislocation-dislocation interactions.

1. Introduction
Due to their versatility, high strength low alloy steels have been widely used for the past few decades in structural, automotive and pipeline applications. Their high strength, combined with toughness and corrosion resistance give these alloys a great advantage over plain carbon steels without an extensive cost rise. Extensive research has been carried out in the past few decades to keep up with these industries requirements and this research field will continue to evolve as industries develop.

Pipeline steels are directly linked to the Bauschinger effect due to the UOE pipe making process. During this process, the wall of the pipe goes through successive steps of compression and tension plastic deformation but, depending on its position, the wall receives a different number of steps, resulting in a strain gradient in its transverse direction. As a consequence, after the UOE process, one side of the pipe will have work hardened and the opposite side will be affected by the Bauschinger effect, thus, presenting a lower yield stress.

The mechanisms that explain the Bauschinger effect are likely to be a combination of the Orowan mechanism and the long range internal stress [1]. According to Sleeswyk et al. [2], during work-hardening, gliding dislocations interact with defects/obstacles and, as they are subjected to stress in the opposite direction, these dislocations overcome the same defects/obstacles more easily. In addition, Mughrabi [3] suggested that the long range internal stress in heterogeneous dislocation arrangements contributed to the Bauschinger effect considering that dislocation walls and cell interiors yield at different stresses, so that, when unloaded, one stresses the other, creating a stress field individual to each dislocation cell, even though the average stress is null.

The aim of this research was to explore the dislocation evolution of a commercial API X70 pipeline steel during Bauschinger testing. The as received material was analysed using TEM and compared to the compressed and compressed-tensioned samples.
2. Experimental methods

2.1. Material

The as-received samples were extracted from a rolled API X70 plate. Table 1 shows the chemical composition of the steel plate.

| C   | Si  | Mn   | P    | S    | Cr  | Mo   | Ni   | Nb   | V    | Ti   | Cu   | B    | Al   |
|-----|-----|------|------|------|-----|------|------|------|------|------|------|------|------|
| 0.042 | 0.23 | 1.53 | 0.0088 | 0.0025 | 0.14 | 0.19 | 0.062 | 0.0039 | 0.014 | 0.16 | 0.0002 | 0.03 |

2.2. SEM and TEM analysis

Samples for SEM analysis were ground and polished before being etched with 3% nitric acid in ethanol for 10 seconds in order to reveal its microstructure. These samples were analysed using a JEOL 7000 operating at 20kV. EDM was used to cut 300 \( \mu m \) slices, from which 3 mm TEM discs were extracted using the same technique. The discs were then thinned to 50 \( \mu m \) before being electropolished using a Tenupol machine at 25 V, in a solution containing 95% ethanol and 5% perchloric acid at -30 °C. A JEOL 2100 operating at 200kV was used to analyse TEM samples.

2.3. Mechanical tests

An ESH Servo Hydraulic Universal Testing machine with a 200 kN load cell was used to perform the compression and Bauschinger tests at a strain rate of 1.6x10\(^{-4}\) s\(^{-1}\). Rectangular test samples of 4x4x8 mm were compressed to 2% plastic strain to simulate the “U” step of the UOE process.

Bauschinger tests consisted of a 2 % plastic strain under compression, followed by a 2 % plastic strain under tension. Cylindrical samples of 3.5 mm diameter and 13 mm gauge length were used for the Bauschinger tests.

The parameters measured to quantify the Bauschinger effect were the stress parameters at 0.2 and 0.5 % strain (\( \beta_{\sigma r_{0.2}} \) and \( \beta_{\sigma r_{0.5}} \) respectively), explained by equation 1 and 2, where \( \sigma_p \) is the value for maximum forward-stress, \( \sigma r_{0.2} \) and \( \sigma r_{0.5} \) are the values for stress in the opposite direction at 0.2 and 0.5 % strain.

\[ \beta_{\sigma r_{0.2}} = (\sigma_p - \sigma r_{0.2})/(\sigma p)^{-1} \]  
\[ \beta_{\sigma r_{0.5}} = (\sigma_p - \sigma r_{0.5})/(\sigma p)^{-1} \]  

3. Results and discussion

3.1. Microstructure

The API X70 alloy presented a predominantly ferritic structure with the average grain size of 2.8 \( \mu m \), and 1.7 and 2.3 % area fractions of perlite and austenite phases respectively. SEM images showed a small amount (0.0873 % area fraction) of relatively large precipitates, with EDX data revealing them to be Ti(C, N). The diameter of these precipitates ranges from 50 to 100 \( \mu m \).

Titanium-type particles start to precipitate at high temperatures, near the liquid-austenite transformation temperature. Therefore, they act mainly as ferrite grain size controllers, refining the microstructure of the plates, but not playing an important role in precipitate strengthening [4].

EDX data from TEM revealed a second type of precipitate, Nb(C, N), of size 25 \( \mu m \), with a much smaller area fraction. No evidence of vanadium type precipitates was found. Because niobium and vanadium particles precipitate at lower temperatures, they tend to be smaller than Ti(C,N)and are able to act as barriers to the movement of dislocations. However, with a small area fraction of the niobium particles and the absence of vanadium particles, very little or no precipitate strengthening is expected.
for this particular alloy and, therefore, none of the Bauschinger effect can be attributed to dislocation-particle interactions.

3.2. Bauschinger test

The results of the Bauschinger test curve are shown in figure 1.

![Figure 1](image)

The Bauschinger parameters $\beta_{0.2}$ and $\beta_{0.5}$ were calculated using equations 1 and 2 from the parameters $\sigma_p$, $\sigma_{0.2}$ and $\sigma_{0.5}$ from the Bauschinger test curve, which values are shown in table 2.

| $\sigma_p$ | $\sigma_{0.2}$ | $\sigma_{0.5}$ | $\beta_{0.2}$ | $\beta_{0.5}$ |
|-----------|----------------|----------------|---------------|---------------|
| 561 MPa   | 405 MPa        | 480 MPa        | 0.278         | 0.144         |

From the tension curve of the Bauschinger test, it is noticeable that plastic deformation starts as early as half of the compression yield stress value and the flow stress changes drastically. However, after 2% tension strain, the stress value has not returned to its maximum forward stress level.

3.3. Dislocation analysis

The typical dislocation microstructure of the as-received, 2 % compressed and Bauschinger tested samples are shown in figure 2. The dislocation density analysis shows that the as-received specimens have a dislocation density of $4.7 \times 10^8$ cm$^{-2}$, the compressed specimens $7.8 \times 10^9$ cm$^{-2}$ and the Bauschinger tested specimens $6.2 \times 10^9$ cm$^{-2}$.

![Figure 2](image)

The dislocation structure of the as-received samples showed a predominantly straight dislocation lines arrangement, with homogeneous spacing in between lines. After 2% compression, the dislocation density increased by over one order of magnitude, starting to present high dislocation density walls but not well defined cells. The Bauschinger tested samples showed a similar dislocation density to the
compressed samples, but the cell structure was much better defined, with the interior of the sub-grains almost dislocation free.

Values for the Bauschinger parameter $\beta_{0.2}$ after 2% plastic strain have been found of up to 0.756 due to the presence of precipitates [5], which, compared to 0.278 of the present X70 alloy, is much more significant. However, as previously mentioned, there was no evidence of dislocation pile-ups at grain boundaries or precipitates pinning dislocations, thus, the Bauschinger effect generated by dislocation-dislocation interactions only may not be neglected.

4. Conclusions
The main findings of this study are:
- The microstructure of the as-received X70 alloy is predominantly ferritic with a small fraction of perlite and austenite grains. The particles identified are the titanium and niobium-type precipitates, although they are either too large or occupy a considerable small area fraction in order to produce any pronounce precipitate strengthening. Vanadium-type precipitates are not present;
- The Bauschinger parameters $\beta_{0.2}$ and $\beta_{0.5}$ are calculated to be 0.278 and 0.144 respectively. After 2% reverse strain, the stress value did not return to the maximum value of forward stress;
- The dislocation density increases after compression but there isn’t any significant change from the compressed state to the Bauschinger tested state. However, there is a significant change in the dislocation structure of the specimens when comparing the as-received, compressed and Bauschinger tested samples. High dislocation density walls are formed after 2% compression, which are transformed into well defined dislocation cells after the Bauschinger test;

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