Mechanical scale-breaking of AISI430 stainless steel

SJ Clowe, M Krzyzanowski, JH Beynon
IMMPETUS, University of Sheffield, Mappin Street, Sheffield, S1 3JD, UK.

ABSTRACT
The optimisation of the industrial scale-breaking process for ferritic stainless steels would lead to a direct improvement in the pickling times. The defect retention of AISI430 ferritic stainless steel, in relation to comparable austenitic grades, means that conventional mechanical scale-breaking techniques such as shot-blasting can leave unacceptable levels of damage in the metal surface. Although this can often be masked by high apparent surface quality in the intermediate sheet, final forming can reveal serious defects.

Division of the aspects of roll-breaking into bend and tension elements led to two areas of laboratory investigation. Bend tests showed the effects of strain on both convex and concave surfaces, and suggested a mechanism for oxide failure. Tension tests illustrated that the failure mechanisms apply over the same range of strain, regardless of the loading state.

Quantitative analysis of the spacings within the crack arrays and the amount of material spalled has led to important conclusions regarding the level of strain which must be imposed on the scale to obtain a minimum mean crack spacing, or for a significant amount of spalling to occur.

1. INTRODUCTION

The surface quality and finish of stainless steels is one of their most important marketable factors. The complete removal of oxide scale, formed during high temperature process steps, is therefore essential. To reduce both process costs and the environmental impact of acid pickling, the most effective method of scale removal is to precede the chemical stage with mechanical fracture of the oxide, providing paths to the chromium depletion layer of the steel where the acid works to undermine the scale1.

Empirical information from the material provider, Outokumpu Stainless, indicated that the ferritic grades are soft enough to retain surface damage caused by shot blasting, with post-processing defects, such as spangling, causing the method to be rejected. In this case roll-breaking is the preferred method of scale fracture, although levels of spalling observed suggest significant behavioural differences to that reported on other steel grades2-4.

The laboratory project was designed to measure the effects of strip tension and bend radius as the main variables during roll-breaking, although the application of simultaneous bend and tension was not possible in the laboratory.

2. EXPERIMENTAL PROCEDURE

2.1 Material
The work has examined AISI430 grade strip exclusively; the chemical composition for the two samples are given in Table 1. Industrially scale-broken sections were used as a datum, with post-anneal hot band sections providing the ideal test material as the oxide was as would be received at the descaling line.
Table 1. Chemical composition of AISI430 strip samples (weight %).

<table>
<thead>
<tr>
<th>Coil</th>
<th>Fe</th>
<th>Cr</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>&gt;80</td>
<td>16.56</td>
<td>0.046</td>
<td>0.46</td>
<td>0.265</td>
<td>0.31</td>
</tr>
<tr>
<td>B</td>
<td>&gt;80</td>
<td>16.55</td>
<td>0.049</td>
<td>0.45</td>
<td>0.282</td>
<td>0.19</td>
</tr>
</tbody>
</table>

All test samples took the form of 25 mm wide strips machined from the as-received material in such a manner as to have the rolling direction along the long axis, varying in length from 150 to 250 mm. Testing was carried out at ambient temperature and humidity. Following deformation, strips from each testing type were sectioned to provide through-thickness sections and microscope-ready planar samples. Sections were mounted in conductive bakelite and prepared to a mirror finish using a succession of silicon carbide papers, diamond pastes and colloidal silica. Microscopic examination of the oxide was carried out in all cases using either a Camscan II or JEOL 6400 scanning electron microscope (SEM), both equipped with energy dispersive X-ray spectroscopy (EDS).

2.2 Bend Tests

Previous work on the behaviour of oxide scale on mild steel rod\(^3\) utilised both free bend and bending round a former to produce the required profile. To enable the separate study of both the concave and convex surfaces of the bent AISI430 material, three point bend tests were employed to introduce a reproducible bend profile with a quantifiable load, where only a small area of the surface would be influenced by contact with the anvils. The tests were conducted such that the bend axis was perpendicular to the original roll direction, as would be the case in an industrial roll-breaker.

The bend profile imposed by the three-point loading provided a variable radius of curvature, symmetrical about the load anvil. Radii at points of interest were determined by superposition of computer-generated contours and then used to determine the surface strain according to Equation 1.

\[
\varepsilon_a = -\varepsilon_b = \ln \left( \frac{2R}{h} + 1 \right)^{-1} + 1
\]  

(1)

where \(\varepsilon_a\) = strain due to circumferential stretch on the convex surface  
\(\varepsilon_b\) = strain due to compression on the concave surface  
R = bend radius  
h = strip thickness

2.3 Tension Tests

Uniaxial tension tests, where the strips were loaded parallel to the original rolling direction, were used to provide a comparison between the two main variables of roll-breaking, bending and tension. Tension tests were also used to expand the range of strain exerted on the oxide, since the geometry of the three-point bend set-up restricts the levels that could be reached by bending alone.

2.4 Quantitative Analysis

Quantitative analysis of the crack spacing for a given radius of curvature, or value of tensile strain, used a lineal intercept method; where features captured by SEM were measured using
KSRun 3.0 image analysis software and the mean spacing calculated according to Higginson and Sellars\textsuperscript{5}. Spalling was also measured from SEM images, using the image analysis software to calculate the operator-selected regions of a field of view as a percentage of the whole.

3. RESULTS AND DISCUSSION

3.1 As-received Material

The hot band sections, taken post-anneal, were examined using scanning electron microscopy to characterise the as-received surface and reveal any cracking present in the scale layer as a result of industrial processing or handling. Figure 1, taken in secondary electron image mode, illustrates the complex nature of an industrial sample though there are few cracks visible.

![Figure 1. As-received post-anneal hot band surface (SEI).](image1)

A through thickness section reveals the inhomogeneous nature of the scale layer, Figure 2. Energy dispersive spectroscopy shows the inhomogeneity is due to the differing concentrations of iron and chromium within the phases, with a chromium-rich layer at the steel-oxide interface, and an iron-rich mixed oxide layer at the oxide-atmosphere interface.

![Figure 2. Through-thickness section of the oxide layer (BEI), with elemental distribution maps for iron and chromium.](image2)

Post scale-break planar sections received from industry displayed a visible fracture network in the oxide layer, Figure 3. Cracks run perpendicular to the rolling direction (RD), marked on the micrograph as a line, and parallel to the axis of the bend imposed by the rolls. At this stage in the processing, the strip has undergone a reverse bend in the roll-breaker, under sufficient tension to maintain roll-strip contact.

![Figure 3. Oxide surface after industrial scale break (BEI). RD marked by a line.](image3)
Through thickness sections reveal that the cracks seen on the outer surface, extend through both oxide types to the steel surface. However two additional crack morphologies can be seen in Figure 4, with the chromium-rich layer presenting finely spaced cracks perpendicular to the steel surface which extend only as far as the oxide phase boundary, with the boundary itself providing the third failure mode as the different oxide types begin to delaminate.

### 3.2 Laboratory Tests

**Three-point Bending**

Scanning electron images of the convex surface of bend test samples reveal an array of cracks similar to those seen on industrial samples, Figure 5, parallel to the bend axis where the axis is marked with a line. The array extends across the sample width, with the spacing consistent for a given radius of curvature and hence strain. The concave surface of the bend tests exhibits a similar morphology, where the crack direction is parallel to the bend axis, although the range of spacings is slightly larger for a given radius of curvature.

The main difference between the two faces is the evolution of the spalling process. In Figure 6 at 0.5 % strain, the oxide scale has cracked and, due to volume constraints, has begun to overlap its nearest neighbour. Figure 7 shows the resulting mixed cracked/spalled structure at 1 % strain where some areas of lifted or overlapped scale have failed at the hinge and become detached from the surface.

---

**Figure 4.** Through thickness section of the as-received scale-broken oxide.

**Figure 5.** Convex surface after three-point bending. Bend axis indicated by a line.

**Figure 6.** Overlapping oxide scale caused by volume constraints on the concave surface of a bend test at 0.5 % strain.

**Figure 7.** Mixed structure of cracks and spalled areas formed on the concave surface of a three-point bend test at 1 % strain.
Uniaxial tension produces a crack morphology that is much the same as that observed in the three point bend tests. Deformation results in a crack network perpendicular to the original rolling direction which in all tension tests was perpendicular to the load axis, marked on the micrographs with a line. Again, crack spacings are consistent for a specific strain, 2 % in the case of Figure 8. Applying higher strains than can be achieved using the bending rig, in this case 10 %, the tensile tests also revealed that spalling occurs when no volume constraints are present, see Figure 9. The proposed spalling mechanism during tension involves the failure of the oxide-oxide interface, allowing the outer phase to delaminate, in agreement with Trull and as seen in the as received material.

The loss of material from the surface of the scale allows observation of the underlying structure, as seen previously in cross-section in the industrial samples.

3.3 Quantitative Analysis

As the morphologies observed on test samples are directly comparable, regardless of whether the sample has been bent or put in tension, it can be concluded that scale fracture will occur when a sufficient surface strain has been reached, irrespective of the loading state. Quantitative analysis is therefore used to relate the surface strain to the crack spacing and the amount of spalled material it produces, with the aim of providing design variables for the industrial process.
Crack Spacing
As the complete descaling of the steel surface is dependant upon the pickling liquor penetrating the scale layer, increasing the number of cracks through the oxide will increase the pickling efficiency.

Figure 11 illustrates the crack spacing decay caused by increasing surface strain for both bend and tension samples. The graph illustrates a law of diminishing returns, where the crack spacing asymptotes out above a strain of 3 %, reaching a limiting range of 10 – 20 µm. As tension alone would be impractical for industrial scale-breaking, and the roll radius of the roll-breaker has finite practical size, the establishment of a minimum effective strain range has important practical implications.

![Crack Spacing Graph](image)

Figure 11. Effect of increasing surface strain on the mean crack spacing observed on the scale surface.

Spalling
The quantitative analysis of the amounts of material spalled from the surface has concentrated on loss caused solely by tensile strain rather than including cases where loss of material is also caused by volume constraints, as seen in the bends tests. Figure 12 illustrates the effect of increasing strain on the mean percentage material spalled from the surface of tension samples.

The significant strain range in this case is 4 – 5 %, where the percentage of material spalled increases seven-fold. The suggested failure mechanism at this stage is shear at the oxide-oxide phase boundary, allowing the delamination of the mixed oxide layer and the exposure of the dense chromium-rich layer which has a higher crack population, as observed previously. As the loss of the outer oxide layer results in the reduction of the acid path, the increase in spalling should increase the efficiency of the pickling process.
Figure 12. Effect of increasing tensile strain on the percentage of material spalled.

Although crack formation and spalling have previously been analysed as separate phenomena, with a significant strain range apparent in both cases due to either the law of diminishing returns or the mechanism activation, it is the comparison of the mean data for tension tests that provides an overall picture of the events at the strip surface. Plotting the crack spacing values and the spall percentage for tension tests on the same graph (Figure 13) illustrates that the separation of cracks in the tension samples drops significantly over a strain range of 4 – 5 %, exactly the same range as the increase in spalled material. Given that the loading state does not influence the minimum crack spacing range, it is therefore reasonable to state that a total surface strain of approximately 5 % will produce the optimum levels of cracking and spalling required to facilitate subsequent acid penetration and hence descaling.

Figure 13. Comparison of the effect of increasing strain on the mean crack spacing values and percentage of material spalled for tension test samples.
The crack spacing series shown in Figure 13 does not exhibit the smooth decay seen for bent samples in Figure 11, where the sudden decrease in crack separation is less visible. It is thought that the crack spacings recorded appear to increase between 1 and 4 % strain as a result of the extension of the substrate, with the crack faces moving further apart before a second series of cracks form at 5 %. Between 5 and 15 % strain, the pattern is repeated. Image analysis software is being used to investigate this hypothesis.

4. CONCLUSIONS

- Microscopic examination of laboratory samples illustrate that surface strain, imposed by either bending or uniaxial tension, generates reproducible parallel crack networks of consistent spacing in the oxides of AISI430 ferritic stainless steel.
- Quantitative analysis demonstrates that mean crack spacing values decay to a lower limit of 10 – 20 μm above 3 % strain.
- The amount of material spalled from the scale surface has been shown to increase significantly at strains of 5 % or more.
- Comparison of the crack spacing and spall percentage data for tension tests have indicated that the significant strain required to produce the optimal combination of path-reducing defects in the oxide scale is of the order of 5 %.

Acknowledgements
The authors would like to acknowledge funding from the Engineering and Physical Sciences Research Council and IMMPETUS, and material from Outokumpu Stainless.

References
1. ROWLANDS, DP. “MSI Pickling and Passivating”, www.msi.co.za.