

Phase Differentiation in Stainless Steel Oxide Scales

Introduction

Stainless steels have applications in areas where surface appearance is an extremely important property. These may include areas such as kitchen work tops, cupboard facings and cladding for building exteriors and interiors. As stainless steel is mainly used for its appearance, any effect of oxidation must be reduced so that it will not be detrimental to the appearance of the final product. Unfortunately, current scale removal processes may not be sufficient to remove all oxides present after slab reheat. These remnants of oxide scale can be pressed into the surface of the material during rolling and cause unsightly defects in the surface, which is unacceptable to both the manufacturer and the purchaser. This study aims to investigate the scales produced on stainless steels during industrial forming. Electron Backscatter Diffraction (EBSD) and Chemical Indexing Scan (ChI-Scan), which combines the structural information measured with EBSD with the complete spectrum chemical information measured with Energy Dispersive Spectroscopy (EDS), has been used to study the microstructure of the scales, as well as possible phase distributions.

Sample Preparation

Samples of 316L stainless steel were oxidized in a box furnace in laboratory air at 1200°C for 4 hours. After oxidation, they were mounted in edge retaining conductive bakelite, metallographically prepared using grades of silicon carbide paper from 80 grit to 2400 grit, 6 and 1 µm diamond polish and then finally polished using colloidal silica solution for at least 25 minutes. It is important to note that the aim of the preparation was to get the ceramic oxide scale ready to be analyzed. The oxide scale is brittle and requires more delicate preparation than a metal sample. The Scanning Electron Microscope (SEM) used to examine samples was a Leo 1530 VP field emission gun with EDAX Energy Dispersive Spectroscopy (EDS) and EBSD systems located at the Loughborough Materials Characterisation Centre. The working distances and operating voltages were varied to give optimum conditions for EDS and EBSD imaging. Figure 1 is an image of a scale taken in backscatter imaging mode.

Phase Identification using ChI-Scan

The backscatter image (Figure 1) of a scale grown on 316L stainless steel after 4 hours at 1200°C shows that the scale has three visible layers. The lowest layer appears porous and fine grained. The middle layer shows a larger grain size, with damage evident from polishing. The upper layer appears less damaged than the middle layer. Although such an image provides some information about the microstructure, an EBSD image quality (IQ) map (Figure 2) provides substantially more detail about the grain structure.

Figure 2 is showing the same scale as that shown in Figure 1. This image clearly indicates that the oxide layer nearest the

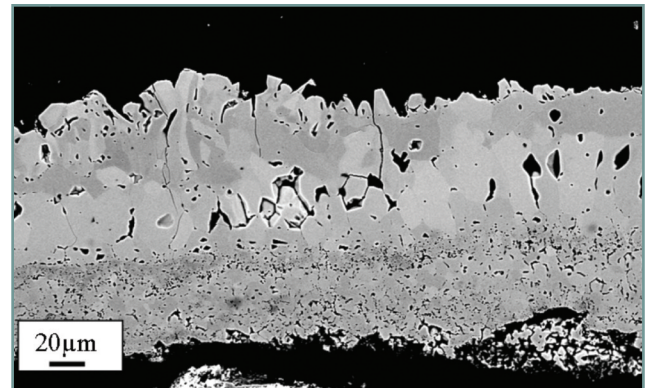


Figure 1. Backscatter electron micrograph of an oxide scale formed on 316L stainless steel after 4 hours at 1200°C.

substrate (layer 1) is very fine grained, the middle layer (layer 2) has larger equiaxed grains and the top layer (layer 3) appears more disordered with some long thin grains at the top edge. However, it is difficult to precisely distinguish between the three layers. Combining this IQ map with EDS data collected simultaneously during the EBSD scan provides information (Figure 3) that is helpful in distinguishing between the layers, and reveals the spatial distribution of phases both between and within the layers.

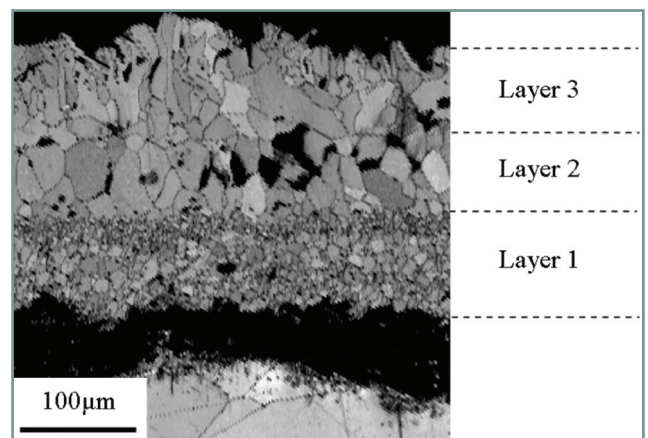


Figure 2. EBSD IQ map of an oxide scale formed on 316L stainless steel after 4 hours at 1200°C in air and cooled in air at room temperature.

From the EDS information shown in Figure 3, it is clear that four discrete chemical phases are contained within the three layers. Figure 3 (a) is a nickel map overlaid on an IQ map. This figure shows that nickel is distributed homogeneously throughout the equiaxed grains of layer 2. However, this is the only layer which shows this type of distribution. Many grains in layer 1 are rich in nickel, but there are also many which contain very little nickel. In the upper layer (layer 3), most of the grains have very low nickel content but there are a number of grains along the upper edge of the scale which show high nickel content. The chromium map (Figure 3 (b)) indicates that only Layer 1 is rich in chromium, while the rest of the scale shows very little chromium. This layer also shows some grains towards the top and bottom of the layer to be higher in nickel content. The iron map (Figure 3 (c)) shows the iron content to increase towards the top edge of the scale. The grains which show a high nickel content on the top edge of the scale also show a low iron content in relation to the rest of layer 3.

For each of the four discrete chemical phases identified in the EDS maps, a corresponding crystallographic phase was selected from a database of diffraction patterns using the chemical composition and EBSD indexing as search criteria. The four discrete phases were identified as hematite and three spinels – Chromium Oxide, Nickel Iron Oxide and Chromium Iron Oxide. The crystallographic structure information (EBSD) and chemical composition data (EDS) were used together (ChI-Scan) to differentiate these phases during a rescan of the simultaneously collected EDS-EBSD data. The austenitic stainless steel underlying the scale was also considered in the rescan.

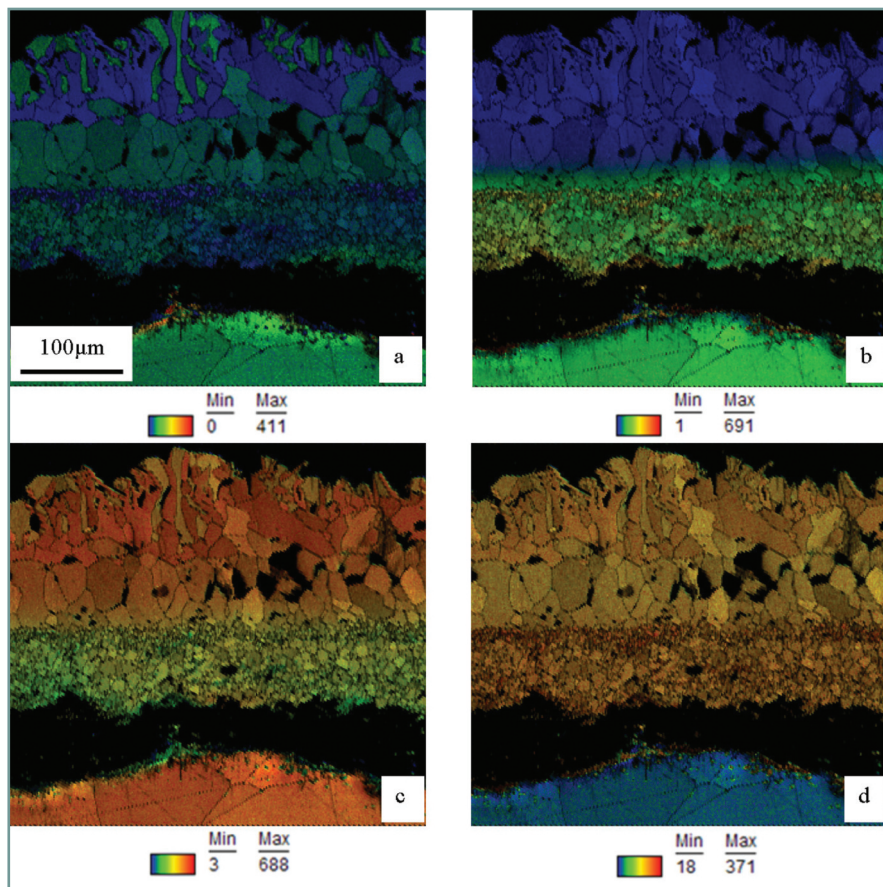


Figure 3. EDS maps of a) nickel, b) chromium, c) iron, and d) oxygen in an oxide scale formed on 316L stainless steel after 4 hours at 1200°C in air and cooled in air at room temperature.

The resulting phase map in Figure 4 shows the four possible phases within the three layers of oxide scale. There is a mixed phase layer closest to the substrate, a nickel iron oxide layer in the middle of the scale and a hematite layer on the top edge of the scale. There are also some nickel iron oxide grains on the top edge of layer 3. If a phase map were to be attempted without ChI-Scan then it would be fairly straightforward to index the hematite layer. This is because it has a different crystal structure (hexagonal) from the rest of the scale of which the majority are different spinel oxides (fcc). In contrast, distinguishing between the individual spinels using their crystallographic data alone would be almost impossible. However, when the EDS information is incorporated into the phase differentiating process using ChI-Scan, the spinels are easily distinguished from one another. The nickel iron oxide is distinguished based on its high nickel content, the chromium oxide by its high chromium content and low iron content and the chromium iron oxide by its high content of both chromium and iron.

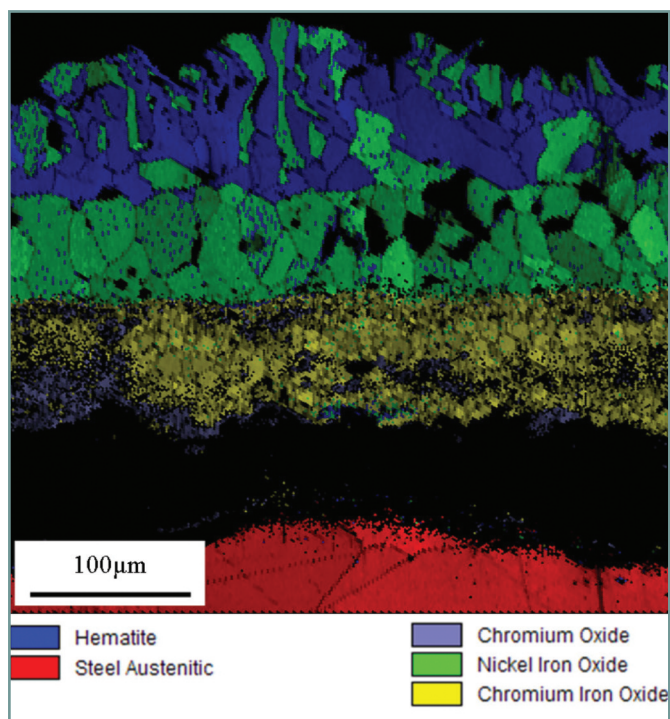


Figure 4. Map of the phases present within a scale formed on 316L stainless steel after 4 hours at 1200°C in air and cooled in air at room temperature.

Conclusions

The capability offered by ChI-Scan to combine both EDS and EBSD analysis results, enables the analyst to differentiate reliably between crystallographically similar and chemically different oxide scales grown on stainless steel samples. The analysis will enable process engineers to eliminate the relevant oxide scale from the final product.

Bibliography

Images taken from:

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