ADDRESSING THE CHALLENGE OF CHARACTERISATION ACROSS THE LENGTH SCALES

One of the biggest issues facing the microscopy of materials is the length scale issue. Components that are used in every day life are typically on the length scale of millimetres to several meters. For example, the aluminium alloy wing skin on a modern passenger aircraft is tens of meters long. The high strength steel bars used for impact resistance to protect passengers in cars are typically around a meter in size. There are, of course, many examples of finer scale artefacts of huge technological importance, including among many, computer chips and catalysts. Irrespective of which artefact is considered, the over-riding conclusion is that the properties are determined by the microstructure; the microstructure that is important is at the atomic or near atomic dimensions. Taking the example of the aluminium wing skin, the strength is derived from strengthening precipitates typically 10-100nm in size. The exact strength obtained is a direct function of the interface between the precipitates and the matrix- i.e. the local atomic structure. So, there is clearly a problem: to understand the properties of the component (say 10 meters long) we need to understand the atomic scale microstructure (10-10 m), i.e. 100 billion times difference in length scale! Is what we examine at 10-10 m in any way representative of all areas of the component? In the absence of a technique that will really allow an answer to this problem, microscopists have had to 'do the best you can'. We remain some way off a complete solution (although techniques such as synchrotron radiation and neutron diffraction help).

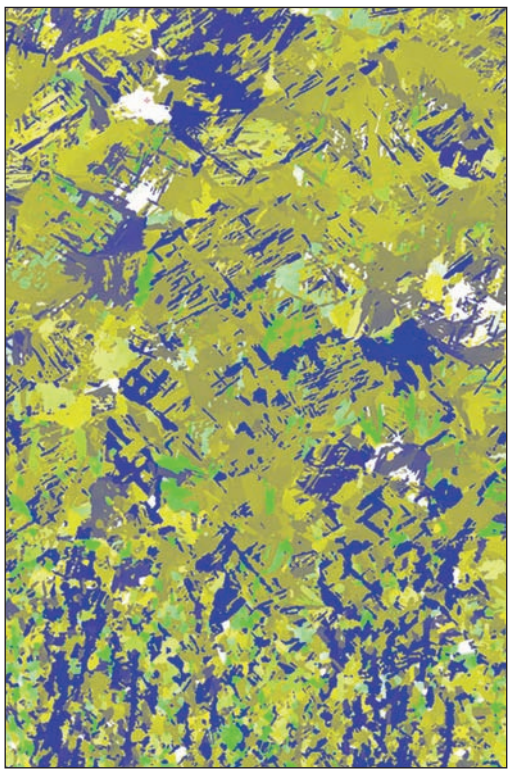

Figure 1. Orientation image map of the thermo-mechanically affected zone in a friction stir weld of Ti-6Al-4V. Image courtesy Dr Matthew Thomas. Sample courtesy TWI.

Microscopy Focus

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However, recent advances in electron back scatter diffraction (EBSD) in the scanning electron microscope is the first technique that allows the bridging of the gap from a few tens of nm up to cm dimensions.

EBSD (electron backscatter diffraction) comes under many names including EBSP (electron backscatter diffraction pattern), OIM (orientation imaging microscopy) etc. The technique itself is far from new, but its applicability has snowballed over the last decade. The first electron backscatter patterns were observed as long ago as 1928 by Kikuchi in the transmission electron microscope, this work being later extended by Von Boersch in 1937. The first use of an SEM and a fluorescent screen to observe the EBSP was undertaken in 1972 by Venables and Harland, which Dingley later developed in the 1980s. The big break through in the technique came in the late 1980s when the first commercial software became available to analyse electron channelling patterns (ECPs) and EBSPs. This led to the development of a commercial system to undertake EBSD automatically in the 1990s, although the rate at which each pattern could be analysed was still relatively low. Substantial increases in the speed of acquisition resulted from the advent of digital cameras in 1999. At the same time the use of field emission gun SEMs delivers high-resolution results, with 10nm resolution being recently demonstrated for twins in copper, for example. It is this combination of excellent spatial resolution, fast pattern identification and automated scanning over long periods of time that add up to the power of the technique. In short, it can provide high resolution (down to 10nm) on large areas of sample (cm possible), which represents a major break through in statistically meaningful high-resolution microscopy.

The best way to understand the power of the technique is through examples, all of which were undertaken to solve specific engineering issues.

Figure 1 shows an orientation image map from a friction stir weld in a titanium alloy (Ti-6Al-4V). Friction stir welding is a break-through technique for joining metals at low temperatures. It employs a tool spinning at high speed that locally heats and deforms the metals in such a way that the two become intermixed and joined. It has been applied extensively to aluminium alloys, but the poor thermal conductivity and high reactivity make it difficult for titanium alloys. However, TWI have recently overcome this problem by using a novel tool design, which represents a major break through.

The nature of the process is such that virtually no in-situ examination is possible; rather, the process can only be understood by studying the resultant microstructure taking a forensic approach to microstructural evolution. EBSD is the ideal tool, as it defines the crystal orientation, which is colour coded as in *Figure 1*, to immediately see the microstructural distribution. In this case, two distinct microstructural regions can clearly be seen (the parent plate structure and the weld itself). Moreover, the technique is very sensitive to small changes in crystal orientation (shown by small changes in colour contrast), which simply would not be seen by any other technique. Although the result is one that has impressive image impact, the real value is in having an image where at every point the crystal orientation is known and therefore the image represents a complete quantification of microstructure.

Figure 2 gives an example of where a microstructure could not be quantified by conventional techniques. It shows a steel structure that has been heated at high temperature (1020ºC) and then transforming to a bainitic structure at 320ºC. Bainitic structures are notoriously difficult to describe because of the rapidly changing heterogeneous structures. Some regions appear almost equiaxed, while others are strongly acicular.

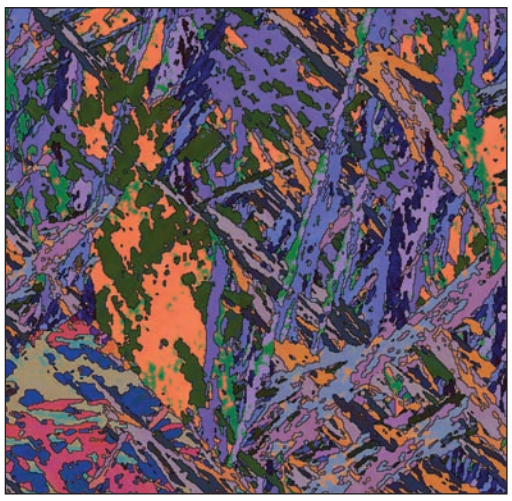

Figure 2. Orientation image map of bainite in a steel. Note the regions exhibiting the same colour that have the same orientation. Image courtesy Dr Pavel Cizek, sample courtesy Dr Mike Green.

The EBSD map in *Figure 2* again shows the local crystal orientation and clearly defines the regions that have the same crystal orientation (e.g. the blue and orange regions). Subtle changes in microstructure are known to give quite significant changes in the manner and speed with which cracks can move through the structure, ultimately resulting in failure. Conventional optical and SEM microscopy do not really allow a correlation between structure and the mechanical properties, such as resistance to crack propagation. However, EBSD provides the ideal tool to identify the parts of the structure that allow easy crack propagation (in this case the orange regions) as opposed to those where crack growth is more tortuous (in this case around the blue regions).

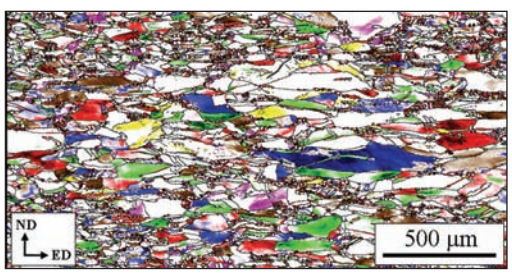

Figure 3. EBSD generated map of a Fe-30%Ni alloy that has been deformed at 950ºC to a strain of 0.6. Grain boundaries are shown in black, while the colours indicate specific texture components (e.g. red is the brass texture, blue is the copper texture). Image courtesy Dr Pavel Cizek, sample courtesy Dr Bai Fang.

Figure 3 shows the power of the technique across the length scales. The image was taken from a hot rolled steel and immediately shows the problems with defining such a structure- how do you even measure a 'simple' variable such as grain size? The image is an EBSD orientation image where specific crystal orientations (texture components comprising combinations of a direction and a crystal plane that contains that direction) have been colour coded. For example, the yellow regions are the so-called Cube orientation. In addition, the grain boundaries have been painted black, while the white regions do not correspond to a specific texture component (but the orientation is exactly known). Thus, the sample crystallographic texture is completely defined. Since the software can label the grain boundaries precisely (by knowing the angle across the boundaries), it can produce an absolute measure of grain size. The deformed structure comprises grains of two different sizes, the larger ones, that tend to be elongated, and finer ones that tend to be equiaxed. The latter are recrystallised grains, which result in a softening of the structure. It is important to know the volume fraction of recrystallised grains, but conventional microscopy gives a significant error, simply because it is difficult to identify which is which. EBSD can define each component much more precisely and give a visual indication of which is which, *Figure 4*. Another similar example is shown in *Figure 5*, taken from a two-phase structure, in this case a steel containing ferrite and austenite, which clearly deform in a very different manner.

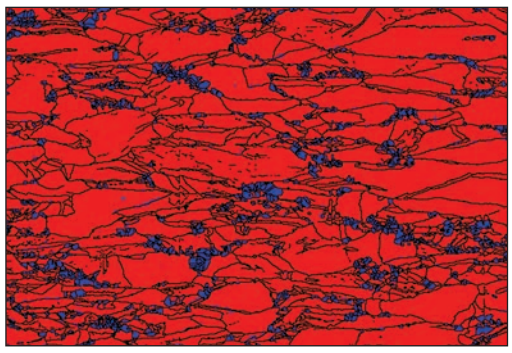

Figure 4. The same area as in Figure 3 (at a slightly greater magnification) with deformed regions labelled as red and recrystallised grains labelled as blue.

The map in *Figure 3* is only a small fraction of the original map that spanned 12mm from left to right- i.e. a significantly large region. However, as noted earlier, the power of EBSD is that one can use small step sizes with a fine electron beam to produce high spatial resolution, but map large areas of the sample, as here. It is then easy to go back and interrogate the fine scale structure at any point. *Figure 6* shows one such point, taken from a specific texture component (in this case the brass texture). Note how the deformation substructure can clearly be seen, with the accumulated dislocation damage leading to distinct dislocation walls and serrations along the grain boundaries.

Such regions can be quantified to show the local crystal misorientation across each dislocation wall, allowing a measure of the stored energy in the structure. The white regions in *Figure 6* have recrystallised, and so the correlation of those with the deformed structure can easily be seen. EBSD can throw up some interesting images, as here, with the lower right region baring an uncanny resemblance to Homer Simpson!

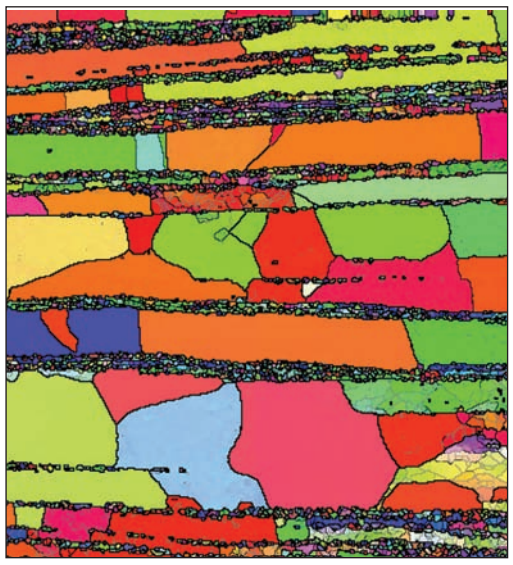

Figure 5. Orientation image map of a ferritic stainless steel showing large ferrite grains and fine austenite grains. Image courtesy Dr John Hinton.

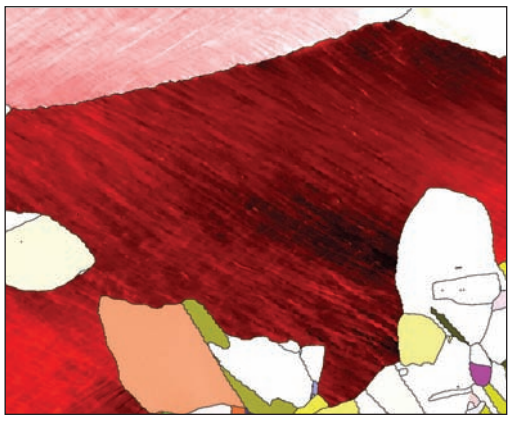

Figure 6. Higher magnification orientation image of a grain exhibiting the brass texture. The dislocation boundaries can clearly be resolved.
Note Homer Simpson in the bottom right recrystallised region!

ACKNOWLEDGEMENTS

The author is very grateful to Drs Brad Wynne, Pavel Cizek, Mat Thomas and John Hinton for the supply of the EBSD maps.

The author is also grateful to EPSRC who provided much of the funding for the work presented here, and to TWI, Corus, Outokumpu and Sheffield Forgemasters for the supply of samples.

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