Fast Orientation Imaging Microscopy

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Orientation Imaging Microscopy is currently the most rapidly growing combined metallographic and crystallographic technique today. The first OIM was recorded by Wright in 1991, and published soon after, Adams *et al.* (1993). The technique is based on the original works on Electron Backscatter Diffraction (EBSD) by Venables and Harland (1973), and Dingley (1984, 1987). By 1994 some number of papers on the subject had been published. At the time of writing the authors are aware of over 600 publications that have utilized the technique and there are in excess of 400 systems in use worldwide.

There are four critical steps in the production of one of these micrographs; 1) rastering the electron beam of the SEM in equal steps over the surface of the sample, 2) the recording at each point of the raster an electron backscatter diffraction pattern, 3) the use of a suitable algorithm to detect the positions of the Kikuchi bands in the pattern and measurement of interband angle and band width, and 4) a comparison of these measured parameters against a table of like parameters calculated from the known crystallography of the sample This leads to the indexing of the bands and the determination of the crystal orientation. When Wright first produced a computer program in which each of these steps was executed in a fully automatic fashion and for thousands of points over the specimen surface, the frequency of measurement was 1 point in two seconds. A typical micrograph contained 30,000 measurements and would take all day and all night in some cases. The operation was limited by computer speed. By 1999, however, computing speed had increased to the point where it was no longer the limiting factor. The limiting step became the rate at which the patterns could be recorded and transferred into computer memory. A parallel advance, by Michael and Goehner (1993) in the analysis of EBSD patterns meant that the technique could be advantageously used for crystal phase identification. The potential for doing this had been explored by Baba-Kishi and Dingley (1987), when the patterns had to be recorded directly on photographic film to ensure high definition. Michael showed that with use of a high resolution slow scan CCD camera, EBSD images could be obtained of almost the same quality as those determined by direct photography. This permitted

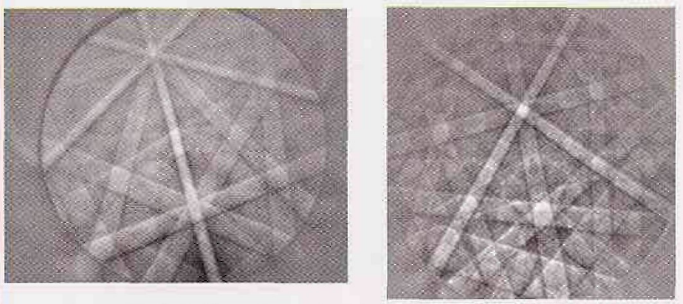


Figure 1a, (Left) EBSD pattern from Al_7Cu_4Ni precipitate in cast Al alloy.
Figure 1b, (Right) EBSD pattern from Si

extraction of greater detail and reliable on-line measurements of lattice spacing. The application of EBSD to phase identification is now an established and accepted procedure.

The most recent advance in the EBSD technology has been the introduction of a new type of CCD camera. In slow scan mode the camera provides the high resolution and high image depth of the Michael camera but have the advantage that they can be switched to a fast imaging mode suitable for OIM data collection. In fast mode the image is binned down both to increase sensitivity and reduce the read out time.

Figures 1a and 1b are EBSD patterns recorded using one of these cameras. The first is from a small precipitate in a cast aluminum alloy and is typical of what is obtained from metallurgical samples. The second is from a silicon crystal and is typical of patterns from very perfect crystals. The images measure 1300 x 1030 pixels and are 12 bits deep. They were recorded using a TSL Digiview 16/12 camera mounted on a Philips XL 30FEGSEM at a beam voltage of 20 kV and beam current of 0.8 nA. The exposure time was 2 seconds in both cases.

When the camera is used in binned mode, several pixels on the image-sensing chip are combined together. For example, at 8x8 binning the signals stored in the 64 pixels in the block are summed together and read out as a single point. The effective gain of the camera is 64 times that when reading out at full resolution. The intensity gain equates to a corresponding reduction in time to collect the image. Thus an exposure of 2 seconds needed for the above image is reduced to 31 milliseconds. If we now enhance the

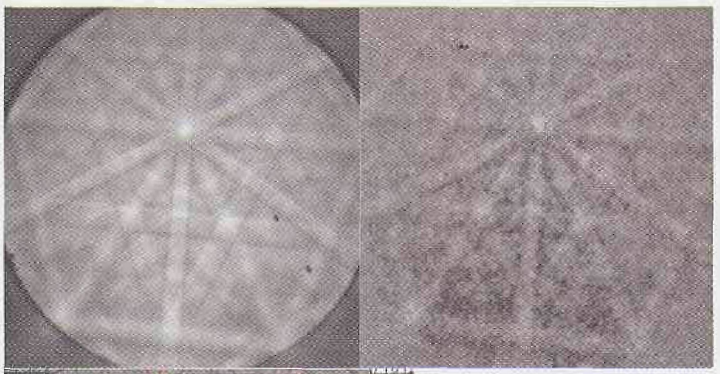


Figure 2a (Top Left) EBSD from TiAl alloy resolution 1030x1003. 2s exposure

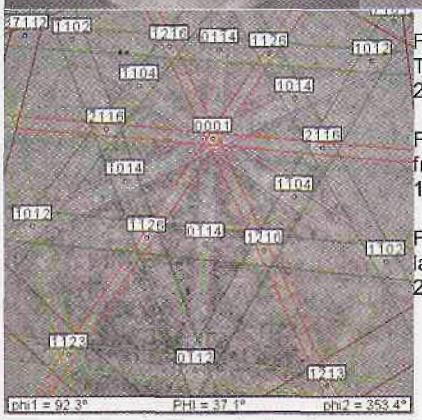
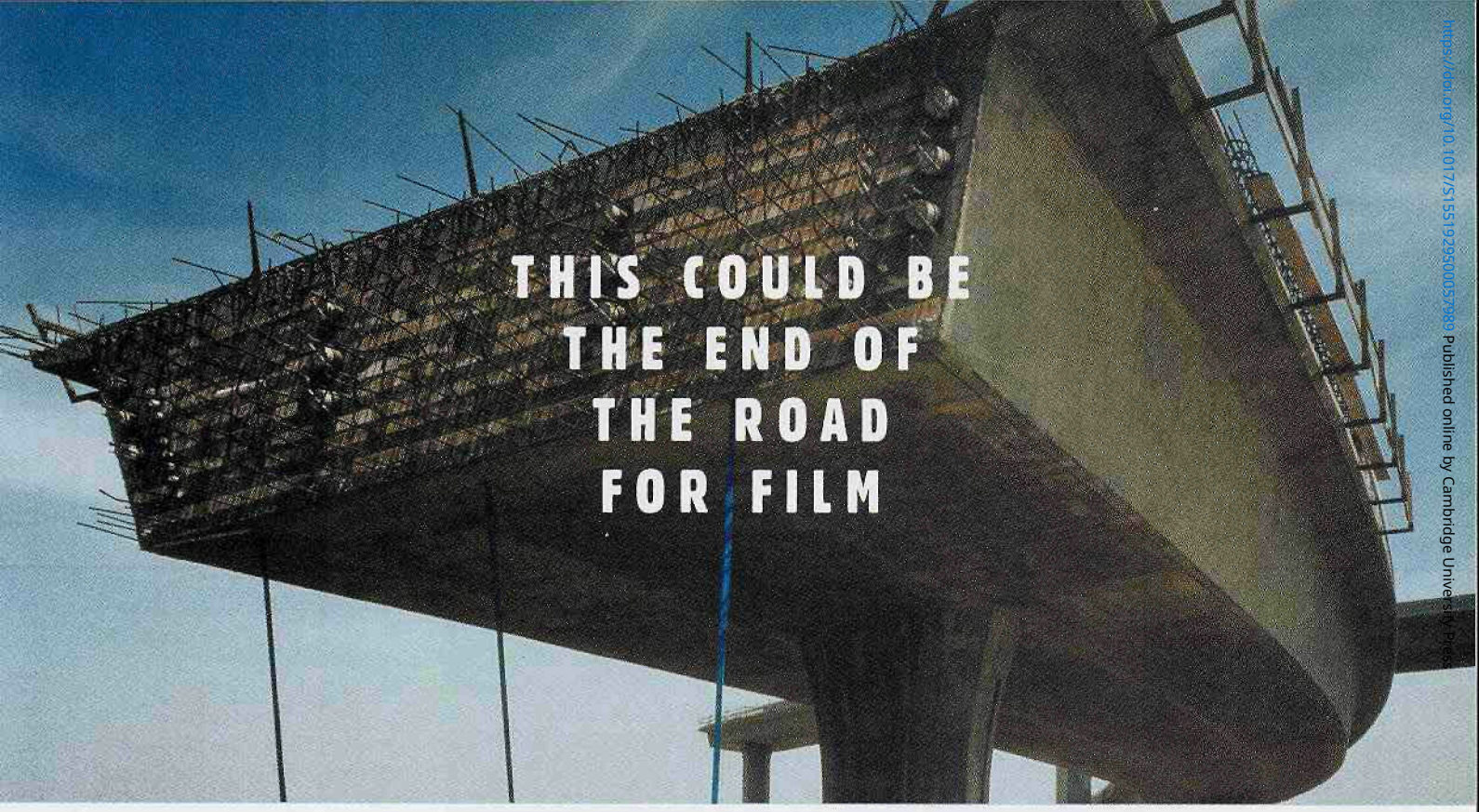


Figure 2b (Top Right) EBSD from TiAl alloy resolution 184x125. 20ms exposure

Figure 2c (Left) Computed overlay on EBSD shown in figure 2b.

image by a further electronic gain of up to 22 decibels, then a greater reduction in exposure time is achieved. In this way patterns of sufficient quality for indexing were collected from the camera at the rate of 64 per second. However, the measured maximum image transfer rate within the computer, though considerably higher than for previous CCD and analogue cameras, limited the data collection speed to 43 points per second. These measurements were made using a computer fitted with a Pentium 4 processor operating at 1.4 gHz and with 500 mbytes of RAM. The images were collected into the computer via a Matrox Meteor Digital video card.

Figures 2a and 2b show high and low resolution images of EBSD patterns recorded from a titanium aluminum sample. The high-resolution image measures 1300 x 1030 at an exposure time of 2s. The low-resolution image measured 184 x 125 pixels and was recorded at an exposure time of 20 ms, 200 times shorter than the high-resolution image. Despite the high noise level in the latter



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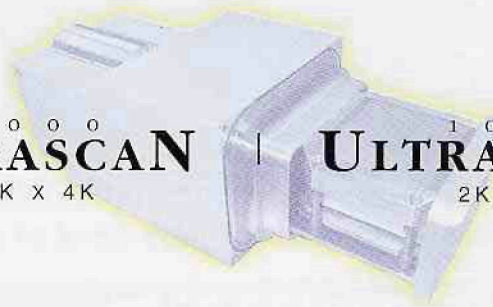

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image, the software by which the Kikuchi band positions are located is still able to detect them. The algorithm used is a Hough transform and is well described in a number of publications, Krieger Lassen (1992). This algorithm in effect sums the intensities of each pixel along a straight line in the image. The addition is repeated for all straight lines that can be drawn in the image at 1° inclinations to each other. When a line passes along the bright center of a Kikuchi band the summed intensity is large. By searching for the largest of the summed intensities the positions of the Kikuchi bands are determined. As the noise in the pattern is random it affects all lines that can be drawn equally and so does not diminish band detection routines as much as one might have thought. Figure 2c shows an overlay of the band center positions computed for the measured orientation. The accuracy of the fit gives a measure of the success of the indexing procedure. The mean angular deviation of all detected bands from the theoretical positions for the measured orientation was 0.8 degrees.

Figures 3a and 3b are OIM maps of the microstructure of the Ti alloy sample from which patterns in figure 2 were recorded. They were obtained at a data collection and indexing rate of 40 points per second. Figure 3a is colored to reveal crystal directions normal to the sample surface. The key is inset. Figure 3b shows the variation in diffraction pattern quality over the sample surface, a parameter that images the grain structure and distortions within the grains extremely well. A measure of confidence in the correctness of the recorded data can be calculated. In the reported scan each EBSD pattern was actually indexed 35 times using different combinations of the detected Kikuchi bands. The confidence index is calculated from the difference between the most frequently found indexing solution and the second most frequent solution normalized to the maximum number of solutions possible. For figure 3, the average confidence index was 0.47. Experiment has shown that any confidence index greater than 0.1 corresponds to the case where more than 95% of the solutions are correct, D.Field (1997). The measured confidence index is high. To test if this could be improved upon a second scan was performed on the same area as shown in figure 3a but with a less extensively binned primary EBSD image and using longer exposure time. The result is shown in figures 3c. This image was obtained under identical microscope conditions to the former result but with the binning set at 4x4 instead of 8x8 and an integration time of 70 ms. Data collection speed was 13 points per second.

Figure 3c can be directly compared with figure 3a and 3d with 3b. Corresponding images are virtually identical. The mean confidence index has improved marginally to 0.56. The total time required to collect the 28,000 points at the higher frame

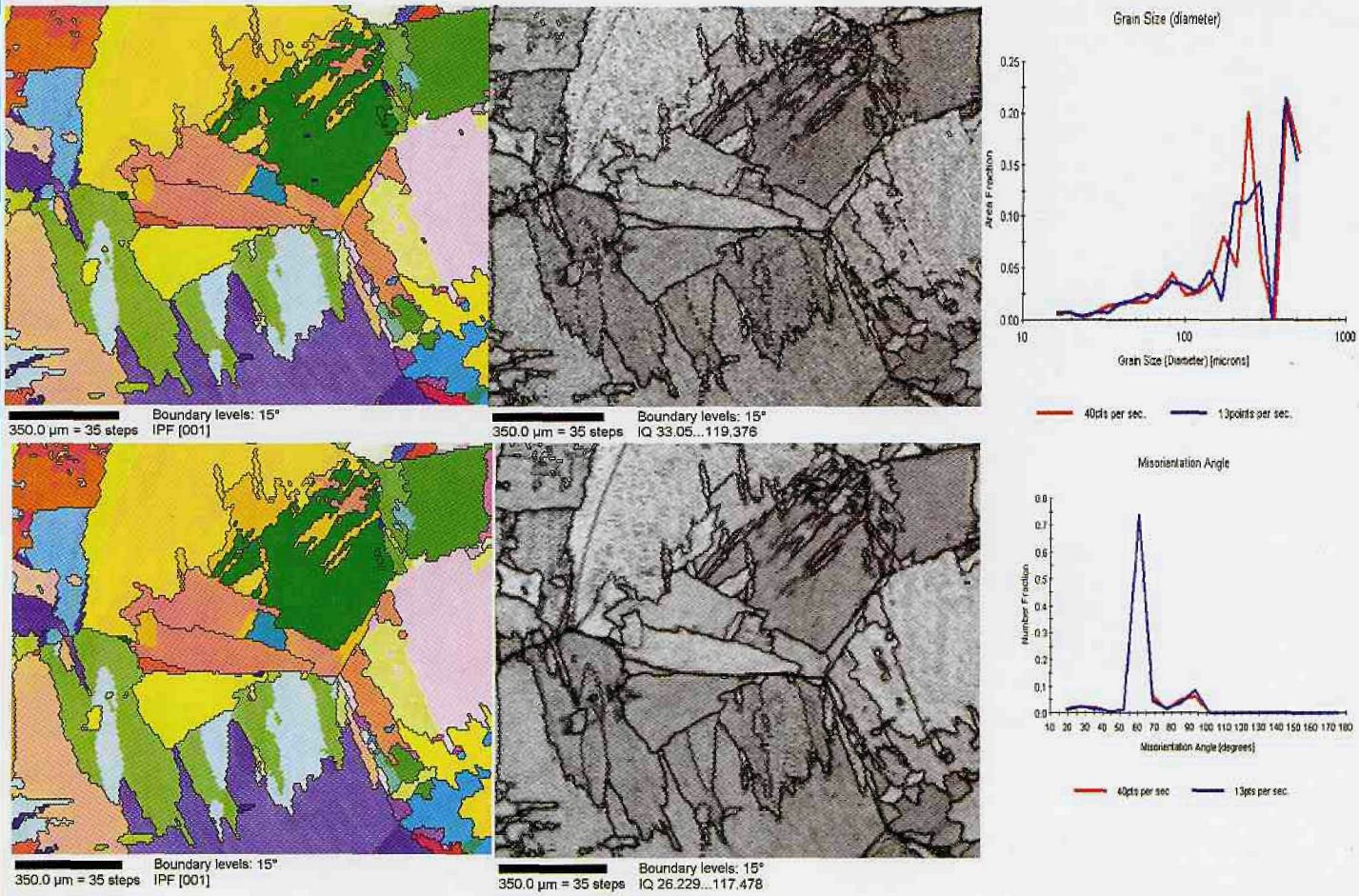
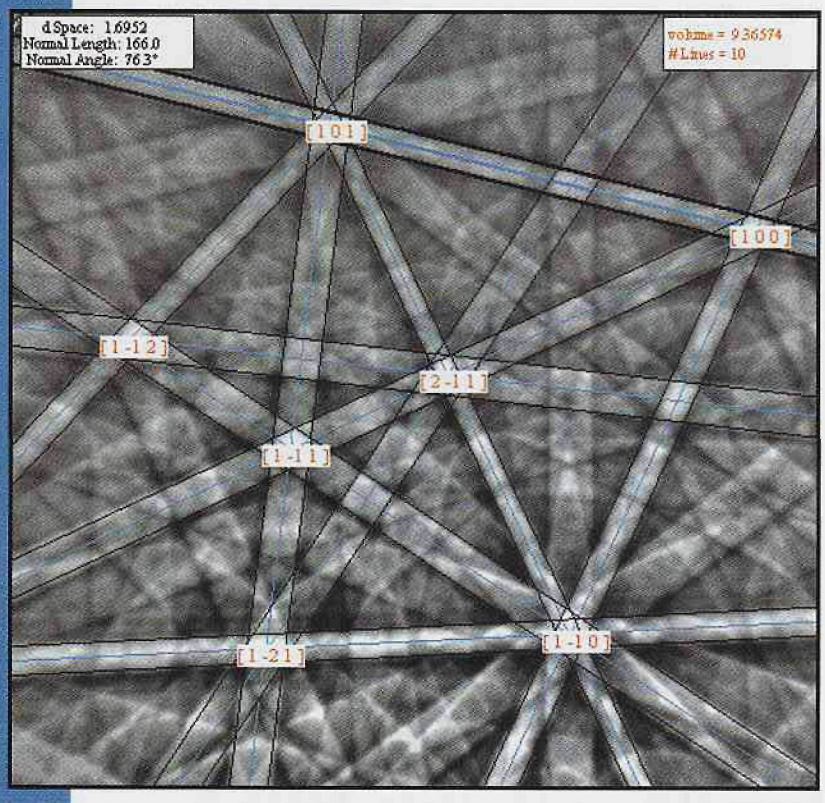


Figure 3a. (Upper Left) Crystal direction OIM of TiAl. Data collection speed 40points per sec.
Figure 3b. (Upper Center) Image quality OIM of TiAl 40 points per sec.
Figure 3c. (Lower Left) Crystal direction OIM of TiAl. Data collection speed 13 points per sec.
Figure 3d. (Lower Center) Image quality OIM of TiAl 13 points per sec.
Figure 4a (Upper Right) Graph of grain size distribution. Red line 13 points per sec. Blue line 40 points per sec.
Figure 4b (Lower Right) Grain boundary orientation distribution. Red line 13 points per sec. Blue line 40 points per sec.

PHASERS SET TO *STUNNING*



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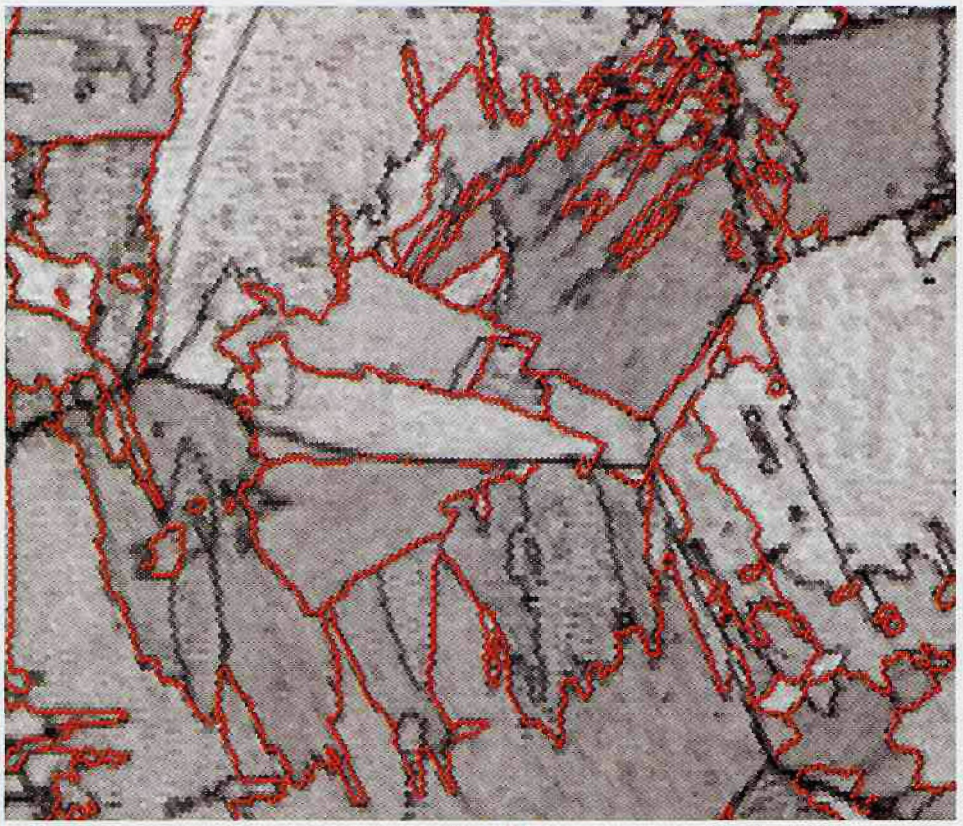

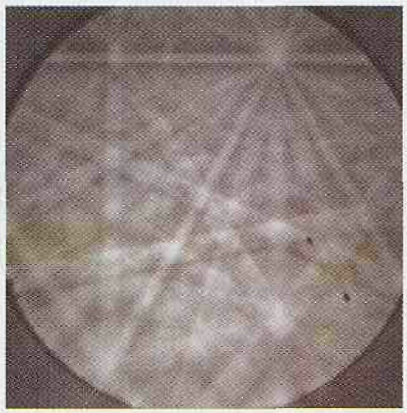

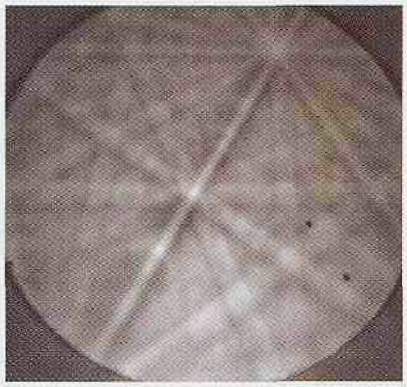

Figure 5a. (Left) Image quality map with 60° boundaries in red
Figure 5b. Above) EBSD patterns taken either side of a 60° boundary

rate was 11 minutes and that at the lower frame rate, 35 minutes. In both cases this represents an enormous speed improvement over what was previously possible and there appears to be no reason why scans at the higher rate of 40 patterns per second should not be the normal mode.

We can make one further quantitative assessment of the micrographs to test statistically for any loss of crystallographic or metallographic information at the higher rate. Figure 4a is a graph of the grain size distribution for both the 40 and 13 point per second data. Figure 3b is a similar plot showing the frequency of occurrence of a particular misorientation angle between grains. In neither case is there a statistical difference between the data sets. The mean grain size for the high speed data is 55 microns and for the slower scan it is 57 microns.

In passing, we note that figure 4b shows that there is a high occurrence of 60 degree boundaries in the micrograph. These boundaries are depicted in figure 5a as red lines. The predominant axis of rotation is close to <11-20>. The unusual nature and high density of this boundary prompted a manual test of the indexing. No errors or ambiguities were found. Figure 5b shows two EBSD patterns recorded either side of one of the 60 degree boundaries.

It is clear the time required to obtain representative OIM images of microstructure has been reduced to tens of minutes rather than the hours needed in earlier work. These times are sufficiently short that the images can be obtained during routine investigation of microstructure, in much the same way as the mapping of chemical information using energy dispersive spectrometry. The principal advantage of OIM is in the degree of quantification of microstructure both from a metallographic and from a crystallographic point of view. ■

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