Another Way to Implement Diffraction Contrast in SEM

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SEM users are familiar with two forms of contrast in SEM images: topographic contrast and atomic number contrast [1]. We can now add a third form of contrast. Contrast can arise due to the different orientation of grains in the sample [2]. However, in normal operation this contrast is very weak, since in the SEM the beam includes a range of incident angles. This has the effect of averaging out diffraction contrast from the different orientations of the grains. This contrast is generally much stronger when the incident beam is an ion beam rather than an electron beam – contrast between the grains is strong in ion-beam images but not in normal SEM images.

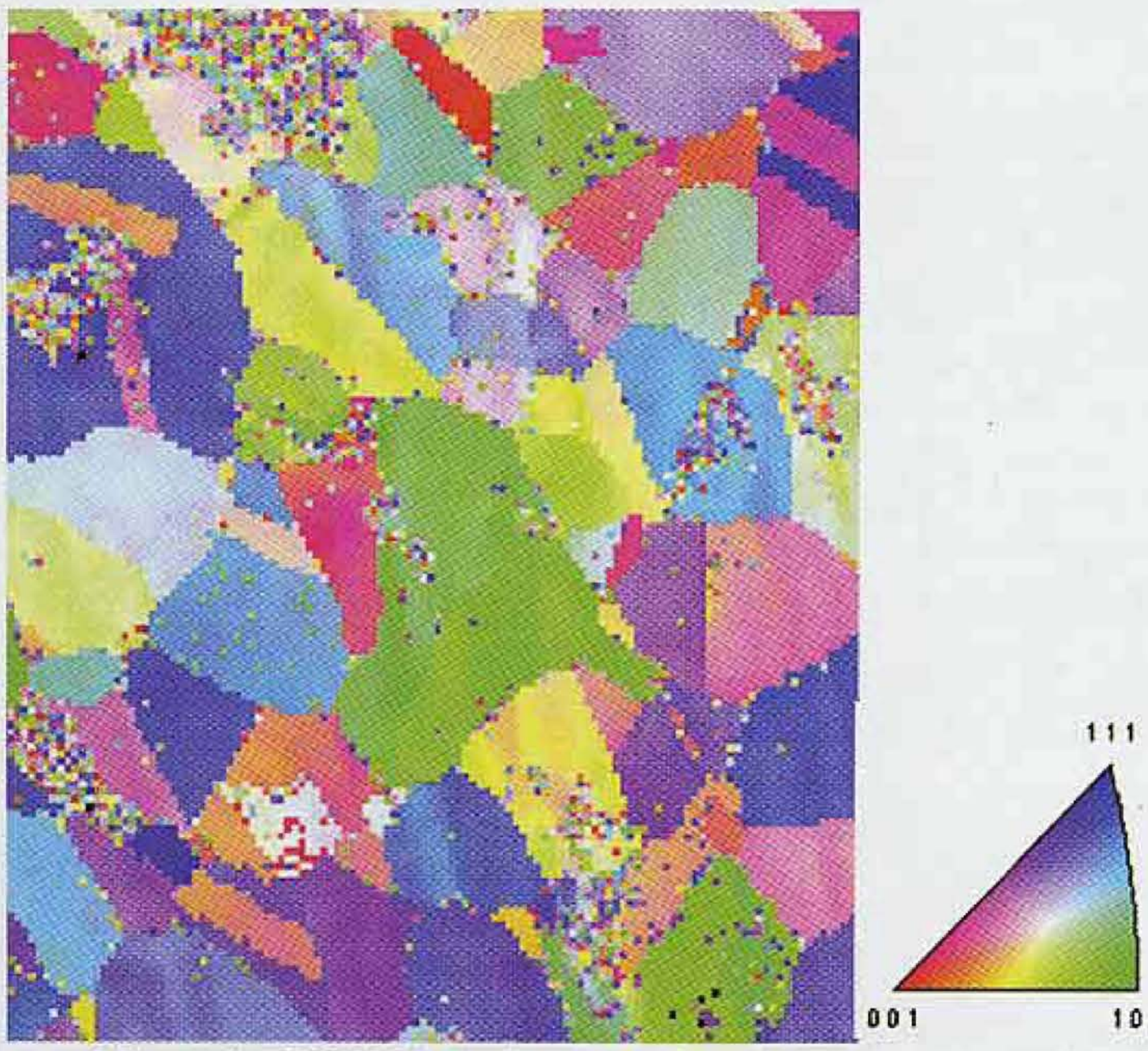
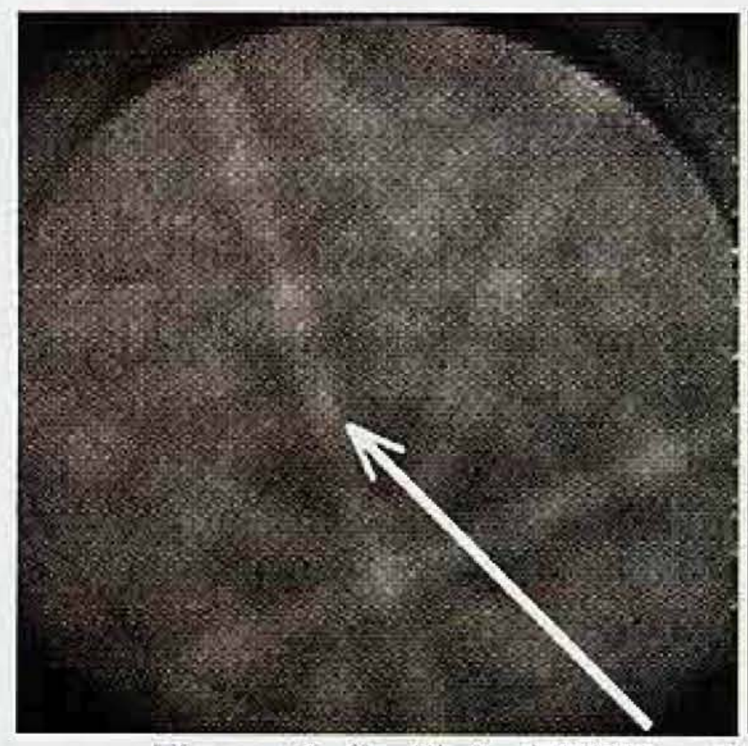


Figure 1. EBSD Orientation map of polycrystalline Ni. FEI XL-30 at 20KV with TSL OIM EBSD system. The step size is 0.5μm.

The advent of EBSD – electron backscattering diffraction - has changed this situation. Figure 1 shows an image of a nickel sample in which the grains have been color coded according to their orientation. The key to the orientations is given in the adjacent stereographic triangle. It is a complicated business to do the calculations that make such a map possible. That is why you paid (or should pay) so much for the EBSD system on your microscope.

We have found that good contrast to reveal the different orientations of the grains can be obtained much more simply. An EBSD pattern is shown in figure 2. The figure is criss-crossed by Kikuchi bands that lie along the projections of the crystal planes in the sample. In making an image like that of figure 1, these Kikuchi bands must be indexed and used to determine the actual orientation of the grain.



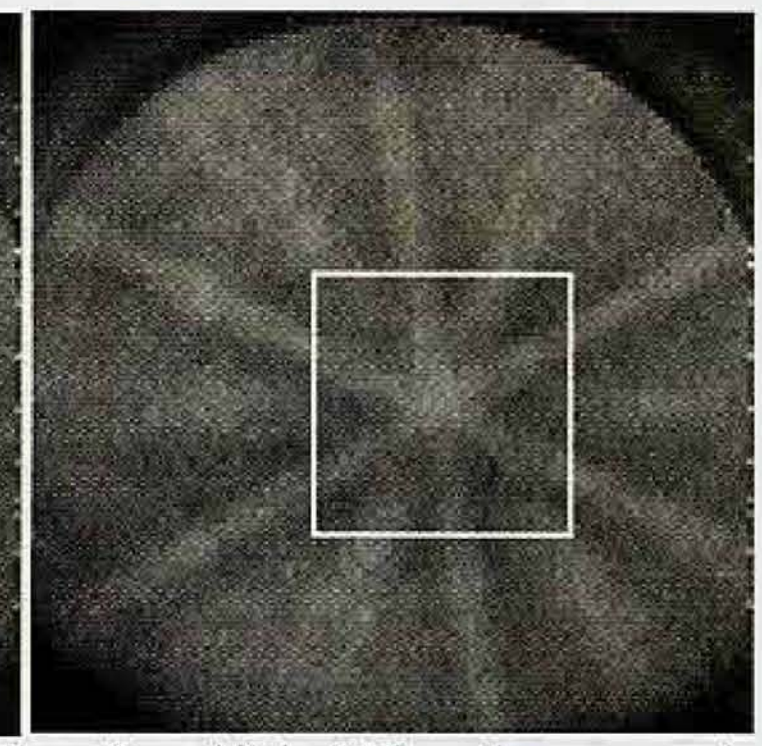


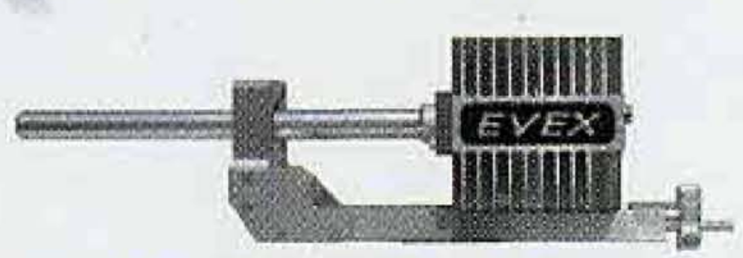
Figure 2. (Left) An EBSD pattern from Nickel. The criss-crossed bands are Kikuchi bands. Band showed by the arrow head is (100)
Figure 3. (Right) The box marks the region around the zone axis used for the mapping

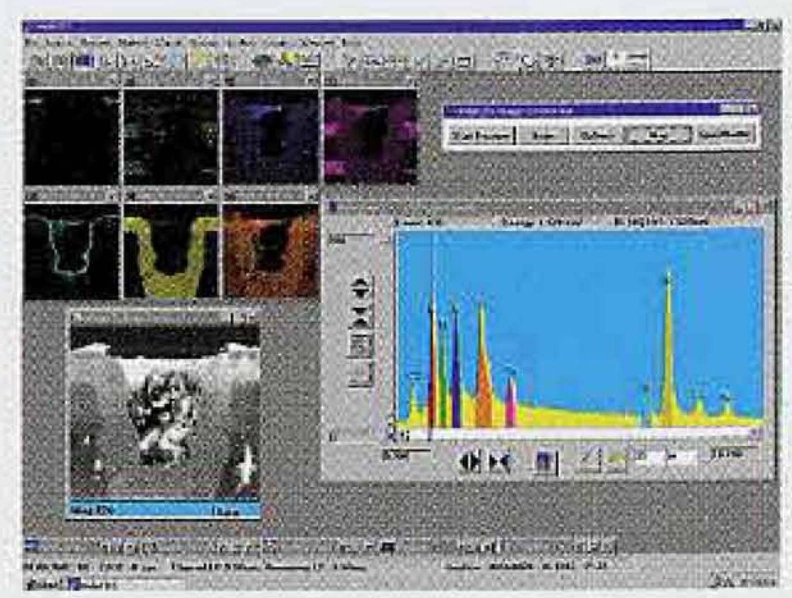
However, when the incident electron beam crosses a grain boundary the EBSD pattern changes dramatically. We do not need to index the pattern to know that it has changed. The change in the pattern across a grain boundary can be monitored in a very simple way by choosing some area of

Figure 4a. Map of the intensity averaged over the box in figure 3.
Figure 4b. Map of the ratio of the intensity in the box to the intensity in the whole pattern.
Figure 4c. Similar to figure 4b but with the box in a different zone axis.

1.008 H 1																	4.003 He 2
6.941 Li 3	9.012 Be 0.108 — 4											10.81 B 0.185 — 5	12.011 C 0.282 — 6	14.01 N 0.392 (Ti) — 7	16.00 O 0.523 (V, Cr) — 8	19.00 F 0.677 (Fe) — 9	20.18 Ne 0.848 — 10
22.99 Na 1.04 (Zn) — 11 —	24.31 Mg 1.254 — 12 —											26.98 Al 1.487 — 13 —	28.09 Si 1.740 (Rb,Sr,Ta,W) — 14 —	30.97 P 2.013 (Zr) — 15 —	32.06 S 2.307 (Mo, Pb) — 16 —	34.45 Cl 2.622 — 17 —	39.95 Ar 2.957 — 18 —
39.10 K 3.313 (Cd, In) — 19 —	40.08 Ca 3.691 0.341 20 —	44.96 Sc 4.090 0.395 21 —	47.90 Ti 4.510 (Ba) 0.452 (N) 22 —	50.94 V 4.952 (Ti, Cr) 0.511 (O) 23 —	52.00 Cr 5.414 (V) 0.573 (O) 24 —	54.94 Mn 5.898 (Cr) 0.637 25 —	55.85 Fe 6.403 (Mn) 0.705 (F) 26 —	58.93 Co 6.929 0.776 27 —	58.70 Ni 7.477 0.851 28 —	63.55 Cu 8.047 0.930 29 —	65.38 ZN 8.637 1.012 (Na) 30 —	69.72 Ga 9.250 1.098 31 —	72.59 Ge 9.885 1.186 32 —	74.92 As 10.542 1.282 33 —	78.96 Se 11.220 1.379 34 —	79.90 Br 11.922 1.480 35 —	83.80 Kr 12.649 1.586 36 —
85.47 Rb 13.393 37 1.694 (Si, Ta, W) —	87.62 Sr 14.163 38 1.806 (Si, Ta, W) —	88.91 Y 14.955 39 1.922 —	91.22 Zr 15.776 40 2.042 (P) —	92.91 Nb 16.617 41 2.166 —	95.94 Mo 17.481 42 2.293 (S, Pb) —	98 Tc 18.368 43 2.424 —	101.07 Ru 19.282 44 2.558 —	102.91 Rh 20.217 45 2.696 —	106.40 Pd 21.180 46 2.838 —	107.87 Ag 22.166 47 2.984 —	112.41 Cd 23.175 48 3.133 (K) —	114.82 In 24.209 49 3.286 —	118.69 Sn 25.272 50 3.443 —	121.75 Sb 26.359 51 3.604 —	127.60 Te 27.471 52 3.769 —	126.90 I 28.615 53 3.937 —	131.30 Xe 29.779 54 4.109 —
132.91 Cs 30.971 55 4.286 —	137.33 Ba 32.196 56 4.465 (Ti) —	L	178.49 Hf — 72 7.898 1.645	180.95 Ta — 73 8.145 1.710 (Si, Rb)	183.85 W — 74 8.396 1.775 (Si, Sr)	186.21 Re — 75 8.651 1.843	190.20 Os — 76 8.910 1.910	192.22 Ir — 77 9.175 1.980	195.09 Pt — 78 9.441 2.050	196.97 Au — 79 9.712 2.122	200.59 Hg 70.806 80 9.987 2.195	204.37 Tl 72.869 81 10.267 2.271	207.2 Pb 74.989 82 10.550 2.346 (S, Mo)	208.98 Bi 77.091 83 10.837 2.423	209 Po 79.272 84 11.129 2.501	210 At 81.513 85 11.425 2.581	222 Rn 83.771 86 11.725 2.662
223 Fr 86.098 87 12.029 2.744	226.03 Ra 88.495 88 12.338 2.911	A															

L	139.91 La 33.441 57 4.650 0.833	140.12 Ce 34.717 58 4.839 0.882	140.91 Pr 36.031 59 5.033 0.929	144.24 Nd 37.358 60 5.229 0.978	145 Pm 38.725 61 5.432 1.032	150.40 Sm 40.118 62 5.635 1.085	151.96 Eu 41.534 63 5.845 1.137	157.25 Gd 42.992 64 6.056 1.190	158.93 Tb 44.476 65 6.272 1.245	162.50 Dy 45.997 66 6.494 1.299	164.93 Ho 47.534 67 6.719 1.353	167.26 Er 49.100 68 6.947 1.409	168.93 Tm 50.730 69 7.179 1.466	173.04 Yb 52.362 70 7.414 1.523	174.97 Lu 54.078 71 7.654 1.581
A	227.03 Ac 88.495 89 12.650 2.911	232.04 Th 93.382 90 12.967 2.996	231.04 Pa 95.886 91 13.288 3.083	238.03 U 98.434 92 13.612 3.171	237.05 Np 100.800 93 13.941 3.260	244 Pu 103.320 94 14.275 3.348	243 Am 105.970 95 14.615 3.437	247 Cm — 96 14.961 —	247 Bk — 97 15.300 —	251 Cf — 98 15.661 —	252 Es — 99 16.018 —	Fm 100	Md 101	No 102	Lr 103





the pattern and monitoring how its intensity changes (using the intensity of the whole pattern as a normalizing factor).

Figure 3 shows another EBSD pattern from a different grain from that of figure 2. We arbitrarily selected the area in the white box, chosen to sit around the major zone axis for a grain at this orientation (in this case a [101] zone axis). Then for all places in the image we calculated the average intensity in the box. This is shown in figure 4a. As can be seen it gives topographic contrast (the little hillocks) but also shows contrast between the different grains. In figure 4b, the intensity in the box is divided by the intensity averaged across the whole area of the pattern. As can be seen it gives good contrast between the different grains in the polycrystalline sample. The topographic contrast is much reduced making the diffraction contrast between the grains clearer. Figure 4c is obtained in a similar way but with the box in a different position in the image. The contrast changes but still shows the grains [3,4]. ■

References:

1. David C. Joy, Dale E. Newbury and David L. Davidson J. Appl. Phys., vol.53, No.8 R81-R122.
2. P.Mortin, M.Pitval, E. Vicario and G.Fontaine, Scanning Vol. 2 217-224 (1979).
3. We are grateful to TSL for allowing us to use their source code.

4. Support from DOE, under grant DE-FG02-00ER45819, is gratefully acknowledged.

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Microscopy Today was initially conceived as a free publication for the US microscopy community financially supported by its advertising. Non-US microscopists paid $80 to $110US for a one year subscription. When the Microscopy Society of America purchased this magazine, and rolled its non-US membership into the subscription rolls, we were faced with the dichotomy of some non-US microscopists paying a great deal of money for essentially a free publication while others, perhaps at the same institution, received the magazine free. MSA has taken a number of steps to eliminate this situation. First: We have redefined the "free to US" rule by adding microscopists in Canada and Mexico to the free eligibility list. Second: We have scanned back issues of MT into computer files, which we now make available for free download anywhere in the world after a six-month delay. The entire issues are downloaded—advertisements and all. Non-North American MSA members may download the current issue with a password provided on request. The magazine was scanned and saved as high-resolution JPEG files that are about 30MB per issue. Very few individuals can download files that large, so we created a second set of PDFs that are about 9MB per issue for downloading purposes. The PDFs are called "low resolution" products in the table below. The 2002 issues are available now at http://www.microscopy-today.com. The most recent five years are scanned and will probably be available for downloading by mid-March 2003. We will scan-in and make available the remaining issues back to 1992 shortly.

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