Effect of Evactron® Cleaning on EBSD Detector Phosphor Screens

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Introduction

The Evactron® cleaning process [1,2] uses a radio frequency plasma source to create oxygen radicals which are then transported through the chamber to be cleaned by a pressure differential between the oxygen radical source (ORS) and the pump that is keeping the chamber under vacuum. These oxygen radicals convert hydrocarbon contaminants into H_2O, CO and CO_2, which are then pumped out of the chamber. Tests have previously shown that the Evactron® cleaning process does not damage or degrade the performance of ultra-thin EDS windows [3]. The results reported here show the effect of the Evactron® cleaning process on the appearance and performance of an EBSD detector phosphor screen.

Experimental Procedure

SEM chamber cleaning was performed using the Evactron® for 20 hours at 14W and 0.4 Torr (the manufacturer-recommended power and vacuum settings). This 20 hour clean represents more than 7 years of SEM chamber cleaning conducted once a week for 3 minutes. During the Evactron® cleaning process, the phosphor screen was mounted on the EBSD detector in its usual (retracted) position within the SEM chamber.

Optical micrographs were taken at 9 different positions (as shown in Figure 1) on the phosphor screen before and after exposure to the Evactron® process to determine if any changes to the screen surface had occurred. The effect of the process on the performance of the phosphor screen was evaluated in two ways. The first was to compare raw EBSD patterns (without any background correction or image enhancement) collected from an {001}<110> Si single crystal. The second was to perform EBSD mapping on the Si silicon crystal and on a duplex steel sample before and after the cleaning. Detailed comparison of the maps was undertaken to determine if any change had occurred in the quality of the EBSD patterns (EBSPs) or the ability of the software to index those EBSPs.

Figure 1: Locations at which optical microscope images were taken on the phosphor screen.

Results

Visual Inspection

The surface of the phosphor screen appeared similar in the optical micrographs taken at each point before and after the Evactron® cleaning process. There were no major changes to the microstructure and the size and distribution of pits in the screen appeared to be the same. However, some reduction in the amount of bright area on the screen was noticed (Figure 2). The micrographs in Figure 2 were taken at point 5 in Figure 1 but are typical of those taken at other positions.

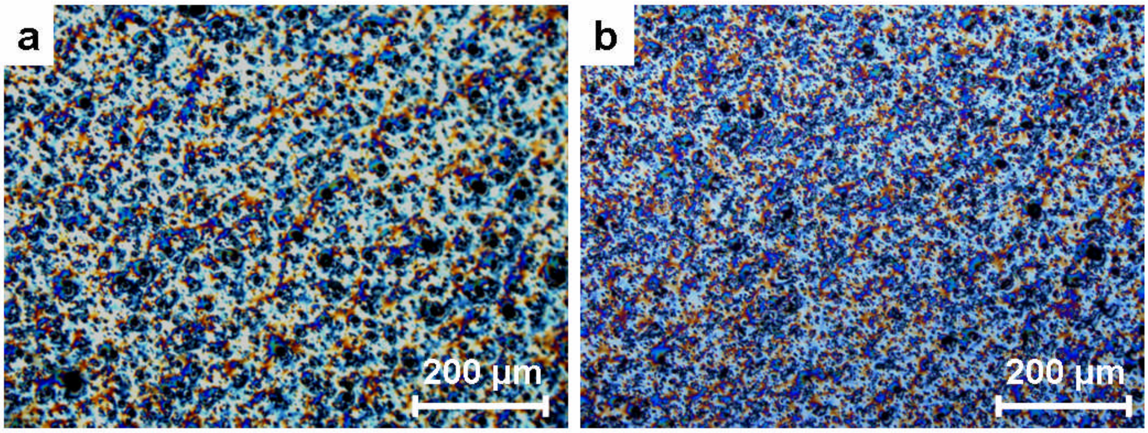

Figure 2: Optical micrographs of the surface of the phosphor screen, (a) before and (b) after exposure to the Evactron® process. These micrographs were taken at point 5 in Figure 1.

<u>Raw EBSD Patterns</u>

No significant difference could be detected between the raw EBSPs taken using the phosphor screen before and after the Evactron® cleaning process (Figure 3).

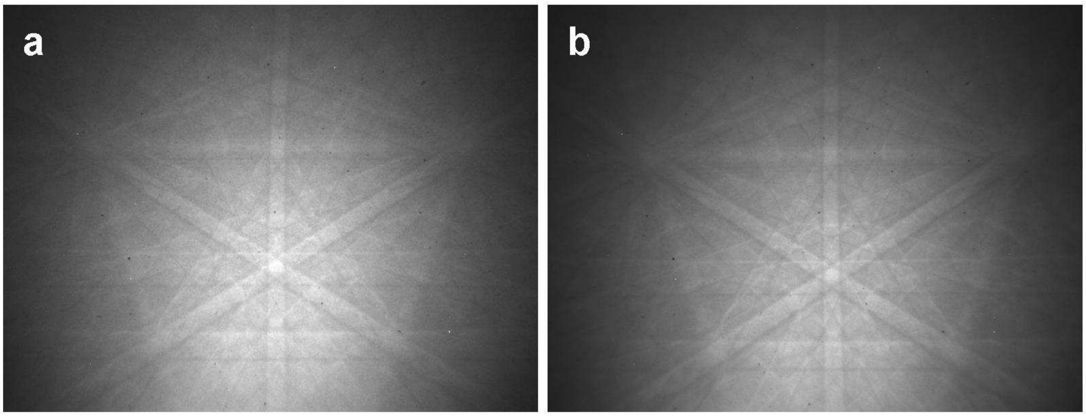

Figure 3: Raw EBSPs collected on a {001}<110> silicon single crystal (a) before and (b) after exposure to the Evactron® process.

<u>EBSD Mapping on a Si Single Crystal</u>

The results of quantitative comparison of the quality of the EBSPs acquired on the Si single crystal before and after SEM cleaning are shown in Figure 4. There was a small decrease in EBSP band contrast (the intensity difference between the bands and the surrounding EBSP) and a small increase in EBSP band slope (the contrast gradient at the edges of the EBSPs). There was a small reduction in the mean angular deviation (a measure of the deviation between the bands detected in the EBSP and the simulated bands of the selected solution). One of these changes (in band contrast) is in the direction of lower EBSP quality, while the other two (in band slope and MAD) are in the direction of higher EBSP quality and higher indexing quality. However, the changes are small and it is concluded that there is no significant difference between the results before and after SEM cleaning.

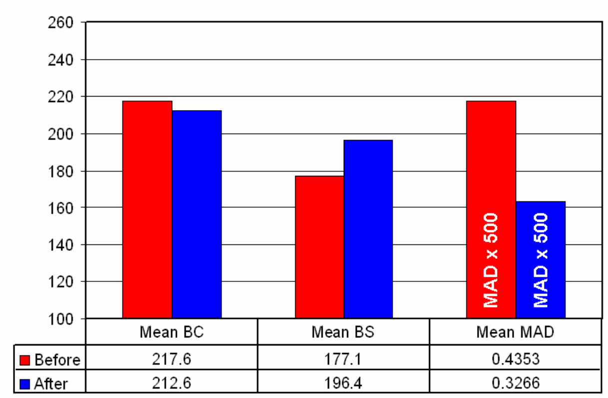

Figure 4: Mean EBSP Band Contrast (BC), Band Slope (BS) and Mean Angular Deviation (MAD) for a 100 pixel EBSD map taken on a Silicon Single Crystal before and after the phosphor screen was exposed to the Evactron® cleaning process.

<u>EBSD Mapping on Duplex Steel</u>

EBSD maps collected on the duplex steel sample before and after SEM cleaning using the Evactron® process are shown in Figure 5. Visually, there is no difference in the quality of the maps, except that some scratches made during the sample polishing process can be seen the region of the sample mapped after the Evactron® cleaning process (c, d). Both maps had a very low percentage of non-indexed points: 1.2% in the case of the "before" map (a, b) and 2.2% in the case of the "after" map (c, d). The non-indexed points were concentrated along grain and twin boundaries and, in the case of the "after" map, along the scratches left over from polishing. The slightly higher percentage of non-indexed points in the map taken after the SEM cleaning can be attributed to the non-indexed points associated with the scratches and the greater number of boundaries (particularly twin boundaries in the fcc phase) in this region of the sample.

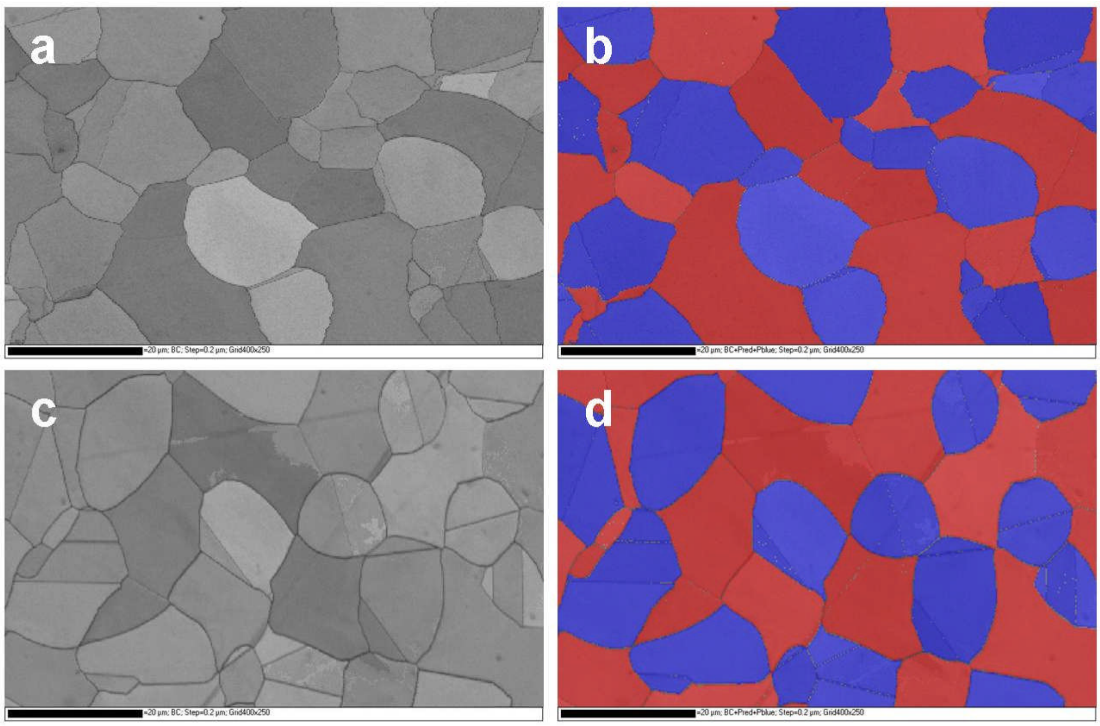

Figure 5: EBSD maps taken on the duplex steel sample before (a, b) and after (c, d) SEM cleaning using the Evactron® process: maps of EBSP band contrast (a, c) and phase maps showing bcc iron in red and fcc iron in blue superimposed on EBSD band contrast (b, d).

The results of a quantitative comparison of the quality of the EBSD patterns before and after SEM cleaning is shown in Figure 6. The mean contrast of the bands used for indexing the EBSPs is shown in Figure 6a, while the mean contrast gradient at the edges of the bands used for indexing the EBSPs is shown in Figure 6b. There is no significant difference between the values before and after SEM cleaning in either case.

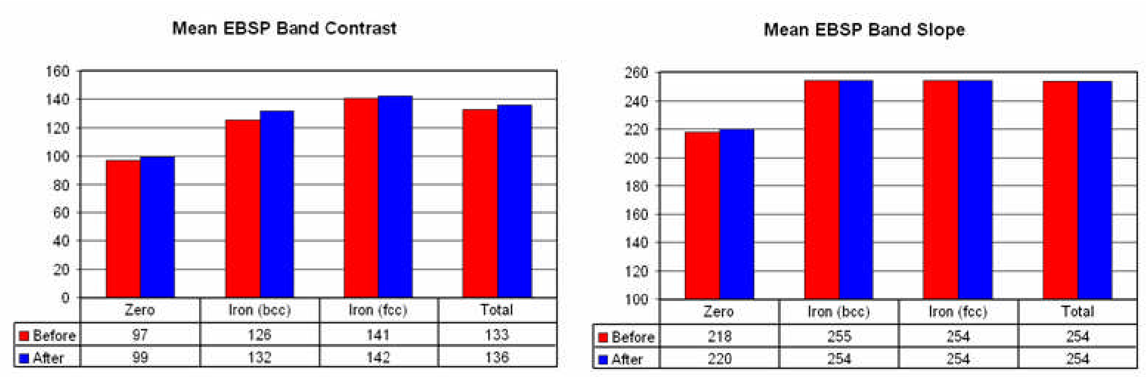

Figure 6: Graphs of mean EBSP band contrast and mean EBSP band slope for the maps taken (a) before and (b) after the cleaning process.

The fit between the bands of the solution EBSPs and the bands detected in the acquired EBSPs is quantified in Figure 7. There is no significant difference between the results before and after SEM cleaning. The small difference is either natural variation or a result of the greater proportion of boundaries and scratches in the area mapped after SEM cleaning.

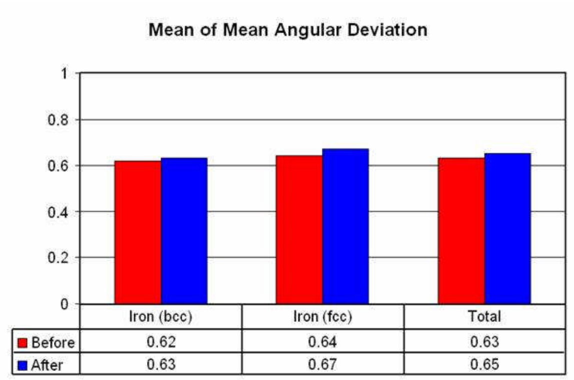

Figure 7: A comparison of the means of the mean angular deviation between the detected bands used for indexing each EBSP and the bands of the selected solution in the duplex steel maps taken before and after SEM cleaning.

Conclusion

Use of the Evactron® process for SEM cleaning at the manufacturer-recommended settings for 20 hours (the equivalent of more than 7 years of SEM chamber cleaning conducted once a week for 3 minutes) caused no significant changes in the quality of the EBSPs or the ability of the EBSD software to index those EBSPs.

References

[1] Vane, R., US Patent 6105589, August 22, 2000.
[2] Vane, R., US Patent 6452315, September 17, 2002.
[3] Vane, R., Roberts, C., and Carlino, V., A Study of the Effects of Evactron® Plasma Cleaning on X-ray Windows, Proc. Microscopy and Microanalysis 2004, Savannah, GA, USA, Aug. 2-5, 2004.