Determining size distribution and shape factor for sub-micron chrome carbides in treated steel piping

S. Scheller[1], H. Nitschke[1,2], D. Hünert[2], and A. Kranzmann[2]

1. Product Management, Bruker AXS Microanalysis GmbH, Schwarzschildstr. 12, 12489 Berlin, Germany.
2. Division V.1 Microstructure of Engineering Materials, BAM Federal Institute for Materials Research and Testing, Unter den Eichen 87, 12205 Berlin, Germany

Samuel.Scheller@bruker-axs.de
Keywords: particle analysis, size distribution, chrome carbides, EDX, SEM

Recent and continuing sets of studies are being conducted into the formation and growth of chrome carbides after aging power plant steels in a corrosive oxyfuel atmosphere H_2O-CO_2. It has been hypothesized that carbides can grow or nucleate during corrosion process. The particular carbides that were measured here were found in a cross section of specially prepared steel piping. The carbides had lengths below 1 µm and widths well below 1 µm.

Challenges with finding and measuring these small carbides existed in their small size and similar atomic number contrast to the matrix. Using a FE-SEM at a moderately high magnification with a frame width of 23 µm, at a 15kV acceleration voltage and using a solid state backscatter (BSE) detector, the carbides were difficult to locate. Using Quantax 800 energy dispersive x-ray spectroscopy (EDS) system, consisting of an XFlash 5000 series EDS detector from Bruker AXS Microanalysis, the Mapping and Feature Analysis software modules were utilized to find, confirm and measure these carbides.

Using the EDS intensity map the carbides were found, but due to the requirement for larger area analysis of their size distribution, this method was too time-consuming. A different approach needed to be employed.

The specimens were re-polished and underwent an etch process, to attempt to lift the carbides a little from the steel matrix. This process allowed the carbides to stand out more in the BSE micrograph and gave the possibility to locate and perform morphological measurements on the carbides via image processing.

Using the Feature Analysis module, the particle detection method was setup with a Binarisation to lift the carbides out of the matrix, followed by some morphological filtering. Performing this analysis on the areas of interest, the carbides could be measured and saved in particle lists and size distribution histograms by length, width, area and other features.

To confirm the bright phases on the BSE image were in fact carbides, a short acquisition EDS map was performed at the center of each region of interest. The regions of interests were set on the treated sides and the untreated sides of the pipes, to be able to compare the carbide concentration and their size distribution via histogram plots of their width and length. These results enabled conclusions to be drawn on the effects of different treatments performed on the steel pipes.

W. Grogger, F. Hofer, P. Pölt (Eds.): MC2009, Vol. 3: Materials Science,
DOI: 10.3217/978-3-85125-062-6-437 , © Verlag der TU Graz 2009

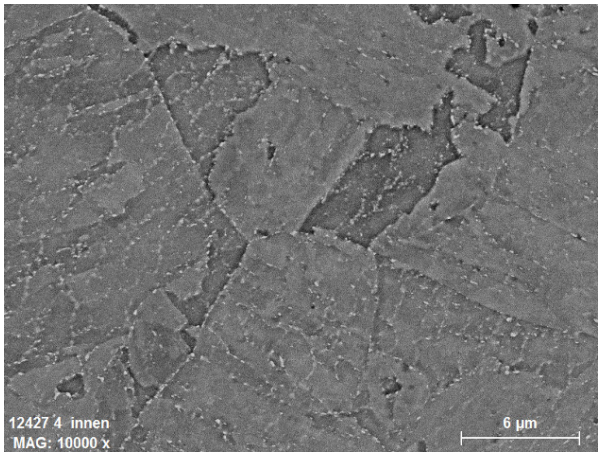

Figure 1. BSE micrograph before corrosion

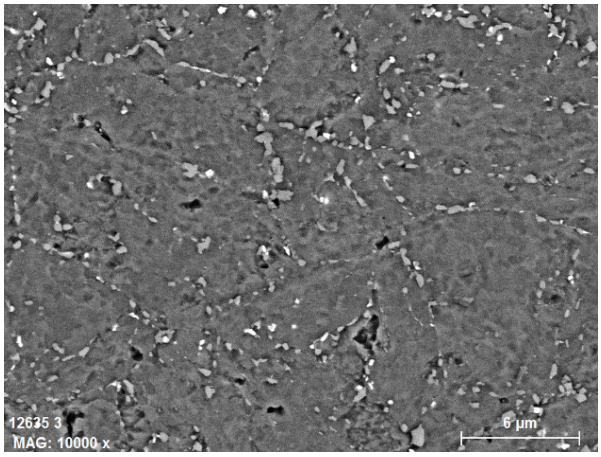

Figure 2. BSE micrograph after corrosion

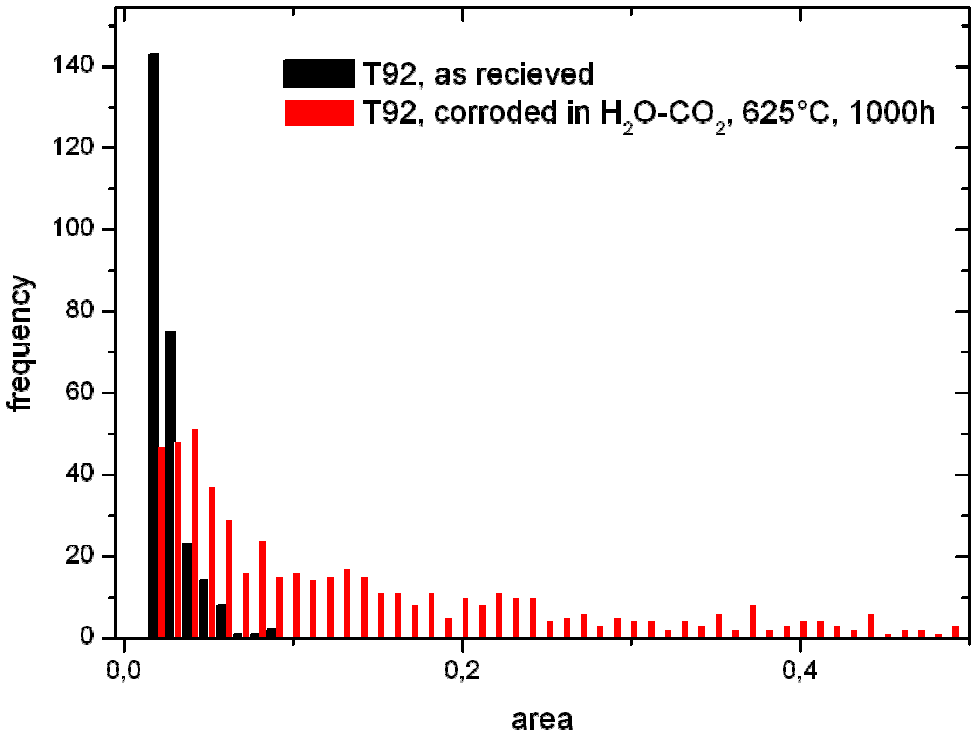

Figure 3. Size distribution for area before and after corrosion

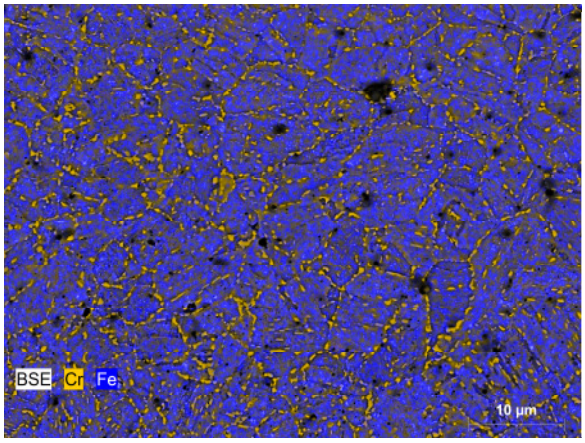

Figure 4. Mapping of carbides in matrix

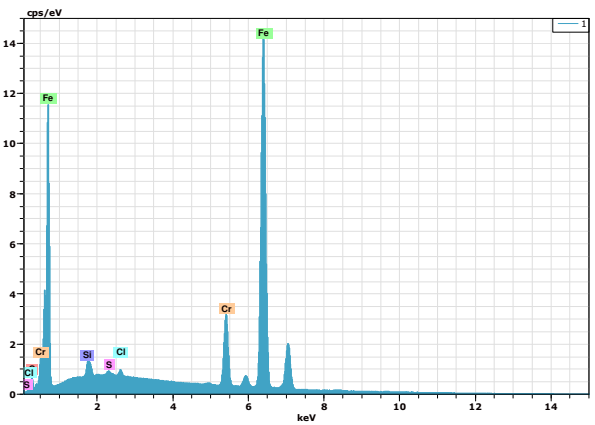

Figure 5. Sum spectra for mapping

W. Grogger, F. Hofer, P. Pölt (Eds.): MC2009, Vol. 3: Materials Science,
DOI: 10.3217/978-3-85125-062-6-437 , © Verlag der TU Graz 2009