Progress Report on

Investigation of the Penetration Depth of Maxon CRS in Metal

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Submitted to

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Problem Statement

Maxon Technologies has developed a new coating to prevent corrosion and have requested that a third party confirm or deny their own experimental results in order to pass on this information to potential consumers.

Purpose

The purpose of this study is to determine the penetration depth of the Maxon CRS into different metals. Maxon Technologies claims that the coating can penetrate into the steel at least 7-9 mils.

Summary of Previous Tests

The Maxon CRS has been applied to plain carbon steel samples and left to penetrate the metal for 2, 5, and 7 days. Samples were prepared for metallography according to ASTM E3 by grinding and polishing to a 1-micron polish. The Maxon CRS coating was not washed off the sample. The sample was then placed in the chamber of a scanning electron microscope (SEM) to focus on electron beam onto the surface. Then an energy dispersive x-ray spectroscopy (EDS) detector was used to identify characteristic x-rays in the sample. First, the Maxon CRS coating was focused onto with the electron beam so that a list of elements present in the coating can be obtained. This produced a spectrum of many elements, including those commonly found in organic compounds and others, such as aluminum (Figure 1). A list of these materials is provided in Table I.

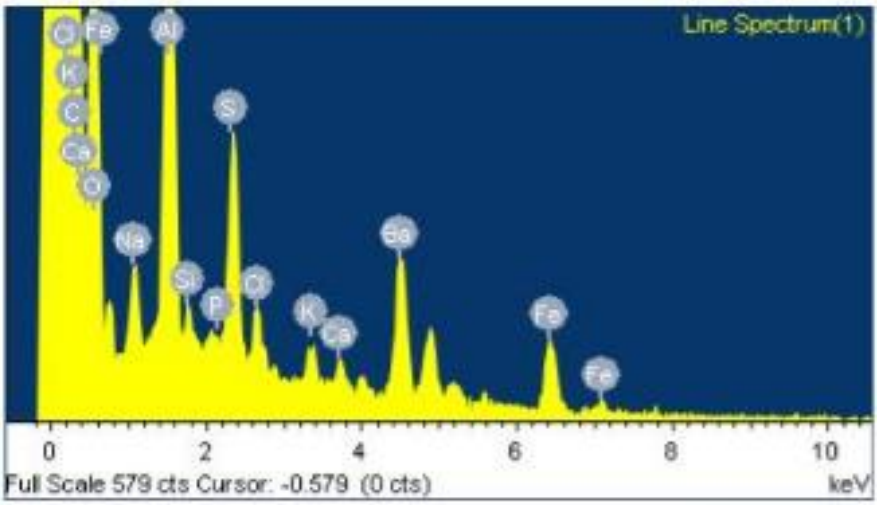

Figure 1. Spectrum of characteristic x-rays gathered from focusing on the thin layer of Maxon CRS coating left on the sample. Height of the peaks represents the intensity of the spectrums, and is used to determine the atomic percentage of the different elements in the material.

Table I. List of elements detected by EDS when focused on Maxon CRS.

Element	Weight%	Atomic%
C K	62.05	72.77
O K	26.68	23.49
Na K	0.53	0.32
Al K	3.44	1.79
Si K	0.16	0.08
P K	0.08	0.04
S K	1.08	0.47
Cl K	0.36	0.14
K K	0.24	0.09
Ca K	0.20	0.07
Fe K	1.33	0.34
Ba L	3.84	0.39
Totals	100.00	

When the electron beam was focused on the steel sample, it produced very inaccurate estimations for the carbon content of the steel. Instead, phosphorous seemed to be a better indicator of the presence of the Maxon CRS in the metal because of its presence in the coating and the ease for the EDS to detect it. The larger the atomic number of an element, the easier it is for the EDS to detect its characteristic X-rays. This is because they produce multiple peaks along the spectrum and at higher energy levels.

Using phosphorous as an indicator the Maxon CRS was estimated to have penetrated about 230 microns after seven days (Table II). This being the first test, it was impossible to know if there was some element of experimental error in the test. Multiple tests could not replicate the detection of phosphorous deep into the metal.

Table II. Summary of Results from Initial Test

Depth (μm)	Maxon CRS elements present?	Specific Maxon CRS elements present	Point of interest label
60	No		Line Spectrum 2(8)
100	Yes	P	Line Spectrum 2(4)
145	Yes	P	Line Spectrum 2(5)
155	Yes	P	Line Spectrum(2)
185	Yes	P	Line Spectrum 2(6)
230	Yes	P	Line Spectrum 2(7)
270	No		Line Spectrum 2(8)
315	Yes	P, C, O, Al	Line Spectrum 2(9)
325	No		Line Spectrum(3)
355	No		Line Spectrum 2(10)
500	No		Line Spectrum(4)
570	No		Line Spectrum(5)

Summary of Newer Tests

The goal of the newer tests has been to determine if there can be a reliable way to determine the amount of penetration from carbon into the metal using different techniques with the SEM to improve its accuracy for elements with smaller atomic numbers.

Several changes were made to the method of gathering data of the SEM in an attempt to improve the reliability of the results. These changes were developed over the course of the quarter, and all found to have a positive effect on the procedure for gathering data.

1. A control sample was used to determine the expected level of carbon content in the metal when Maxon CRS was never applied to the surface.
2. Multiple replications of the same depth were gathered at the same time by gathering data from an array of points along the cross section of the metal (Figure 2).
3. Two different accelerating voltages were used to try to determine if there is an improvement in precision of the carbon content when reducing the accelerating voltage.
4. A new tidy-up procedure was performed before starting work on the machine so that the EDS is completely re-calibrated before each use.

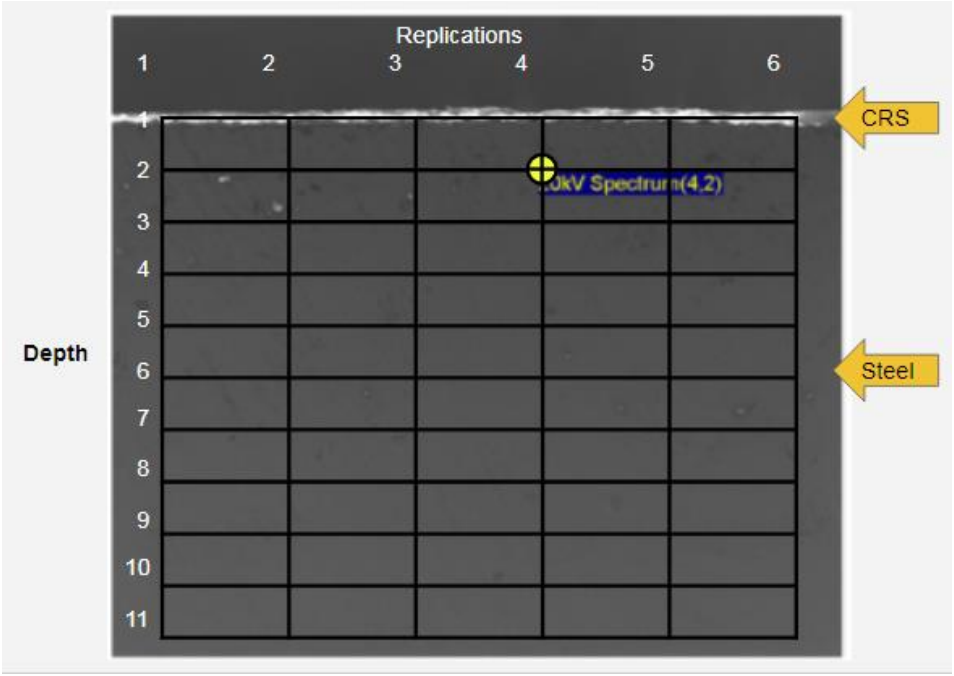

Figure 2. Array of points of interest that the electron beam was focused on. 11 depths, separated at a distance of about 70 microns were observed. For each depth, six replications were measured at different points. This figure has a yellow cross-hair symbol indicating that the electron beam was focused at point (4,2). This translates to replication 4 for depth 2.

As expected, the accelerating voltage has a significant effect on the detected carbon content of the material. The 5kV accelerating voltage produced a smaller weight percentage estimation of carbon than the 20kV setting (Table III). Surprisingly, however, there was not a significant reduction in the standard deviation when reducing the accelerating voltage. There was actually a small increase in the standard deviation. This indicates that the smaller accelerating voltage is not an inherently better method at improving the precision of EDS detector on elements with a smaller atomic number.

Table III. Weight percent of carbon in non-coated steel samples from EDS.

Accelerating Voltage	Average	Standard Deviation	Sample Size
20kV	2.102	0.793	16
5kV	1.497	0.833	16

This study was able to provide some more reliable results for determining the carbon content, however. The tidy-up procedure improved the tests so that negative percentages of carbon were no longer detected by the EDS (a result of there being lower than expected amount of background radiation at the energy level that one would expect to see carbon).

This test did detect large amounts of carbon at the shallowest depth measured, at about 70 microns (Figure 3). However, this was not detected for every replication. This resulted in a high variance for Depth 2 (70 microns) (Figure 4). This may be due to the mechanism that the Maxon CRS penetrates into the metal. If the Maxon CRS penetrates into the metal by preferentially travelling along grain boundaries as opposed to traversing across the grains of the metal, then it would be reasonable to expect that the presence of the Maxon CRS would be highly heterogeneous. This may be preferred because oxidation would occur on the outside of the grains of the metal, so a higher concentration of the Maxon CRS at grain boundaries would be more effective than an even distribution throughout the metal.

What this could mean, however, is that it may be difficult to detect the Maxon CRS. The SEM cannot detect where the grain boundaries of the metal are, so any electron beam can be focused on the center of a grain or directly on a grain boundary and the operator would have no way of knowing. This could mean that tests may have to be done multiple times, and the failure to detect carbon at certain depths could potentially be a result of the electron beam not being focused on any grain boundaries and not because of failure of the Maxon CRS to penetrate into the metal.

If this is in-fact the mechanism for penetration of Maxon CRS, then it may be very worthwhile to investigate the penetration depth of metals with various grain sizes. Smaller grain size would mean more grain boundaries, and this would likely have a significant effect on improving the penetration depth of the Maxon CRS.

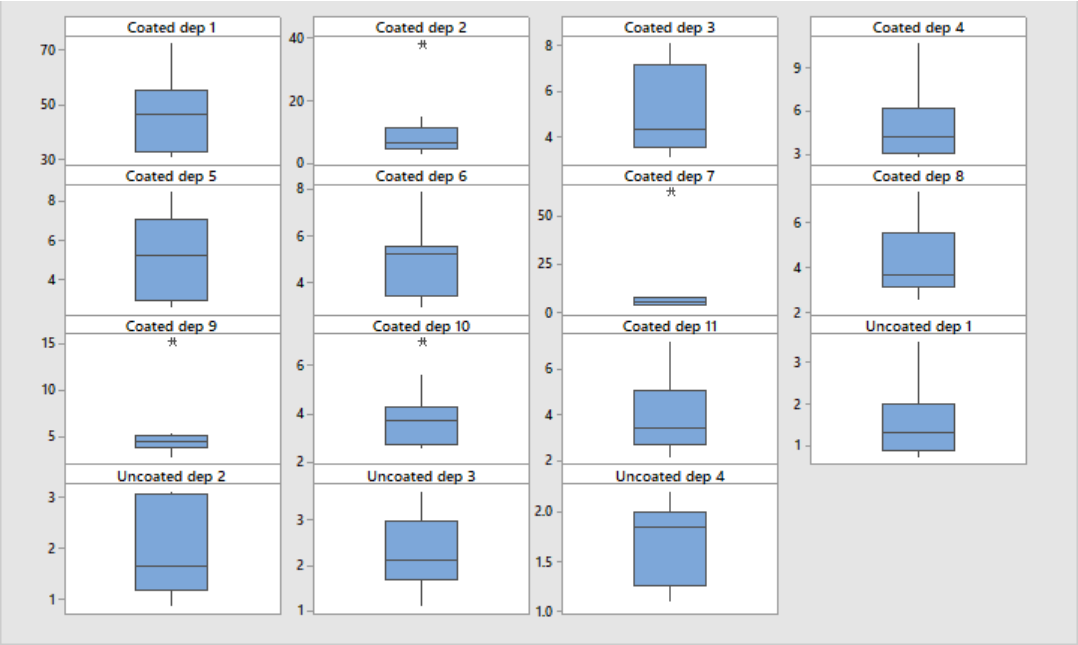

Figure 3. Box plots of the coated and uncoated samples at an accelerating voltage of 5kV. The coated sample at depth 7 (coated dep 7) has an unusually high carbon content for one of its replications because it detected a contaminant such as dust on the surface of the metal.

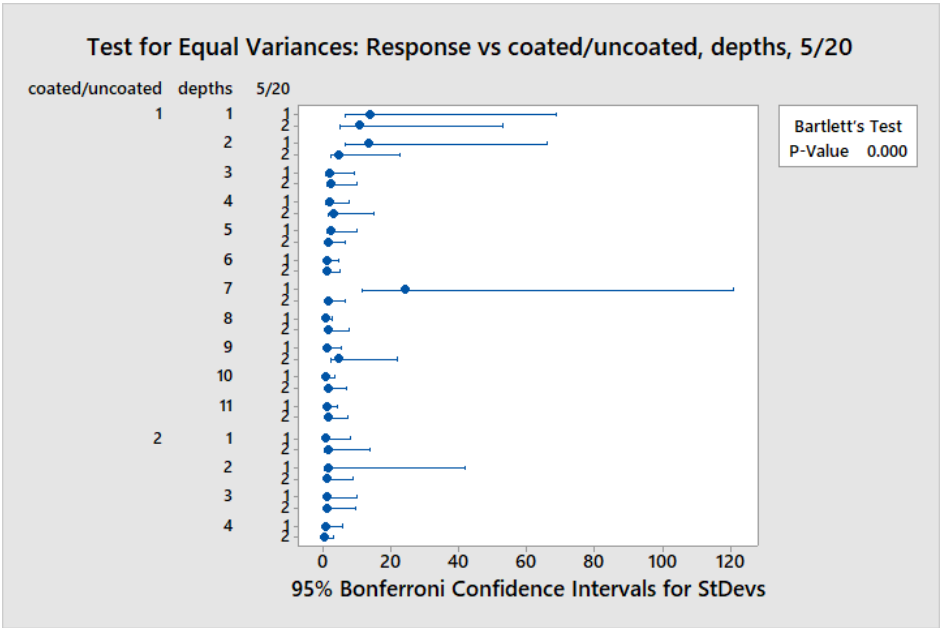

Figure 4. Test for equal variances. This test failed, so a 3-way ANOVA test was unable to be performed. However, this table is a good visualization of the large amount of variation shallow depths (1 and 2). Depth 1 is the Maxon CRS, and Depth 2 is 70 microns into the surface of the metal.