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Effect of Surface Pretreatments On Chromate Conversion Coatings

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Beamline(s): X10C

Introduction: Hexavalent chromium is considered to be the active species in CCC that promotes 'self healing'[1-3]. Zhao et al. have shown the migration of chromate species from a CCC film to an initially untreated alloy sample[1]. Hexavalent chromium has also been shown to repassivate scratches formed on the coating[4]. Hence the amount of hexavalent chromium in these coatings itself plays an important role in the protection offered to the substrates. Chemical surface pretreatment performed on AA2024-T3 prior to conversion coating have been shown to result in significant changes with respect to hexavalent chromium content (6%) in the outer surface[5] as compared to the coatings formed on polished alloy surfaces[6] shown to have a very high hexavalent chromium (60%).

Methods and Materials: The following pretreatments were performed on aluminum alloy AA2024-T3 in as received conditions (i) mechanical polishing (MP), (ii) pickling in 8% KOH for 2 min. followed by immersion in 50% HNO_3 for 15 seconds (KH), (iii) treatment with 30g/l sodium borate in 10% HNO_3 at 50^{o}C for 15 min. (JB) and (iv) the untreated sample (as received condition – AR) acted as a control. The pretreated and untreated AA2024-T3 were conversion coated using Alodine 1200S (Parker Amchem™) for 1min & 5min. These coatings were then analyzed for the amount of hexavalent chromium using X-ray Absorption Near Edge Spectrum (XANES).

Results: The normalized spectrum obtained from XANES is given in Figure below. The chromium edge is at 5989eV. The Cr K absorption edge falls in an energy range where the lifetime broadening is small and the spectrometer resolution is high so that the spectra are well defined. A unique pre-edge feature, when present, indicates the presence of chromium in octahedral coordination or in other words of hexavalent chromium. The height of the pre-edge on the normalized spectra gives the ratio of hexavalent chromium to total chromium in the coating[7-8]. This is shown in detail in the figure.

Total hexavalent chromium content of chromate conversion coatings on various pretreated AA2024-T3 alloy and formed using Alodine solution for 1 min and 5 min are given in table below. It can be seen that time of exposure to conversion coating solution has negligible effect on the hexavalent chromium content. The hexavalent chromium content is 30 $\pm$ 3%. Kendig et al[9] have observed an increase in the amount of Cr(VI) with time of exposure of AA2024-T3 in Alodine coating for up to 5 min and final values in range of 22% have been observed. However Lytle et al.[10] observed no significant changes in the hexavalent chromium content for similar coatings on AA2024-T3 for 1 min and 3 min exposures. The Cr(VI) content was closer to 28%. The results obtained in this study are consistent with the previous observations made by Lytle et al[10].

Pretreatment	**Cr (VI) / Cr (Total)**	
	1 Min	**5 Min**
As Received **(AR)**	-	27.5
Mechanically Polished **(MP)**	34.2	34.0
KOH + HNO_3 **(KH)**	32.1	33.5
$NaBrO_3$ **(JB)**	31.8	30.0

Conclusions: The variation in CCC composition with surface pretreatment might be significant. However, further tests are being performed to evaluate changes in properties.

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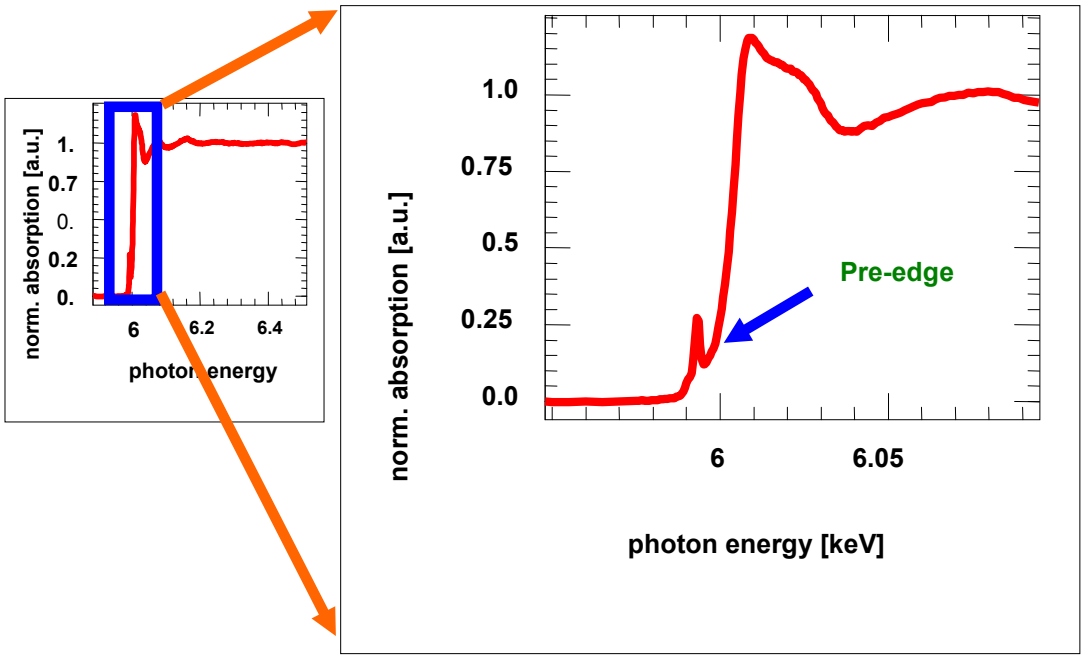

Fig 1: The height of the pre-edge on the normalized spectra gives the ratio of hexavalent chromium to total chromium in the coating. This is shown in detail.