

While attempting to deduce the mechanism of the photosensitivity of these compounds brought about by ultra-violet radiation and heat, a severe explosion occurred as the chromium compound was being irradiated, and perhaps overheated, in a Vycor tube attached to the mass spectrometer. Further investigation showed that the controlled thermal decomposition following ultra-violet radiation resulted in the oxidation of the ligand with the evolution of nitrogen, carbon dioxide and water vapour.

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Sealing of Aluminium Oxide Anodic Films

THE formation of boehmite on the surface of anodized aluminium by immersion in boiling water has been investigated by Hart¹. In particular, studying ammonium borate anodic films on aluminium, he found by electron diffraction that the pseudo-amorphous structure of the surface layer was converted to boehmite, but that this change did not occur when the detached film was immersed in boiling water. Hart therefore suggested that the mechanism of the process was an outward movement of aluminium ions from the metal, passing through the oxide layer, and combining with hydroxyl ions to form boehmite in the pores of the existing oxide layer and on the outer surface.

Similar experimental results were also reported by Kerr², again after an electron-diffraction study of ammonium borate anodized films, when it was concluded that boehmite was formed only in the presence of metallic aluminium. Spooner³, however, making an electron-diffraction study of sulphuric acid anodic coatings, found that conversion of the aluminium oxide layer to boehmite occurred in films attached to, and detached from, their aluminium bases, which confirmed that in this case the process was one of hydration of the existing oxide and did not require the presence of metallic aluminium.

It may therefore be of interest to report that working with boric acid anodic coatings we also found that conversion to boehmite occurs in films, detached from their aluminium bases, when sealed in boiling water.

Aluminium foil (99.99 per cent) of 0.012 mm. thickness was anodized at 550 V. in boric acid solution. After sealing by immersion in boiling water, the anodic film was stripped from the metal using saturated mercuric chloride solution and washed in distilled water. The resulting film was too thick to allow an electron diffraction transmission pattern to be obtained; but it was found that an outer layer, ~100 Å. in thickness, could be obtained by evaporating a thin coating of aluminium on to the sealed film while still attached to the foil, and freeing this with a chromic-phosphoric acid solution⁴. An electron diffraction transmission pattern of this exceedingly thin aluminium-supported layer showed the presence of boehmite, the reflexions being com-

pared and found to be in good agreement with published results⁵.

An X-ray powder diffraction pattern of the whole film as separated by mercuric chloride, which was carefully wrapped on to a glass fibre, showed a predominantly γ -alumina composition with evidence of only a small amount of boehmite present. In a further study the film was removed from an anodized but unsealed foil, using the mercuric chloride technique, and boiled in distilled water for 20 min. An X-ray powder specimen was prepared as before, and the resultant diffraction pattern again showed the presence of boehmite and γ -alumina. In this case, however, a study of the relative intensities of the X-ray reflexions showed that an appreciable amount of γ -alumina had been converted to boehmite. This suggested that the greater surface area of the detached film had contributed significantly to the extent of hydration.

These results show that water-sealing of boric acid anodic films is a hydration process and occurs independently of the presence of metallic aluminium.

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Electrode Potentials and Compound Formation in the Palladium-Platinum-Hydrogen System

ELECTRODE potentials and electrical resistances of palladium wires alloyed with 5, 10, 20 and 25 per cent platinum have been measured while they absorbed hydrogen (dissolved under a pressure of 1 atm.) from aqueous solutions of 2 N sulphuric acid. Apparatus and techniques were similar to those previously employed¹. The wires were 0.027 cm. in diameter and were prepared by directly alloying the metals. The measurements reported were made on 15-cm. lengths of wire in the cold-drawn condition, which were preactivated by discharging oxygen at their surfaces by electrolysis^{2,3}. Potentials differing from zero were positive with respect to a platinum hydrogen electrode in the same solution.

Selected experiments using the method of vacuum outgassing^{1,3} indicated that the relative resistance (R/R_0) of the alloys was proportional to the hydrogen content, as has been established in detail for the palladium-hydrogen system⁴. Plots of R/R_0 against electrode potential for the alloys with 5, 10 and 20 per cent platinum revealed that they behaved like palladium, showing the general relationship illustrated by I in Fig. 1a, although with increasing platinum content the step BC showed increasing departure from the horizontal with respect to the R/R_0 axis. The behaviour of the 25 per cent platinum alloy is shown by II in Fig. 1a. As was found for the palladium-hydrogen system, the equilibrium potential (D in Fig. 1a) is zero with respect to the platinum-hydrogen electrode.