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TWO ZONES OF "IMPURITIES" OBSERVED AFTER PROLONGED ELECTROLYSIS OF DEUTERIUM ON PALLADIUM

J. O'M. Bockris and Z. Minevski, Department of Chemistry, Texas A&M University, College Station, TX 77843-3255

In experiments carried out recently in which hydrogen was electrolyzed from water in contact with a palladium electrode, the concentration and depth of impurities were measured as a function of electrolysis time. It was found that after 3 weeks, two different sets of impurities could be observed, one set within 50Å of the surface and another set different chemical spaces, about 1μ inside the metal.

Thus, the XPS measurements (Table 1) showed materials which correspond roughly to elements found in the solution after three weeks by ICP measurements (see Fig. 1). The concentration of platinum found in the palladium, for example, rose to a maximum at about 8-11 Å from the surface and decreased exponentially so that by 50Å from the surface no further platinum could be seen. Similar concentration profiles were observed for silicon and zinc.

From these XPS determinations it can be concluded that the impurities measured, which spread no further than a few tens of Å inside the Pd, originated in the solution by means of electrochemical deposition or adsorption on the electrode surface and subsequent diffusion into the electrode.

In confirmation of this, the information in Fig. (1) shows materials in solution as a function of the time of electrolysis.

Energy dispersive spectroscopy was also used to seek material which might be further into the metal. In EDS, the maximum intensity of the signal arises from a greater depth than that of XPS (L). In fact, for palladium, for a 20 kV X-ray beam, the escape depth maximizes at about 2m inside the palladium and the signals found both after one hour's electrolysis, and then after three week's electrolysis, are shown in Figures 2 and 3, respectively. It is seen that new nuclei have appeared at this depth in the presence of hydrogen at high fugacity (overpotential more than 0.5 volts).

The concentration of the new atoms is in the range 1-10 atomic percent. The materials shown in Table 2 have no relationship to the impurities in the solution (further, diffusion of the materials cited in Figures 2 and 3, into depth of ~1μ could not occur in three weeks).

Thus, protons at high fugacity in Palladium may give rise to new nuclei (“proton capture”). These results are similar to those of Karabut, Kucherov and Sammatinova (2) who found many new materials formed in palladium after electrolysis. Similar results, according to T. Passed (3), were found by K. Wolf after examination of his palladium electrodes subject to

similar electrolysis (radioactivity was also observed in this study). T. Ohmori and Enyo (4) have reported the formation of new iron nuclei in gold and palladium cathodes; J. Dash (5) has described the formation of Au and Ag on Pd electrodes after hydrogen evolutions. Correspondingly, Bush and Eagleton (6) have observed new strontium during the electrolysis of platinum in a rubidium containing solution; whilst Notoya (7) has found that new Ca++ forms K+ in solution. Such results are, to some degree, rationalized by models of the excited nucleus discussed by Greiner et al (8).

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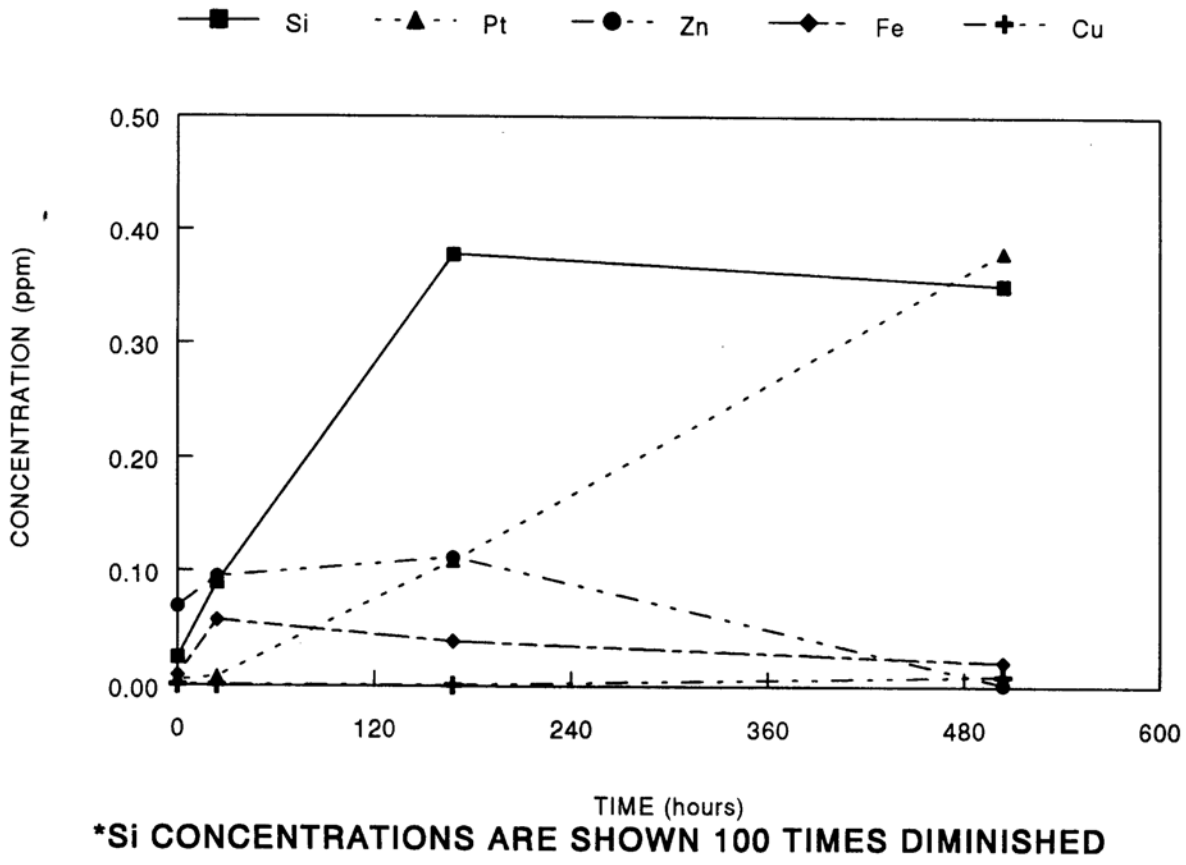

Figure 1. Concentration time dependence of Pt, Zn, Fe, Cu and Si from ICP measurements during a 3-week period

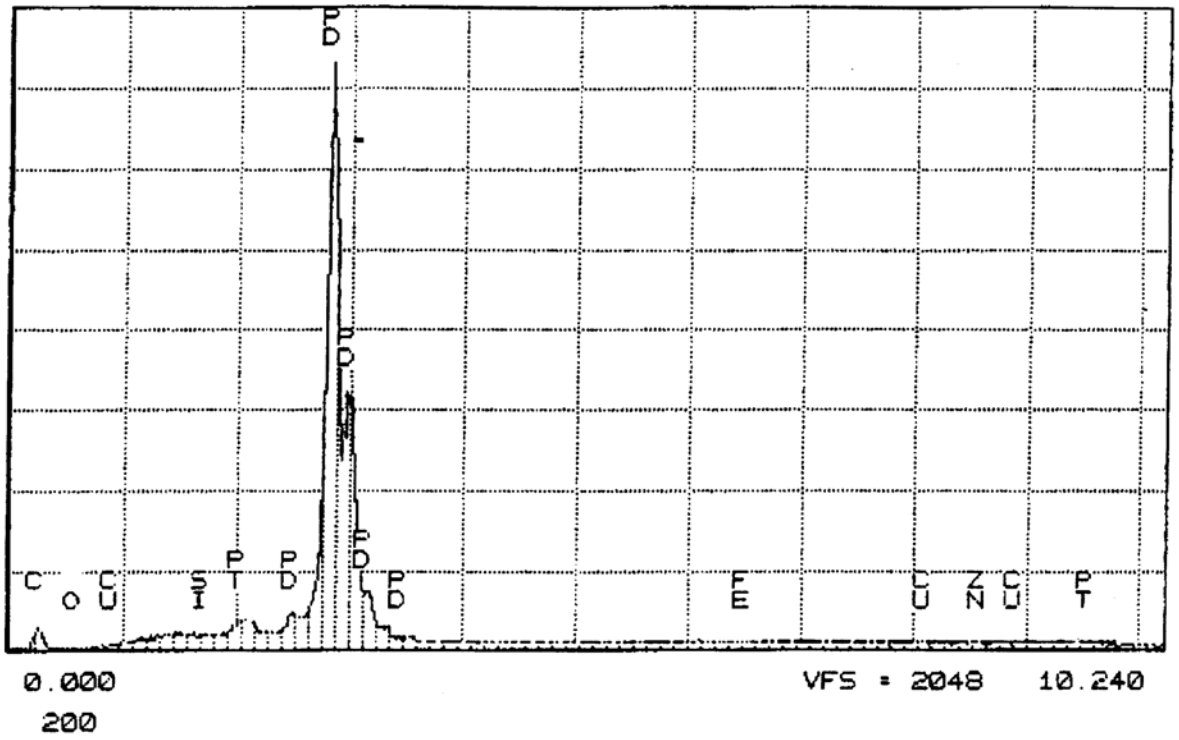

Figure 2. EDS scan of Pd electrode after one hour of electrolysis.

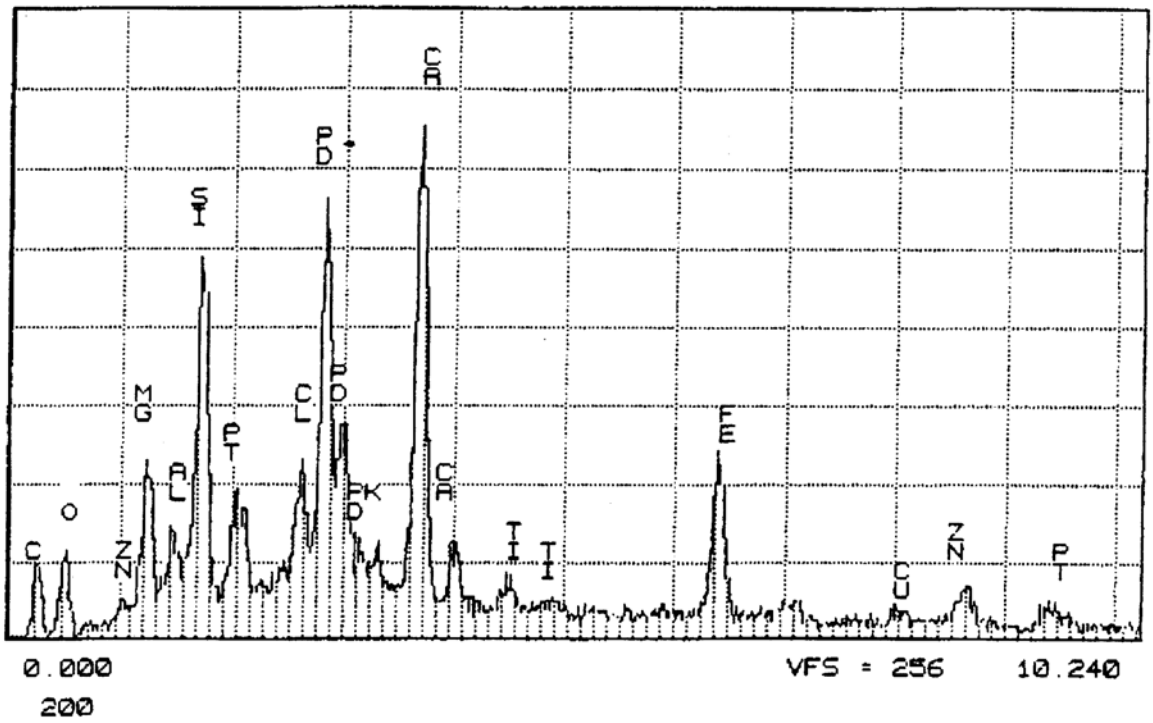

Figure 3. EDS scan of Pd electrode after three weeks of electrolysis.

DEPTH (Å)	ELEMENTS ON THE ELECTRODE SURFACE (ATOMIC %)			
	Pd	**Pt**	**Si**	**Zn**
5.0	0.39	49.16	20.02	30.42
7.5		78.93	13.98	7.08
10.0	0.34	78.38	11.35	9.94
12.5	6.90	65.53	12.38	15.19
15.0	40.99	40.19	6.40	12.41
25.0	84.36	5.63	1.87	8.13
35.0	93.16	2.12	2.36	2.36
45.0	96.87	1.54	0.51	1.07
55.0	96.81	1.06	1.06	1.06

Table 1. Atomic weight percent of elements found on the electrode surface after three weeks of electrolysis (XPS).

ELEMENT	ELEMENTS AT 1μm DEPTH (ATOMIC %)		
	VIRGIN Pd		ELECTROLYZED Pd 3 weeks/EDS
	JOHNSON MATTHEY/ICP	PRESENT WORK/ED	
Mg	$<1.0 * 10^{-4}$	- *	6.7 ± 1.0
Ag	$<1.0 * 10^{-4}$	- *	1.9 ± 1.0
Si	$8.0 * 10^{-4}$	- *	10.2 ± 1.0
Cl	-	- *	3.0 ± 1.0
K	$9.0 * 10^{-4}$	- *	1.1± 1.0
Ca	$3.5 * 10^{-3}$	- *	19.9 ± 1.0
Ti	$<3.0 * 10^{-4}$	- *	1.6 ± 1.0
Fe	$<4.0 * 10^{-4}$	- *	10.5 ± 1.0
Cu	$4.5 * 10^{-3}$	- *	1.9 ± 1.0
Zn	$<4.0 * 10^{-4}$	- *	4.2 ± 1.0
Pt	$1.0 * 10^{-2}$	- *	7.1 ± 1.0
Pd	99.80	98.10 ± 1.0	31.9 ± 1.0

* Lower than measuring limit of EDS

Table 2. Concentrations of impurities (Atomic weight percent) found in virgin Pd after three weeks of electrolysis (EDS)