METHOD OF SEPARATE DETERMINATION OF HYDROGEN IN AN ELECTROLYTIC COATING AND BASE

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When studying hydrogen absorption and hydrogen embrittlement of steel arising upon electrodeposition of zinc and cadmium, it is important to have data on the quantity of hydrogen absorbed not by the coating but by the base metal, since this hydrogen affects its mechanical properties.

In this work we attempted to develop a method of separate determination of hydrogen absorbed by the galvanic coating and the base.

The main difficulty in solving the problem stated obviously consists in selecting such a method of removing the cadmium or zinc coating in which the hydrogen content in the base (steel) does not change. We can assume beforehand that the removal of the coating by mechanical and electrochemical methods or by etching it off in acids lead to a change of the hydrogen content in the base.

Chemical etching of cadmium from steel can be done in a solution of ammonium nitrate. Cadmium is etched off rather slowly in the 15% ammonium nitrate solution usually used in industrial practice. In our work we used a 30-40% aqueous solution of NH_4NO_3 for etching off the cadmium; for removing zinc we used a solution of the following composition (g/liter): NH_4NO_3 100; $NH_4OH_{conc.}$ 550; triethanolamine 50.

A lot of cylindrical specimens made of steel U-8A, 30 mm long and 5 mm in diameter, after degreasing with French chalk and washing in distilled water, was polarized cathodically (hydrogen adsorbed) in a 0.1 N H_2SO_4 solution for 1 h at a current density of 25 mA/cm^2 and temperature of 25°. The anode was a platinum plate. Some of these specimens immediately after cathodic polarization were washed in water and acetone and heated in vacuum (10^{-6} mm Hg) at 400°C to determine the quantity of absorbed hydrogen. Another portion of the specimens that absorbed hydrogen was covered with a 10 μ thick cadmium deposit in an electrolyte containing (g/liter): $CdSO_4 \cdot 8/3H_2O$ 380; H_3BO_3 15; carpenter's glue 1 g/liter at D_c 5 A/dm^2 and temperature 35° or with a 10 μ thick zinc deposit in an electrolyte containing (g/liter): $ZnSO_4 \cdot 7H_2O$ 500; Na_2SO_3 50; $Al_2(SO_4)_3$ 30; temperature 25° and $D_c = 8$ A/dm^2, with agitation. The yield of metal in both cases was 100%, and therefore we can assume that additional hydrogen absorption of steel does not occur during electrodeposition of cadmium and zinc.

After application of the coating, washing in water, and its chemical removal, we determined the hydrogen content in the specimens by the vacuum heating method.

Determination of the quantity of hydrogen after cathodic polarization in sulfuric acid showed that the average hydrogen content in the specimens was 2.55 ± 0.05 cm^3 per 100 g steel. After cathodic polarization, cadmium plating, and removal of the deposit the average hydrogen content was 2.45 ± 0.05 cm^3. Similar results were obtained upon removing the zinc coating.

These experiments showed that removal of cadmium or zinc from steel in the solution indicated above does not change noticeably the hydrogen content in the base.

This method permits determining with sufficient accuracy the content of hydrogen absorbed upon electrodeposition of metal by the coating and base. Thus, upon deposition of a cadmium layer 10 μ thick on steel U-8A from a cyanide electrolyte containing (g/liter): CdO 45; NaCN 120; Na_2SO_4 50; $NiSO_4 \cdot 7H_2O$ 1.5 at $D_c = 1.75$ A/dm^2, the total quantity of hydrogen in the base and coating was 2.5 cm^3 hydrogen per 100 g metal

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(the sum of the weight of the specimen and deposit). On determining hydrogen in the base after removing the coating we obtained only 1 cm^3 hydrogen per 100 g metal. As we see from these data, the determination of total hydrogen gives a result overstated by a factor of 2.5 in comparison with the true quantity of hydrogen absorbed by the base metal upon electrodeposition of cadmium.