DISTRIBUTION OF CATHODICALLY PRODUCED HYDROGEN IN STEEL SURFACE LAYERS

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Studies of the mechanism of permeation of cathodically polarized metals by hydrogen and the elucidation of some specific features of hydrogen embrittlement of steel require a certain knowledge of the qualitative and quantitative variation of the hydrogen concentration in specimens undergoing electrolytic charging.

It is known [1-3] that hydrogen, in the case of cathodically polarized steel, tends to concentrate in the metal surface layers; the initial ductility of hydrogen-embrittled steel can be restored by removing these layers (mechanically or chemically). The indirect methods of determining the thickness of hydrogen-charged layers, used in the studies cited above, give no information on the quantitative distribution of hydrogen concentration at various distances from the metal surface. Some information of this nature, relating to certain specific conditions of current density and polarization time, has been obtained with the aid of the anodic dissolution method [4-6].

The aim of the present investigation was to study the variation in hydrogen concentration distribution in steel surface layers as a function of the duration (30 to 540 min) of hydrogen charging.

Experimental Method and Apparatus

The hydrogen distribution in cathodically-charged steel specimens was determined by anodic dissolution of consecutive surface layers with the aid of a modified version of an apparatus designed by Beloglazov [5] (Fig. 1).

The electrical contact with the steel specimen to be analyzed 1 was made through a threaded copper bolt 7 passing through a stopper 8 into a glass U-tube 9, which housed the lead connecting the specimen to the current source; this construction was found to be quite convenient and reliable in use. A copper-sheet cylinder 3, placed in a glass vessel 4, served as cathode. To ensure uniform (in both longitudinal and radial directions) dissolution of the steel, the specimen was placed immediately underneath the funnel of a small buret 5 in which hydrogen was collected. The small angle of taper of the funnel and the application of an electromagnetically operated vibrator 6 ensured that all the hydrogen evolved was collected in the measuring part of the buret.

A porous ceramic vessel 2 (0.75 l capacity, 3 mm wall thickness) was used as the diaphragm separating the anodic and cathodic compartments (E. B. Goyan was in charge of the preparation of the ceramic diaphragms).

Since diaphragms of this type have to meet exacting requirements, they were made by a method developed by Berkman [7] from a mix of the following composition (%): kaolin - 1.5; clay - 20; quartz - 8; alumina - 19; kaolin grog - 38; gas black - 3; starch - 2; waterglass - 0.4. Vessels made in this way met all the specifications for diaphragms used in acid and alkaline electrolytes and had the following properties: water permeability coefficient - $0.81 \times 10^{-5} \text{m}^3 \text{cm}/\text{m}^2 \text{mm} \text{H}_2\text{O} \text{hr}$; the maximum pore diameter - 0.86-88 $\mu$; total and open porosity - approx. 50%; electrical resistance coefficient, i.e., the ratio of the resistance of an electrolyte-saturated diaphragm to the resistance of an electrolyte layer of equal thickness - 2.5-2.6; and sufficiently high mechanical strength. The diaphragms effectively separated the anodic and cathodic compartments and prevented mixing of the anodic and cathodic gases.

The anodic dissolution of steel specimens was carried out in the electrolyte recommended in [4] (150 g NaCl + 25 g KNaC_4H_4O_6 in 1 l H_2O), at an anodic current density of $D_a = 30 \text{a/dm}^2$. Both the temperature and pH of the electrolyte were controlled. At the potential required to give the experimental current density the possibility of oxygen evolution and oxidation of the anode was precluded. Numerous check analyses of the anodic gas showed that the oxygen and nitrogen concentration did not exceed the magnitude of the experimental error.*

*V. P. Shportko participated in the analytical work.
It may therefore be assumed that under our experimental conditions all the hydrogen present in the dissolved steel layers was collected in the measuring buret.

Mark 20 steel (0.18% C, 0.47% Mn, 0.17% Si), annealed at 860–870°C, was used as the experimental material. Hydrogen was introduced into the steel by cathodic polarization in a 20% H₂SO₄ solution (pure grade), a thermostat temperature-regulated chamber being used to maintain the electrolyte temperature at 18°C. To ensure a high ion concentration on the polarized specimen surface, a high cathodic current density (Dₐ = 50 a/dm²) was employed.

In a series of preliminary experiments calibration curves were constructed showing the thickness and weight of the dissolved steel layers as a function of the anodic dissolution time. The thickness of the dissolved layers was determined by measuring the specimen diameter with the aid of an instrument microscope, which gave results correct to ± 1 micron. These calibration curves enabled us to determine the hydrogen concentration in consecutive surface layers of a specimen without interrupting the anodic dissolution process.

The quantities of hydrogen evolved during anodic dissolution of specimens in the untreated condition were so small that a layer 200 μ thick had to be dissolved to measure the average hydrogen content (1.4 ml/100 g) in this material.