Summary: Section 3

Electrochemical Sensor for the Determination of Hydrogen in Metals by Potential Methods

This method employs a three-part electrode consisting of palladium hydride as a reference electrode, a solid proton conducting electrolyte (hydrogen uranyl phosphate tetrahydrate), and a thin layer of palladium sputtered over the end of the electrolyte.

The determination of hydrogen is based on the increase of voltage from the electrode when the electrode is in contact with the sample material through a contact liquid. The localized hydrogen content is calculated by finding the net voltage increase attributable to hydrogen activity.

The electrochemical sensor offers long-term stability from a few minutes to a few months. The method has been applied to hydrogen problems in the petrochemical, welding, and galvanic industries.

The Barnacle Electrode Method to Determine Diffusible Hydrogen in Steel

The Barnacle Electrode Method is an adaptation of the electrochemical permeation method for hydrogen diffusion. In this method the sample material is the anode of the circuit, the cathode is a nickel, nickel oxide plate enclosed in a chamber filled with a 0.2 *M* sodium hydroxide solution. Mobile hydrogen leaving the surface of the sample is converted to moisture by the sodium hydroxide at the nickel, nickel oxide electrode interface. The hydrogen in the form of water causes a current shift in the electrode relative to the surface mobile hydrogen concentration.

The analysis method is a qualitative method with applications for monitoring hydrogen uptake in material processing or corrosion.

Determination of Hydrogen Content in Molten Steel Using an Inert Gas Bubbling Probe

The Inert Gas Bubbling Method for determining hydrogen offers an on-line determination directly from the molten bath. Experimental data has been accumulated by bubbling argon, nitrogen, and hydrogen mixtures at set flow rates through a precisely sized nozzle immersed at a precise depth into 900 g of molten iron. The gas bubble collector was immersed above the nozzle. Nozzle size, gas flow rate, and positioning of the nozzle and collector tube are extremely important to this method. The gas bubbles were sensed by a mass spectrometer. The experimental data was obtained by calculating the frequency of hydrogen bubble formation using Sieverts law. There was a good correlation of the determined hydrogen contents to the theoretical injected volumes.

A Study of the Effect of Voids on the Hydrogen Diffusion Through ESR Steel

Samples of ESR steel were annealed at 900°C for 1 h. After annealing, the grain size was determined to be 9 ASMS. The samples were then attacked with hydrogen at 600°C for time periods ranging from 120 to 432 h. Afterward the samples were polished and micrographically examined.

By this method it was possible to count the number of voids in the samples and determine the average size of the voids. The volume of the voids was calculated based on the average size. The diffusion of hydrogen and the level of hydrogen contained in the voids was measured electrochemically and correlated to the theoretical volume. Through this study it was found that microvoids will retard the evolution of hydrogen but hydrogen is not permanently retained in microvoids. The diffusivity of hydrogen is relative to microvoid volume. As microvoid volume increases, the diffusion rate decreases.

The Simultaneous Determination of Diffused and Residual Hydrogen

Comparison studies of sampling with a dual-chambered metal sampler, a diffused hydrogen sample carrier, and standard hydrogen studies have been made. Correlative data are obtained using any of the three sampling methods.

Though values accumulated with all three sampling methods are correlative, sampling and sample handling with the metal tube sampler is not totally foolproof. Strict guidelines for sampling immersion time and sampler stripping procedures must be followed. Either of the dual hydrogen sampling methods used appears to provide a more consistent and less tedious method of hydrogen sampling and sample handling than the present standard method.

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