Standards Topics

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Hydrogen Embrittlement Revisited: Part II

This month we continue where we left off in July's column, and elaborate on the details of the hydrogen embrittlement standards initially listed in February's "Standards Topics."

Cleaners & Plating Processes

One of the first test methods concerning hydrogen embrittlement prepared by ASTM Committee F07 on Aerospace and Aircraft was F 519, initially published in 1977. F 519, Mechanical Hydrogen Embrittlement Testing of Plating Processes and Aircraft Maintenance Chemicals, uses specimens fabricated from AISI 4340 steel, an alloy chosen because of its very high susceptibility to hydrogen embrittlement. Five specimen-type geometries are described, four of which are notched.

For testing cleaners and paint strippers, specimens are prepared by degreasing and cleaning by abrasive blasting. The specimens are rinsed free of any abrasive grit, and cadmium-plated from a low-embrittlement cadmium cyanide solution. Following plating, the specimens are chromated, rinsed, dried and baked at 375 °F ± 25 degrees, for 23 hr.

Three specimens are assembled and loaded, according to the requirements of their specimen type, and placed in the solution to be tested for 150 hr. If two or more specimens fail within that time, the stripper or cleaner is considered embrittling.

Plating processes are tested by substituting them for the cadmium cyanide bath in the procedure described previously. The plated specimens are placed under load, according to their specimen type, and held for 200 hr. If two or more of the plated specimens fail within the 200-hr period, the process is considered embrittling.

This method has found wide use in the aerospace and other high-technology industries. The ISO/TC 107 Working Group on Hydrogen Embrittlement has requested that the U.S. submit this method for balloting as a New Work Item proposal.

Cadmium Plating Processes

ASTM F 326, Electronic Hydrogen Embrittlement Test for Cadmium-Electroplating Processes, was issued in 1977. The method describes an electronic hydrogen detection instrument procedure for measurement of plating permeability to hydrogen. A metal-shelled vacuum probe is used as an ion gauge to evaluate electrodeposited cadmium characteristics relative to hydrogen permeation.

After calibration, a section of the probe shell is electroplated at the lowest current density encountered in the cadmium electroplating process. During the subsequent baking of the probe at a closely controlled temperature, the probe ion current, proportional to hydrogen pressure, is recorded as a function of time. From these data and calibration data of the probe, a number related to the porosity of the electroplated metal relative to hydrogen is obtained. The method is used to control cadmium-plating processes where the porosity of the plating relative to hydrogen is critical, such as cadmium on high-strength steel.

The method initially was developed at the Boeing Airplane Company, and at last report continues to be successfully used by Boeing and its suppliers, as well as a number of other aircraft manufacturers.

Diffusible Hydrogen in Steel

ASTM F 1113, Electrochemical Measurement of Diffusible Hydrogen in Steel (Barnacle Electrode), was issued in 1987. The method originally was developed at the U.S. Naval Air Development Center, and has been accepted as a New Work Item for development into an International Standard by ISO/TC 107.

The method is limited to carbon and alloy steels, excluding austenitic stainless steels. It is also geometry-limited to flat specimens or actual parts to which the cell can be attached, or to slightly curved specimens and parts, as long as the gasket defines a reproducible area.

The procedure involves removing the plating from the test area, placing the cell over the bare substrate and clamping a Ni/NiO electrode at the center of the cell. The Ni/NiO electrode becomes the cathode of the system, with the steel being the anode. An electrometer and recorder are made part of the circuit. The cell is filled with 0.2 M NaOH and, within one min of filling the cell, the cell switch and timer are turned on and recorded for 30 min. The current is calculated from the voltage drop across a standard resistor using Ohm's law. A measurement higher than background indicates the presence of diffusible hydrogen; higher current densities indicate higher hydrogen concentrations.

The method is particularly applicable as a quality control tool for processing (*e.g.*, to monitor plating and baking). It is applicable to large parts, and also to small parts, such as fasteners.

Substrate Evaluation

ASTM F 1459, Determination of the Susceptibility of Metallic Materials to Gaseous Hydrogen Embrittlement, was issued in 1993. The method is a departure from the mainstream of hydrogen embrittlement testing in that it evaluates the susceptibility of the base material to hydrogen damage. The test can be used to select materials with no or a very low sensitivity to hydrogen embrittlement as a means of avoiding the problem. Its results can also alert the user to the need for close processing controls when the test shows high susceptibility.

The test was initially developed in France in 1969, and had its first English publication in ASTM STP 543 in 1974. The method has been extensively studied and has been the subject of numerous publications. It has been accepted as a New Work Item for development into an International Standard by ISO/TC 107.

A thin disk of the metallic material to be tested is introduced as a membrane in a test cell and clamped in place. The cell is pressurized with helium gas until the disk bursts. The fracture of the disk will be caused only by mechanical overload and, because of the inert nature of helium, no physical-chemical secondary action is involved.

An identical disk of the same material is introduced into the same test cell and submitted to hydrogen pressure until that disk bursts. Metallic materials susceptible to hydrogen embrittlement will burst at a pressure below the helium burst pressure. Materials not susceptible will fracture under a pressure identical to the helium pressure.

The method has very good reproducibility, typically within ±5 percent, and has been acepted as a New Work Item for development into an ISO standard by ISO/TC 107.

Inclined Wedge Test

ASTM B 839 (ISO/DIS 10537), Residual Embrittlement in Metallic Coated, Externally Threaded Articles, Fasteners, and Rods: Inclined Wedge Method, was initially developed at the IBM Corporation. The work started in 1972 as an effort to refine the Flat Plate test of MIL-STD-1312. Although the fastener lots passed the embrittlement test, they were plagued with failures. The best repeatability obtained with the flat plate test was ±30 percent. The underside of the fastener heads were eventually examined, and showed out-of-flatness as a wide-spread condition. It was immediately recognized that this out-of-flatness created a wedge-like condition, and that the wedge angle varied as to the degree of out-of-flatness. Experimentation with wedge angles showed that the best repeatability—about five percent—was obtained with a six-degree wedge.

The method uses the Sampling Plan for Destructive Tests found in ASTM B 602 (ISO 4519), and this type of plan is not found in MIL-STD-105. This plan uses a sample size of 32 pieces for lots of 1,201–35,000 parts, rather than sample sizes of 125–315 pieces for that range of parts.

The test pieces are placed in close-toleranced holes in the wedge plate and torqued to their seating torque with nuts. Varying lengths are accommodated with filler pieces. The close tolerance of the hole is to prevent canting of the fastener in the hole with its consequent bending moment.

The torqued test pieces are held for a set time—48, 96, or 200 hr. The test time is specified by the purchaser, depending on the confidence level required. The greater the test hours used, the higher the confidence level that can be assumed. Unfortunately, there is no test that can guarantee complete freedom from hydrogen embrittlement.

The test can be used for lot acceptance or for process control, again depending on the purchaser's need for confidence that the fasteners are free of any residual embrittlement. ❑