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Determination of diffusible hydrogen in deposited metal

熔敷金属中扩散氢测定方法

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1 Scope

The standard lays down the mercury method and the thermal conductivity method as the basic methods for determining the diffusible hydrogen content of deposited metal.

It applies to the determination of the diffusible hydrogen content of martensitic, bainitic and ferritic weld metal produced by shielded metal arc welding with covered electrodes, submerged arc welding, gas shielded arc welding with solid wire and flux cored arc welding.

2 Overview

A single linear weld bead is deposited on the test assembly with the welding consumable by an arc welding process. After treatment, the diffusible hydrogen is collected under given conditions of temperature and time, and the collected hydrogen is measured from the volume of mercury it displaces (mercury method) or from the thermal conductivity (thermal conductivity method). The result is converted into the volume of diffusible hydrogen per 100 g of deposited metal at standard conditions (0 °C, 101.325 kPa), expressed in mL/100 g.

In this standard the mercury method collects the hydrogen for several days at room temperature or for 72 h at 45 °C, and applies to type B and type C test blocks.

The thermal conductivity method uses a thermal conductivity detector and is divided into carrier gas hot extraction and hydrogen collection; it normally employs gas chromatography and applies to type A, type B and type C test blocks. In carrier gas hot extraction the specimen is heated to a higher temperature, up to 400 °C, and collection and analysis run continuously so that the diffusible hydrogen is determined quickly; in the collection variant

the specimen is heated to a moderate temperature, generally 45 °C to 150 °C, to collect the diffusible hydrogen, which is analysed once collection has ended.

3 Preparation before welding

3.1.1 The test blocks shall be of non-alloy killed steel with C not greater than 0.18 % and S not greater than 0.02 %.

3.1.2 The test assembly is made up of a run-on plate, a centre test block and a run-off plate, with the dimensions shown in Figure 1. Each test series includes at least 3 test assemblies; 4 assemblies are used for arbitration.

3.1.3 Before machining, the test assembly shall be given a hydrogen removal treatment at (650 $\pm$ 10) °C held for 1 h and cooled in the furnace; the treatment may also be carried out after machining, in vacuum or in dry inert gas. If dehydrogenation is done in air, the oxide scale shall be removed. The test blocks shall be kept under dry conditions.

3.1.4 The centre test block and the run-on and run-off plates shall be surface ground in one operation, so that the width is uniform and the surfaces smooth and clean, and the mating faces shall be square, to make sure the assembly is firmly clamped in the copper fixture.

3.1.5 The back face of the centre test block shall be stamped with an identification mark and the block weighed, the mass being recorded as m_1 , to 0.1 g for a type A test block and to 0.01 g for a type B or type C test block.

Figure 1 gives the size of the three test assemblies in millimetres: type A, centre block length 80, width 25, thickness 12, run-on and run-off plate length not less than 50; type B, 30, 15, 10 and not less than 50; type C, 15, 30, 10 and not less than 50. The footnotes to the figure state that the heat input of any welding process shall not exceed 3 kJ/mm and give the formula for it, that the tolerance on the width is $\pm$ 0.25 mm, that the type A centre block may be replaced by two blocks 40 mm long, and that the run-on and run-off plate length is not less than 25 mm for shielded metal arc welding. The figure also carries a grid showing which welding processes and which analysis methods apply to each type of block; the tick marks in that grid could not be read with certainty on the scan and are not reproduced here.

3.2.1 For covered electrodes, when the test is made for product classification, the electrode diameter and the welding parameters shall be those used to prepare the mechanical test specimens. Where nothing is laid down, a 4.0 mm electrode shall be used and the welding current shall be 15 A below the maximum current recommended by the manufacturer, or 90 % of that maximum, held within $\pm$ 10 A. The welding speed is adjusted so that (10 $\pm$ 1.5) g of deposited metal is obtained on a type A centre block or (4 $\pm$ 0.5) g on a type B centre block, which normally corresponds to 12 mm to 13 mm of electrode consumed for every 10 mm of weld.

3.2.1 continued: when the test is made for product inspection, the welding parameters shall follow the recommendation of the manufacturer and, where nothing is laid down, the welding current shall be 90 % of the maximum current recommended by the manufacturer. The electrode covering shall be free from cracks and damage; when electrodes are tested as received, the packaging shall be checked to be properly sealed so that they do not pick up moisture before welding. Where re-baking before welding is called for, it shall be done at the temperature and for the time set by the manufacturer, the middle value being taken if the schedule gives a range. A calibrated baking oven shall be used, holding only the electrodes for the test, which shall touch neither each other nor the oven walls. After baking, the electrodes shall be cooled to room temperature in a sealed drying container and held there for at most 1 h; re-baking for reuse is not allowed.

3.2.2.1 For submerged arc wire, when the test is made for product classification the wire size and the welding parameters shall be those used to prepare the mechanical test specimens, and the welding speed is adjusted so that (21 ± 5) g of deposited metal is obtained on a type A centre block or (4 ± 0.5) g on a type C centre block. When the test is made for product inspection the welding parameters shall follow the recommendation of the manufacturer, the middle value being taken if a range is given; solid wire held at 650 °C for 1 h in vacuum or in inert gas helps to show the influence of the welding parameters, of the flux type and of the baking schedule on the hydrogen content of the weld. The welding parameters shall be chosen so that the heat input does not exceed 3 kJ/mm.

3.2.2.2 For submerged arc flux, where re-baking before welding is called for the baking schedule shall follow the product standard when the test is for classification, or the recommendation of the manufacturer when it is for inspection. A calibrated oven shall be used holding only the flux for the test, and the heap of flux in an open tray shall be not more than 15 mm high. After baking the flux shall be cooled to room temperature and used at once, or cooled and held in a sealed container; used flux shall not be reused.

3.2.3.1 For gas shielded solid wire and gas shielded or self-shielded flux cored wire, when the test is made for product classification the welding parameters, stick-out included, shall be those used to prepare the mechanical test specimens, and the welding speed is adjusted so that (10 ± 1.5) g of deposited metal is obtained on a type A centre block or (4 ± 0.5) g on a type B centre block. When the test is made for product inspection the welding parameters shall follow the recommendation of the manufacturer, the middle value being taken if a range is given.

3.2.3.2 For the shielding gas, when the test is made for product classification the gas and the flow rate shall meet the requirements of the product standard; when it is made for product inspection they shall follow the recommendation of the manufacturer, and if required the gas may be dried to remove moisture, in which case its moisture content shall be measured and recorded.

3.3.1 The copper fixture shown as an example in Figure 2 is used to align and clamp the

test assembly. It applies a uniform clamping force so as to give good contact and good heat removal, and it releases the assembly quickly once the arc has been extinguished. A water cooling channel helps to complete each test series quickly, and the temperature of the cooling water shall be controlled so that no condensation forms on the faces where the fixture and the assembly meet.

3.3.2 A thin copper sheet about 1 mm thick, of suitable length and height, should be placed between the test assembly and the copper fixture to prevent the fixture from being burned; in submerged arc welding the sheet shall be high enough to hold the flux. After annealing and water quenching, the thin copper sheet may be freed of oxide in dilute nitric acid (10 %), then washed with distilled water and dried.

4 Preparation of the test specimen

4.1.1 When the test is made for product classification, the absolute humidity of the surroundings during welding shall be at least 3 g of water vapour per 1 kg of dry air, which corresponds to a relative humidity of about 54 % or more at 5 °C, about 38 % or more at 10 °C and about 20 % or more at 20 °C. A sling hygrometer or another calibrated instrument shall be used for the measurement.

4.1.2 The flux is heaped to a thickness of 25 mm, or as laid down by the manufacturer; the specified heap thickness may be reached by adjusting the height of the thin copper sheet.

4.1.3 The outer layer of the wire shall be discarded. Before welding, the wire feed rolls shall be cleaned and the gas remaining in the torch and in the wire guide shall be purged, together with any moisture, by repeated gas flow. In continuous welding the cleaning step may be omitted.

4.1.4 The test assembly shall be cleaned in acetone or alcohol and then arranged in the copper fixture in the length direction, run-on plate, centre block and run-off plate in that order, and clamped tight. Before each weld bead is laid, the surface temperature of the copper fixture shall be above room temperature but not more than 25 °C above it.

4.1.5 The arc is struck on the run-on plate along the centre line and a single linear bead is laid. If the arc breaks during welding, the test assembly is discarded.

4.1.6 No arc may be struck to condition an electrode before welding; a short arc shall be used and kept steady. The length of deposit on the run-on plate shall not exceed 25 mm.

4.1.7 For welding consumables other than covered electrodes the length of deposit on the run-on plate is not laid down; the striking position shall be such that both the arc and the bead shape are steady by the time the centre block is reached.

4.1.8 Attention shall be paid to the deposition time; the crater on the run-off plate shall be not more than 25 mm from the centre block, as shown in Figure 1.