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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 23603-2024

Replacing GB/T 23603-2009

**Test method for titanium and titanium alloy surface
contamination**

钛及钛合金表面污染层检测方法

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3 Terms and definitions

3.1 alpha case: an alpha stabilised surface layer enriched in oxygen, nitrogen and carbon.
Note: the alpha case usually forms on exposure to air at high temperature, is usually hard and brittle, is regarded as harmful and is also called the surface contamination layer.

3.2 thickness of alpha case: the maximum depth of the continuous alpha case at the surface of titanium and titanium alloys together with the discontinuous alpha case extending from it.

Clause 4 states that the symbols defined in GB/T 38982 apply.

5 Principle

A metallographic sample retaining the original surface of the product is prepared and etched with a specific etchant so that the alpha enriched layer stands out from the matrix structure, on which basis the presence of an alpha case is judged. The thickness of an alpha case that is present is then measured under the microscope, which is the metallographic method. Alternatively the thickness of a thicker alpha case is determined through the specific relationship between the hardness of the alpha case and that of the matrix, measured with a microhardness tester, which is the microhardness method.

6 Reagents

Unless otherwise stated, only reagents of confirmed chemical purity and distilled or deionised water, referred to below as water, are used in the test.

6.1 Hydrofluoric acid, density 1.15 g/mL. 6.2 Nitric acid, density 1.40 g/mL. 6.3 Ammonium bifluoride solution, 20 g/L: weigh 20 g of ammonium bifluoride, dissolve it in 1 L of water and mix.

7 Apparatus

7.1 The metallographic microscope used for observation and measurement shall provide magnifications from 50 times to 500 times, and the microscope and the image measuring software shall have been calibrated and be within their calibration period.

7.2 The microhardness tester used for measurement shall have been calibrated and be within its calibration period.

8 Samples

8.1 Sampling. Any face of the blank to be examined shall be perpendicular to the test face, that is, the face on which an alpha case may be present; sampling shall keep the test face intact and representative. Figure 1 shows sampling for bar, tube and plate, its key naming the diameter of round products, the outside diameter and wall thickness of ring products and the thickness of flat products, all in millimetres. The recommended blank size is a test face area of less than 400 mm² and a sample height of 15 mm to 20 mm, less than the transverse dimension. Overheating during sampling shall be avoided so as not to contaminate the blank.

8.2 Sample preparation. All samples shall be mounted, without damaging the test face and without letting the alpha case spall or break away. The test face shall be kept perpendicular to the table of the mounting press, and a mounting material with good edge retention shall be chosen. After mounting, the result is examined by eye; if there is a gap between mounting material and sample the sample shall be re-mounted until its border is complete and gap-free.

8.2.2 Samples are ground and polished as required by GB/T 5168, automatically or by hand; electrolytic polishing shall not be used. Damage to the sample edge and to the original microstructure shall be prevented, and after grinding and polishing the quality shall be checked under the microscope to confirm that the border is complete and gap-free.

8.2.3 For samples used for judgement and thickness measurement by the metallographic method, the etchant and its conditions of use shall follow Table 1. Table 1 has six columns, number, etchant, etching method, time, temperature and field of application, and fixes three etchants, all used at room temperature. Etchant 1, 2 mL hydrofluoric acid (6.1) plus 100 mL water, immersion for 5 s to 10 s, applies to judgement of the alpha case in all titanium alloys. Etchant 2, 1 mL hydrofluoric acid (6.1) plus 4 mL nitric acid (6.2) plus 195 mL water,

swabbing for 2 s to 5 s, together with the ammonium bifluoride solution (6.3) by immersion for 15 s to 20 s, applies to judgement and thickness measurement of the alpha case in pure titanium and near-alpha titanium alloys. Etchant 3, 2 mL hydrofluoric acid (6.1) plus 4 mL nitric acid (6.2) plus 94 mL water, swabbing for 5 s to 10 s, together with the ammonium bifluoride solution (6.3) by immersion for 15 s to 20 s, applies to judgement and thickness measurement of the alpha case in alpha plus beta and beta titanium alloys.

Where immersion is used, the test face shall face upwards, the sample shall be fully immersed and the etchant shall be stirred gently. The effectiveness of the etchant shall be assured and may be verified on a sample known to carry an alpha case. After etching, the sample is rinsed under running clean water and dried so that no etchant remains on it.

8.2.4 Samples for the microhardness method are generally not etched; where necessary they may be swabbed with etchant 2 of Table 1 to leave the surface clean and bright.

9 Determination of the alpha case

For judgement by the metallographic method the whole edge structure of the sample is scanned and observed under the metallographic microscope at magnifications of 50 times to 500 times, and the presence of an alpha case is judged by reference to Annex A.

10 Measurement of alpha case thickness

10.1 Metallographic method. It applies to samples whose alpha case is not thinner than 0.001 mm. The whole test face is observed at 50 times to 500 times and the identified alpha case is measured with the eyepiece micrometer or on captured images with image analysis software. For a thickness below 0.030 mm the magnification shall be not less than 200 times; for 0.030 mm to 0.25 mm not less than 100 times; above 0.25 mm not less than 50 times. Where the purchaser has special requirements, these are agreed between supplier and purchaser. The three fields with the most severe alpha case are selected and measured separately, and the largest of the three measurements is taken as the result.

10.2 Microhardness method. It applies to samples whose alpha case is not thinner than 0.050 mm. Vickers hardness testing is carried out on a properly prepared sample on a Vickers microhardness tester as required by GB/T 4340.1, with a test force of 1.961 N (0.2 kgf). Testing follows Figure 2 on the test face; the face shall be perpendicular to the indenter axis and adjacent indentations shall be more than 2.5 times the indentation diagonal apart.

10.2.4 At least three hardness readings are taken well away from the alpha case, in the matrix structure, and their mean is taken as the matrix hardness of the sample. A value 50 above the matrix hardness is set as the defined hardness value; the original does not state the unit of that increment.

10.2.5 Two parallel columns of Vickers indentations perpendicular to the test face are made alternately, columns A and B, the parallel spacing S being not less than 2.5 times the

indentation diagonal. Adjacent indentations within a column shall be equally spaced. The perpendicular distance d_1 from the centre of indentation A1 to the test face is half the matrix hardness indentation diagonal plus 10 micrometres. The difference between the perpendicular distances of A1 and of B1 from the test face, d_2 minus d_1 in Figure 2, shall be not greater than 0.1 mm.

10.2.6 Measuring along the two parallel lines progressively away from the test face, when two adjacent measuring points bracket the defined hardness value the thickness D of the alpha case is obtained by linear interpolation with Formula (1). The formula reached this workstation with its division rule lost and is not reproduced. Its key reads: n , the number of Vickers hardness test points; d_n , the distance from the test face of the point exceeding HS , in millimetres; d_{n+1} , the distance from the test face of the point falling short of HS , in millimetres; H_n , the hardness measured at depth d_n ; HS , the defined hardness value; H_{n+1} , the hardness measured at depth d_{n+1} . The key of Figure 2 reads: d , the perpendicular distance from the centre of a Vickers indentation to the test face, in millimetres; A , the first column of test points; B , the second column, parallel to the first; S , the parallel distance between columns A and B .

10.2.7 At least two sets of alpha case thickness measurements are made on the test face within the examined face and the largest value is reported.

10.3 Data processing. Results are rounded as specified in GB/T 8170: for thicknesses of 0.001 mm to 0.100 mm the calculated result is kept to three decimal places, and above 0.100 mm to two decimal places.

10.4 Arbitration. Where the metallographic method and the microhardness method give results in doubt, the metallographic method is the arbitration method.

11 Test report

The test report shall contain at least the material name, grade, batch number and specification; the sample number; the number of the document; the method used; for the metallographic method, the magnification and the etchant; and the test result.