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Test method for the oxygen concentration in silicon materials - Inert gas fusion infrared detection method

硅材料中氧含量的测试 惰性气体熔融红外法

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1 Scope

This Standard specifies the method that uses inert gas melting and infrared technology to test the oxygen concentration in silicon materials.

This Standard is applicable to the tests of oxygen content in silicon single crystal and polycrystalline silicon with different conductivity types and different resistivity ranges. The test range is $2.5\times10^{15}cm^{-3}$ (0.05ppma) ~ $2.5\times10^{18}cm^{-3}$ (50ppma).

NOTE: The oxygen content in silicon materials is measured in the number of atoms per cubic centimeter.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 1557, *Test method for determining interstitial oxygen content in silicon by infrared absorption*

GB/T 14264, *Semiconductor materials - Terms and definitions*

3 Terms and definitions

For the purposes of this document, the terms and definitions defined in GB/T 14264 apply.

4 Method principle

Place the pre-weighed sample in a high-purity double-layer graphite crucible. The sample is heated and melted at high temperature under the protection of inert gas to release oxygen, nitrogen and hydrogen. The oxygen in the sample combines with the carbon in the graphite crucible to form carbon monoxide.

Nitrogen and hydrogen are released as nitrogen and hydrogen, respectively. Depending on the test instrument, the content of carbon monoxide can be directly measured by a non-dispersive infrared detector. It is also possible to oxidize carbon monoxide through a heated rare earth copper oxide catalyst under the transportation of inert gas to generate carbon dioxide, and the content of carbon dioxide is measured by infrared testing equipment. The test instrument performs blank deduction based on the content of carbon monoxide or carbon dioxide. Combined with the weight of the sample, the oxygen content in the tested sample is finally obtained.

5 Disturbing factors

5.1 Inert gas (helium or argon) is as the carrier gas in the test instrument. The impurities contained therein may adsorb the oxygen released by the silicon material sample, thereby affecting the test results. Therefore, it is recommended to use high purity gas with a purity (volume fraction) not less than 99.99% to improve the accuracy of the test. At the same time, sodium hydroxide (super-grade pure) can be used to absorb the residual carbon dioxide in the inert gas. Anhydrous magnesium perchlorate ($MgClO_4$) is used to absorb water in inert gas. Copper chips are used to absorb oxygen in the inert gas. Reduce the influence of the inert gas on the test results.

5.2 The use of single-layer graphite crucible will cause temperature fluctuations, which will affect the test results, so double-layer graphite crucibles shall be used.

5.3 The oxygen in the graphite crucible will also continue to be released during the test. Therefore, crucibles made of high-purity graphite shall be used. Confirm the purity of the graphite crucible before the test to reduce the impact on the test results.

5.4 The calibration curve is the key to the reliability of oxygen content testing. The establishment of the silicon single crystal calibration curve is based on the test results of the interstitial oxygen content in the silicon single crystal sample by the method specified in GB/T 1557. Therefore, it is necessary to ensure that the test values of the samples used to establish the calibration curve are accurate and reliable.

5.5 This method uses the test results of GB/T 1557 as the calibration curve, which indirectly reflects the interstitial oxygen content in silicon. The oxygen content value measured by this method is directly related to the gap oxygen content calibration factor selected by the calibration curve.

5.6 The detection limit of this method is related to the blank value of the instrument. The difference of the test equipment and the difference in the test process will have an impact on the detection limit.

9 Drawing of calibration curve

Use at least 2~3 silicon single crystal reference samples with traceable oxygen content and 1 blank reference sample. Test according to GB/T 1557 and 10.4.1~10.4.5 respectively. Establish a calibration curve. The oxygen content range of the reference sample shall cover the oxygen content of the sample to be tested.

10 Test steps

10.1 Instrument preparation

Check the working status of the instrument. After the instrument is fully warmed up, perform leak detection and analysis to ensure that the instrument has no air leakage.

10.2 Blank test

Use an empty double-layer graphite crucible to perform a blank test according to 10.4.2~10.4.5. Test at least 3 times in parallel. Take the average value until the standard deviation of the blank value is not more than $5\times10^{13}cm^{-3}$ (0.001ppma). Enter the average value of the blank value and establish a blank deduction.

10.3 Instrument calibration

Use 1~2 samples with known oxygen content. Test according to 10.4.1~10.4.5. Verify whether the test value conforms to the calibration curve. Set the drift calibration value according to the bias of the test value.

10.4 Determination

10.4.1 Weigh 0.3g~1.0g (to the nearest of 0.001g) of sample and put them into the loading port of the instrument.

10.4.2 Place an empty double-layer graphite crucible on the lower electrode of the instrument and raise it to the test position.

10.4.3 According to the operation process of the instrument, start the crucible exhaust process.

10.4.4 The sample is transferred to the crucible. After the in-situ degassing cycle removes the oxide layer on the surface of the sample (refer to 5.11 for details), the heating and melting process of the sample is started.

10.4.5 The oxygen in the sample is released and converted into carbon monoxide or carbon dioxide. Its content is measured by infrared test method.

10.4.6 After correcting according to the calibration curve, the instrument automatically calculates according to the input sample weight, and directly outputs the oxygen content.

11 Precision

11.1 This method combines the infrared test method specified in GB/T 1557, which is generally recognized in the industry, to draw the calibration curve. Therefore, the comparison with GB/T 1557 directly affects the accuracy of this method. In the same laboratory, select 3 silicon single wafers with a diameter of 150mm. In the diameter direction of the same silicon wafer, select a test point every 1cm. Use infrared spectrometer. Test the interstitial oxygen content in accordance with the method specified in GB/T 1557. Take the average value of the oxygen content of all gaps with a difference of not more than $5.0\times10^{16}cm^{-3}$ (1ppma) as the nominal value of the gap oxygen content of the silicon single wafer. Select the corresponding diameter range as the test sampling area of this method. Compare the test results of this method and GB/T 1557. The difference between the test results of the two methods is not more than 6% of the nominal oxygen content of the silicon wafer.

11.2 Select 18 silicon single wafers with a diameter of 150mm~200mm and an oxygen content ranging from $5.5\times10^{17}cm^{-3}$ (11ppma) to $1.1\times10^{18}cm^{-3}$ (22ppma). In the same laboratory, sample each silicon single wafer and perform 4~10 tests according to this method. The standard deviation of the test results is less than $5.5\times10^{16}cm^{-3}$ (1.1ppma).

11.3 Select 8 silicon single wafers with a diameter of 150mm~200mm and an oxygen content ranging from $5.0\times10^{17}cm^{-3}$ (10ppma) to $1.1\times10^{18}cm^{-3}$ (22ppma). Respectively sample from different laboratories in the center and adjacent areas of the same silicon wafer. Test according to this method. The difference between the test results of the two laboratories is not more than $5.0\times10^{16}cm^{-3}$ (1ppma).

12 Test report

The test report shall contain the followings:

a) Equipment name, operator's name and test date;

b) Sample name and material;

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