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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 24578-2024

Replacing GB/T 24578-2015

Test method for measuring surface metal contamination on semiconductor wafers - Total reflection X-Ray fluorescence spectroscopy

半导体晶片表面金属沾污的测定 全反射X射线荧光光谱法

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Relationship to previous standards

This document replaces GB/T 24578-2015 and GB/T 34504-2017.

3 Terms and definitions

The terms defined in GB/T 14264 apply, together with four terms defined here.

3.1 Total reflection: the phenomenon in which light travelling from an optically denser medium towards an optically thinner medium loses its refracted ray entirely and only the reflected ray remains, once the angle of incidence exceeds the critical angle. A note observes that for X-rays most solids are optically thinner than air.

3.2 Glancing angle: the angle between the plane of the sample surface and the virtual plane containing the X-rays incident on that surface. A note explains that when the X-rays strike the wafer surface at a small glancing angle they undergo total reflection and the angle of reflection equals the glancing angle.

3.3 Critical angle: the angle of incidence at which X-rays can undergo total reflection; below that glancing angle the surface under test totally reflects the incident X-rays. A note adds that if the angle of incidence is small enough the X-rays are reflected without passing through the sample, and the angle of incidence at the boundary between the refracted and reflected rays is called the critical angle.

3.4 Angle scan: the measurement of the emitted fluorescence signal as a function of the glancing angle.

4 Principle of the method

4.1 Monochromatic X-rays from an X-ray source strike the mirror surface of the wafer at a glancing angle below the critical angle and undergo total reflection. The evanescent wave passes into the wafer surface, excites the atomic levels to fluorescence levels and produces characteristic X-ray fluorescence spectra corresponding to the atomic numbers present; this energy dispersive spectrum is collected by a solid state detector such as a silicon-lithium detector. The evanescent wave decays exponentially in the process and the decay strength depends on the total electron density of the wafer surface or of its native surface oxide. A note states that for silicon wafers of all resistivity ranges the exponential decay length is about 5 nm. The principle is shown in Figure 1, whose key identifies the monochromator, the detector, the sample stage with free movement in X, Y and Z, and the X-ray source.

4.2 A calibration sample with a certified areal density of a given element above 10 to the eleventh atoms per square centimetre is used, the integrated count rate under the fluorescence peak being linear with the certified areal density. The calibration sample carries at least one element of known areal density within the measurement area; the total reflection X-ray fluorescence spectrometer analyses it and obtains the integrated fluorescence count rate corresponding to the known areal density. One or more samples are then measured under the same conditions, and the relative sensitivity factor (RSF) associated with each certified element allows the integrated fluorescence count rate of the elements in the sample under test to be determined. If the X-ray source is changed, a different set of RSF values shall be used.

5 Interfering factors

5.1 Factors of the TXRF method. The choice of glancing angle should take account of the main type of metal contamination on the sample surface, that is whether the contamination lies in the oxide layer, including the native oxide, or as particles on the surface; strictly the two types call for different glancing angles, and for particle contamination too low a glancing angle introduces a large error. Deviations in the RSF of a fluorescence line introduce a bias in the result. If the X-ray beam is diffracted by the wafer under test and the diffracted beam enters the detector, it excites metal in the detector window or in the detector itself and produces instrument peaks that affect the result. Where the fluorescence signal is not linear with the areal density of the impurity, detector dead time can arise at high total count rates. The degree of smoothing of the fluorescence curve affects the accuracy of the measured value. For elements below atomic number 16, such as sodium, magnesium and aluminium, the detection limit is higher, usually more than 10 to the eleventh atoms per square centimetre and sometimes higher still.

5.2 Factors of the TXRF equipment. The interferences known in X-ray fluorescence spectrometry all apply, including but not limited to overlapping fluorescence lines, overlap of escape peaks and sum peaks, calibration drift of the energy gain, stability of the X-ray source and instrument background peaks; the equipment is not required to correct for secondary fluorescence or matrix absorption. Common interferences from the software and the calculation can be estimated by comparing data systems, as described in A.2. The detection limit depends on atomic number, excitation energy, photon flux of the exciting X-rays, instrument background, integration time and blank value; a note adds that for constant instrument parameters the interference-free detection limit is a function of atomic number and varies by more than two orders of magnitude, and refers to Annex A for the relationship between repeatability and detection limit. If the calibration of the glancing angle is not repeatable, variability is introduced; if it is incorrect, a bias is introduced. Mechanical vibration lowers the energy resolution of the detector and may affect the detection limit. Different targets suit different elements, and whether the target is fixed or rotating also affects the detection limit. The vacuum level in the X-ray target chamber affects the accuracy. To prevent particle contamination and its accumulation, the nitrogen used to purge the main chamber shall meet the requirement for high purity nitrogen in GB/T 8979-2008, otherwise the chamber may be contaminated and the result affected.

5.3 Factors of the sample surface. Different glancing angles shall be set according to the type of metal contamination, since particles standing proud of the surface and particles inside the oxide layer call for slightly different angles; where the angle scan of the known element on the calibration sample differs from that of the element on the sample under test, for instance particle contamination on the sample against metal in the native oxide on the calibration sample, a bias in the measured value is introduced. Differences in surface roughness and waviness can also interfere: a surface that has not been chemically and

mechanically polished lowers the detection capability, shifts the value and increases the variability, and differences in roughness or waviness produced by different cleaning processes can also interfere. A note states that when polished sapphire wafers are measured with an atomic force microscope the surface roughness Ra within a 5 μm x 5 μm area is not more than 3 nm, and that the effect of the surface roughness of other materials on the semi-quantitative TXRF result has not yet been determined. A bias in the certified areal density of the element on the calibration sample leads to a bias in the areal density measured by TXRF. Surface contamination introduced during handling or measurement biases the result where the contaminating element is one of the elements measured. Non-uniform surface contamination can give different results at different positions, which matters in particular when the method is compared with others; a note states that the method is non-destructive and complements other methods, and refers to B.1 for the comparison.

6 Test conditions

Testing shall be carried out at a temperature of (23 +/- 5) °C, with a temperature fluctuation during the test of not more than 2 °C; at a relative humidity of not more than 60 percent; in air of a cleanliness class not lower than class 5 of GB/T 25915.1-2021; and with the instrument placed where there is no appreciable vibration.

7 Apparatus and data system

7.1 The TXRF spectrometer shall provide a monochromatic X-ray source, a handling device for the sample under test, an energy dispersive X-ray detector, software for background subtraction, peak integration, RSF calculation and analysis, developed by the instrument manufacturer and stored in the instrument computer program, and an argon-free analysis environment such as a vacuum of 1.33 Pa or helium. It shall provide a method for calibrating the glancing angle and an attenuation routine for subtracting escape peaks so that the escape peak signal can be removed.

7.2 The data system for the calibration samples shall use statistical tools to study and confirm the repeatability of the instrument. Repeatability and detection limit are covered in A.1.

8 Samples

The sample surface shall be flat and clean. The surface to be measured shall have been chemically and mechanically polished to a mirror finish, or shall be a mirror finish carrying an oxide layer.

9 Calibration