NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 24582-2023

Test method for measuring surface metal impurity content of polycrystalline silicon - Acid extraction-inductively coupled plasma mass spectrometry method

多晶硅表面金属杂质含量测定 酸浸取-电感耦合等离子体质谱法

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Foreword

This document was issued on 6 August 2023 by the State Administration for Market Regulation; Standardization Administration of the PRC and takes effect on 1 March 2024.

It is a GB/T standard: recommended rather than compulsory, but it is the text a Chinese reviewer applies when assessing a submission.

1 Scope

GB/T 24582-2023 covers the measurement of the metal impurities left on the surface of polycrystalline silicon: the surface is leached with acid and the leachate is measured by inductively coupled plasma mass spectrometry. The method principle is a timed leach in a mixture of nitric acid, hydrofluoric acid and deionised water, after which the leachate is carried into a high-temperature plasma source through the sample introduction system. Because the amounts being weighed are far below what ordinary laboratory handling keeps clean, a whole clause is given to interfering factors: sampling in a cleanroom, clips and collection bags proved free of contamination, and samples taken off-site sealed in double bags and kept intact in transit. The reagents clause fixes the grade of water and acid, the apparatus clause the mass spectrometer and the analytical balance, and the samples clause the mass range and the requirement for at least six parallel specimens, three with the outer skin and three without. Data processing, precision and the test report close the

method. Metal picked up on the surface follows the silicon into the crystal puller and ends up as electrically active contamination in the wafer, where it costs far more to find than at incoming inspection. It is used by polysilicon producers, wafer makers checking incoming material, and testing laboratories.

This document describes a method for leaching metal impurities from the surface of polycrystalline silicon using acid and quantitatively determining the content of these impurities using inductively coupled plasma mass spectrometry (ICP-MS).

This document is applicable to the determination of the content of impurity elements - including alkali metals, alkaline earth metals, and first-series transition elements (such as sodium, potassium, calcium, iron, nickel, copper, zinc, and aluminum) - on the surface of solar-grade and electronic-grade polycrystalline silicon. The determination limit is 0.01 ng/g.

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 11446.1, Electronic Grade Water

GB/T 25915.1-2021, Cleanrooms and associated controlled environments -- Part 1. Classification of air cleanliness by particle concentration

3 Terms and definitions

This document contains no terms and definitions requiring definition.

4 Method principle

The test material is leached for a specified period in a mixture of nitric acid, hydrofluoric acid, and deionized water. The resulting leachate is introduced into a high-temperature plasma source via the sample introduction system, where it undergoes evaporation, dissociation, atomization, and ionization within the high-temperature torch. The vast majority of metal ions are converted into singly charged ions. These ions pass through the interface cones into the mass analyzer, where they are separated based on

their mass-to-charge ratios. The content of the target metal elements is then determined using an inductively coupled plasma mass spectrometer (ICP-MS).

5 Interfering factors

5.1 Sampling shall be performed in a cleanroom. Ensure that sampling clips, sample collection bags, and similar items are free from contamination. If sampling is conducted off-site, seal the samples in double-layered bags and ensure they remain intact during transport. Prevent sample contamination during the sampling process.

5.2 The stability of the testing equipment shall be verified prior to testing. The cleanliness of the test chamber environment must meet the ISO Class 6 requirements specified in GB/T 25915.1-2021.During the test, operators shall be mindful of the impact of their actions to avoid affecting the test results.

5.3 Ensure that the vessels used for testing are free from contamination. Test them using inductively coupled plasma mass spectrometry (ICP-MS) prior to use. Proceed only after confirming their cleanliness.

5.4 Samples must be representative. Since surface contaminants are not distributed uniformly, the size and number of samples selected shall accurately represent the entire batch. If the sample size is too small or the quantity insufficient, the samples shall fail to represent the batch, resulting in excessive deviation between parallel samples.

5.5 If the impurity content in the sample is high, the detection curve shall be adjusted. Where necessary, the sample may be diluted prior to analysis to prevent instrument contamination, which could otherwise compromise the instrument's detection limit and precision.

5.6 Factors such as diatomic ions, polyatomic ions, matrix effects, background noise, inter-element interferences, cross-contamination, and instrument drift can affect test results.

6 Reagents and materials

6.1 Deionized water. Type EW-I deionized water as described in GB/T 11446.1, or deionized water of equivalent quality.

6.2 Nitric acid (rho = 1.42 g/mL). The content of the metal impurities to be determined is below 10 ug/L.

6.3 Hydrofluoric acid (rho = 1.19 g/mL). The content of the metal impurities to be determined is below 10 ug/L in all cases.

6.4 Leaching solution. The volume ratio of nitric acid, hydrofluoric acid, and water is 1.1.10.

6.5 Sample vials and clips. The sample vials come with caps, and the clips are made of PTFE, PFA, or similar polymer materials that are resistant to hydrofluoric acid corrosion and can be cleaned.

7 Apparatus and equipment

7.1 ICP-MS. Inductively coupled plasma mass spectrometer with a dynamic reaction cell. Mass resolution is better than 0.8 amu.

7.2 Analytical balance. The scale interval is 0.001 g.

7.3 Acid-resistant electric heating plate.

8 Samples

The sample mass ranges from approximately 20 g~360 g. For arbitration purposes, the dimensions range from (3 cm x 3 cm x 1 cm) ~ (3 cm x 3 cm x 3 cm). At least six parallel samples shall be taken. three with the outer skin and three without. The mass of an individual sample is approximately 120 g.

9 Test steps

9.1 Apparatus and equipment preparation

Turn on the ventilation system. Adjust the flow rate of the gas to be analyzed and the pressure of the cooling water circulation system. Power on the instrument and allow it to warm up to meet operational requirements. Before testing, select the appropriate detection mode for the inductively coupled plasma mass spectrometer (ICP-MS) and perform tuning to achieve optimal testing conditions.

9.2 Parallel test