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

GB/T 32996-2025

Replacing GB/T 32996-2016

**Surface chemical analysis - Analysis of metal oxide films by
glow discharge optical emission spectrometry**

表面化学分析 辉光放电发射光谱法分析金属氧化物膜

(English Translation)

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Foreword

This document is in accordance with the provisions of GB/T 1.1-2020 "Guidelines for standardization work Part 1: Structure and drafting rules for standardization documents" Drafting.

This document replaces GB/T 32996-2016 "Surface chemical analysis of metal oxide films by glow discharge optical emission spectrometry" and Compared with GB/T 32996-2016, in addition to structural adjustments and editorial changes, the main technical changes are as follows.

- Added the metal element Zr in the oxide range (see Chapter 1);
- Added terms and definitions (see Chapter 3);
- Changed 5.1.1 Overview and modified the instrument description, including the use of

CCD and CID, COMS type solid-state array detectors Spectrometer (see 5.1.1, 4.1.1 of the 2016 edition);

--- Added a 2.5mm anode size commonly found in commercial instruments (see 5.1.3.1);

--- Changed 6.1 Overview to introduce the most common working modes of RF sources and the differences between RF sources and DC sources, and emphasized the importance of forming a good vacuum seal between the test sample and other samples (see 6.1, 5.1 of the 2016 edition);

--- Changed the optical system inspection to include instruments equipped with array detectors (see 6.1, 6.2.1.1, 5.1, 5.2.1.2 of the 2016 edition);

--- Changed "Set detector high voltage" to "Set detector sensitivity parameters" and changed these settings to accommodate array detectors Notes (see 6.1, 6.2.1.1, 6.2.2, 6.3.2, 6.3.3, 6.3.4, 2016 edition 5.1, 5.2.1.2, 5.2.2, 5.3.1, 5.3.2, 5.3.3);

--- Added 6.2.3 constant voltage and gas pressure mode, which sets the DC source discharge parameters by constant voltage and gas pressure mode.

Number (see 6.2.3);

--- Changed the description of optimized crater shape from mandatory to optional (see 6.2.1.3, 6.2.2, 6.2.3, 6.3.2, 6.3.3, 6.3.4, 5.2.1.4, 5.2.2, 5.3.1, 5.3.2, 5.3.3 of the 2016 edition);

--- Added 6.4.4 Control lamp cleanliness and starting performance, and introduced the method to verify whether the starting performance of the lamp meets the requirements for analysis Procedure for ultra-thin films (see 6.4.4);

--- Changed the overview of 8.2.1 to change the minimum number of samples required to establish the working curve of each element from 5 to 3 (see 8.2.1, 7.2.1 of the 2016 edition);

--- Changed 8.4 Determination of sputtering rate of working curve sample and confirmation sample. The calculation of absolute sputtering rate should be in accordance with the manufacturer's the method provided calculates the spatter volume of each crater, compensating, if possible, for craters with non-flat bottoms (see 8.4, 2016 edition).

This document is equivalent to ISO/T S25138.2025 "Surface chemical analysis of metal oxide films by glow discharge optical emission spectrometry".

The file type was adjusted from ISO 's technical specifications to my country's national standards.

1 Scope

This document describes the determination of metal oxide film thickness, mass per unit area, and chemical properties using glow discharge optical emission spectroscopy.

Composition method.

This method is suitable for measuring oxide films with a thickness of 5nm~10000nm on metals. The metal elements of the oxides include iron, chromium, One or more of nickel, copper, titanium, silicon, molybdenum, zinc, magnesium, manganese, zirconium and aluminum. Other detectable elements include oxygen, carbon, nitrogen, hydrogen, phosphorus and sulfur.

2 Normative references

The contents of the following documents constitute the essential clauses of this document through normative references in this document.

For referenced documents without a date, only the version corresponding to that date applies to this document; for referenced documents without a date, the latest version (including all amendments) applies to this document.

ISO 14284 Steel and iron - Sampling and preparation of samples for determination of chemical composition Note. GB/T 20066-2006 Steel and iron - Sampling and preparation of samples for determination of chemical composition (ISO 14284:1996, IDT) Note. GB/T 19502-2023 Surface chemical analysis - Glow discharge optical emission spectroscopy - General rules (ISO 14707:2021, IDT)

3 Terms and Definitions

There are no terms or definitions that require definition in this document.

4 Principles

Glow discharge emission spectroscopy analysis includes the following steps.

a) Prepare the sample to be tested, which is generally in the shape of a flat plate or disc (circular or rectangular with a width greater than 5 mm) suitable for the instrument or analysis requirements.

Sample, usually 20mm~100mm);

b) In a DC or RF glow discharge source device, causing cathode sputtering on the surface of a metal oxide;

c) In a glow discharge device, analyte atoms are excited in a plasma;

d) Spectral measurement of the emission intensity of the atomic characteristic lines of the object being measured over time (qualitative depth profiling);

e) Through the working curve, the change of intensity with time in the depth profile is converted into the change of mass fraction with sputtering depth (quantification).

Measure samples of known chemical composition and sputtering rate to establish a system working curve.

5.1.1 Overview

The required apparatus shall consist of a Grimm type [4] or similar glow discharge source (DC or RF) and a multi-channel