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Methods for chemical analysis of titanium sponge, titanium and titanium alloys - Part 29: Determination of aluminum, carbon, chromium, copper, iron, manganese, molybdenum, nickel, silicon, tin, vanadium and zirconium contents - Spark atomic emission spectrometry

海绵钛、钛及钛合金化学分析方法 第29部分：铝、碳、铬、铜、铁、锰、钼、镍、硅、锡、钒、锆含量的测定 光电直读光谱法

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1 Scope

This document describes a method for determining the aluminium, carbon, chromium, copper, iron, manganese, molybdenum, nickel, silicon, tin, vanadium and zirconium contents of titanium sponge, titanium and titanium alloys by spark atomic emission spectrometry.

This document applies to the determination of the contents of several elements in titanium sponge, titanium and titanium alloys. The determination range of each element is given in Table 1. Where the determination range overlaps that of another part of GB/T 4698, this method is not used as the referee method.

Table 1 gives, for each element, the determination range as a mass fraction in percent: aluminium from 0.014 to 7.80; carbon from 0.010 to 0.18; chromium from 0.006 to 2.92; copper from 0.003 to 0.46; iron from 0.020 to 0.55; manganese from 0.004 to 4.70; molybdenum from 0.006 to 6.11; nickel from 0.004 to 0.85; silicon from 0.006 to 0.46; tin from 0.009 to 3.20; vanadium from 0.007 to 14.90; zirconium from 0.012 to 4.08.

2 Normative references

The contents of the following documents constitute indispensable provisions of this document through the normative references made to them in the text. For dated references, only the version corresponding to that date applies to this document; for undated references, the latest version (including all amendments) applies to this document.

GB/T 2524-2019, Titanium sponge.

GB/T 8170, Rules of rounding off for numerical values and expression and judgement of limiting values.

GB/T 14203, General rules for spark discharge atomic emission spectrometric analysis.

GB/T 31981, Method of sampling and sample preparation for chemical composition analysis of titanium and titanium alloys.

3 Terms and definitions

The terms and definitions given in GB/T 14203 apply to this document.

4 Principle

In an argon atmosphere the prepared analytical sample discharges against the counter-electrode under the action of a spark source; the electric spark melts the surface of the test piece, which then evaporates and is excited so that it emits light. When the atoms or ions of the element being determined are excited, the electrons move between the different energy levels within the atom, and when they fall back from a higher to a lower level they give out the characteristic spectral lines. The spectral intensity of the selected analytical lines and internal standard lines is measured, and from the relationship between the line intensity, or the intensity ratio, of the element in the analytical sample and the content of that element, the content is calculated from the working curve.

5 Test conditions

The ambient conditions of the spark atomic emission spectrometer shall meet the following requirements: a temperature of 18 degrees Celsius to 30 degrees Celsius, with the room temperature varying by no more than 5 degrees Celsius within one standardization cycle; a relative humidity of 20 percent to 80 percent; and freedom from electromagnetic interference and from vibration.

6 Reagents or materials

6.1 Gas: argon, of a purity not lower than 99.995 percent by volume.

6.2 Certified reference samples: the reference samples used to draw the working curve shall be a series of certified spectrographic reference samples. Their chemical composition shall cover the content range of the elements being determined and shall be spaced at suitable intervals.

6.3 Standardization samples. 6.3.1 The standardization samples are used to correct the departure of the instrument readings from the working curve caused by various factors; they shall be homogeneous and shall give a stable line intensity ratio. 6.3.2 The standardization samples may be the certified reference samples, or other samples that meet the basic requirements and are of suitable content, homogeneous, stable and well reproducible. Where two-point standardization is used, the contents are chosen near the upper limit and near the lower limit of the working curve of each element.

6.4 Control samples: the control samples shall have a chemical composition and a structure close to those of the analytical sample and are used to correct further the result read for the analytical sample from the working curve. A control sample may be a certified reference sample or an in-house control sample.

7 Apparatus

7.1 Instrument: a spark atomic emission spectrometer, which shall comprise an excitation source, a spark stand, an argon system, a counter-electrode, a dispersing system and a measuring system, and which shall meet the wavelength, sensitivity and precision required by the determination.

7.2 Sample preparation equipment: a lathe or a milling machine able to meet the machining requirements of the analytical sample.

8 Samples

8.1.1 Titanium sponge samples. The analytical sample of titanium sponge is taken and prepared according to Annex B of GB/T 2524-2019. The sample is taken between the filament turning sample and the Brinell hardness sample.

8.1.2 Titanium and titanium alloy samples. The analytical sample of titanium and titanium alloys is taken according to GB/T 31981 or by another agreed method. The sample may be plate, bar, strip, an extrusion, a casting or another wrought form or shape. The thickness of the sample shall be such that it is not burnt through during excitation. For strip and bar samples the following sizes may be taken as a guide: a) strip samples, thickness not less than 0.25 mm, length not less than 30 mm, width not less than 20 mm; b) bar samples, diameter not less than 6 mm and a suitable length.

8.2 Preparation of the sample. Warning: titanium is a flammable metal, and the high temperature and sparks produced momentarily during light machining readily set titanium swarf alight. No flammable cooling medium is used during turning.

The analytical sample, the certified reference samples (6.2), the standardization samples (6.3) and the control samples (6.4) shall be prepared under the same conditions. The excitation face shall be clean and flat, free from pores, inclusions and cracks, and its surface roughness Ra shall be not greater than 3.2 micrometres.

When the sample is turned, the lathe speed shall be kept at 450 r/min and the milling machine at 475 r/min. Cemented carbide and coated tools are recommended. For samples that are not suited to turning, the excitation face may be prepared with abrasive paper, an abrasive paper disc or a belt grinder, but such methods may affect the results for elements present at low content.

9 Calibration methods

9.1 Working curve method. Under the selected working conditions, a series of certified reference samples (6.2) is excited, in principle using reference samples at five or more levels, each sample being excited at least three times; the working curve is drawn using the mean emission intensity, or intensity ratio, of each element to be determined against the concentration of that element in the reference sample, and the content of the element in the analytical sample is determined from that curve. A first-order or a second-order regression curve is usually chosen. If the second-order regression curve is strongly curved, the curve shall be calibrated in segments, each segment using reference samples at five or more levels. Where necessary, correction for interfering elements shall be applied.

9.2 Control sample method (type standardization method). Because the analytical sample and the reference samples used to draw the working curve differ in metallurgical history and in structure, the result often departs from the true value. To avoid the effect of that difference, a control sample whose metallurgical history and structure are close to those of the analytical sample is usually determined at the same time. Under the same working conditions the control sample and the analytical sample are determined together, and the difference between the result found for the elements in the control sample and its certified value is used to correct the result found for the analytical sample.

10 Test procedure

10.1 Analytical conditions. The choice of analytical conditions depends on the model of the spectrometer and on its software. The recommended internal standard lines, analytical lines and possible interfering elements are given in Annex A.

10.2 Preparation for analysis. 10.2.1 The noise, the dark current and the lamp intensity of the spectrometer shall be checked periodically to confirm that the systems concerned are working normally. 10.2.2 The instrument shall be profiled periodically and the result compared with the error the instrument allows. 10.2.3 The electrode gap shall be checked and adjusted periodically, by the method given in the instrument manual. 10.2.4 Suitable