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Methods for chemical analysis of magnesium and magnesium alloys - Part 21: Determination of element content - Photoelectric direct reading atomic emission spectrometry

镁及镁合金化学分析方法 第21部分：元素含量的测定 光电直读原子发射光谱法

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7 Sampling and Sample Preparation

7.1 When sampling from the molten state, use a preheated cast iron or steel mold for casting. The sample should be free of burrs, inclusions, porosity, and cracks. When taking samples from ingots, castings, and processed products, samples should be taken from representative parts.

7.2 No lubricant shall be used during the processing of the test surface of the sample. The test surface shall be free of oxidation, pores, and other foreign substances, and shall not... There are oil stains and other dirt.

7.3 The sample size specifications should cover the excitation aperture of the excitation stage.

7.4 Standardized samples, control samples, and test samples should preferably be prepared simultaneously under the same conditions. The determination should be completed within 2 hours after sample preparation.

8 Spectrometer Testing Conditions

8.1 Calibration of the spectrometer The spectrometer should be calibrated regularly, with a calibration cycle not exceeding 24 months.

8.2 Adjustment of the excitation system

8.2.1 Counter electrode The counter electrode should be cleaned and replaced periodically according to actual conditions. During non-excitation periods, the excitation hole should be covered to protect the counter electrode.

8.2.2 Electrode gap The gap between the sample and the counter electrode should be checked periodically using a distance gauge.

8.2.3 Gas Supply System The argon gas supply system should be as short as possible, with no leaks at the connections. Adjust the gas flow rate to ensure that the excitation flow rate meets the spectrometer's specified argon flow rate. The air flow rate should be maintained at the minimum during non-working periods.

8.3 Adjustment of the optical system

8.3.1 Focusing lens or quartz protective film When the focusing lens or quartz protective plate is contaminated, it should be cleaned. The degree of contamination can be judged by the decrease in the intensity of the element being measured.

8.3.2 Indoor Pressure The pressure of the vacuum optical chamber should meet the instrument requirements. The gas-filled optical chamber should be protected with high-purity inert gas to ensure the pressure within the gas-filled optical chamber is within acceptable limits. To maintain the purity of the protective gas, the pressure of the gas inside the light chamber should be slightly higher than atmospheric pressure and kept constant.

8.3.3 Tracing By tracing and adjusting the position of the incident slit, the intensity of the spectrum emitted from the light source entering the slit is maximized.

8.4 Adjustment of the Metering System

8.4.1 Pre-combustion time The appropriate pre-ignition time is determined through preliminary tests.

8.4.2 Integrating Time The integration time was determined through experiments based on the accuracy requirements such as time and analytical line strength.

8.4.3 Test Intensity Range Based on the content range of the analyzed elements and the characteristics of the spectral lines, the test intensity range is determined by adjusting the signal amplification factor of each detector.

8.5 Setting Test Parameters

8.5.1 Analysis Line Select the analytical line from the emission spectrum of the element to be measured, which is less affected by interference from other spectral lines and has a high signal-to-noise ratio.

8.5.2 Internal Standard Elements with relatively small variations in content within magnesium and magnesium alloys are selected as internal standard elements; matrix magnesium elements are typically used. The spectral lines of the internal standard element are then analyzed. Choose an appropriate analytical line as the internal standard line.

8.5.3 Spectral line interference and correction Spectral line overlap and background differences caused by variations in sample composition can affect quantitative analysis results. Therefore, it is important to consider the interactions between coexisting elements. Correct for interference or background interference.

8.5.4 Recommended internal standard and analytical line Recommended internal standards, elemental analysis line wavelengths, and possible interfering elements are shown in Table 2.

8.6 Excitation Point

8.6.1 The excitation point should be 5mm to 10mm away from the edge of the sample, and the excitation points should not overlap.

8.6.2 After excitation, this point should be a relatively deep depression surrounded by a black ring.

9 Calibration

9.1 Calibration Curve Method Under the selected working conditions, a series of standard samples are excited, ideally using more than five standard samples with different concentrations, and plotted... A calibration curve is generated comparing the spectral intensity (or intensity ratio) of the element to be measured with its content (or content ratio), thus determining the elemental content in the sample. Calibration curve The lowest point of the line should not be higher than the lower limit of the element determination range, and the highest point should not be lower than the upper limit of the determination range.

9.2 Original Calibration Curve Method First, establish a calibration curve using the calibration curve method. When the calibration curve shifts due to factors such as temperature, humidity, or vibration, or... When the calibration curve deviates due to changes in luminescence intensity, drift correction is performed to restore the elemental intensities to the levels when the curve was initially established. The intensity. If the spectrometer undergoes significant changes or the original calibration curve drifts beyond the correction range, a new calibration curve should be established.

10 Experimental Procedure

10.1 Preparation of the spectrometer Before using the spectrometer, it should be powered on for an appropriate period of time and adjusted until it is in a stable working state.

10.2 Pre-excitation The spectrometer should be pre-excited 2 to 5 times before use.

10.3 Calibration of the calibration curve

10.3.1 Overall Drift Correction (Overall Normalization) When using the original calibration curve method, the original calibration of the same matrix for multiple test procedures is periodically performed by measuring multiple drift correction samples. The curve is corrected across its entire range. The overall drift correction period depends on the stability of the spectrometer.

10.3.2 Type Standard Correction (Type Standardization) When applicable, before testing a sample, it is advisable to select a standard sample or sample whose chemical composition, tissue structure, and processing method are consistent with or similar to the sample to be tested. Control samples and perform type standard calibration to correct for differences caused by matrix or tissue structure.

10.4 Confirmation of Calibration Curve Before testing samples, it is advisable to select a certain number of standard samples or control samples to verify the calibration curve. The difference between two determinations should not exceed the repeatability limit (r) specified in Table 5, and the difference between the average value (x) of the determination and the standard value (μ0) should not exceed the limit specified in Table 5. If the drift is greater than the critical difference (CD0.95) or the critical value (C), the cause should be investigated and the drift correction should be performed again. The critical difference (CD0.95) is calculated according to formula (1). The value can be calculated or found in Table A.1 of Appendix A; when the standard sample gives the uncertainty (U), the critical value (C) is calculated according to formula (2).

10.5 Sample Determination

10.5.1 Place the sample on the excitation platform of the spectrometer and excite the sample at least twice. If the test results are not used, additional excitations should be performed. Number. Test results should not be deleted.

10.5.2 When the test result deviation exceeds the repeatability limit, or when the excitation point after excitation is abnormal, the number of excitations can be increased by 1 to 2 times, and the total excitation... The number of times should not exceed 8.

10.5.3 The instrument software provides the mass fraction of each element based on the calibration curve.

10.6 Reconfirmation of Calibration Curve When abnormal test results are found, or when multiple samples are tested consecutively, or when the instrument has been idle for an extended period of time, the calibration curve should be adjusted. Reconfirm the line. If the confirmation does not meet the requirements, proceed according to steps