INTERNATIONAL STANDARD

ISO 7625

Third edition
2012-11-15

Sintered metal materials, excluding hardmetals — Preparation of samples for chemical analysis for determination of carbon content

Matériaux métalliques frittés, à l'exclusion des métaux-durs — Préparation des échantillons pour analyse chimique en vue du dosage du carbone

Reference number
ISO 7625:2012(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 7625 was prepared by Technical Committee ISO/TC 119, *Powder metallurgy*, Subcommittee SC 3, *Sampling and testing methods for sintered metal materials (excluding hardmetals)*.

This third edition of ISO 7625 cancels and replaces the second edition (ISO 7625; 2006), which has been technically revised in Clause 6.

Introduction

The chemical analysis of sintered metal materials, excluding hardmetals, is carried out as it would be for solid metals, by using existing standard methods. However, as these sintered materials very often contain carbonaceous constituents, the correct determination of carbon content (free or total) requires that certain precautions be taken when preparing the sample for analysis from components.

Samples prepared in accordance with this International Standard may also be suitable for chemical analysis for other elements.

Sintered metal materials, excluding hardmetals — Preparation of samples for chemical analysis for determination of carbon content

1 Scope

This International Standard specifies methods for preparing a sample from one or more sintered parts to be analysed for free or total carbon content. Combined carbon is determined as the difference between total and free carbon. This standard covers the preparation of samples for the determination of carbon by a chemical method, i.e. combustion in oxygen and measurement of the carbon dioxide produced, in accordance with ISO 437. It does not cover the preparation of samples for carbon determination by physical methods, such as metallography or spectroscopy.

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.

ISO 437, *Steel and cast iron — Determination of total carbon content — Combustion gravimetric method*

ISO 2738, *Sintered metal materials, excluding hardmetals — Permeable sintered metal materials — Determination of density, oil content, and open porosity*

3 Principle

Eliminate any impregnated or surface carbon using the procedures in Clause 5. Prepare samples for analysis by fragmentation or machining, taking care not to lose any free carbon.

4 Materials

This International Standard applies to parts in which carbon is uniformly distributed and present in the forms shown in Table 1. It does not apply to parts in which the carbon is not uniformly distributed throughout the part, including those parts with carburized or decarburized surfaces.

In instances where the carbon is not uniformly distributed, for example a case-hardened part, the method of selecting the sample should be agreed upon between the supplier and the purchaser.

The presence of carbonaceous materials in the pores or on the surface of the part to be analysed interferes with the determination of carbon, see Table 2.

Table 1 — Forms of carbon that can be determined by chemical analysis

Form of carbon	Typical materials	Comments
Combined carbon (as carbides or in solid solution)	Carbon-containing steels and super-alloys	Determined as the difference between total and free carbon
Free carbon	Bronze and steel containing graphite; material impregnated with a graphite-containing liquid	Determined by combustion of the residue of selective dissolution of the metal in the sample.
Total carbon		Determined by direct combustion of the sample

Table 2 — Origins of carbonaceous material and feasibility of carbon determination by chemical methods

Carbonaceous material	Origin	Feasibility
1 Liquid or semi-liquid organic substances on the surface or in the pores	Sizing lubricant, quenching oil, impregnating lubricant, cutting fluid, grease	Carbon determination is possible only when these organic substances are totally eliminated by the method described in Clause 5
2 Waxes and wax-like substances	Sizing lubricant, corrosion protective	
3 Deposits of carbon on the parts	Sintering, heat treatment	
4 Solid organic substances, for example, plastics materials on the surface or in the pores	Sealant material	In general, it is not possible to eliminate these substances, and determination of carbon is not possible

5 Procedure

5.1 Removal of interfering carbonaceous material

5.1.1 General

Carbonaceous materials (see Table 2) that affect the carbon determination shall be removed from the part or parts to be analysed as specified in 5.1.2 and 5.1.3.

5.1.2 Impregnated organic substances in the pores or on the surface (see Cases 1 and 2 in Table 2) shall be removed by Soxhlet degreasing with an appropriate solvent, in accordance with ISO 2738. The solvent used shall be stated.

NOTE When the impregnated liquid contains fine carbon particles, for example colloidal graphite, it is possible that not all of these particles are removed by Soxhlet degreasing. In such a case, the determination of free carbon and total carbon is invalid. Nevertheless, the calculation of combined carbon is still valid if the free carbon and total carbon are determined on the same part or parts after degreasing.

5.1.3 Surface deposits of carbon (see Case 3 in Table 2) shall be removed by mechanical treatment.

5.2 Final preparation

The sample for analysis shall consist of small fragments of a size appropriate to the analysis method to be used, prepared by one of the following methods:

— crushing in a mortar made of a material which does not alter the sample composition;

— drilling, milling or turning. Such machining shall be performed dry by using hardmetal or ceramic tools. Care shall be taken to avoid overheating, oxidation and contamination of the fragments. Where drilling is used, the part shall be drilled through completely and the distribution of drilling holes shall be uniform over the whole surface of the part. If the part is too thick, the holes shall be drilled to the middle from both faces of the part.

Where crushed or machined fragments contain fine particles (for example free graphite), care shall be taken not to lose these particles during handling.

Where segregation is expected to be a problem, the entire sample should be analysed after being divided into a suitable number of test portions. The carbon content is then calculated as the average of the results from each portion.

In general, a sufficient quantity of fragments, representative of the part or parts, shall be prepared. The samples for analysis shall be kept in a container that is tightly sealed, in order to avoid contamination.

6 Precision

When the degreasing is carried out according to ISO 2738 as stated in 5.1.2, the carbon content after oil removal can be considered suspect if the repeatability is greater than 5 % and the reproducibility is greater than 10 % at the 95 % confidence level.

ICS 77.160
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