INTERNATIONAL STANDARD

ISO 4491-2

Second edition
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Metallic powders — Determination of oxygen content by reduction methods —

Part 2:
Loss of mass on hydrogen reduction (hydrogen loss)

Poudres métalliques — Dosage de l'oxygène par les méthodes de réduction —

Partie 2: Perte de masse par réduction dans l'hydrogène (perte dans l'hydrogène)

Reference number
ISO 4491-2:1997(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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.

International Standard ISO 4491-2 was prepared by Technical Committee ISO/TC 119, *Powder metallurgy*, Subcommittee SC 2, *Sampling and testing methods for powders (including powders for hardmetals)*.

This second edition cancels and replaces the first edition (ISO 4491-2:1989), table 1 and clause 7 of which have been technically revised.

ISO 4491 consists of the following parts, under the general title *Metallic powders — Determination of oxygen content by reduction methods*:

— *Part 1: General guidelines*

— *Part 2: Loss of mass on hydrogen reduction (hydrogen loss)*

— *Part 3: Hydrogen-reducible oxygen*

— *Part 4: Total oxygen by reduction-extraction*

Annex A forms an integral part of this part of ISO 4491.

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Metallic powders — Determination of oxygen content by reduction methods —

Part 2:
Loss of mass on hydrogen reduction (hydrogen loss)

1 Scope

This part of ISO 4491 specifies a method for the determination of the relative loss of mass which a metallic powder undergoes when heated in a stream of pure dry hydrogen under specified conditions.

The purpose of this test is to evaluate a chemical powder characteristic which is of importance to the powder metallurgical industry. The test is not intended as a means for the determination of the content of specific elements. (See annex A and ISO 4491-1.)

The test method is applicable to unalloyed, partially alloyed and completely alloyed powders of the metals listed in table 1 (see 6.1). It is not applicable to lubricated powders or to mixtures of metal powders.

The results can be influenced by the presence of reducible, oxidizable or volatile metals, metalloids or compounds (see annex A). The results obtained on such powders shall be used with caution and their interpretation shall be subject to agreement between supplier and user.

This part of ISO 4491 shall be read in conjunction with ISO 4491-1.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this part of ISO 4491. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this part of ISO 4491 are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 4491-1:1989, *Metallic powders — Determination of oxygen content by reduction methods — Part 1: General guidelines.*

3 Reagents and materials

3.1 Hydrogen, with a maximum oxygen content of 0,005 % (m/m) and a dew point not higher than – 45 °C.

3.2 Nitrogen or **argon**, with a maximum oxygen content of 0,005 % (m/m) and a dew point not higher than – 45 °C.

(See also 6.3, third paragraph.)

4 Apparatus

An example of suitable test arrangement is shown schematically in figure 1.

4.1 Laboratory balance, of sufficient capacity, and capable of weighing to an accuracy of 0,1 mg.

4.2 Electrically heated tubular furnace, that can be continuously operated at the appropriate temperatures given in table 1 and that has a control system capable of maintaining the temperature in that part of the tube containing the boat (4.5) to within the temperature tolerance stated in table 1.

NOTE — When testing magnetic powders, it is recommended that wire-wound furnaces shall be wound non-inductively.

4.3 Gas-tight tube, of quartz or refractory material (for example dense alumina). The inside diameter of the tube shall be between 25 mm and 40 mm and its length such that it extends about 200 mm beyond each end of the furnace.

When a large number of hydrogen loss determinations is to be carried out, a larger furnace than that described in this part of ISO 4491, and one which permits several test portions to be tested simultaneously, may be used, provided that the temperature and time conditions shown in table 1 are fulfilled and the results obtained are in agreement with those obtained when the test is carried out with the preferred apparatus.

4.4 Totally enclosed thermocouple, for example platinum/platinum-rhodium, and an **indicating or recording instrument**, permitting the measurement of temperature with an accuracy of 5 °C.

If for some reason it is desirable to place the thermocouple outside the reduction tube, this is acceptable. But in this case, a preliminary calibration shall be made with a second thermocouple placed inside the tube in order to ascertain that the temperature of the test sample is in accordance with the values and tolerances specified in table 1.

4.5 Boat, preferably of high-alumina ceramic with a polished surface. Other materials, for example quartz and nickel, may be used when test conditions allow. The boat shall be of such dimensions, for example 75 mm long and 12 mm wide, that the thickness of the powder, when uniformly distributed, does not exceed 3 mm.

New boats shall be pretreated in a stream of hydrogen at the test temperature and stored in a desiccator.

A boat may be used more than once, provided that it is always used for testing the same metal powder or type thereof and provided that it is carefully cleaned by mechanical means between determinations and stored in a desiccator.

4.6 Supply unit for hydrogen and either nitrogen or argon, with pressure gauges and flow meters to control the flow of gas.

5 Sampling

5.1 The powder shall be tested in the as-received condition.

5.2 The loss in mass shall be determined on two test portions.

5.3 The mass of the test portion shall be approximately 5 g, except that for powders of low apparent density it may be reduced to comply with the requirements of 4.5 and 6.2.