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Pulvermetallurgi – Het isostatisk pressning – Argondetektion med gaskromatografi- och masspektrometriteknik

Powder metallurgy – Hot isostatic pressing – Argon detection using gas chromatography and mass spectrometry techniques

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Information about the content of the standard is available from the Swedish Standards Institute (SIS), telephone +46 8 555 520 00. Standards may be ordered from SIS, who can also provide general information about Swedish and foreign standards.

Standarden är framtagen av kommittén för Pulvermetallurgi, SIS/TK 133.

Har du synpunkter på innehållet i den här standarden, vill du delta i ett kommande revideringsarbete eller vara med och ta fram andra standarder inom området? Gå in på www.sis.se - där hittar du mer information.

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Introduction

The argon levels in Powder Metallurgical Hot Isostatic Pressing (PM HIP) products are important to control because argon is present in the PM HIP product as gaseous voids which seriously affects the performance of the PM HIP product, most notably the mechanical properties. Since argon is used as pressure builder in the HIP furnace any problems with the welded can(ister) will result in imperfections caused by argon being entrapped in the PM HIP product.

Argon gas atomization will result in argon being trapped within the powder particles which will lead to higher levels of argon in the PM HIPed product compared to nitrogen atomized powder.

1 Scope

This standard specifies a gas chromatography- and a mass spectrometry method of detecting the presence of argon in metal powder produced components, consolidated by hot isostatic pressing.

This standard specifies the calibration and functionality test for the equipment covered. It also specifies methods for sampling, sample preparation and sample test procedure of PM HIP components to detect argon presence.

Components produced by additive manufacturing are not covered in this standard.

NOTE It is the responsibility of the purchaser of the PM HIP service to specify in the purchase order if an argon detection test shall be made and if so, the agreed argon limit shall be specified.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
alcohol
the term “alcohol” shall, in this document, be understood as ethyl alcohol (ethanol, C_2H_5OH)

[SOURCE: ISO 4805:1982, 3.1.1]

3.2
blank test
test performed without sample in the same manner as, and parallel with, a test using an analytical sample

[SOURCE: ISO 11323:2010, 8.13]

3.3
calibration
operation that, under specified conditions, in a first step, establishes a relation between the quantity values with measurement uncertainties provided by measurement standards and corresponding indications with associated measurement uncertainties and, in a second step, uses this information to establish a relation for obtaining a measurement result from an indication

Note 1 to entry: A calibration may be expressed by a statement, calibration function, calibration diagram, calibration curve, or calibration table. In some cases, it may consist of an additive or multiplicative correction of the indication with associated measurement uncertainty.

Note 2 to entry: Calibration should not be confused with adjustment of a measuring system, often mistakenly called “self-calibration”, nor with verification of calibration

Note 3 to entry: Often, the first step alone in the above definition is perceived as being calibration.

[SOURCE: ISO/IEC Guide 99]

3.4
can
capsule
canister

container used to encapsulate the powder during the pressure consolidation process; it is partly or fully removed from the final part

3.5
detection limit
smallest actual amount of an analyte that can be detected by a measuring method

[SOURCE: ISO 20553:2006, 3.14, modified measurand replaced by analyte]

3.6
functionality test
assessment of the performance of a measuring system, based on specific parameters

[SOURCE: ISO/TS 14907-1:2015, 3.10, modified measurand replaced by analyte]

3.7
gas chromatograph
device that physically separates components of a mixture in the gaseous phase and measures them individually with a detector which signal is processed

[SOURCE: ISO 14532:2014, 2.4.3]

3.8
mass spectrometer
instrument which separates ionized particles of different mass/charge ratios and measures the respective ion currents

[SOURCE: ISO 3529-3:2014, 2.5.1]

3.9
PM HIP
powder metallurgy hot isostatic pressing
process for simultaneously heating and forming a compact in which the powder is contained in a sealed formable enclosure usually made from metal and the so-contained powder is subjected to equal pressure from all directions at a temperature high enough to permit plastic deformation and consolidation of the powder particles to take place

[SOURCE: ASTM A988/A988M – 15A]

3.10
reference sample
material or substance which property values are sufficiently homogeneous and well established to be used for the functionality test of an apparatus, the assessment of a measurement method, or for assigning values to materials

4 Equipment for argon detection

4.1 Gas chromatography

4.1.1 Principle

Gas chromatography is a separation technique where the mobile phase is gaseous. When GC is used as analytical technique, a known amount of sample is evaporated and dissolved into a mobile phase, also called the carrier gas. The sample compounds are carried by this mobile phase through a chromatographic column and further through detectors. Due to different specific interactions between the sample compounds and stationary phase, the sample compounds are retained individually and “travel” through the column with different velocities. This leads to a separation of individual components in time. The so-called retention time is the amount of time that elapsed from injection of the sample to the recording of the peak maximum of the component band (peak). When using non-specific detectors, the retention time is the only indication in chromatography for the correct identification of an individual sample component.

4.1.2 Apparatus

The carrier gas for argon detection shall be helium with purity ≥ 99,9999 %. Measurement starts with fusion of a test portion in a graphite crucible under helium gas at a temperature of about 2200 °C. The instrument samples a known volume of gas which subsequently enters the column(s) where the gases are separated. The apparatus shall, by an integrated function, correctly identify the signal which belongs to the separated argon. As an example, in Figure 1 a time relayed valve ejects all other gases to leave-out argon as the only analyte producing a signal. However, the chromatograph column may not be able to separate argon from all gases that may originate from the test sample. In that case, it may be necessary to integrate into the apparatus a separation and/or chemical conversion step prior to the gas stream entering the chromatograph column. The gases are ionized by an ionization source in a chamber and are detected and quantified by the instrument. An example of an apparatus is shown in Figure 1.

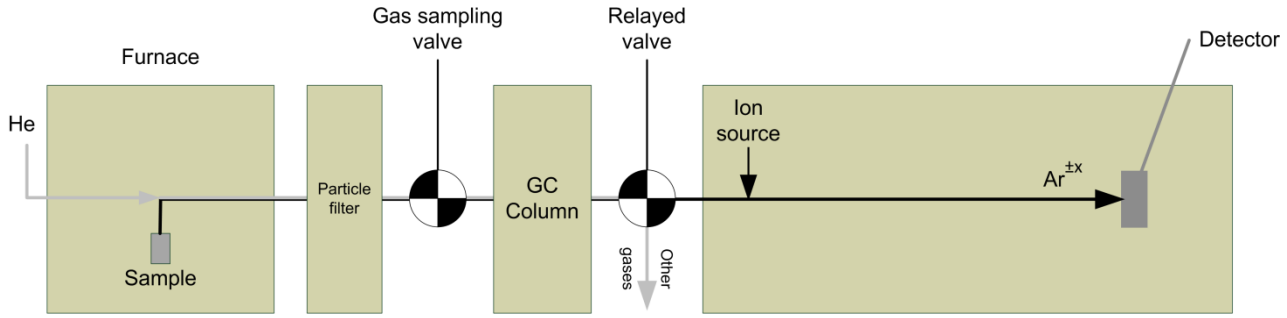

Figure 1 - Principle of gas chromatography

4.1.3 Equipment detection limit

The detection limit of the equipment shall at least correspond to a content of 0,02 µg/g (0,02 ppm by mass) Ar in a test sample prepared according to clause 6.

Note The detection limit does not correspond to the lowest level that can be quantified with statistical certainty.