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BSI Standards Publication

Nanotechnologies — Characterization of carbon nanotube samples using thermogravimetric analysis

National foreword

This Published Document is the UK implementation of ISO/TS 11308:2020. It supersedes DD ISO/TS 11308:2011, which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee NTI/1, Nanotechnologies.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Contents

Page

Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 229, *Nanotechnologies*.

This second edition cancels and replaces the first edition (ISO/TS 11308:2011), which has been technically revised. The main change compared with the previous edition is as follows:

— a generalization has been made from single-walled carbon nanotubes to all forms of carbon nanotubes (including multi-wall).

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Carbon nanotubes (CNTs) are allotropic forms of carbon with cylindrical nanostructures. As a result of their geometric structures, these materials exhibit unique mechanical, thermal and electrical properties[1][2][3][4][5]. CNTs are synthesized by several different methods, including pulsed laser vaporization, arc discharge, high pressure disproportionation of carbon monoxide and chemical vapour deposition (CVD)[6][7][8]. These processes typically yield a heterogeneous mixture of CNTs and impurities, often requiring post-synthesis purification. Commonly observed impurities include other forms of carbon [e.g. fullerenes, amorphous carbon, graphitic carbon, single-wall carbon nanotubes (SWCNTs) and multi-wall carbon nanotubes (MWCNTs) outside the desired size or chirality range], as well as residual metallic catalyst nanoparticles. Purification can be accomplished using gaseous, chemical or thermal oxidation processes[9][10][11][12].

Thermogravimetric analysis (TGA) measures changes in the mass of a material as a function of temperature and time, which provides an indication of the reaction kinetics associated with structural decomposition, oxidation, pyrolysis, corrosion, moisture adsorption/desorption and gas evolution. By examining the reaction kinetics for a given sample, the relative fraction of different constituents present can be either quantitatively or qualitatively determined.

TGA is one of a number of analytical techniques that can be used to assess impurity levels in samples containing CNTs[14][15][16][17][18][19][20][21][22]. For CNT-containing samples, TGA is typically used to quantify the level of non-volatile impurities present (e.g. metal catalyst particles). TGA is also used to assess thermal stability of a given sample, providing an indication of the type(s) of carbon materials present. Recent advances in TGA instrumentation enable better resolution during analysis. However, TGA alone is not specific enough to conclusively quantify the relative fractions of carbonaceous products within the material. Therefore, the information obtained from TGA should be used to supplement information gathered from other analytical techniques in order to achieve an overall assessment of the composition of a CNT sample[23][24][25][26][27][28][29].

Nanotechnologies — Characterization of carbon nanotube samples using thermogravimetric analysis

1 Scope

This document gives guidelines for the characterization of carbon nanotube (CNT)-containing samples by thermogravimetric analysis (TGA), performed in either an inert or oxidizing environment. Guidance is provided on the purity assessment of the CNT samples through a quantitative measure of the types of carbon species present as well as the non-carbon impurities (e.g. metal catalyst particles) within the material.

In addition, this technique provides a qualitative assessment of the thermal stability and homogeneity of the CNT-containing sample. Additional characterization techniques are required to confirm the presence of specific types of CNT and to verify the composition of the metallic impurities present.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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/TS 80004-3, *Nanotechnologies — Vocabulary — Part 3: Carbon nano-objects*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/TS 80004-3 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
primary oxidation temperature
T_{ox}'
temperature at which the most intense peak occurs in the first derivative thermogravimetric curve

3.2
thermal stability
dimensional stability of a solid material heated under specified conditions

Note 1 to entry: Thermal stability can be affected by the relative fraction of material *constituents* (3.4) in the sample as well as physical characteristics of the nanotube materials, such as diameter, length, defect state or surface treatment.

3.3
homogeneity
degree to which a property or a *constituent* (3.4) is uniformly distributed throughout a quantity of material

EXAMPLE Primary oxidation temperature.

