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Nanotechnologies — Characterization of single-wall carbon nanotubes using thermogravimetric analysis

Nanotechnologies — Caractérisation des nanotubes en carbone monofeuillet par analyse thermogravimétrique

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Foreword

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An ISO/PAS or ISO/TS is reviewed after three years in order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn. If the ISO/PAS or ISO/TS is confirmed, it is reviewed again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.

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/TS 11308 was prepared by Technical Committee ISO/TC 229, *Nanotechnologies*.

Introduction

Single-wall carbon nanotubes (SWCNTs) are an allotropic form of carbon which exhibit unique mechanical, thermal and electronic properties respective to the geometric structure[1][2][3][4][5]. SWCNTs can be synthesized by several different methods, including pulsed laser vaporization, arc discharge, high pressure disproportionation of carbon monoxide, and chemical vapor deposition[6][7][8]. These processes often yield a heterogeneous mixture of SWCNTs and impurities, requiring post-synthesis purification. Commonly observed impurities include other forms of carbon [e.g. fullerenes, amorphous carbon, graphitic carbon and multiwall carbon nanotubes (MWCNTs)], as well as residual metallic catalyst nanoparticles. Purification can be accomplished using gaseous, chemical and/or thermal oxidation processes[9][10][11][12].

Thermogravimetric analysis (TGA) is one of a number of techniques that can be used to assess impurity levels in as-produced and purified samples containing SWCNTs[14] to [22]. TGA measures changes in mass as a function of temperature and is widely used to assess reaction kinetics associated with structural decomposition, oxidation, corrosion, moisture adsorption/desorption, and gas evolution. By evaluating the reaction kinetics for a given sample, the relative fraction of different constituents present can be either quantitatively or qualitatively determined. For SWCNT-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 (a measure of the type or types of carbon present). However, TGA alone cannot conclusively quantify the relative fractions of carbonaceous products within the material. Therefore, the information obtained from TGA is used to supplement information gathered from other analytical techniques in order to achieve an overall purity and quality assessment of a SWCNT-containing sample.

Additional uses of TGA include process and quality control[23] and the characterization of MWCNTs[24][25][26][27][28] and few-walled carbon nanotubes[29].

Nanotechnologies — Characterization of single-wall carbon nanotubes using thermogravimetric analysis

1 Scope

This Technical Specification provides guidelines for the characterization of SWCNT-containing samples by the use of TGA, performed in an air environment. Guidance is provided on purity assessment of SWCNT samples through a quantitative measure of the non-carbon impurity (i.e. metal catalyst) level within the material.

In addition, this technique can provide a qualitative assessment of the thermal stability and homogeneity of the SWCNT-containing sample. Additional characterization techniques are required to confirm the presence of SWCNTs and to verify the composition of the metallic impurities present.

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/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.

3.1
primary oxidation temperature
temperature at which the most intense peak occurs in the derivative thermogravimetric curve

3.2
thermal stability
temperature at which the major carbon component oxidizes in an air (i.e. oxygen-containing) environment, represented by the primary oxidation temperature

3.3
homogeneity
measure of how uniformly distributed all constituents (nanotubes as well as impurities) of SWCNT material are throughout a larger sample, as determined by measuring repeated smaller samples using TGA

3.4
constituents
different components present in a SWCNT-containing sample

NOTE A SWCNT-containing sample is often comprised of different carbon and non-carbon materials and is identified by oxidation peaks in the TGA curve and by residual weight.