ISO 19749:2021 (E)

Nanotechnologies — Measurements of particle size and shape distributions by scanning electron microscopy

Contents

Foreword

Introduction

1 Scope

2 Normative references

3 Terms and definitions

3.1 General terms
3.2 Core terms: image analysis
3.3 Core terms: statistical symbols and definitions
3.4 Core terms: measurands and descriptors
3.5 Core terms: metrology
3.6 Core terms: scanning electron microscopy

4 General principles

4.1 SEM imaging
4.2 SEM image-based particle size measurements
4.3 SEM image-based particle shape measurements

5 Sample preparation

5.1 Sample preparation fundamental information
5.2 General recommendations
5.3 Ensuring good sampling of powder or dispersion-in-liquid raw materials
5.3.1 Powders
5.3.2 Nanoparticle dispersions in liquids
5.4 Ensuring representative dispersion
5.5 Nanoparticle deposition on a substrate
5.5.1 General
5.5.2 Nanoparticle deposition on wafers and chips of silicon or other materials
5.5.3 Nanoparticle deposition on TEM grids
5.6 Number of samples to be prepared
5.7 Number of particles to be measured for particle size determination
5.8 Number of particles to be measured for particle shape determination

6 Qualification of the SEM for nanoparticle measurements

7 Image acquisition

7.1 General
7.2 Setting suitable image magnification and pixel resolution

8 Particle analysis

8.1 Particle analysis fundamental information
8.2 Individual particle analysis
8.3 Automated particle analysis
8.4 Automated particle analysis procedure example

9 Data analysis

9.1 General
9.2 Raw data screening: detecting touching particles, artefacts and contaminants
9.3 Fitting models to data
9.4 Assessment of measurement uncertainty

9.4.1 General
9.4.2 Example: Measurement uncertainty for particle size measurements
9.4.3 Bivariate analysis

10 Reporting the results

Annex A (normative) Qualification of the SEM for nanoparticle measurements

A.1 Background
A.2 General
A.3 Measurement of spatial resolution
A.4 Measurement of drifts
A.5 Measurement of electron beam-induced contamination
A.6 Measurement of scale and linearity
A.7 Measurement of noise
A.8 Measurements of primary electron beam current

Annex B (informative) Cross-sectional titanium dioxide samples preparation

B.1 Background and objectives
B.2 Cross-sectional sample preparation

Annex C (informative) Case study on well-dispersed 60 nm size silicon dioxide nanoparticles

C.1 Background and objectives
C.2 ILC
C.3 ILC participants
C.4 Summary of the protocol in the ILC Phase 1
C.5 Details of sample preparation and observing conditions in ILC Phase 1
C.6 Size distribution measurement results in ILC Phase 1
C.7 Summary of the protocol in ILC Phase 2
C.8 Details of sample preparation and image acquisition conditions in ILC Phase 2
C.9 ILC Phase 2 size distribution measurement results
C.10 Remarks

Annex D (informative) Case study on 40 nm size titanium dioxide nanoparticles

D.1 Background and objectives
D.2 ILC
D.3 ILC participants
D.4 Protocols in the ILC Phase 1
D.5 Details of sample preparation and image acquisition conditions in ILC Phase 1
D.6 Size distribution measurement results in ILC Phase 1
D.7 Protocols in the ILC Phase 2
D.8 Details of sample preparation and image acquisition conditions in ILC Phase 2
D.9 ILC Phase 2 size distribution measurement results
D.10 Remarks

Annex E (informative) Example for extracting particle size results of SEM-based nanoparticle measurements using ImageJ

Annex F (informative) Effects of some image acquisition parameters and thresholding methods on SEM particle size measurements

F.1 General
F.2 Effect of slight electron-beam induced contamination
F.3 SEM parameters that can impact particle size measurements
F.4 Effect of the number of pixels representing particles

Annex G (informative) Example for reporting results of SEM-based nanoparticle measurements

Page count: 71