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Nanotechnologies — 3D image reconstruction of rod-supported nano-objects using transmission electron microscopy

Nanotechnologies — Reconstruction d'images 3D de nano-objets soutenus par des tiges à l'aide de la microscopie électronique à transmission

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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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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This document was prepared jointly by Technical Committee ISO/TC 229, *Nanotechnologies*, and Technical Committee IEC/TC 113, *Nanotechnology for electrotechnical products and systems.*

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.

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Introduction

Electron tomography, in transmission electron microscope (TEM), has impact on nanotechnology and nanomaterial metrology like that of computer tomography in medicine. For example, industries using nanotechnologies have requirements to verify materials, processes and products. Quantitative measurement at the nanoscale, including three-dimensional (3D) image reconstruction of nano-objects using TEM, responds to this need.

TEM, a two-dimensional (2D) imaging instrument, can provide 2D projection images of materials at the nanoscale, in the length range from below 1 nm to above 100 nm. From multiple 2D TEM images collected at suitable tilt increments, the 3D shape, size and volume parameters can be determined. This document describes sample preparation, instrumentation setup, data acquisition and processing for 3D image reconstruction of nano-objects using TEM, from which dimensional parameter values can be determined and interpreted. Variation in methodology for use with scanning transmission electron microscopy (STEM) is included in an informative annex.

The method described herein is limited to samples dispersed on or within an electron-transparent rod-shaped support. This method is particularly useful when the detailed shape of a limited number of objects, such as nanoparticles, is sought. For example, when 2D measurements yield a non-uniform distribution of objects, 3D image reconstruction can be used applied to study a small number of the objects in more detail. A variant of sample preparation is described that allows 3D reconstruction to be used in conjunction with 2D TEM analysis of a sample area of interest, such as an area containing outliers.

Potential applications for 3D image reconstruction of nano-objects using TEM are broad and might include validation of metrological artefacts, such as polystyrene latex nanoparticles, and site-specific analysis of interfaces buried within devices, and measurement of individual objects such as nanoparticles. The method might also be utilized to obtain detailed shape of non-symmetric nano-objects such as nanorods and nanocrystals.

Other applications include calibration for a variety of nanoscale characterization tools, particularly nanoscale characterization instruments and artefacts, to ensure that they are applied in a consistent way.

Case studies are provided in informative annexes, including variations of sample preparation, data acquisition, alignment and reconstruction methods. It is noted that placing of alternative data acquisition, alignment and reconstruction methods in annexes does not imply that a method is inferior to the one described in the main body of the document. Conversely, such might be the subject of future revisions of this document. However, the process, from sample preparation on a rod-shaped support to extraction of measurands, has been tested in accordance with the steps described in this document and tested on samples described in the annexes.

Figure 1 summarizes the procedure steps in this document. Normative aspects are highlighted in red. Informative aspects are highlighted in blue and appear in annexes. Additional annexes not listed in this figure are also included.

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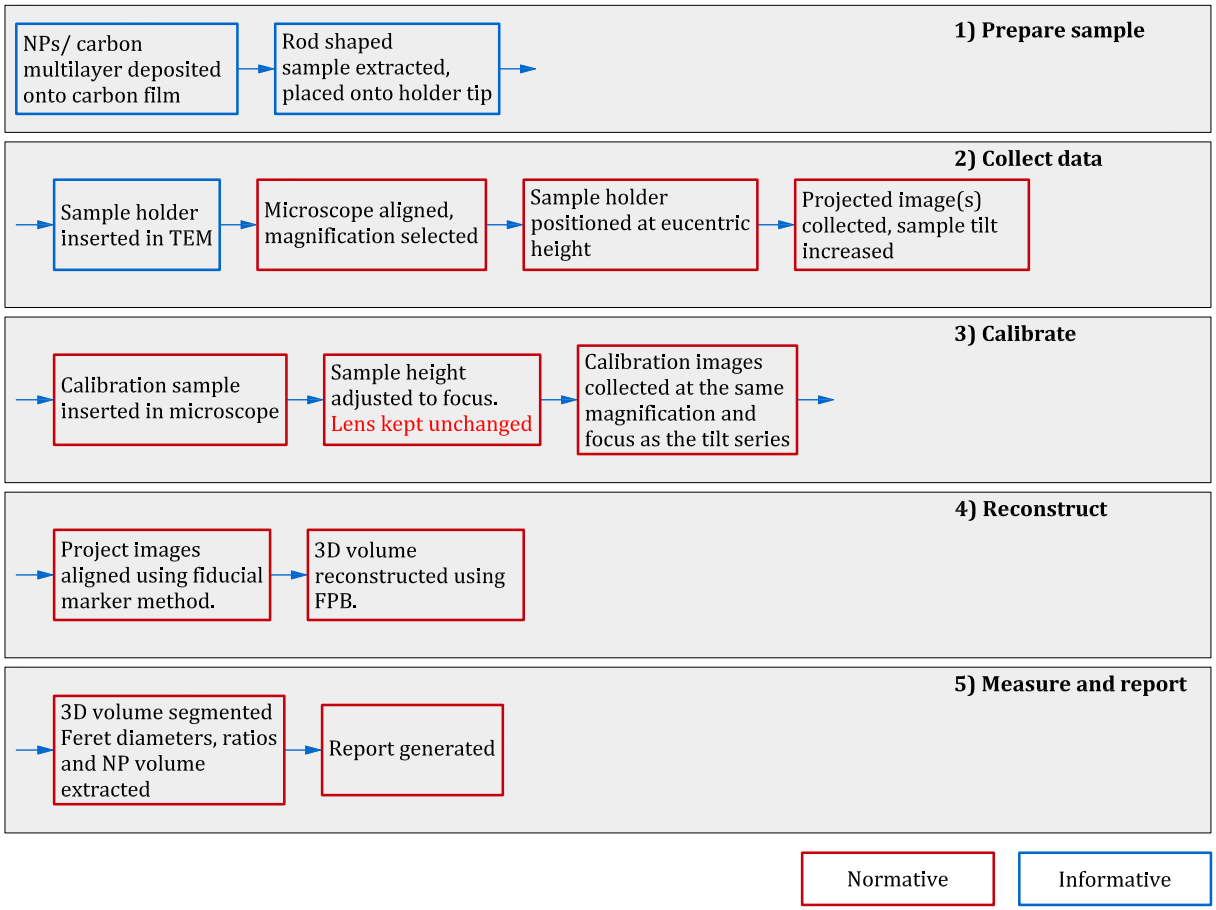

Figure 1 — Procedure steps

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Nanotechnologies — 3D image reconstruction of rod-supported nano-objects using transmission electron microscopy

1 Scope

This document provides guidance for sample preparation, data acquisition by transmission electron microscopy, data processing, and three-dimensional image reconstruction to measure size and shape parameters of nano-objects on rod-shaped supports. The method is applicable to samples dispersed on or within an electron-transparent rod-shaped support.

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/IEC Guide 99:2007, *International vocabulary of metrology — Basic and general concepts and associated terms (VIM)*

ISO/TR 945-2:2011, *Microstructure of cast irons — Part 2: Graphite classification by image analysis*

ISO/TS 10797:2012, *Nanotechnologies — Characterization of single-wall carbon nanotubes using transmission electron microscopy*

ISO 21363, *Nanotechnologies — Measurements of particle size and shape distributions by transmission electron microscopy*

ISO/TS 24597:2011, *Microbeam analysis — Scanning electron microscopy — Methods of evaluating image sharpness*

ISO 26824:2013, *Particle characterization of particulate systems — Vocabulary*

ISO/TS 80004-1:2015, *Nanotechnologies — Vocabulary — Part 1: Core terms*

ISO/TS 80004-2:2015, *Nanotechnologies — Vocabulary — Part 2: Nano-objects*

ISO/TS 80004-6:2021, *Nanotechnologies — Vocabulary — Part 6: Nano-object characterization*

ISO/TS 80004-8:2020, *Nanotechnologies — Vocabulary — Part 8: Nanomanufacturing processes*

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