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TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Key control characteristics – Part 4-3: Nano-enabled electrical energy storage – Contact and coating resistivity measurements for nanomaterials

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IEC Central Office
3, rue de Varembé
CH-1211 Geneva 20
Switzerland

Tel.: +41 22 919 02 11
Fax: +41 22 919 03 00
info@iec.ch
www.iec.ch

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FOREWORD

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Technical Specifications are subject to review within three years of publication to decide whether they can be transformed into International Standards.

IEC 62607-4-3, which is a Technical Specification, has been prepared by IEC technical committee 113: Nanotechnology standardization for electrical and electronic products and systems.

The text of this Technical Specification is based on the following documents:

Enquiry draft	Report on voting
113/239/DTS	113/263A/RVC

Full information on the voting for the approval of this Technical Specification can be found in the report on voting indicated in the above table.

This publication has been drafted in accordance with the ISO/IEC Directives, Part 2.

A list of all parts in the IEC 62607 series, published under the general title *Nanomanufacturing – Key control characteristics*, can be found on the IEC website.

The committee has decided that the contents of this publication will remain unchanged until the stability date indicated on the IEC website under "http://webstore.iec.ch" in the data related to the specific publication. At this date, the publication will be

- transformed into an International Standard,
- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

A bilingual version of this publication may be issued at a later date.

IMPORTANT – The 'colour inside' logo on the cover page of this publication indicates that it contains colours which are considered to be useful for the correct understanding of its contents. Users should therefore print this document using a colour printer.

INTRODUCTION

The future utilization of renewable energy technologies including e-mobility for individual transportation significantly depends on the development of efficient systems for energy storage. From today's perspective, lithium-ion batteries, supercapacitors and their derivative concepts are regarded as the most promising innovative candidates.

A high energy density for the desired power and a long life time (recharge characteristics) are the two most important criteria for electrode materials. Because many electrochemically active materials such as metal oxides show an inherently lower and insufficient conductivity for the electron transport, composite materials with carbon nanomaterial content are used for optimization of the current flow in the electrodes of a battery. The electrochemical reactions and the ensuing energy density of the battery cells are influenced by the movement of electrons in a composite. Furthermore, the electronic contact resistivity between the electrode material and the metal collector is important to realize a low ohmic internal resistance of the battery or capacitor device.

This part of IEC 62607 provides standard methods to measure coating and contact resistivity of nano-enabled electrode materials and to evaluate the best combinations of the composite material recipes and fabrication technologies for carbon containing coatings of such nano-enabled electrodes. Following this method will allow comparison of the results of different research groups.

This standardized method is intended for comparing the contact and coating resistivity of composite materials with carbon nanomaterial content in the study stage, not for evaluating the electrode in end products.

The method is applicable for nano-enabled materials exhibiting function or performance only possible with nanotechnology, intentionally added to composite materials for measurable and significant improvement of the current flow in the electrodes of electrical energy storage devices.

In this context it is important to note that the percentage content of nanomaterial of the device in question has no direct relation to the applicability of this part of IEC 62607, because minute quantities of nanomaterial are frequently sufficient to improve the performance significantly.

The fraction of nanomaterials in electrodes, electrode coatings, separators or electrolyte is not of relevance for using this method.

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 4-3: Nano-enabled electrical energy storage – Contact and coating resistivity measurements for nanomaterials

1 Scope

This part of IEC 62607 provides a standardized test method for the measurement of contact and coating resistivity of nano-enabled electrode materials. This method will enable a customer to:

a) decide whether or not a coating composite material is usable, and

b) select best combinations of coating composite material with fabrication technologies suitable for their application.

This part of IEC 62607 includes:

– definitions of terminology used in this part of IEC 62607,

– recommendations for sample preparation,

– outlines of the experimental procedures used to measure and calculate the contact and coating resistivity,

– methods of interpretation of results and discussion of data analysis, and

– a case study.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

3 Terms, definitions, acronyms and abbreviations

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/TS 80004-1 and the following apply.

3.1.1
electrode nanomaterial
material used in nano-enabled energy storage devices such as lithium-ion batteries or super-capacitors which contains a fraction of nanomaterial and exhibits function or performance made possible only with the application of nanotechnology

Note 1 to entry: Electrodes used in lithium-ion batteries or supercapacitors consist of mixed raw material powders (e.g. electrochemical active and carbon based nanomaterial powders) in a solvent with binder which forms a casting slurry. These slurries are coated by doctor blade process on thin metal collector foils, dried and subsequently calendar compressed to the final electrode. The electrode shows a multilayered layout, built up of (1) an aluminium or copper current collector and (2) the electrode material layer. This material layer consists of the active phase (cathode – lithium containing mixed oxides or phosphate, e.g. LCO, NCA, NCM, and LFP; anode, e.g. graphite and supercap – activated carbon), a conducting phase (e.g. carbon nanomaterials like CB, carbon nanotubes or fibres) and an organic binder (e.g. PVDF or SBR).

3.1.2
coating resistivity
resistance to the passage of an electric current through the electrode material layer

Note 1 to entry: It is expressed as electrical resistivity.

Note 2 to entry: The electric resistivity of the electrode material layer depends on several factors such as raw materials, the slurry processing step and the final electrode fabrication technology. Differences in the nanomaterial carbon content, the fabrication technology and the density or porosity of the layer can significantly influence its resistivity. It is possible to evaluate the resistivity by preparing a thin coating of electrode material on an isolator substrate. In the attached case study a sample design based on 5 cm^2 ceramic substrates is shown.

3.1.3
contact resistivity
electrical contact resistance between the metal current collector and the electrode material layer for a contact area of 1 cm^2

Note 1 to entry: During the life time of a battery the contact resistance influences the degradation stability (e.g. rise in internal resistance due to delamination), capacity loss during cycling or heating and rise of the internal temperature of the cell. The contact resistivity depends on the microstructure of the interface between metal collector and electrode material layer. The material and the electrode processing steps such as choice and pre-treatment of the metal collector or the calendaring process have an important influence on the contact resistivity. It is possible to evaluate the contact resistivity by preparation of a thin coating of electrode material on an isolator substrate. The method is derived from a “transmission line method” (TLM) used for characterization of contact resistivity of metal-semiconductor interfaces in the field of photovoltaics [1]. In the attached case study a sample design based on 5 cm^2 ceramic substrates is shown. The measurement of coating resistivity is carried out using a 4-point probe method.

3.1.4
calendaring
process where the electrode foils pass under rollers at a high pressure

Note 1 to entry: Calendaring is an important step during the electrode manufacturing process, because by this method the final electrode microstructure and thickness is formed. Methods like rolling or lamination are used to densify the electrode material layer to a desired degree of thickness and porosity.

3.2 Acronyms and abbreviations

LCO lithium cobalt oxide, $LiCoO_2$

NCA lithium nickel cobalt aluminium oxide, $Li(Ni,Co,Al)O_2$

NCM lithium nickel cobalt manganese oxide, $LiNi_{1/3}Co_{1/3}Mn_{1/3}O_2$

LFP lithium iron phosphate, $LiFePO_4$

CB carbon black

PVDF polyvinylidene difluorite

SBR styrene-butadiene rubber

EDLC electrical double-layer capacitor

TLM transmission line method

4 Sample preparation methods

4.1 General

The preparation of electrode nanomaterial samples consists of the following steps:

a) mixing a casting slurry;
b) assembly of metal collector strips on isolator carrier substrates;
c) casting of the slurry on these carrier substrates; and
d) drying and densification of the samples.

4.2 Reagents

4.2.1 Casting slurry

An electrode casting slurry is prepared in steps by dispensing and mixing different powders with solvent and binder. The choice of material recipe and the procedure of slurry preparation depend on the user and can be carried out similar to the industrial processes. The viscosity of the casting slurry should be in the range 0,5 Pa·s to 6 Pa·s (at low shearing rate 1/20 s). In this way, the slurry can be cast by doctor blade.

4.2.2 Isolator substrates

An isolator substrate serves as a carrier of the electrode coating. The substrate should be non-conductive, show a high accuracy in thickness homogeneity and flatness, a low roughness and a proper wettability with the casting slurry. Ceramic based thick film substrates like alumina with a thickness of (650 ± 5) mm, a flatness of < 10 mm per sample (50 mm × 50 mm substrate area) and a roughness R_a < 1 µm are recommended.

4.2.3 Metal collector strips and sample layout

Metal strips are cut out from the original current collector foil in the geometry of 2 mm width by 70 mm length. For the measurement of the coating resistivity four of these strips are bonded with glue based on cyanoacrylate in four-probe geometry (inner distance between metal strips contacts is 30 mm) on the isolator substrate. For the measurement of the contact resistivity 10 strips are bonded an equal distance (3 mm) from each other. Figure 1 shows the sample layouts. The choice of current collector material depends on the user and can be similar to industrial processes. Typical collector thicknesses are in the range 9 mm to 40 mm for aluminium and 10 mm to 20 mm for copper current collectors.

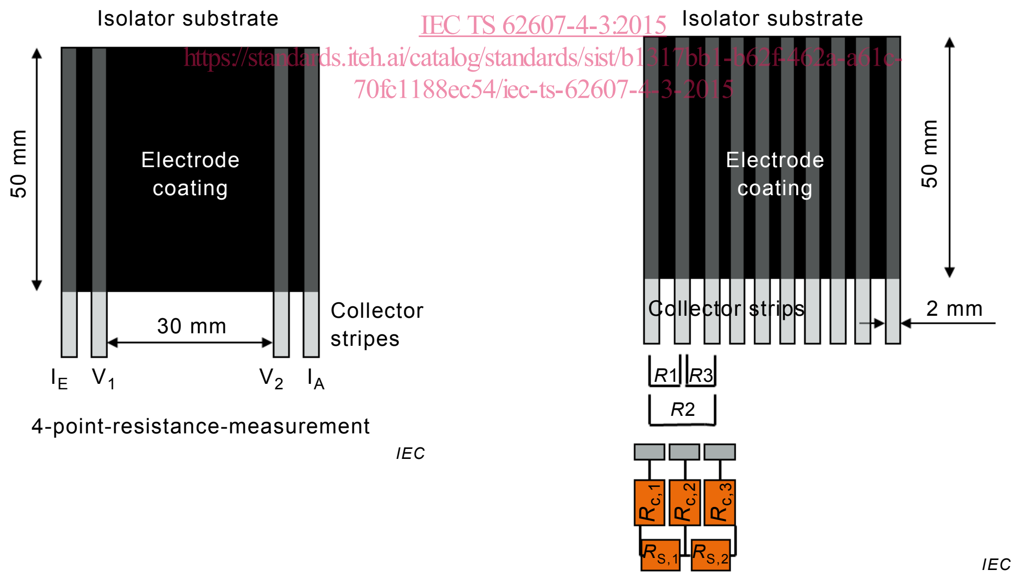

Figure 1 – Layout of the coating (left) and contact (right) resistivity measurement

4.3 Preparation of the electrode nanomaterial test samples

The slurry is cast by hand with a film applicator (doctor blade) on the isolator substrates which forms after drying the electrode layer. To set an accurate layer thickness the substrates are fixed on an aluminium carrier panel with cavities of the sample size of 50 mm × 50 mm. A doctor blade with a 500 µm slot is used. To prepare a series of samples of the same type and quality it is possible to arrange several substrates on the carrier and to coat them with a single coating step.