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# Determination of modulation period of nano-multilayer coatings by low-angle X-ray methods



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# Foreword

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This document was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 9, *Physical vapor deposition coatings.*

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

Nano-multilayer coatings by physical vapor deposition (PVD), which possess high coating-substrate adhesion, high hardness and good wear resistance, corrosion resistance and conductive resistance, have been widely applied on tools, moulds, microelectronics and other important parts to improve their service life. Nano-multilayers formed by depositing two materials alternately at nanometer scale have attracted considerable interest due to their superior physical and chemical properties. The modulation period refers to the thickness of these two alternate layers.

Based on the chemical compositions, the main nano-multilayer coatings involve metal/metal, metal/ceramic and ceramic/ceramic systems such as Cu/Ni, Cu/W, Cu/Ag, Ti/TiN, Cr/CrN, Zr/ZrN, TiN/CrN, CrN/AlCrN, TiC/TiCN and CrAlN/AlCrTiSiN. The key factor influencing the properties of nano-multilayer coatings was previously the modulation period, which has an important effect on properties including hardness, toughness, electromagnetic and optical property. For example, as the modulation period decreases, the hardness of the nano-multilayer coatings increases. At present, the high-resolution projection electron microscope (HR-TEM) and the X-ray methods including the X-ray reflectivity (XRR) and glancing incident X-ray diffraction (GIXRD) are the two common methods for determining the modulation period of the nano-multilayer coatings. X-ray methods are more suitable for determination of the modulation period due to the advantages of being non-destructive, statistical, convenient and accurate compared with HR-TEM.

However, there is not yet any standard to qualify the modulation period of these nano-multilayer coatings, which limits their further development.

Thus, the motivation of this document is to prescribe the calculation of the modulation period of the nano-multilayer hard coatings and the measurement conditions of X-ray methods. The modulation period is an important technical indicator of the nano-multilayer coatings, which can also provide the communication bridge for customers who want to use the coatings, tool coater and analytic service provider. This document can be used for quality assurance of products with nano-multilayer coatings.

# Determination of modulation period of nano-multilayer coatings by low-angle X-ray methods



## 1 Scope

This document specifies the substrate conditions and testing of the modulation period (including the principles for low-angle X-ray methods, the requirements of the coatings, the requirements for X-ray measuring apparatus, the calibration of apparatus and samples, and the testing conditions and calculation process) of nano-multilayer coatings by low-angle X-ray methods including X-ray reflectivity (XRR) and glancing incident X-ray diffraction (GIXRD).

## 2 Normative references

There are no normative references in this document.

## 3 Terms and definitions

No terms and definitions are listed in this document.

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

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

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

## 4 Substrate conditions

Considering that the roughness of industrial tools or moulds is normally not the roughness required, it is necessary to supply testing samples with the same substrate and surface condition. The roughness of the substrate of testing samples should be less than 50 nm of peak-to-valley or less than 5 nm RMS (route-mean-square) before the coating process. The substrate of testing samples should be cleaned by using the ultrasonic agitation procedure: immersion in a correct solution to remove hydrocarbons and others surface contaminants.

## 5 Testing of modulation period

### 5.1 Principle for low-angle X-ray methods

#### 5.1.1 General

The X-ray methods in this document consist of X-ray reflectivity (XRR) and glancing incident X-ray diffraction (GIXRD).

#### 5.1.2 XRR method

When X-rays are irradiated on to the sample at very low angles, and the angle of irradiation is gradually increased beyond a certain angle called critical angle, X-rays are reflected from the interfaces of the sample and give rise to interference fringes. The periodicity of the fringes is proportional to the thickness of the modulation period. The modulation period can then be calculated by a specific calculation. Figure 1 shows the typical schematic diagram of the XRR method.