STRUCTURE INVESTIGATION OF HARD COATINGS

BY TOTAL PATTERN ANALYSIS

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INTRODUCTION

X-ray diffraction is used in structure investigation of hard coatings as a complementary method to the complicated techniques of electron microscopy and electron diffraction. However, the routine X-ray methods are in many cases insufficient for a complete characterization of microstructure of thin films and unusual results are often said to be confusing. The main reason for these confusions is the fact that we are facing a new type of polycrystalline microstructure which is unknown in bulk materials. The only way how to understand the structure of these coatings is to use methods which are as complex as possible and which are accurate enough. The method of total pattern analysis (TPA), based on an analytical approximation of the whole diffraction spectrum, can fulfil these demands if it is accompanied by other methods for direct investigation of stresses and texture in the layers. Application of these methods for TiN coatings is presented here as an example.

TiN coatings are intensively studied during last few years for their great industrial importance. They are applied as wear resistant coatings on cutting tools, anticorrosive and antiabrasive coatings, selectively transmitting coatings on architectural glass, diffusion barriers in integrated circuits and in jewellery industry. Microstructure of these coatings strongly depends on deposition conditions and their properties can be accordingly modified. TiN coatings deposited by magnetron sputtering were investigated in our laboratory and new aspects of these coatings, namely an occurrence of large strains and stresses, were observed. Further, a dependence of microstructure of the coatings on their thickness was found together with a new type of inhomogeneity which is dependent on grain orientation.

Deposition Conditions

TiN coatings of thickness ranging from 1 μm to 12.5 μm were reactively sputtered onto polished substrates from 12 % Cr steel at substrate temperatures 50 °C or 150 °C. The sputtering was carried out in an atmosphere of argon and nitrogen at a total pressure of 0.3 Pa in a planar magnetron with a circular Ti-target. The deposition rate was in the range from 0.1 to 0.5 μm/min. Composition of the coatings was controlled by changing the N₂/Ar flow rate ratio and thickness by time of deposition.

J. Hašek (ed.), X-Ray and Neutron Structure Analysis in Materials Science
Fig. 1. Total pattern analysis of TiN coatings. Profiles of Kα₁ components of individual reflections are also shown.

EXPERIMENTAL

The coatings were investigated by X-ray diffraction using the Bragg-Brentano goniometer and monochromatized CuKα radiation. The angular position, integral breadth and integrated intensity of the Kα₁ components of individual reflections were determined by fitting analytical functions to the measured diffraction profiles (see Fig. 1). Most measurements were carried out with the flat specimen in symmetrical reflection position and thus only diffraction from crystallographic planes parallel to the specimen surface was registered. An asymmetric reflection position was used for the study of residual stress and preferred orientation only. Line broadening was analysed using the Williamson-Hall plots

$$B = \frac{1}{D} + \frac{4}{\lambda} e \sin \Theta,$$

where $B$ is the line broadening expressed in reciprocal space units ($B = \beta \cos \Theta / \lambda$, $\beta$ is line broadening in radians), $D$ is the domain size and $e$ strain ($e = \Delta d / d$).