X-ray analysis of protective coatings

Zoestbergen, Edzo

1 Introduction

The main objective of surface modification is to enhance the surface properties, while maintaining the bulk properties of the substrate. Deposition of a thin film on bulk material is one of the techniques to achieve this goal. In this thesis Physical Vapour Deposited (PVD) coatings are studied that are aimed at protecting a material against wear. The microstructure, crystallographic texture and the state of stress of these coatings may have a considerable influence on their performance in practical use and thus on their protective qualities. Examination of these characteristic properties may give an insight to the reason why some systems show excellent wear protection and how this property may be improved [1].

Roughly speaking, the thesis consists of three parts. In the first part, chapter 2, the theoretical background is presented. The second part, chapter 3 and 4, is focused on the effect of the deposition parameters on the characteristic properties of magnetron sputtered TiN coatings. The research presented in the final part, chapter 5 and 6, is devoted to the investigation of commercially produced PVD coatings. Three different systems are investigated: TiN, Ti(C,N) and a multi-layer system of alternating TiN and TiAlN sub-layers and the influence of the preliminary treatment on the properties of these systems is studied.

In chapter 2 the fabrication process of the magnetron sputtered and the commercial PVD coatings are described. Besides this, some of the physical and mechanical properties of the ceramic coating materials, investigated in this thesis, are summarised. Furthermore this chapter provides the necessary theoretical background for the interpretation of the experimental results obtained using X-ray diffraction and electron microscopy techniques [2].

In many cases it is important to control accurately the crystallographic orientation, surface texture and macro residual stresses. This is only possible when the physical controlling phenomena and the influence of the deposition parameters are understood [3,4]. In chapter 3, three deposition parameters: the bias voltage, the concentration of sputtered materials and the deposition times are varied. The effect of these deposition parameters on the microstructure and the crystallographic texture of the deposits is studied in the first part of this chapter. The state of stress of the deposits is also strongly related to these deposition parameters. Finally, the relation between the microstructure, the state of stress and the crystallographic texture is examined.

In chapter 4 the X-ray diffraction analysis of the magnetron sputtered coatings is treated in more detail. Determining the macro residual stress is only possible if linear elasticity is applicable. However, the diffraction experiments of chapter 3 showed a deviation from this linearity. In some cases the non-linearity seems to be related to the crystallographic texture that is present in the specimens. In the first section of chapter 4 the phenomena responsible for the texture and residual stress developments are studied in detail. Besides this non-linearity, it is found that the deformation strain in the specimens is very high and comparable to that of cold worked metals. To investigate both phenomena a commercially fabricated TiN film is isolated from the substrate and the change in residual stress and deformation strain is investigated. In the final section of chapter 4 a theoretical survey into the origin of deformation strain is performed. Furthermore, annealing experiments are carried out on the magnetron sputtered specimens to study the change in macro residual stress and deformation strain as a function of the heat treatments.

The last part of this thesis is concerned with the investigation of commercially produced PVD coatings. These coatings are manufactured according to a fixed procedure. Three different systems are studied: TiN, Ti(C,N) and a multi-layer system of alternating TiN and TiAlN sub-layers.

The PVD process enables the production of coatings composed of multi-layers, and in doing so it combines the advantages of the separate components into a single coating. The TiN/TiAlN multi-layer system is an example of this and combines the higher oxidation resistance, hardness and abrasive resistance of TiAlN at higher temperatures with the superior performance of TiN at lower temperatures [5,6]. In chapter 5 a study is presented of the microstructure, the morphology and the state of stress of TiN and the relatively new multi-layer system of TiN/TiAlN.

The treatment of a substrate prior to the deposition may largely influence the quality and performance of a coating. This preliminary treatment usually consists of two stages: a polishing and a cleaning stage. In chapter 6 the influence of the substrate roughness, prior to deposition, on the coating properties is investigated. In the first part the coatings are characterised and in the second part in-situ bending experiments are carried out to assess the mechanical properties of the coatings [7,8]. The mechanical properties are then studied in relation with the characteristic features of the coatings.

References

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