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X-ray analysis of protective coatings

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Summary

The main objective of the research described in this thesis is to acquire novel information about the microstructure of vapour deposited films in relation with the deposition parameters. Furthermore, an attempt is made to try to connect this knowledge to the mechanical properties of the films and to investigate the bonding behaviour of the protective coatings.

Deposition techniques make it possible to modify the surface of a material and to combine the surface properties of a coating with the properties of a substrate. In addition, different processes make it possible to manufacture various kinds of exotic materials and even very thin multi-layer structures. One of the most commonly used deposition methods is physical vapour deposition. This technique produces a vapour by bombarding a target with noble gas ions. When this vapour condenses a film is formed. It is possible to vary the chemical composition of the deposit by using different targets and adding reactive gases to the deposition chamber. PVD enables the production of films with a thickness ranging from a few nano-metre to a few micro-metre.

In order to increase the deposition rate of the target material on the substrate it is possible to apply a bias voltage between the deposition chamber and the substrate. This voltage accelerates positively charged ions towards the growing film and due to collision with uncharged atoms these ions increase the deposition speed. Furthermore, also the kinetic energy of the atoms and ions arriving at the deposit changes, which will influence the properties of the deposit.

The target material, the reactive gasses and the bias voltage all have a noticeable influence on the characteristic properties of the deposit and consequently the deposition is a very complex process.

In the first part of this thesis, the effects of three deposition parameters: the bias voltage, the deposition time and the concentration of the chemical elements, on the properties of TiN films are investigated. The results show that the bias voltage has a significant influence on the state of residual stress, the deformation strain and the crystallographic texture of the films. When the bias voltage is zero or approximately zero the macro residual stresses in the coatings have a thermal origin. The product of the difference between the linear expansion coefficients of the substrate and film, and the temperature difference between deposition and room temperature yields the magnitude of the stress. Increasing the negative bias voltage results in a densification of the film and an

increase in compressive residual stresses. This is a consequence of the higher kinetic energy of the atoms and ions, which removes loosely bonded particles from the surface and makes it possible that atoms penetrate the film. Due to the penetration of atoms the film will expand, which is constricted by the substrate. This will result in a compressive state of stress in the film. An indication that this so called atomic peening is indeed present, is visible in the specimens with a biaxial texture. The biaxial texture originates from the fact that the atoms arrive at the growing film with a preferential direction. Atoms will only penetrate aligned grains that correspond with this preferential direction and will damage the misaligned grains. The aligned grains will grow faster than the misaligned grains, which explains the biaxial texture. However, this will also result in a difference in the stress state between aligned and misaligned grains. This is also observed experimentally with X-ray diffraction. When the bias voltage is increased even more the crystallographic texture change from a biaxial into a {111} fibre texture.

Varying the deposition time makes it possible to change the thickness of the obtained deposit. However, this also has an influence on the magnitude of the compressive residual stresses. The compressive stresses are lower for thicker films and it is found that the magnitude of the residual stress is inversely proportional to the distance between the position in the film and the interface.

The second part of this thesis concerns the characterisation and mechanical testing of commercially manufactured coatings. Three different coating systems are investigated: TiN, Ti(C,N) and a multi-layer system of alternating layers of TiN and TiAlN. These three systems are deposited onto a tool steel substrate of which the surface roughness is varied. The coatings are characterised using X-ray diffraction and electron microscopy and are tested inside an Environmental Scanning Electron Microscope (E-SEM).

The TiN and Ti(C,N) coatings have a resemblance with the films fabricated at the highest bias voltage, i.e. high compressive residual stresses and a {111} fibre texture. Although, the quality of these commercially manufactured coatings is inferior to the non-commercial coatings. The texture of the multi-layer system is a {200} texture and the state of stress of the TiN sub-layers is different from that of the TiAlN sub-layers. The stress in the TiAlN sub-layers has only a thermal origin whereas the atomic peening stress is present for the TiN sub-layers. The compressive stress in the TiN sub-layers is also much higher than that of the single TiN films, this may be a consequence of the stress gradient that is present in these layers.

Increase of the substrate roughness does not only result in a decrease of the compressive stresses of the films but also the state of stress changes. The state of stress is not simply biaxial anymore, but tensile stresses perpendicular to the

interface develop. These stresses have a large influence on the film quality. Furthermore, the substrate roughness also affects the crystallographic texture. An increase of the roughness results in a decrease in diffraction intensity and therefore the texture is less pronounced.

To determine the mechanical properties of the coatings in-situ bending experiments are carried out in an electron microscope. During the bending experiments the coatings are loaded in compression and tension. During compressive loading only the coatings with the highest interface roughness show signs of failure both in the coating and at the interface. The reason that only for the specimens with the roughest substrate failure occurs is probably due to the tri-axial state of stress of these specimens. Loading of the coatings under tension results in cracks in the coating perpendicular to the interface. A crack will only propagate in the substrate when at the interface a brittle chromium carbide particle is present. Failure of the coatings starts after a certain offset bending. This is a result of the compressive residual stresses that are present in the coatings. TiN has the best crack resistance and Ti(C,N) the worst and there is no clear influence of the interface roughness visible. Therefore the influence of the substrate roughness is only visible in the loading under compression. Loading the coating in tension results in cracks in all the three system and in all systems local delamination of the coatings arises.