

Report on Doctoral thesis of Martin Daněk entitled "Design of Wear Resistant Coatings for High Temperature Industrial Applications"

Martin Daněk has carried out his doctoral research in a very important industrial topic, that of the design of wear resistant coatings for high temperature industrial applications. Many of the current coating compositions and architecture have been derived by trial and error and the present thesis tries to put the design of wear resistant coatings on a more scientific basis. However, as with many studies on complex systems where many factors affect the performance of the coatings, trying to rationalise the performance on scientific principles is difficult and thus incomplete. That is especially true for, for examples the explanations given for the change in residual stresses present upon thermally cycling the coatings designed for use in Aluminium High Pressure Die Casting. Nevertheless, the author has made a good attempt and thus the thesis is relevant to the current needs of industry.

The objectives of the thesis, although not explicitly stated, but discussed in chapter 4 and 5.3 are to design improved more temperature resistive coatings for cutting tools and for the dies used in high pressure die castings, the aim being that these tools can then be used for longer under given operating conditions or can be used under more extreme conditions where higher temperatures will be present. The objectives proposed to achieve this aim have largely been fulfilled especially for the case of improved coatings for cutting tools. The coatings developed, TiAlN with chromium additions, show an improvement in performance over the base line TiAlN coatings when machining at high speeds when the ability to withstand high temperatures in an oxidising atmosphere is the most important property. The conclusion is that the coating $\text{Ti}_{0.28}\text{Al}_{0.31}\text{Cr}_{0.51}\text{N}$ is a very good candidate for high speed dry cutting. This improvement was demonstrated in both laboratory tribological tests but also in practical tests on drills. In the case of the coatings developed for high pressure die casting laboratory tests suggested that the nitrided steel with $\text{AlCrTiN}-(\text{CrAl})_2\text{O}_3$ coatings performed well but this does not seem to have been confirmed by industrial results (page 181).

The methods used in the thesis are appropriate and appear to have been executed well. I would have liked to see error limits placed on the measured properties, e.g. hardness, Youngs modulus, residual stresses.

The thesis itself consists of six chapters which are in general well presented. The industrial aspects of the thesis are introduced in chapter 1, followed by two literature review chapters, chapter 2 being an overview of hard film preparation and chapter 3 on thin film growth. These chapters are detailed and contain an insight into the many variables associated with magnetron sputtering as a method of fabricating thin coatings and of the problems associated with the industrial production of these coatings. Chapter 2 shows that the author has read widely in this area and that is very familiar with the development and production of PVD coatings. Chapter 3 relates to the science behind the production of these coatings. The chapter is detailed but some aspects, for example the importance of the E/H ratios could have received greater attention. Chapter 4 gives a useful state of the art of industrial coatings where the aims of this research can be deduced. The experimental methods are introduced in Chapter 5. Many of these methods are standard methods used in tribology and thus a relatively brief introduction to these techniques is enough. The main experimental results and contributions of this work are contained in Chapter 5, section 5.2 on the development of

temperature resistive coatings for cutting tools and section 5.3 on the development of coatings for aluminium high pressure die castings. A model is presented in section 5.2.1 to assess the effect of sample rotation on the microstructure and composition of the coating produced by an industrial coating apparatus. I found this interesting work as it addressed the formation of the coatings from a very practical aspect. Details of the samples prepared are given. A rather annoying feature is in the nomenclature of the coatings. Page 126 states that "for easy coating identification, from now on, the coatings will be designated as $Ti_xAl_yCr_zN$ where x, y, z denotes the Ti, Al and Cr content in the films." The content of these elements in the films is then given in Table 5-2. This designation is used on that same page but in the figure caption for Figure 5-30 on the following page and elsewhere in the thesis a different notation is used. The test results on the coating evaluation are described well and the useful conclusions generated. The results from this experimental programme warrant publication in an appropriate scientific journal.

Chapter 5.3 contains the results on the development of coatings for aluminium high pressure die casting. Here the important properties are the thermal cycling resistance, adhesion of aluminium to the coatings, abrasion resistance and resistance against aluminium corrosion. Four chromium containing coatings with different morphologies and mechanical properties are chosen as the base layer deposited on nitrided and non-nitrided tool steel. The chemical composition of these base layer coatings is presented together with their adhesion and a stress analysis of the coatings both as deposited and after thermal cycling. An attempt is made to explain the changes in residual stress after thermal cycling and relate this to the E/H ratio. However, only tentative explanations can be provided. Various shield layers were studied and tested principally by pin-on-disc sliding against aluminium at ambient temperature and aluminium and alumina at high temperature. Based on these results web diagrams are provided from which the best base layer was an AlCrTiN coating on a nitrided steel while the best shield layer was a $(CrAl)_2O_3$ coating.

Chapter 6 provides a useful summary of the work.

The work adds to our knowledge in this area and is a useful contribution and provides a good basis for continued scientific work in this area, especially regarding the spinoidal phase transformation present in TiAlN and TiAlCrN coatings. The thesis most definitely satisfies the conditions of creative scientific work

The author of the thesis proved to have an ability to perform research and to achieve scientific results. I recommend the thesis for presentation with the aim of receiving a Ph.D. degree.



Dr Brian G Mellor

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