2003년도 한국표면공학회 춘계 학술발표회 논문 초록집

TiN/CrN 나노 박막의 기계적 특성에 관한 연구
Study on the mechanical properties of nanostructured TiN/CrN coatings

김광석*, 이상율 (한국항공대학교)
한준희 (한국표준과학연구원)
한전건 (성균관대학교)

1. *Introduction*

PVD hard coatings have been successfully applied to molds, punch, cutting tools and other machine parts to improve their lifetime for over two decades. The current interest in the hard coatings has been concentrated on the nanostructured coatings with hardness above 40GPa, so called superhard coating. The nanostructured coatings could be generally divided into two groups according to the difference in their structure. The first group is superlattice coatings, which are nanometer-scale multilayers composed of two different alternating layers with a superlattice period, such as TiN/VN, TiN/NbN, CrN/AlN, CrN/NbN and ZrN/CN and their hardening effect is attributed to the resistance to dislocation glide across interface between two layers. The second group is nanocrystalline composite (nanocomposite) coatings such as nc-MeN/a-nitride (a-nitride = a-Si3N4, a-TiB2, etc) and nc-MeN/metal (metal = Cu, Ni, Y, Ag, Co, etc) formed from isolated nanocrystal imbedded in thin amorphous matrix; here nc- and a- denote the nanocrystalline and amorphous phase, respectively, and Me is an element such as Ti, W, V, Mo, Zr, etc. to form hard nitride. The main hardening mechanism of nanocomposite coatings is attributed to the decrease of grain size (Hall-Petch effect) and the formation of dense and hard grain boundaries between the two phases. In these nanostructured coatings, it is well known that their mechanical properties are closely connected to the structure, crystal phase and chemical composition.

In this work, the nanostructured TiN/CrN coatings with various atomic concentration ratio of X=Cr/(Ti+Cr) were synthesized using a closed field unbalanced magnetron sputtering method with separate Ti and Cr targets and their structure, crystal phase and mechanical properties as a function of the X value in the nanostructured TiN/CrN coating system were investigated by glow discharge optical emission spectroscopy

(GDOES), X-ray diffractometry (XRD), transmission electron microscopy (TEM), nanoindentation and wear tests.

2. Experimental details

The nanostructured TiN/CrN coatings were deposited on silicon wafer of (100) orientation and nitrided AISI H13 steel using a closed-field unbalanced magnetron sputtering technique with separated Ti and Cr targets (round planar target: diameter 100 mm, t=10 mm). Prior to deposition of coating, the substrates were cleaned by the conventional cleaning process and the base pressure of sputtering chamber was pumped down to less than 3×10^{-5} Torr. In the deposition conditions of coating, the power of Ti target was maintained at pulsed DC 3kW (frequency 25 kHz and duty cycle 50%), while the power of Cr target was varied between DC 66W (power supply current setting: 0.2 A) and 0.63 kW (1.5 A) to produce coating with various atomic concentration ratio of X=Cr/(Ti+Cr). Other deposition conditions such as the distance of target-to-substrate, substrate bias voltage and substrate rotation speed were fixed to 60 mm, DC -100 V and 7 rpm, respectively. During the deposition process, the Ar pressure was set initially at 2.4×10^{-3} Torr and the reactive gas of N2, which is controlled by a feedback signal a mass spectrometer to a controller, was subsequently added to obtain desired gas composition, maintaining a total working pressure at 2.8×10^{-3} Torr. For all the coatings the deposition sequence started with the deposition of a Ti adhesion layer followed by a TiN bonding layer to promote coating adhesion. This was obtained while the substrates were held stationary above the Ti target.

3. Conclusions

In this work, the nanostructured TiN/CrN coatings with various atomic concentration ratios of X=Cr/(Ti+Cr) (from 0.15 to 0.59) were synthesized using a closed field unbalanced magnetron sputtering method and their mechanical properties were evaluated. The main results could be summarized as follows:

1. The single TiN coating has a preferred orientation of (111) at $2\theta=36.3^\circ$ and this peak has a strong tendency of decreasing the intensity and broadening the width with increasing the Cr content (or X value). This tendency indicates that the grain

size of TiN(111) gradually decreases.

2. The low-angle and high-angle XRD experiments show that the coating with $X=0.15$ is not a multi-layered film but a composite film, a mixture of TiN and CrN, with a grain size of approximately 16 nm.
3. The hardness and elastic modulus of nanostructured TiN/CrN coatings decrease with increasing the X value and were measured to be in a range from 31 to 41 GPa and from 390 to 440 GPa, respectively. The maximum hardness of approximately 41 GPa was observed from the coating with $X=0.15$, this value is approximately 1.6 times higher than that of the single TiN coating (approximately 27 GPa).
4. The maximum values of plastic deformation resistance (H^3/E^2 : 0.36 GPa) and elastic recovery (We : 66 %) were shown at the coating with $X=0.15$, which has an excellent wear resistance property than that of the single TiN coating. This enhancement on the mechanical properties could be attributed to the CrN doping effect, which has reduced the TiN grain size.

Reference

1. S. Veprek and M. Jilek, Vacuum, 67 (2002) 443-449.
2. J. Musil, Surf. & Coat. Technol., 125 (2000) 322-330.
3. P. Holubar, M. Jilek and M. Sima, Surf. & Coat. Technol., 133-134 (2000) 145-151.
4. William D. Sproul, Surf. & Coat. Technol., 86-87 (1996) 170-176.
5. Philip C. Yashar and William D. Sproul, Vacuum, 55 (1999) 179-190.
6. J. Musil, P. Karankova and J. Kasl, Surf. & Coat. Technol., 139 (2001) 101.
7. A.A. Voevodin, J.P. ONeill and J.S. Zabinski, Surf. & Coat. Technol., 116-119 (1999) 36.
8. S. Veprek, A. Niederhofer, K. Moto and et al., Surf. & Coat. Technol., 133-134 (2000)152.
9. L.S. Birks and H. Friedman. J. Appl. Phys. 17 (1946) 687.
10. C. Kim, S.B. Qadri, M. Twigg and A.S. Edelstein J. Vac. Sci. Technol. A **8** (1990) 3466.
11. T.F. Page, G.M. Pharr, J.C. Hay et al. Riester, Mater. Res. Soc. Symp. Proc. (1998) 522.
12. A. Leyland and A. Matthews, Wear 246 (2000)1-11.