Deposition of TiAlSiN hard film by cathodic arc plasma evaporation using a single target combined with a shield filter

Pham Van Vinh and Tran Vu Dien Ngoc
1Hanoi National University of Education, 136 Xuan Thuy Road, Cau Giay District, Hanoi, Vietnam
2Hanoi University of Technology, 1 Dai Co Viet Road, Hanoi, Vietnam

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Films of TiAlSiN were deposited on AISI H13 tool steel substrate by a cathodic arc-plasma deposition system using a single TiAlSi alloy as target. Presence of TiN crystal phase was found on films, but no XRD peaks related to Al and Si were found. Hardness, morphology and adhesion of films changed with different deposition parameters. Maximum hardness (36.29 GPa) of films was found on sample deposited at 300°C with 3x10^-3 Torr and cathode arc current of 45 A.

Keywords: Cathodic arc plasma, Hardness, Scratch, Shield filter, Single TiAlSi alloy target, TiAlSiN, Wear

Introduction

TiN is known as a hard coating material but it gives limited oxidation resistance at high temperatures during cutting process. In comparison with TiN, TiAlN coatings have better oxidation resistance and remain stable at higher temperature. Ti_{x}Al_{y}Si_{z}N has an oxidation temperature of 700°C, whereas TiN begins to oxidize at 500°C. Shizhi et al successfully deposited superhard TiSiN films, thus adding silicon into TiAlN films to improve mechanical properties. In general, dislocation with some mobility, microcracks and other structural defects of grain boundaries, formed during deposition, results in decrease of strength and hardness of materials. Materials, in which nanocrystalline TiAlN embedded into amorphous Si_{3}N_{4} matrix, can prevent dislocation within nanocrystals from sliding through amorphous grain boundary matrix even under highly applied stress. When silicon is added to films by using a multitarget approach with at least one target being a silicon-alloy, TiAlSiN films deposited exhibit very good mechanical properties. However, depositing of composite films has required a complicated configuration system with many targets. Münz et al showed that number and size of droplets would increase if melting point of targets was low. Mechanical properties of TiAlSiN films deposited by cathodic-arc evaporation using dual targets have been studied.

In this work, to retain simplicity of a single target while minimizing formation and affect of droplets, a shield filter was introduced, resulting in a simplified process with enhanced mechanical properties. Also, properties of TiAlSiN composite films deposited by cathodic arc evaporation using a single TiAlSi alloy target were studied.

Experimental Section

TiAlSiN films were deposited on AISI H13 tool steel substrate by typical cathodic arc-plasma deposition equipment (Fig. 1). A single target (diam, 63 mm) made of Ti_{x}Al_{y}Si_{z} alloy (Ti, 65; Al, 30; and Si, 5 wt %) is installed on chamber wall. Substrates were placed in front of target (distance, 280 mm). Small metal sheet between target and substrate acts as shield filter to reduce droplets on films. Substrates were manually ground with SiC papers, polished with Al_{2}O_{3} powder using a low-speed polishing machine, cleaned ultrasonically in pure alcohol and etched in argon gas with a current (0.6 A) for 10 min. Crystalline phases were determined by an X-ray diffractometer (Rigaku, RAD-3C). Compositional analysis was carried out by electron probe microanalyzer (EPMA). Morphology was observed using a scanning electron microscope (SEM) and optical microscope. Mechanical properties were studied using Vickers hardness, a wear tester and a scratch tester.
Results and Discussion

TiAlSiN films deposited (pressure, $3.0 \times 10^{-3}$ Torr; temp., 300°C; and bias voltage, -150V) were used for microanalysis. XRD pattern (Fig. 2) shows presence of TiN crystalline peaks. Although compositional analysis shows contents of Al (12.715%) and Si (2.897%), no peaks related with Al and Si were found, indicating that nitrides of Al and Si in films were amorphous. Two types of clusters corresponding to crystalline and amorphous phases are clearly visible in FE-SEM cross sectional image (Fig. 3a). It is therefore possible for crystalline grains to be grown as a columnar grain mixed with amorphous clusters. These results confirmed that TiAlSiN films are a composite of crystalline TiN and amorphous aluminum and silicon nitride. Looking into FE-SEM image of surface of films (Fig. 3b), droplets and clusters make surface rough, thereby adversely affecting mechanical properties of films. In arc-cathodic method, microparticles (size, 0.1-100 µm) transferred from target to substrate may cause this problem. It is possible to improve surface morphology by using a filter. Surface morphology of films deposited with shield filter shows significant droplet reduction (Fig. 3c).

Deposition temperature influences strongly the reaction constant, diffusion of defects, surface free energy etc. By increasing substrate temperature, mobility of atoms is also increased, leading to a structure with less imperfection. Films deposited at 300°C exhibited maximum hardness (Fig. 4a). Also, film surface showed smooth morphology. Keeping temperature (300°C) and
bias voltage (-150 V) constant, pressure was varied (0.5x10⁻³ - 7x10⁻³ Torr) for each sample (Fig. 4b) and observed that hardness increases with increase of pressure. However, further increasing pressure decreased hardness. Optical micrographs of these films (Fig. 5) show that width of scratch tracks increase as a function of pressure. Broken track dentations, found on samples deposited at higher pressure, result from bad adhesion. At low pressure during reactive deposition, low nitride concentrations result in increasing defects in films that reduces hardness. Samples deposited at pressure below 3x10⁻³ Torr (optimal pressure) showed low hardness. Above this pressure, hardness starts to fall again. Increase in nitrogen pressure leads to an increased nitride density that decreases kinetic energy of particles so inhibiting transfer to substrate. Low kinetic energy particles will adhere weakly to substrate leading to bad films. This was confirmed by an optical microscope that revealed some cracks on samples. Cracked films gave rise to cracked dentations track and decreased hardness. Hardness of films deposited at 300°C and 3.0 x 10⁻³ Torr as a function of bias voltage (Fig. 6) increases from 23.54 GPa to 34.32 GPa with increasing bias voltages from 0 V to -150 V; beyond this, hardness decreases. Under a bias voltage, ions of nitride and particles are driven to substrate. Ions’ bombardment of sample surface removes weakly adhering particles and transfers energy to substrate where film grows. Therefore, films deposited with a bias voltage show better adhesion and hardness than those without bias voltage. Hardness decreased beyond -150 V. Here ionic bombardment resulting from
high bias voltage impinges on substrate resulting in damage to films and a decrease in hardness.

Scratch tracts change with increasing bias voltage (Fig. 7). Scratch track of films deposited without bias voltage is rougher than with a bias voltage. Films deposited without bias also have rough surface and bad adhesion. Hardness increased with an increase of arc current (Fig. 8). Hardness reached 36.29 GPa in films deposited with arc current of 45 A. Increase of arc current increases not only deposition rate but also energy of particles. Films deposited with high arc current had high particle energy leading to improved transfer to substrate giving more hardness. Wear tracks of films (Fig. 9) show broadened wear track width on the sample deposited at arc current of 35 A and narrowed track on the sample deposited at an arc current of 45 A, indicating that sample deposited with arc current of 45 A exhibits a good wear resistance.

Comparing with reported\textsuperscript{17} hardness gained, hardness gained in present work is a little lower. However, with simple technique, these results are acceptable.

**Conclusions**

TiAlSiN composite films with mixed crystalline and amorphous phases were deposited successfully on AISI H13 tool steel by using an arc cathodic technique with a single TiAlSi alloy target. Droplets and consequential film degradation were significantly reduced when a shield filter was used in deposition process. Optimum hardness, best adhesion and wear resistance were found on samples deposited at: temp., 300°C, pressure, 3x10\textsuperscript{-3}Torr; bias voltages, -150V; and arc current, 45A.

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