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Study on the anti-wear and anti-impact property of composite CrAlTiSiN and TiAlN multilayer coatings

Tung-Sheng Yang *, Shang Yi Chang, Yin You Chang, Zong Han Xie

Department of Mechanical and Computer-Aided Engineering, National Formosa University, 64, Wunhau Rd., Huwei, Yunlin 632, TAIWAN.
*Corresponding author: tsyang@gs.nfu.edu.tw

KEYWORDS

Composite thin film
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ABSTRACT

The demand for multi-element and multilayer coatings composed of various materials has been growing steadily to enhance their mechanical and tribological properties. This study aims to: (1) deposit CrAlTiSiN composite coatings, TiAlN/TiN multilayers, and TiAlN/CrN multilayers using cathodic arc evaporation; (2) characterize the cross-sectional microstructure and adhesion properties of these coatings through electron microscopy and indentation testing; and (3) evaluate the wear resistance and impact resistance of the three coatings via wear and reciprocating impact tests. Results indicate that the deposited CrAlTiSiN composite, TiAlN/TiN multilayer, and TiAlN/CrN multilayer exhibited HF2, HF2, and HF3 fracture modes, respectively. Both the CrAlTiSiN composite and TiAlN/TiN multilayer demonstrated good film adhesion, while the TiAlN/CrN multilayer showed acceptable adhesion. In terms of wear resistance, the wear volumes of CrAlTiSiN, TiAlN/TiN, TiAlN/CrN, and CrAlTiSiN coatings were 310,778 μm^3, 271,948.8 μm^3, respectively. Regarding impact resistance, the CrAlTiSiN coating could withstand 60,000 cycles under a 98 N load, while the TiAlN/TiN and TiAlN/CrN coatings could withstand 20,000 cycles under 70 N and 30 N loads, respectively. Therefore, compared to TiAlN/TiN and TiAlN/CrN multilayer coatings, the CrAlTiSiN composite coating exhibited superior wear resistance and impact resistance.

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1.0 INTRODUCTION

TiN, CrN, and TiAlN thin films are widely employed as protective hard coatings due to their exceptional mechanical properties. However, single-phase coatings exhibit limitations in functionality. To overcome these limitations, researchers have actively explored multi-element alloyed, multiphase, and multilayer systems. By integrating materials with complementary properties, these novel coatings offer the combined advantages of their constituents while retaining the core functionalities of traditional coatings. This synergistic approach leads to significantly enhanced mechanical performance of the coatings. Multilayer thin films offer a unique combination of properties that meet the demanding requirements of cutting tools. This advantage stems from several factors: (1) they leverage the desirable characteristics of individual constituent materials within a single structure. (2) The presence of numerous interfaces, parallel to the substrate surface, impedes dislocation motion, enhancing both hardness and toughness of the coating. (3) Multilayer films can achieve significant thickness (>10 μm), fulfilling the wear resistance needs of cutting tools. (4) The layered architecture allows the film to dissipate stress effectively, mitigating peak stress at the surface and subsurface, ultimately leading to a higher load-bearing capacity. Nano-multilayer films, specifically, consist of alternating layers of two or more materials with nanoscale thickness. They inherit the beneficial properties of individual layers, while additionally boasting exceptional hardness, adhesion, wear resistance, and toughness.

Hard coatings are essential for surface processing as they significantly influent the working environment. To meet the demands of high efficiency and precision in the global market, these coatings must exhibit exceptional hardness, strength, wear resistance, and impact resistance. Such properties are crucial for extending component lifespan and enhancing product quality. The well-known Rockwell C indentation test, as prescribed by the VDI 3198 standard (Figure 1), is a destructive method for assessing the quality of coated materials (Archard, 1953). Figure 1 illustrates acceptable (HF1-HF4) and unacceptable (HF5-HF6) film adhesion. Nanoindentation has been employed to characterize the mechanical properties, such as elasticity and hardness, of thin films with varying thicknesses and compositions.

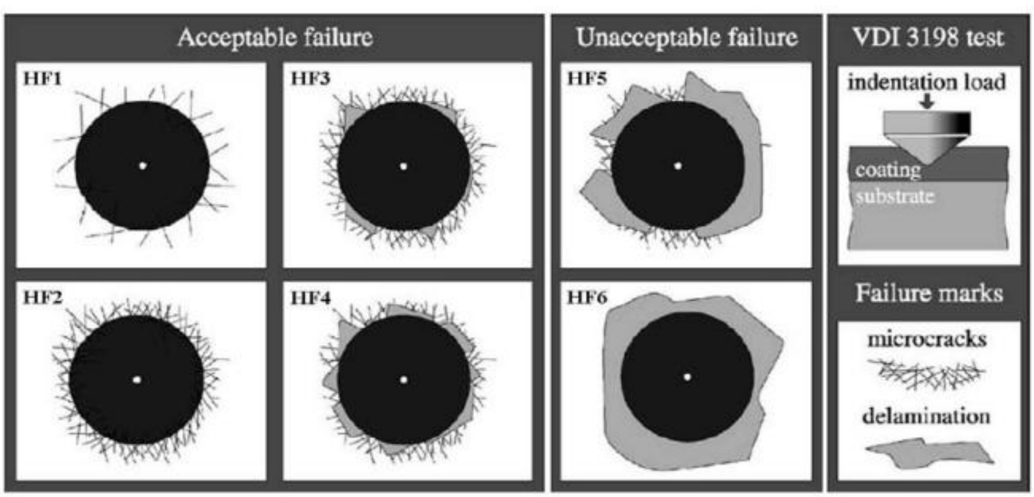


Figure 1: VDI 3198 norm.

TiAlN coatings have been widely employed as hard coatings due to their exceptional wear resistance, hardness, and oxidation resistance (Bressan et al. 2001, Yoon et al. 2002, Barshilia et al. 2009). TiAlN exhibits an oxidation initiation temperature of approximately 800 °C, leading to the formation of an Al_2O_3 oxide layer that contributes to its enhanced high-temperature wear resistance (Ohnuma et al. 2004). By incorporating an optimal amount of Si into TiAlN to form TiAlSiN, researchers have attributed the superior hardness, wear resistance, and oxidation resistance of TiAlSiN to its unique nanostructured composite. This composite comprises TiO_2 nanocrystals embedded within an amorphous SiO_2 matrix (Xie et al. 2012). Additionally, tribo-chemical reactions generate SiO_2 and $Si(OH)_2$, forming a lubricating friction layer that reduces the coefficient of friction and wear rate of TiAlSiN (He etal., 2016). Furthermore, (Fei et al., 2019) deposited three distinct Si-containing AlCrSiN gradient coatings via multi-arc ion plating for advanced cutting tool applications. The optimized AlCrSiN gradient coating exhibited superior adhesion strength, high hardness, and low elastic modulus. (Lim et al., 2023) investigated the microstructure and wear resistance of CrAlSiTiN coatings produced by co-alloying TiAlN with Si and Cr. The tribological behavior of these coatings, as assessed through reciprocating sliding and machining tests, was correlated with their microstructural features and surface oxidation mechanisms. The study concluded that SiO_2 offers superior lubrication and wear protection compared to Cr_2O_3. In another study, (Chen et al., 2021) isothermally annealed AlCrTiSiN nano-multilayer coatings at temperatures ranging from 800 to 1100 °C. The primary focus was on meticulously examining thermal stability and microstructural evolution to elucidate the thermal dissolution mechanism. Recent studies have demonstrated the promising oxidation resistance, thermal stability, and tribological performance of TiAlCrSiN coatings (Alhafian et al., 2023; Bobzin et al., 2020; Schulz et al., 2023).

In recent years, nano-multilayer coatings have emerged as a promising approach to enhance the hardness, wear resistance, thermal stability, and corrosion resistance of materials (Nordin et al., 1999; Barshilia et al., 2004). These coatings typically consist of alternating nanometer-scale layers of different transition metal nitrides, such as TiAlN/TiN, TiAlN/CrN, and TiAlN/ZrN (Knutsson et al., 2008; Povstugar et al., 2013; Li et al., 2007). By combining the complementary properties of single layer, these multilayer coatings offer superior mechanical, wear, and thermal performance compared to single-layer coatings. Consequently, they have found widespread applications in various industrial sectors (Knutsson et al., 2011; Ducros et al., 2006; Yu et al., 2017). Magnetron sputtering has been a key technique for depositing high-quality multilayer coatings. For instance, magnetron-sputtered TiAlN/CrN multilayers have exhibited exceptional hardness and oxidation resistance (Barshilia et al., 2005). In addition, Wei et al. (2011) demonstrated that TiN/TiAlN multilayer coatings significantly enhance the surface hardness, achieving up to 2.7 times the hardness of the substrate material. Zheng et al. (2018) further confirmed the superior wear resistance of TiN/TiAlN multilayer coatings compared to single-layer coatings on steel substrates.

Typically, mechanical property assessments of coatings and substrates often rely on static loading techniques, such as Rockwell hardness testing and nanoindentation. However, practical applications involve dynamic loading conditions, necessitating dynamic load testing for accurate evaluation of coating performance and interfacial integrity. (Bouzakis et al. 2004) pioneered the use of reciprocating impact testing to assess the impact resistance of micrometer-thick hard coatings by defining failure zones and analyzing crack propagation. This methodology is directly applicable to high-speed precision impact forming tools and components. (Lugscheider et al., 1999) found that nitride coatings exhibited superior impact fatigue durability compared to carbonitride coatings. TiN coatings have demonstrated enhanced fatigue life and contact fatigue strength compared to uncoated substrates (Xu et al., 2013). The formation of a hard TiN layer with high residual compressive stress through PVD processes can further improve tensile strength and fatigue resistance (Gopkalo and Rutkovskyy, 2011). Multilayer coatings, such as TiN/CrN and TiN/TiAlN, have shown significant advantages in impact fatigue resistance compared to single-layer coatings, especially on tool steel and cemented carbide substrates (Mendibide et al., 2006; Skordaris et al., 2014). However, Bobzin et al. (2023) emphasized the complex interplay between chemical composition, morphology, residual stress, and elasto-plastic deformation behavior, which can influence the impact fatigue performance of coatings, particularly thicker coatings.

In recent decades, TiAlN coatings have gained significant attention as hard coatings due to their exceptional wear resistance, hardness, and oxidation resistance. Recent studies have demonstrated that both multi-elemental CrAlTiSiN composite coatings and multilayer structures such as TiAlN/TiN and TiAlN/CrN can further enhance wear resistance and impact resistance. Consequently, this study aims to: (1) deposit CrAlTiSiN composite coatings, TiAlN/TiN multilayers, and TiAlN/CrN multilayers using cathodic arc evaporation; (2) characterize the cross-sectional microstructure and adhesion properties of these coatings through electron microscopy and indentation testing; and (3) evaluate the wear resistance and impact resistance of the three coatings via wear and reciprocating impact tests.

2.0 EXPERIMENTAL METHODS

2.1 Microstructure and Adhesion Experiment

Composite CrAlTiSiN, multilayer TiAlN/TiN, and multilayer TiAlN/CrN coatings were deposited on the polished surface of SKD61 die steel using a cathodic arc evaporation system. Cr, Ti, and pre-alloyed targets of CrAl (70 at.% Cr, 30 at.% Al), TiSi (80 at.% Ti, 20 at.% Si), and TiAl (50 at.% Ti, 50 at.% Al) were utilized for deposition. Table 1 summarizes the deposition parameters (pressure, time, current, bias voltage, substrate temperature, etc.) for each coating. To deposit the composite CrAlTiSiN coating, both CrAl and TiSi targets were evaporated in a nitrogen atmosphere, resulting in CrAlTiSiN formation on SKD61 die steel. TiN and CrN were used as interlayers within the multilayer TiAlN/TiN and TiAlN/CrN coatings, respectively. The specimen temperature was precisely controlled during deposition using a nearby thermocouple. The coatings were deposited on specimens mounted on a rotating substrate holder with controlled rotation speeds. An adequate cathode current was established, and Ar and N2 gases were introduced through a dedicated channel around the target to facilitate plasma reactions.

To investigate the cross-sectional structures of deposited composite CrAlTiSiN, multilayered TiAlN/TiN, and multilayered TiAlN/CrN coatings, a high-resolution field emission scanning electron microscope (JEOL JSM-7000F) equipped with secondary electron imaging and a backscattered electron imaging detector was employed. Backscattered electron micrographs revealed distinct contrasts between the various coatings, highlighting the differences in their microstructures. The adhesion of the coatings was evaluated using a Rockwell-C indentation hardness tester equipped with a standard Rockwell-C spherical top indenter, and the microstructure of the deformation zone was then examined using scanning electron microscopy (SEM). The deposited coatings were subjected to an indentation force of 1471 N. Following the VDI 3198 norm (Verein, 1991), the well-known Rockwell C indentation test was used as a destructive method to assess the quality of adhesion for coated compounds (Figure 1). The results indicated good, acceptable, and poor film adhesion for HF1-HF2, HF3-HF4, and HF5-HF6, respectively.

Table 1: Deposition parameters of the composite CrAlTiSiN coatings.

Coatings	**CrAlTiSiN**	**TiAlN/TiN**	**TiAlN/CrN**
Deposition parameters	Value	Value	Value
Deposition pressure (Pa)	3×10^{-2}	2.7×10^{-2}	2.7×10^{-2}
Deposition time (min)	60	85	85
Distance between cathode and substrate (mm)	150	145	145
CAE target	Cr, $Ti_{80}Si_{20}$, $Cr_{70}Al_{30}$	Ti, Cr, $Ti_{50}Al_{50}$	Cr, $Ti_{50}Al_{50}$
Cathode current (A)	Cr =70, $Ti_{80}Si_{20}$=90, $Cr_{70}Al_{30}$=70	Ti=60, Cr=60, $Ti_{50}Al_{50}$=60	Cr=60, $Ti_{50}Al_{50}$=60
Bias voltage at ion cleaning stage (V)	-800	-800	-800
Bias voltage at coating stage (V)	-100	-150	-150
Substrate temperature (°C)	200-230	230-250	230-250
Reaction gas	Ar, N_2	Ar, N_2	Ar, N_2
Rotation (rpm)	5	2.5	2.5

2.2 Friction and Wear Test

Tribological properties were evaluated using a UNMT-1 tribometer (CETR, USA) under dry sliding conditions at room temperature. A reciprocating pin-on-flat and surface contact wear mode was employed. The bottom sample was dynamically loaded, while the top pin remained stationary. Tests were conducted for 15 minutes under a normal load of 2.5 N, a wear track length of 18 mm, and a spindle speed of 200 rpm. WC pins (WC-6 wt% Co) with a diameter of 0.5 mm were used. Each specimen measured 40 mm × 40 mm × 10 mm. CrAlTiSiN, TiAlN/TiN, and TiAlN/CrN coated samples (substrate: SKD11) were evaluated for coefficient of friction and wear. A force sensor continuously measured the friction force between the pin and sample, with data recorded by a computer. The coefficient of friction was calculated as the ratio of friction force to applied normal force. White light interferometry was used to quantify the wear track on the test samples. The wear volume of each coating was calculated based on the wear cross-sectional area obtained under identical test conditions. Each test was repeated twice, and the results were averaged.

2.3 Reciprocating Impact Test

This study employed a cyclic loading device to evaluate the impact resistance of the deposited coating through an impact test. A schematic diagram of the reciprocating impact test apparatus is shown in Figure 2. The test utilized a continuously oscillating tungsten carbide-cobalt (WC-Co) carbide spherical indenter with a diameter of 1.5 mm impacting the coated specimen. The impact test was conducted at varying loads ranging from 9.8 N to 98 N and a fixed frequency of 8 Hz. The indenter was maintained at a constant distance of 2 mm from the specimen surface. The impact force and number of impacts were monitored to assess the development and extent of cracking on the coatings under different test conditions. Figure 3 presents a photograph of the assembled reciprocating impact testing machine.

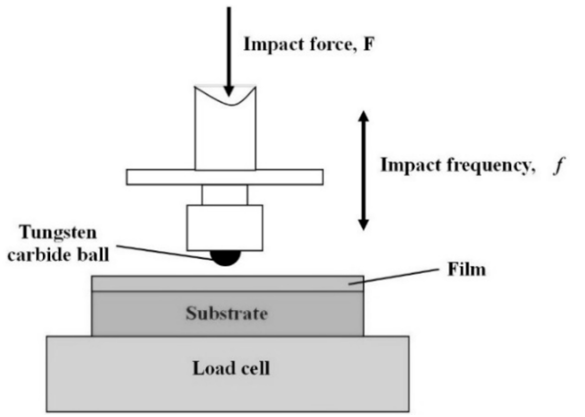


Figure 2: Schematic diagram of the reciprocating impact test device.

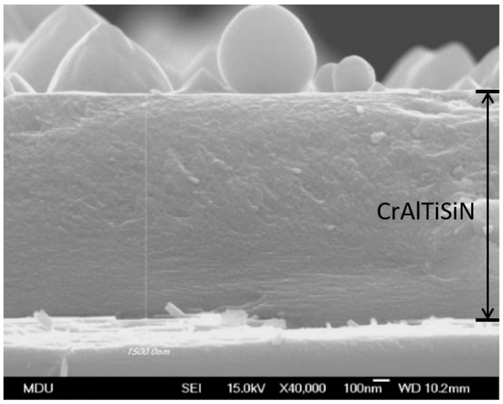
Figure 3: Reciprocating impact tester.

3.0 RESULTS AND DISCUSSION

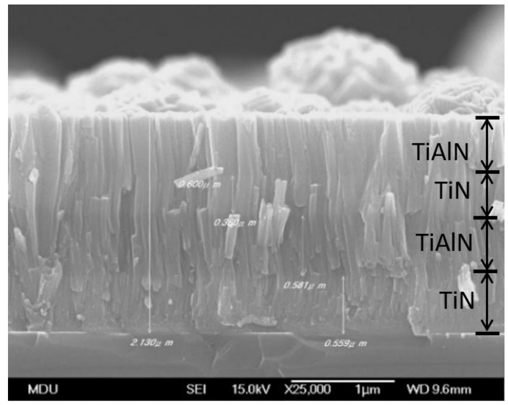
3.1 Microstructure and Adhesion Analysis

Figure 4 shows cross-sectional SEM images of the deposited coatings: composite CrAlTiSiN, multilayer TiAlN/TiN, and multilayer TiAlN/CrN. The thicknesses of these coatings were 1.5 μm (CrAlTiSiN), 2.13 μm (TiAlN/TiN), and 1.957 μm (TiAlN/CrN), respectively. Figure 4(a) presents Cross-sectional SEM diagrams for Composite CrAlTiSiN. Previous studies (Gui et al., 2021; Tu, Duan, Xu, & Li, 2014) have shown that TiAlCrN ceramic coatings exhibit superior mechanical properties and thermal stability compared to TiAlN and CrAlN, primarily due to grain refinement. The composite CrAlTiSiN was produced by co-evaporating CrAl and TiSi targets in a nitrogen atmosphere. Unlike traditional coatings, the CrAlTiSiN composite lacks a typical columnar structure, indicating that the co-evaporation process of CrAl and TiSi effectively inhibited columnar growth, resulting in a more compact structure(Bobzin, Kalscheuer, & Tayyab, 2024). Figure 4(b) shows that the multilayer TiAlN/TiN coating is composed of two layers of TiN and two layers of TiAlN. Similarly, as shown in Figure 4(c), the multilayer TiAlN/CrN coating has a comparable multilayer structure. Typical columnar structures were observed in the multilayer TiAlN/TiN, and multilayer TiAlN/CrN coatings.

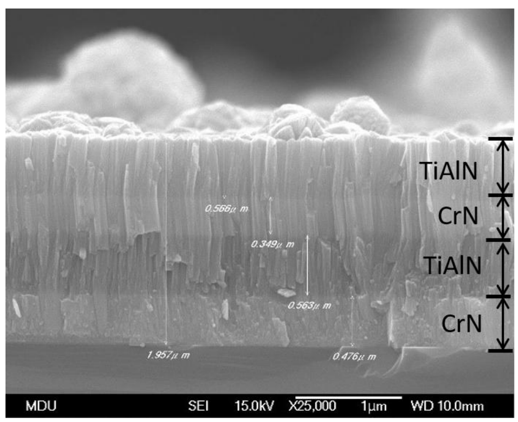
An indentation load of 1471 N was applied during the test, and this method caused plastic deformation and local damage near the indentation. Backscattered electron micrographs revealed distinct contrasts between the various coatings, highlighting the differences in their microstructures. As shown in Figure 3, the deposited CrAlTiSiN composite, multilayered TiAlN/TiN, and TiAlN/CrN coatings exhibit varying degrees of damage. To observe the fracture mode more clearly, a quarter of the model part is taken for observation. The failure mode and adhesion strength of these coatings were assessed based on the condition of the area surrounding the indentation crater. Radial cracks and some delamination (visible as white areas) are observed in the indented craters of the deposited CrAlTiSiN composite and multilayered TiAlN/TiN coatings. In contrast, the TiAlN/CrN coating shows more pronounced delamination. Interfacial adhesion is described as a complex behavior, where hard coatings deposited under specific conditions and multilayer structures demonstrate strong adhesion with well-designed layered (TiAlN/TiN) and composite (CrAlTiSiN) coating structures. However, the alternative structure (TiAlN/CrN) exhibited significantly lower adhesion compared to other tested configurations. (Chang, Yang, & Weng, 2020; Özkan et al., 2021). According to VDI 3198 (Vidakis, Antoniadis, & Bilalis, 2003), the deposited CrAlTiSiN composite, multilayered TiAlN/TiN, and TiAlN/CrN coatings exhibited HF2, HF2, and HF3 fracture modes, respectively. Both the CrAlTiSiN composite and multilayered TiAlN/TiN coatings demonstrated good film adhesion (HF2), while the multilayered TiAlN/CrN coating exhibited acceptable adhesion (HF3).



(a) Composite CrAlTiSiN coating

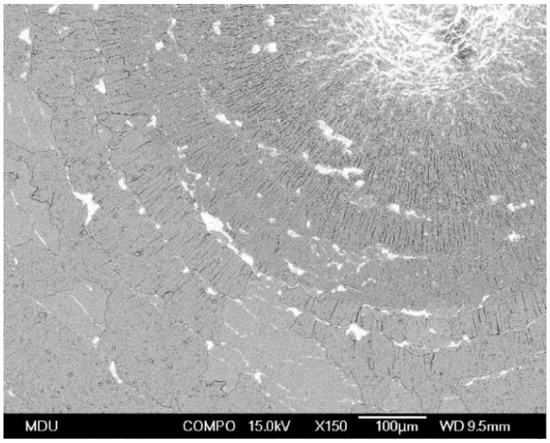


(b) Multilayered TiAlN/TiN coating

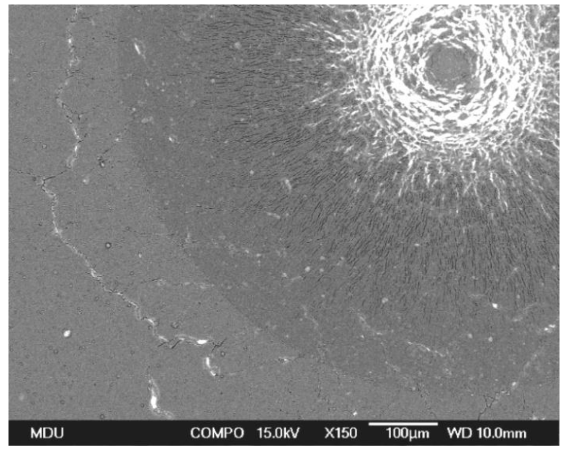


(c) Multilayered TiAlN/CrN coating

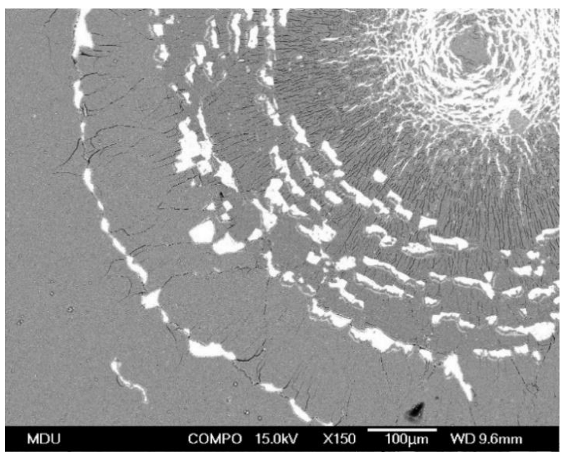
Figure 4: Cross-sectional SEM diagrams for Composite CrAlTiSiN, multilayered TiAlN/TiN, and multilayered TiAlN/CrN coating.



(a) Composite CrAlTiSiN coating (HF2)



(b) Multilayered TiAlN/TiN coating (HF2)



(c) Multilayered TiAlN/CrN coating (HF3)

Figure 5: Adhesion SEM diagrams for composite CrAlTiSiN, multilayered TiAlN/TiN, and multilayered TiAlN/CrN coating.

3.2 Friction and Wear Test

Friction and wear behavior were investigated for pins sliding against specimens coated with CrAlTiSiN, TiAlN/TiN, and TiAlN/CrN. Figure 6 illustrates the evolution of friction coefficients for the three coatings (CrAlTiSiN, TiAlN/TiN, and TiAlN/CrN) during testing. The TiAlN/TiN coating exhibited the highest average friction coefficient, followed by the TiAlN/CrN coating, while the CrAlTiSiN coating demonstrated the lowest average friction coefficient. The average friction coefficients for CrAlTiSiN, TiAlN/TiN, and TiAlN/CrN coatings as 0.67, 0.83, and 0.73, respectively. To evaluate the cumulative wear of specimens induced by fretting, specimen surfaces were scanned, and the volume wear of each specimen was measured. As shown in Figure 7, the wear volumes for CrAlTiSiN, TiAlN/TiN, and TiAlN/CrN coatings were 310,778 μm^3, 2,719,488 μm^3, and 4,219,463 μm^3, respectively. The TiN interlayer enhanced the adhesion strength of the deposited TiAlN/TiN coating, resulting in this multilayer structure achieving better anti-wear performance compared to TiAlN/CrN (Cui, Zhu, Man, & Yang, 2005). The CrAlTiSiN coating shows a lower friction coefficient and the smallest wear volume compared to the other two multilayer coatings, highlighting its superior wear resistance. With a dense, compact structure, the CrAlTiSiN composite coating displays minimal strain, a stable friction curve, and the lowest friction coefficient. Furthermore, it meets strong HF2 adhesion criteria and shows no significant delamination, achieving the best overall tribological performance (Lim et al., 2023).

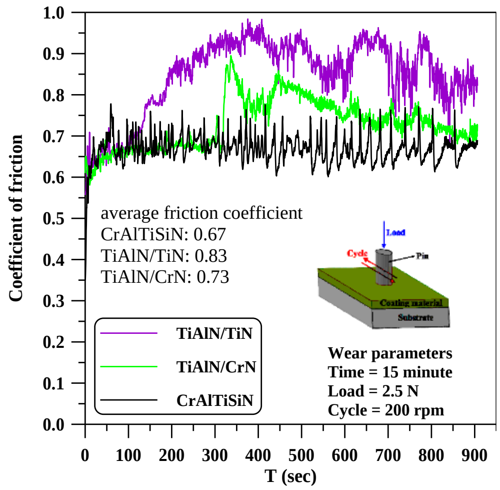


Figure 6: Relationship between friction coefficient and time for the three coatings. (Total distance = 108000 mm).

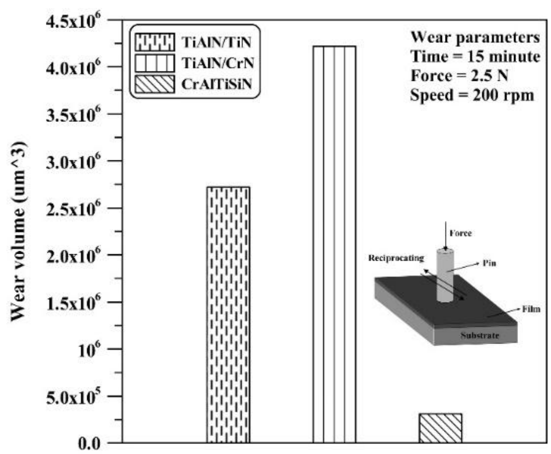


Figure 7: A comparison of the wear volume for the three coatings. (Total distance = 108000 mm).

3.3 Reciprocating Impact Test

Figure 8 presents the scanning electron microscopy (SEM) observations of the crack patterns formed on the CrAlTiSiN coating after a constant impact frequency of 8 Hz but varying impact forces. No fractures were observed at any of the tested forces (9.8, 30, 49, and 70 N) except for the case with 98 N force and 80,000 impacts. The crack patterns of different TiAlN/TiN and TiAlN/CrN coatings were also observed using the same method.

Figure 9 presents the crack resistance of CrAlTiSiN coatings under varying impact forces (9.8, 30, 49, 70, and 98 N) and impact cycles (20,000 to 100,000). Coatings remained intact without cracks when the impact force was below 49 N, even after 100,000 cycles. However, cracks were observed in coatings subjected to 70 N after 100,000 cycles and at 98 N after only 80,000 cycles. Figure 10 depicts the cracking behavior of the TiAlN/TiN coating under different impact forces and cycle numbers. Coatings withstood impacts up to 100,000 cycles without cracking when the force was less than 30 N. At 49 N, cracks appeared after 60,000 cycles, while at 70 N, cracking occurred after 40,000 cycles. When the force reached 98 N, cracks were evident even after 20,000 cycles. Figure 11 illustrates the cracking behavior of TiAlN/CrN coatings under various impact conditions. The coatings remained uncracked up to 100,000 cycles at 9.8 N. At 30 N, cracks formed after 40,000 cycles. For forces exceeding 45 N, cracking occurred even after 20,000 cycles.

Figures 9–11 reveal that CrAlTiSiN coatings demonstrated superior impact resistance compared to both TiAlN/TiN and TiAlN/CrN coatings. The CrAlTiSiN coatings sustained 60,000 impacts at a force of 98 N, while TiAlN/TiN coatings endured 20,000 impacts at 70 N, and TiAlN/CrN coatings withstood only 20,000 impacts at a force of 30 N. To enhance the bonding of hard coatings, such as the CrAlTiSiN composite and multilayer TiAlN/TiN and TiAlN/CrN coatings, to the metallic SKD61 tool steel substrate, composite coating structures and interlayers that are chemically compatible with both the coating and substrate have proven effective in improving adhesion, tribological performance, and resistance to repetitive impact. In this study, multilayer TiAlN/TiN and composite CrAlTiSiN coatings were used to increase the load-carrying capacity of soft metallic substrates, outperforming the TiAlN/CrN coating (Bouzakis, Anastopoulos, Asimakopoulos, Michailidis, & Erkens, 2006; Fang, Wang, Cai, Chen, Gyawali, & Zhang, 2024). Previous studies (Bouzakis et al., 2006; Chang & Chao, 2021) have shown that incorporating binary nitrides at the coating-substrate interface allows for adjustments to contact stiffness, thereby enabling control over adhesion strength and impact resistance. These results indicate that CrAlTiSiN coatings exhibit the most favorable anti-impact properties, followed by TiAlN/TiN coatings. TiAlN/CrN coatings demonstrated the poorest impact resistance among the three materials tested. The enhanced tribological performance and resistance to reciprocating impacts can be attributed to the increased adhesion strength in the multilayer coatings with TiN interlayers. Additionally, the composite CrAlTiSiN coatings demonstrated strong resistance to plastic deformation and maintained good adhesion strength. The composite CrAlTiSiN and TiAlN/TiN multilayer structures effectively inhibited the development of cracks in the subsurface.

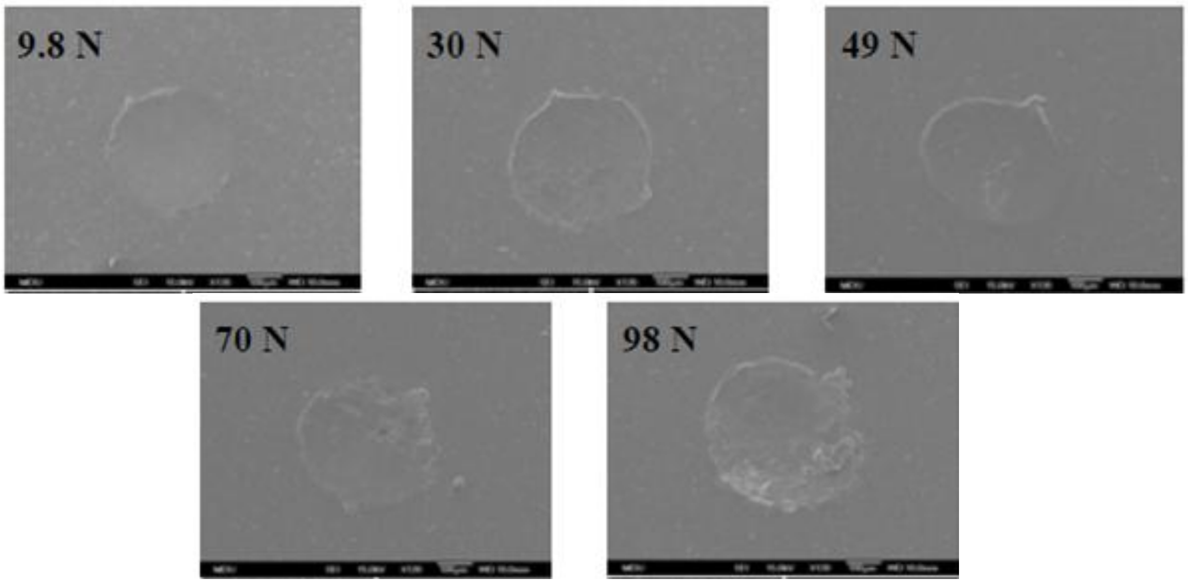


Figure 8: Surface topography under various impact loads of the CrAlTiSiN coating.

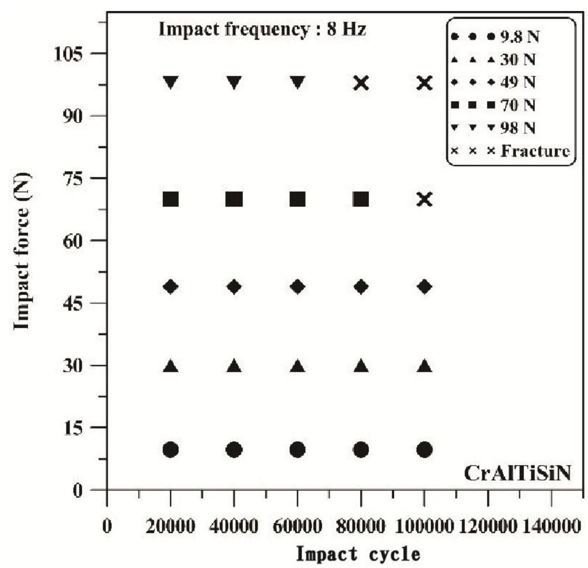


Figure 9: Crack conditions of the CrAlTiSiN coatings under various impact parameters.

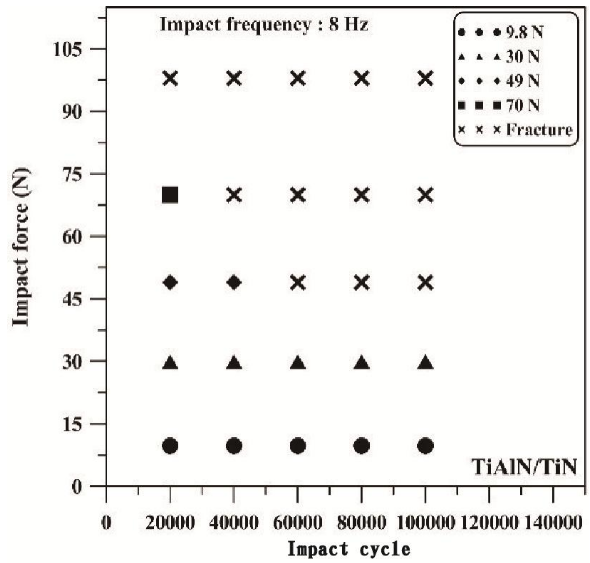


Figure 10: Crack conditions of TiAlN/TiN coating under various impact parameters.

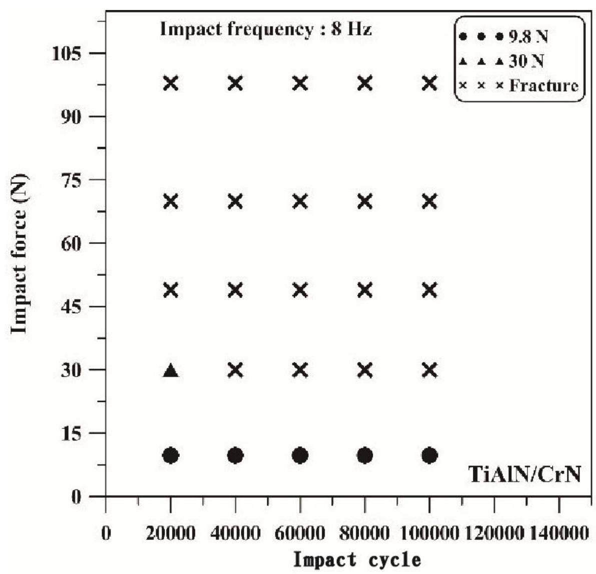


Figure 11: Crack conditions of TiAlN/CrN coating under various impact parameters.

CONCLUSIONS

Surface treatments and coatings play a crucial role in enhancing the performance of manufacturing tools by mitigating wear and improving impact resistance. This study investigates the mechanical and tribological properties of CrAlTiSiN composite and TiAlN multilayer coatings deposited via cathodic arc evaporation. Adhesion analysis revealed good bonding for CrAlTiSiN composite coating and TiAlN/TiN multilayer coating, exhibiting HF2 fracture modes. The TiAlN/CrN multilayer coating displayed acceptable adhesion with an HF3 fracture mode. Wear testing showed average friction coefficients of 0.67, 0.73, and 0.83 for CrAlTiSiN, TiAlN/TiN, and TiAlN/CrN coatings, respectively, with corresponding wear volumes of 310,778 μm^3, 271,948.8 μm^3, and 421,946.3 μm^3. In terms of impact resistance, the CrAlTiSiN coating could withstand 60,000 cycles under a 98 N load, while TiAlN/TiN and TiAlN/CrN coatings endured 20,000 cycles under 70 N and 30 N loads, respectively. These results demonstrate that CrAlTiSiN composite coatings exhibit superior wear resistance and impact resistance compared to TiAlN/TiN and TiAlN/CrN multilayer films.

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