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Structure and mechanical performance for TiAlN films that are grown with a low Al composition

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Abstract
An orthogonal array L9 (34) is used with the grey Taguchi design to optimize the performance of TiAIN films that are grown by direct current magnetron co-sputtering, using a low Al concentration. The structure, mechanical performance and the cutting properties of a coated TiAIN tool for which the films are deposited using various parameters are determined. For the optimal deposition conditions, the (111) diffraction peak intensity increases, which demonstrates an improvement in the crystallinity of the TiAIN films. A TiAIN film coated cutter insert is used for the dry milling of a Cr-Mo alloy steel workpiece and it is shown that the tool flank wear decreases and there are fewer surface defects proved. In the confirmation cutting tests the surface roughness is decreased by 26.08% and the flank wear is 16.15% less. The TiAIN films exhibit good mechanical performance, using grey relational analysis, the improvement rate in hardness H is 14.74%, in friction coefficient is 45.88%, in elastic modulus E is 9.56%, in H/E is 9.09% and in H3/E2 is 25.92%.

1. Introduction

Transition metal carbide films and nitride films that are coated by physical vapor deposition (PVD) or other methods have been the subject of much study [1]. PVD produces good coating characteristics, in terms of film density, structure and mechanical properties [2, 3]. Ternary nitride coatings (TiWN, SiWN, ZrWN, CrWN and TiAIN [4]) are used to enhance the characteristics of forming and cutting tools and find various industrial applications where there is an abrasive environment [5]. TiAIN coatings exhibit high surface hardness, reduced wear and high thermal stability, and are used for cutting tools and engineering components [6, 7]. Bouzakis et al [8] reported the effect of PVD parameters (deposition rate, duration time and substrate bias voltage) on the microstructure and performance of TiAIN hard films. Elmkhah et al [9] studied the effect of bias voltage on the structure, the mechanical and tribological properties, the surface roughness and the residual stress of TiAIN coatings. TiAIN films with good hardness (31 GPa) and superior tribological properties were obtained. Chang et al [10] produced TiAIN coatings using high-power impulse magnetron sputtering. The effect of the composition of the TiAIN films on the structure, hardness and corrosion behavior was determined. The fabricated Ti1−xAlxN film can have a polycrystalline, nanocomposite or amorphous structure when alloy targets with a different ratio of Ti-Al are used.

The Taguchi method is a systematic approach to reduce the number of experimental trials and optimize designs [11] and is used to improve product quality, and to resolve manufacturing problems [12]. The Taguchi method uses an orthogonal array to determine the important coating factors. Deng [13] used grey relational analysis to compensate for imperfect, uncertain and little information. Chen et al [14] deposited CrWN films coatings by direct current (DC) magnetron reactive co-sputtering system to enhance the performance of a cutting tool, and showed that the multiple functional characteristics of CrWN hard nanocomposite films are

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optimized using the grey Taguchi approach. Senthilkumar et al. [15] optimized cutting parameters using grey Taguchi analysis and achieved lower flank wear and surface roughness and a higher material removal rate, which clearly demonstrates that the performance is significantly enhanced using the grey Taguchi method. Wu et al. [16] used a grey relational Taguchi design to study the effect of coating conditions for nitride films on the structure and mechanical performance of the films and on the machining characteristics of coated cutting tools. The study shows that this method gives the optimal solution for complex multiple features.

TiAlN performs better than TiN because the Al in TiAlN layers forms a superficial coating of composite ceramic, which significantly increases resistance to oxidation, gives excellent wear resistance and prolongs service life [17]. The resistance of TiAlN films to oxidation depends on the Al content. There are few studies of TiAlN coatings with an Al concentration of less than 5 at%. Most studies use atomic concentrations of Al of more than 10 ~ 50 at% [9, 10].

This study uses TiAlN films with an Al concentration of less than 3.3 at%. TiAlN coatings are deposited on a glass and a NX2525 cermet insert by reactive DC co-sputtering. The grey relational approach is used to determine the multiple function characteristics of the Taguchi design to give an optimum coating process using a limited number of experiments [18]. The effect of the deposition conditions for TiAlN films on a tool that is used for the dry milling of SCM415 alloy steel is determined. The structure, the surface morphology, the coefficient of friction, the hardness and the elastic recovery of the TiAlN films are determined.

2. Experimental details

TiAlN hard films with Al concentrations of less than 3.3 at% were deposited on glass and cermet cutter inserts (NX2525, Mitsubishi) by reactive DC co-sputtering. Pure Ti (76.2 mm diameter) and Al (50.8 mm diameter) targets and an Argon and Nitrogen gas mixture were used. The composition of the TiAlN films was changed by adjusting the DC power and the diameter of the metal target. Grey Taguchi design was used to determine the effect of the deposition conditions on a coated TiAlN film cutting tool. Table 1 lists the coating parameters for TiAlN thin films and the factors and levels. An orthogonal array L9 (34) is used for the Taguchi design of experiment. An analysis of variance (ANOVA) and the signal to noise ratio (S/N) are used to determine the properties of the film coatings. Optimum values are obtained by using the highest values of the average S/N ratios [19].

Milling is a common machining method that is used in the production of many components. The dry milling of chrome-molybdenum alloy steel (JIS SCM415) requires coated tools. Typical mechanical properties and chemical compositions for SCM415 steel are given in [20]. Cermet inserts (with and without TiAlN films coated) were placed in a conventional vertical milling machine (figure 1) to mill a JIS SCM415 steel workpiece. The face milling head has a diameter of 50 mm, with a single tooth cutter and the cutting speed is 188 m min−1. The workpiece is 200 mm long and 50 mm wide. The total depth of cut is 2.5 mm and the feed rate is 5.4 mm rev−1. Table 2 lists the dry milling parameters.

After deposition, the morphology of the film was measured using a field emission scanning electron microscope (JEOL JSM-6500F, SEM). Energy dispersive x-ray spectroscopy (EDS) was used to determine the semi-quantitative chemical composition of the coatings. Film thickness was measured using a surface profilometer (AMBIOS XP-1, α-step). The phase structure of the film was measured using x-ray diffractometry
X-ray Generator Rigaku-2000 with Cu Kα radiation (0.1541 nm). The coefficient of friction and the wear behavior of the TiAlN film were measured using a ball-on-disk tribometer (CSM Instruments, Switzerland). The hardness, the elastic modulus and the elastic recovery of the films were determined using a nanoindenter (ASMEC UNAT). The surface roughness of workpiece and the flank wear for the cutter were measured as performance indices, in order to determine the quality of the dry milling process. Flank wear is used to analysis the service life of a cutter. The surface roughness Ra is measured using a profilometer (Suftest-402 Mitutoyo) [13]. SEM morphology was used to measure flank wear.

2.1. Grey relational analysis
Grey Taguchi relational analysis is used to determine the complicated relationships between multiple performance characteristics [21]. The grey relational coefficient is written as [13]:

$$r(x_i(k), x_k(k)) = \frac{\min\min x_0(k) - x_i(k) + \zeta \max\max x_0(k) - x_i(k)}{x_0(k) - x_i(k) + \zeta \max\max x_0(k) - x_i(k)}$$

where $x_i(k)$ is the normalized value of the $k$th performance index in the $i$th experiment and $\zeta$ is a distinguishing coefficient: $\zeta \in [0, 1]$. The value of $\zeta$ is adjusted according to the actual requirements of the system. For this study, the coating process parameters for the TiAlN films are equally weighted so the value of $\zeta$ is 0.5.

The grey relational grade is a weighting-sum of the grey relational coefficient. It is defined as [13]:

$$r(x_0, x_i) = \frac{1}{n} \sum_{k=1}^{n} r(x_0(k), x_i(k))$$

where $n$ is the number of characteristics. The value of the grey relational grade ranges from 0 to 1. The optimum level for the coating condition is the level that produces the best grey relational grade [21].
Table 3. Elemental constitution of the coated TiAlN films, measured using EDS analysis.

| Factor          | Atomic %   | Film thickness (nm) |
|-----------------|------------|---------------------|
| No.             | A | B | C | D | Ti | Al | N₂ | Total |                  |
| 1               | 1 | 1 | 1 | 1 | 57.51 | 1.85 | 40.64 | 100 | 356 |
| 2               | 1 | 2 | 2 | 2 | 59.43 | 2.34 | 38.23 | 100 | 392 |
| 3               | 1 | 3 | 3 | 3 | 57.47 | 2.67 | 39.86 | 100 | 378 |
| 4               | 2 | 1 | 2 | 3 | 62.31 | 2.81 | 34.88 | 100 | 396 |
| 5               | 2 | 2 | 3 | 1 | 62.81 | 2.74 | 34.45 | 100 | 408 |
| 6               | 2 | 3 | 1 | 2 | 64.70 | 3.01 | 32.29 | 100 | 418 |
| 7               | 3 | 1 | 3 | 2 | 63.88 | 2.55 | 33.57 | 100 | 399 |
| 8               | 3 | 2 | 1 | 3 | 65.42 | 3.14 | 31.44 | 100 | 346 |
| 9               | 3 | 3 | 2 | 1 | 63.74 | 3.26 | 33.00 | 100 | 337 |

Note: A = Ti DC Power (W), B = Al DC Power (W), C = flow rate of N₂/(N₂ + Ar) ratio (%), D = Substrate bias (−V), Ti target diameter = 76.2 mm, Al target diameter = 50.8 mm.

Table 4. Experimental results and the corresponding S/N ratios for surface roughness and flank wear, for coated and uncoated cermet tools that are used for dry milling (three trials for each instance).

| Factors | Flank wear (μm) | The lower the better S/N (dB) | Surface roughness, Rₐ (μm) | The lower the better S/N (dB) |
|---------|-----------------|-------------------------------|-----------------------------|-------------------------------|
| No.     | A | B | C | D | F1 | F2 | F3 | Ave. | Stdev. | S1 | S2 | S3 | Ave. | Stdev. |
| 1       | 1 | 1 | 1 | 1 | 8.3 | 9.0 | 9.1 | 8.80 | 0.436 | −18.89 | 0.48 | 0.56 | 0.33 | 0.46 | 0.117 | 6.74 |
| 2       | 1 | 2 | 2 | 2 | 9.8 | 8.4 | 9.8 | 9.33 | 0.808 | −19.40 | 0.37 | 0.16 | 0.24 | 0.26 | 0.106 | 11.70 |
| 3       | 1 | 3 | 3 | 3 | 8.8 | 8.4 | 9.4 | 8.87 | 0.503 | −19.96 | 0.44 | 0.41 | 0.25 | 0.37 | 0.102 | 8.64 |
| 4       | 2 | 1 | 2 | 3 | 7.3 | 9.1 | 8.8 | 8.40 | 0.964 | −18.49 | 0.32 | 0.13 | 0.24 | 0.23 | 0.095 | 12.77 |
| 5       | 2 | 2 | 3 | 1 | 8.4 | 8.2 | 8.9 | 8.50 | 0.361 | −18.59 | 0.22 | 0.16 | 0.27 | 0.22 | 0.055 | 13.15 |
| 6       | 2 | 3 | 1 | 2 | 7.8 | 7.0 | 7.5 | 7.43 | 0.404 | −17.42 | 0.24 | 0.23 | 0.21 | 0.23 | 0.015 | 12.77 |
| 7       | 3 | 1 | 3 | 2 | 8.1 | 7.6 | 8.6 | 8.10 | 0.500 | −18.17 | 0.25 | 0.28 | 0.25 | 0.26 | 0.017 | 11.70 |
| 8       | 3 | 2 | 1 | 3 | 11.9 | 10.5 | 11.0 | 11.13 | 0.709 | −20.93 | 0.35 | 0.27 | 0.29 | 0.30 | 0.042 | 10.46 |
| 9       | 3 | 3 | 2 | 1 | 10.8 | 10.1 | 11.8 | 10.90 | 0.854 | −20.75 | 0.66 | 0.57 | 0.46 | 0.56 | 0.100 | 5.04 |
| Uncoated | | | | | 15.2 | 14.3 | 14.7 | 14.7 | 0.451 | 1.21 | 1.17 | 1.29 | 1.22 | 0.061 | |

3. Results and discussion

This study determines the effect of each of the coating conditions on the chemical composition, the mechanical behavior and the surface morphology of a TiAlN film. Table 3 lists the EDS compositional analysis for the TiAlN films, which shows the presence of elemental Ti, Al and N₂. The Ti content is 57.47 ~ 65.42 at%, the Al content is 1.85 ~ 3.26 at% and the N₂ content is 31.44 ~ 40.64 at%. The film thickness ranges from 337 to 418 nm.

To determine the effect of the TiAlN film coating conditions on the characteristics of cutter inserts in terms of wear behavior during milling, experiments used a conventional vertical milling machine. No coolant is used for dry machining. Cutting processes that do not involve pollutants decrease environmental damage [22]. Table 4 shows the experimental results for the flank wear and surface roughness for coated and uncoated cermet cutter inserts that are used for dry milling and the relevant S/N ratio. Experimental result show that the flank wear ranges from 7.43 to 11.13 μm and the surface roughness ranges from 0.22 to 0.56 μm. A TiAlN film that is coated on the cutter inserts results in a significant reduction in surface roughness and flank wear. All of the coated cutters have a much longer tool life than uncoated tools. The standard deviation (Stdev.) for the flank wear is 0.361 to 0.964 and the surface roughness varies from 0.015 to 0.117. A small variance indicates that the experimental outcomes are close to the mean value.

Table 5 lists the results of an ANOVA for flank wear and surface roughness. The Ti DC power and the substrate bias have the greatest effect on flank wear and surface roughness. An appropriate Ti DC sputtering power and substrate bias gives TiAlN film coatings with the best mechanical performance. This is similar to the results of Liu et al. [23], in that an appropriate substrate bias voltage increases the adhesive strength between the coating and the substrate and results in smaller grains, so cutting tools that are coated with TiAlN perform significantly better than uncoated tools.
The surface roughness and flank wear for the dry milling of SCM415 steel using TiAlN coated tools are the multiple response performance characteristics. The grey relational grade and its order for the optimally coated cermet tools for the dry milling process is calculated using equations (1) and (2), as shown in Table 6. A higher grey relational grade means that the corresponding parameter combination is closer to the optimal value [18]. Parameter set No. 6 \([A_2B_3C_1D_2]\), which signifies a Ti DC power of 120 W, an Al DC power of 180 W, a flow rate ratio for \(\text{N}_2/(\text{N}_2 + \text{Ar})\) of 10% and a substrate bias of \(-50\) V gives the best multiple performance characteristics for the nine experiments. The S/N response graph for the grey relational grade for TiAlN coatings is shown in Figure 2. The plot
uses the predicted optimal combination of parameters, $A_2B_1C_3D_2$ [which represents a Ti DC power of 120 W, an Al DC power of 60 W, a flow rate ratio of $N_2/(N_2+Ar)$ of 30% and a substrate bias of $-50$ V]. To validate this result, a confirmation run was performed using the predicted optimal levels for the parameters.

Table 7 shows the results of the confirmation run for the multiple performance characteristics, which are the orthogonal array and the optimal predicted conditions, respectively. A comparison of the results of the orthogonal array (No.6, $A_2B_3C_1D_2$) and the grey Taguchi (optimal condition, $A_2B_1C_3D_2$) shows that the flank wear is reduced from 7.43 to 6.23 $\mu$m and the surface roughness decreases from 0.23 to 0.17 $\mu$m. These results are consistent with the SEM images in figure 3. The dry cutting parameters are listed in table 2. Using the grey Taguchi method increases the performance of the TiAlN films. The machined surface is smoother (compare figures 3(a) and (c)) and the flank wear for the coated cutter is lower (compare figures 3(b) and (d)).

Table 7. Results of the confirmation run for the multiple performance characteristics using the orthogonal array (No.6) and grey Taguchi (optimal condition) for cutting performances of the TiAlN films.

|                        | Orthogonal array $A_2B_3C_1D_2$ | Optimal condition $A_2B_1C_3D_2$ | Improvement rate (%) |
|------------------------|----------------------------------|-----------------------------------|----------------------|
| Flank wear, $R_a$ ($\mu$m) | 7.43                             | 6.23                              | 16.15                |
| Roughness ($\mu$m)     | 0.23                             | 0.17                              | 26.08                |

Note: No. 6 [$A_2B_3C_1D_2$, which signifies a Ti DC power of 120 W, an Al DC power of 180 W, a flow rate ratio for $N_2/(N_2+Ar)$ of 10% and a substrate bias of $-50$ V].
For the uncoated tools, the flank wear and surface roughness are 14.7 μm and 1.22 μm (figures 3(e), (f)), respectively.

Optimal condition \([A_2B_1C_3D_2]\), which represents a Ti DC power of 120 W, an Al DC power of 60 W, a flow rate ratio of \(\text{N}_2/(\text{N}_2 + \text{Ar})\) of 30% and a substrate bias of \(-50\) V.

Figures 4(a) and (b) shows the SEM surface morphology of the TiAlN coatings for the orthogonal array parameter \(A_2B_3C_1D_2\) (No.6) and the grey Taguchi parameter \(A_2B_1C_3D_2\) (optimal condition), respectively. The surface of the coating exhibits a granular structure. The optimal condition for the coating (figure 4(b)) give a notably more refined structure, which indicates a higher film density, and a smoother surface structure. Cross-sectional SEM images of the film are shown in figure 5. The TiAlN films are dense, homogeneous, adhere perfectly to the substrate and have no defects or voids. A Ti content of \(~63.26\) at%, a \(\text{N}_2\) content of \(~34.89\) at% and a lower Al content of \(~1.85\) at%, give the optimal multiple performance characteristics for the TiAlN coatings.

Table 8 lists the mechanical properties of the TiAlN films. TiAlN films that are grown using the optimal condition \([A_2B_1C_3D_2]\) exhibit good mechanical performance, with a coefficient of friction of 0.46, a hardness \(H\) of 25.61 GPa, an elastic modulus \(E\) of 221.1 GPa, a \(H/E\) ratio (plasticity index) of 0.12 and a \(H^3/E^2\) ratio (resistance to plastic deformation) of 0.34 GPa. Table 9 shows comparison the improvement rate between orthogonal array (No.6) and grey Taguchi (optimal condition) for mechanical properties of the TiAlN films.

A ball-on-disk tribometer was used to determine the tribological properties of the films. Figure 6 shows that the coefficient of friction is decreased for the optimal condition. A low coefficient of friction results in decreased film deformation of the wear track [24].

Figure 7 shows the XRD patterns for TiAlN coatings that are deposited using orthogonal array No. 1 to No. 9 and the optimal condition \([A_2B_1C_3D_2]\). The TiAlN films exhibit a NaCl-type face-centered cubic structure, which is typical of titanium base nitrides. These XRD patterns show the orientation in crystallographic planes \((111), (200\) and \((220). These results are in good agreement with those for other studies of TiAlN coatings [25]. The TiAlN coating film exhibits a strong \((111)\) preferred orientation. A comparison of the experimental results for parameter sets nos. 1, 2 and 3 shows that as the Al atomic concentration increases from 1.85 to 2.67%, the
The intensity of the (111) peak decreases, because there is an increase in the bombardment energy of the ions [26]. For a film that is deposited using the optimal condition, the intensity of the diffraction peak increases, because there is an improvement in the crystallinity of the TiAlN films.

The nano-indentation test is a standard means of measuring the mechanical properties of thin films. The load–displacement data for a complete loading–unloading cycle during the nano-indentation test was obtained using the method of Oliver and Pharr [27]. The nanomechanical properties of the films are determined in terms of the hardness, coefficient of friction, and elastic modulus.
of indenter penetration depth at maximum load \( h_{\text{max}} \), the depth of the residual indent \( h_r \) and the elastic recovery \( R_e \) [28]. The \( R_e \) value shows that the films resist plastic deformation. The value is calculated as [28]:

\[
\%R_e = \frac{h_{\text{max}} - h_r}{h_{\text{max}}} \times 100\%
\]  

Figure 8 shows the load–displacement curves for nano-indentation for TiAlN films. The maximum load is 2 mN for the orthogonal array (No.6) \( h_{\text{max}} = 42.92 \text{ nm}, h_r = 22.22 \text{ nm}, R_e = 48.22\% \) and the optimal condition \( h_{\text{max}} = 35.96 \text{ nm}, h_r = 16.85 \text{ nm}, R_e = 53.14\% \). As a result, the optimum TiAlN coating has the highest values for hardness, elastic modulus, \( H/E, H^3/E^2 \), elastic recovery and good cutting performance.

4. Conclusions

TiAlN films that are deposited using DC magnetron co-sputtering with an Al concentration of less than 3.3 at% are shown to have superior mechanical properties. Pure Ti (76.2 mm diameter) and Al (50.8 mm diameter) metal targets are used. An orthogonal array \( L_9(3^4) \) and the grey Taguchi method are used to determine the performance characteristics for coating operations. The TiAlN film thickness ranges from 337 to 418 nm. The
TiAlN films exhibit a face-centered cubic structure and the x-ray diffraction patterns show orientations in crystallographic planes (111), (200) and (220). The TiAlN films are highly compact, homogeneous and adhere perfectly to the substrate.

A TiAlN film that is coated on cutter inserts gives a significant reduction in the surface roughness and flank wear. Cutting tools that are coated with TiAlN perform significantly better than uncoated tools. The experimental results show that an appropriate substrate bias voltage increases the adhesive strength between the coating and the substrate. The optimum TiAlN coating has the highest values for hardness, elastic modulus, H/E, H'/E^2 and elastic recovery. In the confirmation runs, the performance of cutter inserts that are coated with the TiAlN films are shown to be better after grey relational Taguchi analysis.

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Figure 8. Load–displacement curves for nanoindentation for TiAIN films: the maximum load is 2 mN.
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