Effect of Substrate Temperature, Biasing and Sputter Cleaning on the Structure and Properties of Nanostructured TiB$_2$ Coatings on High Speed Steel

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Fabrication and development of TiB$_2$-based nanostructured coatings was investigated in the present work. By varying the sputter-target power density, substrate temperature, deposition time, substrate-to-target distance, substrate biasing and substrate sputter cleaning, the relationship between the sputtered structure, properties and sputtering conditions were established. The experimental results showed that the target-to-substrate distance played a major role in the coating structure and properties. Sputter cleaning of substrate helped to improve TiB$_2$ coating hardness and adhesion. The deposition process could be controlled to produce a TiB$_2$ coating with both high hardness and good adhesion strength. This was achieved by introducing substrate sputter-cleaning and then biasing for the early stage of deposition, followed by deposition without biasing. [doi:10.2320/matertrans.MC200906]

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1. Introduction

In recent years, nanostructured materials have been attracting growing scientific and industrial interests due to their novel properties. In terms of mechanical properties, nanostructured materials show extraordinarily high hardness, tribological performance and yield strength. These unusual properties may provide great potentials for the application of this new class of materials in many fields, including mechanical, automobile, aerospace and microelectronic engineering.$^{1-3}$ Increasing applications have been found for thin, hard, wear resistant (ceramic) coatings in metal cutting and metal forming tools. The invention of low temperature (below 500$^\circ$C) physical vapour deposition (PVD) processes such as magnetron sputtering has made possible the deposition of various hard and wear resistant coatings onto commonly used tool steels. When applied properly, protective coatings on cutting tools and other tribological components can extend component lifetimes. It is usually desirable to have coating with high hardness and low internal stress. Protective coatings with high hardness provide better wear resistance of coated tool steel against abrasion at high contact pressures.$^4$

Among many materials of interest, titanium diboride (TiB$_2$) has notably been chosen. TiB$_2$ possesses many interesting physical, mechanical and chemical properties, such as high hardness, high melting point, good chemical stability and good thermal and electrical conductivity. There have been increasing interests in fabrication of this material in thin film and coating forms for many potential applications, for example to reduce wear and corrosion in engineering components and particularly in material processing tools and dies.$^5,6$ Although TiB$_2$ coatings have been widely studied by many researchers, their real applications have been very limited. The existing problem of TiB$_2$ coating is that its adhesion is poor for the coating-substrate system.

In the present investigation, attempts have been made to fabricate TiB$_2$-based nanostructured engineering coatings on high speed tool steel substrate under various parameters such as sputter-target power density, substrate temperature, deposition time, substrate-to-target distance, substrate biasing and substrate sputter cleaning. The characterization of their structures and mechanical properties for the resultant coatings has been carried out.

2. Experimental Procedure

In this study, high speed steel (HSS), SECO WKE45 (Sweden), in fully hardened and tempered condition was chosen as substrates. The specimen surface was manually ground and polished. The HSS substrates were then ultrasonically cleaned with acetone and ethanol before charging into the deposition chamber. A planar magnetron sputtering system supplied by the Coaxial Company (UK) was used for depositions. The system consists of a cylindrical chamber with three 3-inch water cooled target holders tilted at approximately 30 degree with respect to the normal of the horizontal substrate holder, which can be heated by graphite heating elements. The substrates can be stationary and rotated and the substrate-target distance can be adjusted from 60 mm to 100 mm for TiB$_2$ and Ti targets. All the experiments were conducted at a constant working pressure of 0.65 Pa and at a total gas flow rate (Ar) of 20 sccm. The substrate temperature can be changed from room temperature (RT) until 400$^\circ$C. A RF power biased to the substrate was used to sputter clean the substrate surface. Both DC and RF sputtering were used in this work by using DC power for the Ti target and RF power for the TiB$_2$ target. The DC and RF power employed in this study was varied from 200–400 W. The details of deposition are summarized in Table 1.

The phase identification of the resultant coatings was examined by Shimadzu X-ray diffractometer with Cu-K$_\alpha$ radiation. The fractured cross-sections of the coatings were imaged using a field emission scanning electron microscope (FESEM), Jeol JSM 6340F.
The coating thickness was measured by making a ball-crater on the coating surface using the Calotest machine available at Nanoshield Co. Ltd. manufactured by CSM, Switzerland. The roughness of surfaces was imaged using an atomic force microscopy (AFM).

Nanoindentation test was performed with a Berkovich diamond indenter. Experiments were performed at a constant loading and unloading rate of 0.05 mN/s. In order to assess the intrinsic mechanical properties of the coatings i.e. hardness and modulus, all specimens were tested at 50 nm penetration depth to avoid any possible effect from the substrate during the indentation process. The unloading curves were used to derive the hardness and reduced modulus values by the analytical technique developed by Oliver and Pharr.7) The reported hardness and modulus values are the average of 10 measurements.

The microscratch test was performed using the single-pass scratch mode with a Rockwell diamond indenter topped as a conical with spherical end form of 25 μm in radius. The scanned length was scratched by applying a linearly increasing load at 5 mN/s after prescanning the initial 50 μm distance under a small initial load of 0.25 mN. During scratching, the friction force on the indenter and the surface profile along the full length of the scratched track were measured continually, such that a friction force versus scratching distance (or load) curve was obtained. The critical load for coating failure (Lc), commonly used to measure of the coating-substrate adhesion strength, was determined by the sudden change in friction force.

3. Results and Discussion

Two sets of experiments were first conducted to study the effect of various parameters on the structure and properties of single layer TiB₂ coatings with a Ti interlayer (Fig. 1). The purpose of depositing a thin Ti interfacial layer by sputtering a pure Ti target for a few minutes is to increase the adhesion between HSS substrate and TiB₂ coating. The first set was performed without substrate cleaning and biasing with the purpose to determine the best combination of other parameters in terms of structure and properties. The second set employed the optimized condition obtained from Set 1 and introduced substrate sputter cleaning and biasing during deposition to further enhance the structure and properties characteristics.

3.1 Set 1 samples

3.1.1 Structural characterization of set 1 samples

Table 1 summarizes the deposition conditions for TiB₂ coatings.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Substrate Temperature (°C)</th>
<th>Ti Power/Time</th>
<th>TiB₂ Power/Time</th>
<th>TiB₂ Target/Substrate Distance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Set-1</td>
<td>RT</td>
<td>150 W/5 min</td>
<td>150 W/2 hours</td>
<td>100 mm</td>
</tr>
<tr>
<td>S1-2</td>
<td>100</td>
<td>150 W/5 min</td>
<td>150 W/2 hours</td>
<td>100 mm</td>
</tr>
<tr>
<td>S1-3</td>
<td>200</td>
<td>150 W/5 min</td>
<td>150 W/2 hours</td>
<td>100 mm</td>
</tr>
<tr>
<td>S1-4</td>
<td>300</td>
<td>150 W/5 min</td>
<td>150 W/2 hours</td>
<td>100 mm</td>
</tr>
<tr>
<td>S1-5</td>
<td>300</td>
<td>300 W/5 min</td>
<td>300 W/2 hours</td>
<td>100 mm</td>
</tr>
<tr>
<td>S1-6</td>
<td>400</td>
<td>350 W/20 min</td>
<td>350 W/3 hours</td>
<td>100 mm</td>
</tr>
<tr>
<td>S1-7</td>
<td>400</td>
<td>200 W/10 min</td>
<td>200 W/3 hours</td>
<td>60 mm</td>
</tr>
</tbody>
</table>

The reported hardness and modulus values are the average of 10 measurements.

The scanned length was scratched by applying a linearly increasing load at 5 mN/s after prescanning the initial 50 μm distance under a small initial load of 0.25 mN. During scratching, the friction force on the indenter and the surface profile along the full length of the scratched track were measured continually, such that a friction force versus scratching distance (or load) curve was obtained. The critical load for coating failure (Lc), commonly used to measure of the coating-substrate adhesion strength, was determined by the sudden change in friction force.

3.1.2 Structural characterization of set 1 samples

Table 2 summarizes the resultant coating properties.

<table>
<thead>
<tr>
<th>Material</th>
<th>Coating Thickness (μm)</th>
<th>TiB₂ Texture</th>
<th>Hardness (GPa)</th>
<th>Elastic Modulus (GPa)</th>
<th>Critical Load (mN)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti</td>
<td>0.05</td>
<td>(101)</td>
<td>8.2</td>
<td>166.6</td>
<td>20.8</td>
</tr>
<tr>
<td>S1-2</td>
<td>0.05</td>
<td>(101)</td>
<td>10.0</td>
<td>184.1</td>
<td>21.1</td>
</tr>
<tr>
<td>S1-3</td>
<td>0.05</td>
<td>(101)</td>
<td>8.1</td>
<td>171.7</td>
<td>26.5</td>
</tr>
<tr>
<td>S1-4</td>
<td>0.05</td>
<td>(101)</td>
<td>7.8</td>
<td>171.6</td>
<td>10.2</td>
</tr>
<tr>
<td>S1-5</td>
<td>0.10</td>
<td>0.065</td>
<td>11.1</td>
<td>189.5</td>
<td>18.1</td>
</tr>
<tr>
<td>S1-6</td>
<td>0.25</td>
<td>0.75</td>
<td>17.1</td>
<td>208.5</td>
<td>65.0</td>
</tr>
<tr>
<td>S1-7</td>
<td>0.15</td>
<td>0.55</td>
<td>28.4</td>
<td>307.2</td>
<td>74.5</td>
</tr>
</tbody>
</table>

Fig. 1 Schematic of single layer TiB₂ coating on HSS with Ti interlayer.

Fig. 2 FESEM image of fractured cross-section of sample S1-6.
highest hardness. The hexagonal TiB$_2$ phase in samples S1-1 to S1-4 did not demonstrate the preferred (001) orientation even with increased temperature to 300°C. It is noted that since the (001) plane has the highest packing factor, energetic adatom are required to create such an orientation. With this in mind, further depositions were conducted by increasing the deposition power from 150 W to 300 W (sample S1-5). Unfortunately, this has little effect on the orientation of TiB$_2$ coating (Fig. 3). Attempts were thus made to further increase the sputtering power to 350 W, substrate temperature to 400°C and deposition time to 3 h (sample S1-6). In this case, the (001) reflection appears in the diffraction pattern, indicating that the coating starts to orient in this direction. It thus appears that increasing sputtering power, substrate temperature and deposition time, favors the evolution of the (001) orientation.

In order to enhance the development of the desired (001) orientations, another deposition was conducted by reducing the TiB$_2$-target-substrate distance from 100 mm to 60 mm (sample S1-7). The sputtering power was reduced to 200 W because it was found that the TiB$_2$ target cracked after several depositions at 300 W. The XRD result shown in Fig. 3 reveals the development of a strong (001) texture in the coating.

### 3.1.2 Coating properties characterization

#### (1) Nanoindentation test

Although nanoindentation has been carried out at various indentation depths, due to the thin coating thickness (Table 2), the hardness and modulus values summarized in Table 2 were obtained from 50 nm depth, which is expected to minimize substrate effect. It can be seen that all the TiB$_2$ coatings with (101) orientations (samples S1-1 to S1-5) exhibit hardness and modulus values lower than those expected for bulk TiB$_2$ and those reported by other investigators. This low hardness would be due to the poor orientation, small thickness and poor adhesion of the coating. Indeed, with increasing coating (001) orientation, the highest hardness of 28.4 GPa was obtained in sample S1-7, which is close to many reported values.

It is also worth to note the response of the coating to large indentation depth. Figure 4 shows the load-displacement curves of sample S1-6 produced at 300 nm depth. It is noted that pop-in event (arrow points at 1) occurs during loading, whilst pop-out event occurs during unloading (arrow points at 2) of some indentations. These non-linear phenomena were observed commonly in the single layer TiB$_2$ coatings (samples S1-1 to S1-7), and is associated with the brittleness of the coating and poor coating/substrate adhesion, which lead to coating cracking or debonding during the indentation process.

In order to gain a better understanding of the indentation deformation and cracking behavior, selected indents produced at large depth were examined by SEM, as shown in Fig. 5 for sample S1-6. Figure 5(a) shows an indent at 500 nm depth, from which it is clearly seen that the sink-in effect takes place. However, the coating is cracked if the indentation is made at 1,500 nm in depth (the coating...
thickness of sample S1-6 is only 950 nm) as shown in Fig. 5(b). This shows that the coating is such brittle that cannot carry a higher indentation load. Further study of imprinted nanoindentation at 700 nm is shown in Fig. 5(c) using AFM. This shows clearly for the indentation and calculate the true area for hardness calculation.

(2) Microscratch test

Coating adhesion was assessed using the microscratch adhesion test. The simplest method for evaluating the critical load for coating failure is to plot friction force vs load. Optical and scanning electron microscopic (SEM) examinations were also used to confirm the results from the friction curve. The results are summarized in Table 2.

As expected, since samples S1-1 to S1-5 have a very thin coating thickness, cohesive failure could not be detected and only adhesive failure could be found. The critical load, \( L_C \), for adhesive failure of these coatings was found to be very low, in the range between 16 mN to 30 mN (Table 2). No correlation has been found between \( L_C \) and substrate temperature and sputtering power. Figure 6 shows typical scratch tracks measured by optical microscope (Fig. 6(a)) and AFM (Fig. 6(b)) in which the mechanism of coating failure can be observed.

From Table 2, it can be seen that the critical load was measured to be 65 mN in sample S1-6, which also showed a higher hardness. Sample S1-7 showed the highest \( L_C \) value of 74.5 mN, which also exhibits the highest hardness and strongest (001) orientation.

3.2 Set 2 samples

The results of Set 1 experiments presented above demonstrated that sample S1-7 possesses the (001) texture, highest hardness and adhesion. However the coating hardness and adhesion achieved are still relatively low compared with those reported in the literature.\(^{11–13}\) Set 2 experiments were thus conducted using the deposition conditions of sample S1-7, but with sputter-cleaning of the substrate at the initial stage, and substrate biasing during deposition. Table 3 summarizes the deposition condition for this set. It should be noted that the only variables in this Set are sputter-cleaning and substrate bias. The other parameters are kept constant (S1-7, Table 1).

![Image](a)

**Fig. 6** Microscratch tracks measured by optical microscope (a) and AFM (b) of sample S1-6.

![Image](b)

**Fig. 7** XRD patterns generated from TiB\(_2\) coatings of sample S2-1 to S2-13.

3.2.1 Structural characterization

XRD patterns are shown in Fig. 7 for all samples of this Set. As discussed previously, for sample S1-7, the TiB\(_2\) coating produced without sputter-cleaning of substrate and biasing has a hexagonal structure with strong (001) preferred orientation. (001) orientation was found in all the coatings produced without substrate biasing (samples S2-1, S2-6, S2-7) and with small biasing (30 W) for the first hour of deposition only, which was followed by 2 h deposition without bias (samples S2-8 to S2-13). It can also be seen that sputter-cleaning of the substrate before deposition seems to reduce the intensity of this orientation (compare sample S2-1 with sample S2-6 and S2-7). However, increasing the substrate sputter cleaning time from 20 min (sample S2-6) to 60 min (sample S2-7) slightly promoted the TiB\(_2\) (001) orientation.

With the introduction of substrate bias for the entire deposition of 3 h (S2-2 to S2-5), the orientation of the TiB\(_2\) coating is significantly changed. From Fig. 7, it can be seen...
that with increasing bias power, the evolution of the (001) orientation decreased and the orientation changed to (101), especially in samples S2-4 (bias 90 W) and S2-5 (bias 120 W). This is in agreement with the work of Weidemann and Oettel.15) The change of orientation may account for the observed decrease in hardness as discussed later.

The morphology and thickness of the deposited coatings were examined under FESEM. The thickness values of the coatings are summarized in Table 3. A thickness between 700 nm to 800 nm is typical of the samples produced without substrate bias, while the thickness of the coatings decreases with applied substrate bias. Figure 8 shows the fractured cross-section of the coating produced with substrate cleaning and substrate biasing at the only first hour of whole 3 hours deposition (S2-8 to S2-13). Indeed, the effect of biasing of 1 h can be seen clearly at the area near the interface between the TiB$_2$ top layer and the Ti interlayer. The affected area from biasing shows the similar thickness to the Ti interlayer (Fig. 8).

However, the feature of this region could not be identified. It can also be seen that this region shows a different structure as compared to the columnar structure of TiB$_2$ top layer. From Fig. 8, the Ti interlayer could be seen and the region produced by biasing can also be found in the TiB$_2$ coating. This indicates that biasing can significantly change the structure of the TiB$_2$ top layer, which may affect the coating properties, in particular the coating adhesion due to the modified structure at the interface. From Table 3, it can be seen that the coating hardness slightly decreases, whilst the coating adhesion increases significantly. This shows that biasing can change the coating structure and helps to enhance the adhesion. However, it is noted that biasing during the whole deposition deteriorates both hardness and

<table>
<thead>
<tr>
<th>Sample</th>
<th>Sputter-cleaning of substrate</th>
<th>Substrate bias (W)</th>
<th>Thickness (nm)</th>
<th>Coating Texture</th>
<th>Hardness (GPa)</th>
<th>Modulus (GPa)</th>
<th>Critical load $L_C$ (mN)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S2-1</td>
<td>No cleaning</td>
<td>No biasing</td>
<td>700</td>
<td>(001)</td>
<td>28.4</td>
<td>307.2</td>
<td>74.6</td>
</tr>
<tr>
<td>S2-2</td>
<td>rf 150 W, 20 min</td>
<td>30 W for 3 h</td>
<td>690</td>
<td>(101)</td>
<td>10.8</td>
<td>212.5</td>
<td>343.2</td>
</tr>
<tr>
<td>S2-3</td>
<td>rf 150 W, 20 min</td>
<td>60 W for 3 h</td>
<td>650</td>
<td>(101)</td>
<td>15.7</td>
<td>220.7</td>
<td>286.3</td>
</tr>
<tr>
<td>S2-4</td>
<td>rf 150 W, 20 min</td>
<td>90 W for 3 h</td>
<td>510</td>
<td>(101)</td>
<td>15.2</td>
<td>215.3</td>
<td>247.3</td>
</tr>
<tr>
<td>S2-5</td>
<td>rf 150 W, 20 min</td>
<td>120 W for 3 h</td>
<td>500</td>
<td>(101)</td>
<td>18.3</td>
<td>207.9</td>
<td>214.5</td>
</tr>
<tr>
<td>S2-6</td>
<td>rf 150 W, 20 min</td>
<td>No Biasing</td>
<td>780</td>
<td>(001)</td>
<td>29.2</td>
<td>297.1</td>
<td>96.4</td>
</tr>
<tr>
<td>S2-7</td>
<td>rf 150 W, 60 min</td>
<td>No Biasing</td>
<td>730</td>
<td>(001)</td>
<td>31.7</td>
<td>327.9</td>
<td>123.3</td>
</tr>
<tr>
<td>S2-8</td>
<td>No cleaning</td>
<td>30 W for the first hour only</td>
<td>650</td>
<td>(001)</td>
<td>19.2</td>
<td>235.5</td>
<td>529.1</td>
</tr>
<tr>
<td>S2-9</td>
<td>rf 150 W, 20 min</td>
<td>30 W for the first hour only</td>
<td>650</td>
<td>(001)</td>
<td>23.5</td>
<td>264.5</td>
<td>322.7</td>
</tr>
<tr>
<td>S2-10</td>
<td>rf 150 W, 30 min</td>
<td>30 W for the first hour only</td>
<td>650</td>
<td>(001)</td>
<td>20.8</td>
<td>242.5</td>
<td>595.4</td>
</tr>
<tr>
<td>S2-11</td>
<td>rf 150 W, 60 min</td>
<td>30 W for the first hour only</td>
<td>600</td>
<td>(001)</td>
<td>23.6</td>
<td>275.1</td>
<td>648.5</td>
</tr>
<tr>
<td>S2-12</td>
<td>rf 150 W, 90 min</td>
<td>30 W for the first hour only</td>
<td>550</td>
<td>(001)</td>
<td>26.4</td>
<td>283.2</td>
<td>875.5</td>
</tr>
<tr>
<td>S2-13</td>
<td>rf 150 W, 120 min</td>
<td>30 W for the first hour only</td>
<td>550</td>
<td>(001)</td>
<td>18.6</td>
<td>271.2</td>
<td>701.1</td>
</tr>
</tbody>
</table>

Fig. 8 FESEM images showing the fractured cross-section of sample S2-10.
adhesion (Table 3). This can be concluded that substrate biasing for a short time during the whole deposition can help to improve the coating adhesion, and still maintain the high hardness.

3.2.2 Nanoindentation for mechanical properties test

In order to assess the intrinsic mechanical properties of the coatings, i.e. hardness and modulus, all specimens were tested at 50 nm (less than 10% of coating thickness) penetration depth to minimize the effect from substrate. Figure 9 shows the typical load-displacement curves of samples S2-1, S2-2 and S2-8. The hardness and modulus values as measured by nanoindentation are summarised in Table 3.

It can be seen that the (101)-oriented coatings produced with substrate bias have relatively low hardness and modulus (Table 3) and experience significant plastic deformation during the indentation process (e.g. sample S2-2 in Fig. 9). The (001)-oriented coatings produced without substrate bias experience significant elastic recovery during the unloading stage (e.g. sample S2-1 in Fig. 9) and possess much higher hardness, around 30 GPa and higher modulus between 300–320 GPa. Substrate sputter cleaning before deposition seems to slightly increase the hardness of the coating, although the degree of (001) orientation is reduced. It is therefore clear that applying substrate bias during the whole deposition period (3 h) causes a drop in coating hardness and modulus, in agreement with the observation of others. In order to achieve reasonably high hardness and modulus, it is essential that the TiB$_2$ coating maintains its (001) preferred orientation. The effect of sputter cleaning time on TiB$_2$ coating hardness and modulus is shown in Fig. 10. Sputter cleaning can slightly increase coating hardness and modulus. However, the hardness and modulus decrease when the sputter cleaning time is more than 90 min as illustrated in Fig. 10.

3.2.3 Microscratch test

Table 3 also summarises the critical load ($L_C$) for coating failure during micro-scratch test. It can be seen that the coatings produced without substrate bias (samples S2-1, S2-6, S2-7), although exhibit high hardness, show low values of $L_C$, which indicate poor coating adhesion strength. In addition, it can be seen that substrate sputter cleaning helps to improve coating adhesion and with increasing sputter cleaning time, the $L_C$ value increases. On the other hand, the coatings produced with substrate bias show high values of $L_C$, particularly with low bias power (30 W), although these coatings have low hardness.

Figure 11 demonstrates the typical scratch curves with linearly increasing load for a TiB$_2$ coating (Sample S2-2). It can be seen that DS profile during scratch increases linearly with increasing load. The profiles in Fig. 11 can be divided into several regions. First, the region from A to B shows the pre-scan (300 µm) under the load of 0.25 mN, where no scratch damage occurred, since BS, DS and AS have the same profile. Second, in the region from B to C, DS profile increases almost linearly with increasing load. Hence, there is no plastic deformation or material loss that has taken place during this period, which showed the elastic recovery of TiB$_2$ coating represented by having the same profile of AS and BS. It is noted that after point C, plastic deformation or material loss starts. From the region C to D, all profiles are relatively smooth until point D. It is obvious that there are fluctuations in DS profile and AS profile at point D. This indicates the damage of TiB$_2$ coating. Therefore, the scratch load at point D can be taken as the critical load ($L_C$) for coating failure (listed in Table 3). Furthermore, from the scratch results, the failure mode of coating could be observed. With biasing the failure mode is changed from compressive spallation (Fig. 12(a)) to micro-chipping (Fig. 12(b)), confirming the improvement of coating adhesion.
In order to achieve both high hardness and good coating adhesion, samples S2-8 to S2-13 were produced under conditions of substrate sputter cleaning, substrate bias for the first hour of deposition and without substrate bias for the last 2 h of deposition. From Table 3, it can be seen that a good combination of high coating hardness and adhesion is achieved. In addition, with increasing sputter cleaning time, the resultant coating exhibits a higher hardness and a high value of $L_C$. However, the $L_C$ decreases when the sputter cleaning time is more than 90 min. This is possibly due to the contamination or impurity layer originating from the deposition chamber, which is in agreement with literature.\(^{16}\)

From these results, it is clear that TiB$_2$ coatings with high hardness and good adhesive strength can be produced with well-designed deposition parameters and process control. It is noted that sputter cleaning of the substrate is necessary prior to deposition and sputter cleaning time of 60 to 90 min gives very good results in coating adhesion, but the sputter cleaning time should not be longer than 90 min.

4. Conclusions

Based on the Set 1 and Set 2 experiments, the main results are summarized as follow.

(1) It was found that in order to increase the hardness of the nanostructured TiB$_2$ coatings, the TiB$_2$ (001) preferred orientation should be promoted, and this can be achieved by reducing the target-to-substrate distance from 100 mm to 60 mm and increasing substrate temperature to 400°C.

(2) The hardness and elastic modulus of the TiB$_2$ coatings are affected by deposition conditions. The coatings exhibiting the enhanced (001) orientation possess the highest hardness.

(3) The TiB$_2$ coatings produced without substrate bias are (001) oriented and have high hardness, but low adhesion with the substrate.

(4) Sputtering cleaning of substrate helps to improve TiB$_2$ coating hardness and adhesion strength.

(5) The TiB$_2$ coatings produced with substrate bias are (101) oriented and possess low hardness, but good adhesion with the substrate.

(6) The deposition process can be controlled to produce a TiB$_2$ coating with both high hardness and good adhesion strength. This is achieved by introducing substrate sputter-cleaning and then biasing for the early stage of deposition, followed by deposition without biasing.

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