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# Effects of Deposition Temperature and Time on the Surface Characteristics of TiN-coated High-speed Steel by Arc Ion Plating

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**Abstract**

We present the effects of both deposition temperature and deposition time on the surface characteristics of TiN-coated high-speed steel(AISI M2) by arc ion plating. The microparticles, surface roughness, microhardness, coated layer thickness, adhesion strength, and atomic distribution of Ti, N, and Fe elements were measured for various deposition temperatures and times. The results demonstrated that a deposition temperature in the range of 400 to 500 °C had a slight influence on the surface characteristics, while a deposition time in the range of 10 to 180 min had a great influence on the microparticles, surface roughness, microhardness, coated thickness, atomic distribution of Ti, and adhesion strength.

*Keywords:* Arc ion plating; Deposition temperature; Deposition time; High speed steel; Surface characteristics

## 1. Introduction

The quality of many products is largely dependent on their surface characteristics, such as roughness, wear, hardness, and corrosion. In particular, surface coating technology plays a decisive role in the competitiveness of a company in forming or machining industries worldwide since the machining tools and forming dies affect not only the life span of the product but also the relative manufacturing productivity.

Chemical vapor deposition (CVD)(Jiménez et al., 1995; Yun et al., 1998; Hu et al., 1998; Ma et al., 2001) and physical vapor deposition (PVD)(Håkansson et al., 1991; Kadlec et al., 1992; Jindal et al., 1999) technologies are widely used for coating metal surfaces to enhance their wear resistance, roughness, hardness, and corrosion characteristics (Caelle et al., 1995). It is well-known that the adhesion strength between a CVD coated layer and the substrate or material to be coated is quite high because diffusion takes place between the coated layer and substrate since the CVD coating is conducted at temperatures of more than 1000 °C. However, the substrate may deteriorate since it is exposed to high temperatures that may cause it to soften. Organometallic compounds can help keep the deposition temperatures as low as possible, but are rarely used because they are very expensive, unstable, and poisonous.

Therefore, many researchers and application engineers have focused on PVD technology since its deposition temperature can be kept below 500 °C. There are several different ways(Ohta et al., 2006; Ben Rabeh et al., 2005; Choi et al., 2004; Yoon et al., 2000) of applying PVD coatings, such as vacuum evaporation, sputtering coating, and arc ion plating (AIP)(Hatto et al., 1986; Freller et al., 1988). An AIP coated layer has the best surface qualities, including high adhesion strength, uniform layer thickness, and

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good mechanical properties due to its high ionization ratio, deposition rate, and ion energy (Maek et al., 1999; Kadlec et al., 1999). The advancement of the AIP coating technique has been led mainly by industrial applications and considerable research (Matsue et al., 1999; Lang et al., 2001; Yoon et al., 2002 ; Oh et al., 2003 ; Matsue et al., 2004 ; Ouyang et al., 2004; Yoon et al., 2004) on this technique has been performed. However, it is not easy for process engineers to determine the optimal working conditions; thus, more systematic research is required to supply the valuable information for working conditions.

In this study, the effects of deposition time as well as deposition temperature on the surface characteristics of TiN-coated layer of AISI M2 high-speed steel were experimentally investigated using an AIP coating equipment.

## 2. Experimental conditions and measurement methods

Figure 1 shows a schematic illustration of the experimental apparatus for arc ion plating. The composition of the AISI M2 test material to be coated is given in Table 1. The diameter and thickness of the specimens were 20 and 10 mm, respectively. The mean hardness of material was 62 on the Rockwell C scale (HRC). Ultrasonic cleaning followed by post-processing was performed for 15 min to detach any contaminating material from the specimens.

The apparatus was evacuated up to a base pressure of $7.5 \times 10^{-3}$ Torr and then heated to the test internal temperature. The specimens were bombarded with arc ions for 10 min in Ar gas at a pressure of $5.0 \times 10^{-4}$ Torr and then in $N_2$ gas at a lower pressure of $9.3 \times 10^{-3}$ Torr before they were coated. Details of arc ion bombardment and TiN deposition conditions are summarized in Table 2.

Two types of tests were performed. The first test revealed the effects of deposition temperature on surface characteristics. Test internal temperatures of 400, 450, and 500°C were used with a fixed deposition time of 120 min. The second test was performed for deposition times of 10, 30, 60, 120, and 180 min with a fixed deposition temperature of 450 °C to determine the effects of the deposition time on the surface characteristics of the coated layer.

The microhardness was measured using a Vickers hardness tester (AKASHI, MVK-H1, load: 25 g, dwell time: 30s). The surface roughness was measured using a stylus roughness tester (Mitutoyo, SV-3000) with a measurement distance and cutoff of 4 and 0.8 mm, respectively. The surface roughness was described by the centerline average roughness (Ra, $\mu$m). The thickness of the coated layer was measured using SEM micrographs of cross sections of TiN-coated specimens.

Energy dispersive spectroscopy (EDS) was used to examine the element distribution of Ti, N, and Fe on the surface of the coated layer in a quantitative manner. For the EDS tests, the acceleration voltage was 20 keV, the measurement distance was 20 mm, and the measurement area was 600×600 $\mu$m. The adhesion strength of the AIP-TiN coatings was evaluated by a Rockwell C indentation test (Kim, 2004) and a scratch test(Ichimura et al., 2000). The Rockwell C indentation test used a diamond indenter at 150 kgf and was performed on the coating surface. The degree of the indenter was 120° and the dwell time was 30 s. The scratch tester (J&L Tech, Scratch test JLST022) used a Rockwell C diamond stylus (cone apex angle of 120°, tip radius of 200 $\mu$m). During the scratch test, the stylus load was increased continuously up to 100 N with a loading rate (dL/dt) of 3 N/s and a translation speed of 0.2 mm/s.

Each test was conducted five times under the same

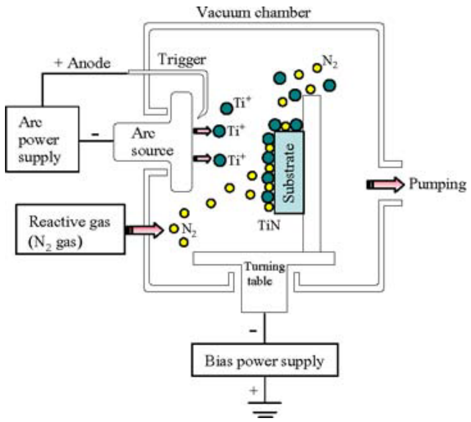


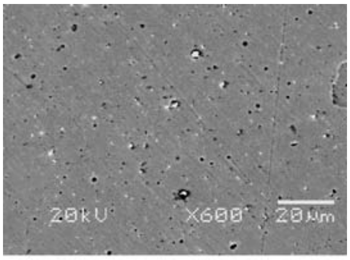

Fig. 1. Schematic illustration of the experimental apparatus for arc ion plating.

Table 1. Chemical composition of AISI M2 steel.

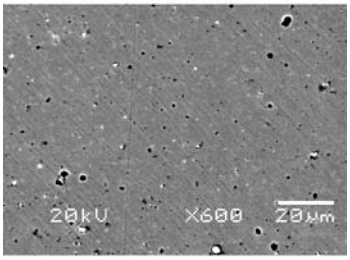
| Chemical composition (wt,%) | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| C | Si | Mn | P | S | Cr | Mo | W | V |
| 0.8–0.9 | <0.40 | <0.40 | <0.03 | <0.03 | 3.8–4.5 | 4.5–5.5 | 5.5–6.7 | 1.6–2.2 |

Table 2. Typical deposition conditions for the TiN coatings applied using the AIP coating system.

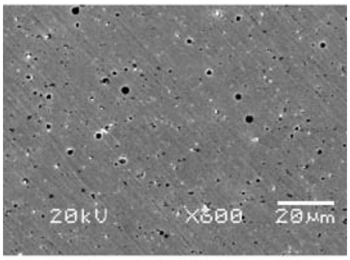
| Process | Variable | TiN |
|---|---|---|
| Arc ion bombardment | Temperature | 450 °C |
| | Bias voltage | –1000 V |
| | Ar gas pressure | $5.0 \times 10^{-4}$ Torr |
| | Time | 10 min |
| Coating | Target | Ti (purity 99.9 %) |
| | Reactive gas | $N_2$ (purity 99.9 %) |
| | Specimen | AISI M2 steel |
| | $N_2$ gas pressure | $9.3 \times 10^{-3}$ Torr |
| | Bias voltage | –125 V |
| | Arc current | 65 A |
| | Deposition temperature | 450 °C |
| | Deposition time | 10, 30, 60, 120, 180 min |




(a) 400 °C




(b) 450 °C




(c) 500 °C

Fig. 2. SEM micrographs of the created microparticles at different deposition temperatures.

conditions to enhance the reliability and reproducibility of the experimental results.

## 3. Results and discussion

### 3.1 Effects of the deposition temperature

#### 3.1.1. Microparticles and surface roughness

Figure 2 shows SEM micrographs of the microparticles created at different deposition temperatures. As shown in the figure, a similar number microparticles was created as the deposition temperature increased. Figure 3 indicates that the surface roughness also did not vary with deposition temperature. Com-parison of the two figures indicates that the surface roughness is much affected by the microparticles (Tai et al., 1990; Boxman et al., 1995).

#### 3.1.2. Microhardness

Figure 4 shows the variation of the microhardness with deposition temperature. The microhardness decreased slightly as the deposition temperature increased, but the variation was small enough to be negligible.

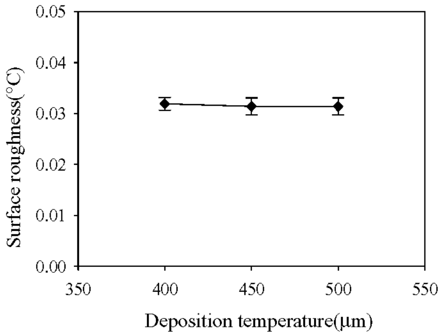



Fig. 3 Variation of the surface roughness with deposition temperature.

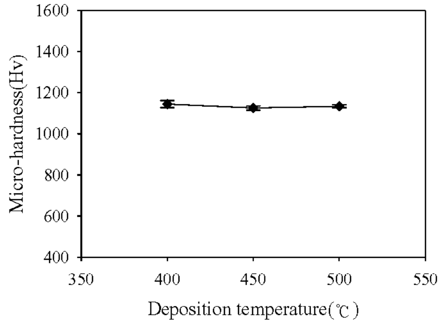
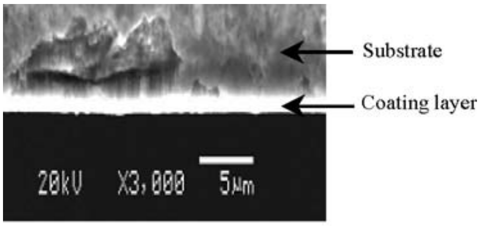


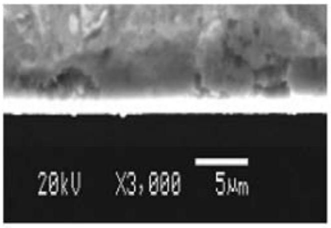

Fig. 4. Variation of the microhardness with deposition temperature.

### *3.1.3. Coated layer thickness*

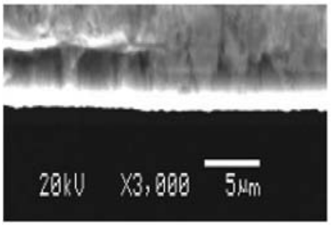
The coated layer thickness was measured using a SEM technique, as shown in Fig. 5. The variation of the coated layer thickness was almost independent of the deposition temperature, as illustrated in Fig. 6.




(a) 400 °C




(b) 450 °C




(c) 500 °C

Fig. 5. SEM cross-sectional view of the TiN-coated layer at different deposition temperatures.

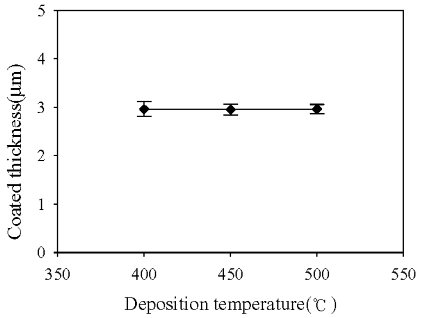


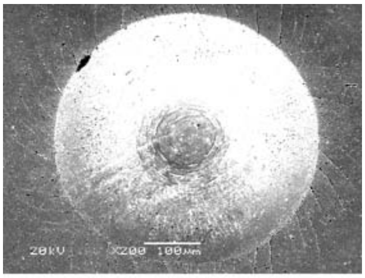

Fig. 6. Variation of the coated layer thickness with deposition temperature.

### *3.1.4. Adhesion*

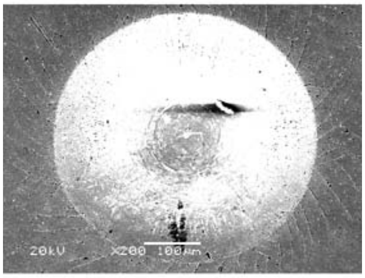
The adhesion was evaluated using Rockwell C indentation tests. The level of adhesion strength can be determined by comparing the micro-crack patterns generated by diamond cone indentations with standard patterns. The rank of adhesion is classified into six levels, *i.e.*, HF1–HF6. A lower level of adhesion denotes a higher rank. Levels HF1–HF4 have adhesion strengths sufficient for commercial metal forming and machining purposes (Kim, 2001).

Figure 7 shows SEM micrographs of the micro-cracks in the TiN-coated layer after the Rockwell C indentation tests. All the micro-crack patterns obtained for the three deposition temperatures can be distinctly observed; these specimens were therefore graded HF2.

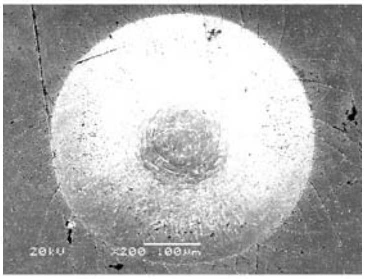
It is seen that the adhesion strength is nearly inde-



(a) 400 °C



(b) 450 °C



(c) 500 °C

Fig. 7. SEM micrographs of micro-cracks in the TiN-coated layer after the Rockwell C indentation tests.

pendent of the deposition temperature in the range of 400 to 500℃.

### 3.2. Effects of the deposition time

The previous section demonstrated that deposition temperatures between 400 and 500 °C only had a slight influence on the surface characteristics of the coated layer, including the microhardness, coated layer thickness, and adhesion strength. Therefore, the deposition temperature was fixed at 450 °C to examine the effects of the deposition time.

#### 3.2.1. Microparticles and surface roughness

Figure 8 shows SEM micrographs of the created microparticles for different deposition times, and Fig. 9 illustrates the effect of the deposition time on the surface roughness. The creation of microparticles increased with the deposition time, and the surface roughness also increased concomitantly with the number of microparticles on the TiN-coated surface.

It is interesting to node that these tendencies discussed herein and in the previous section indicate that the surface roughness is much affected by the microparticles.

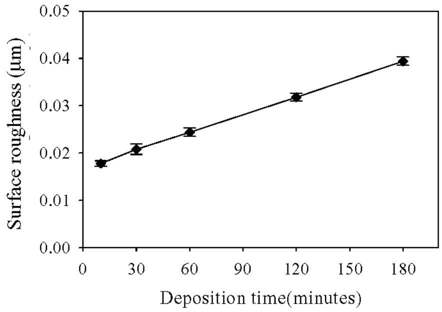



Fig. 9. Variation of the surface roughness with deposition time.

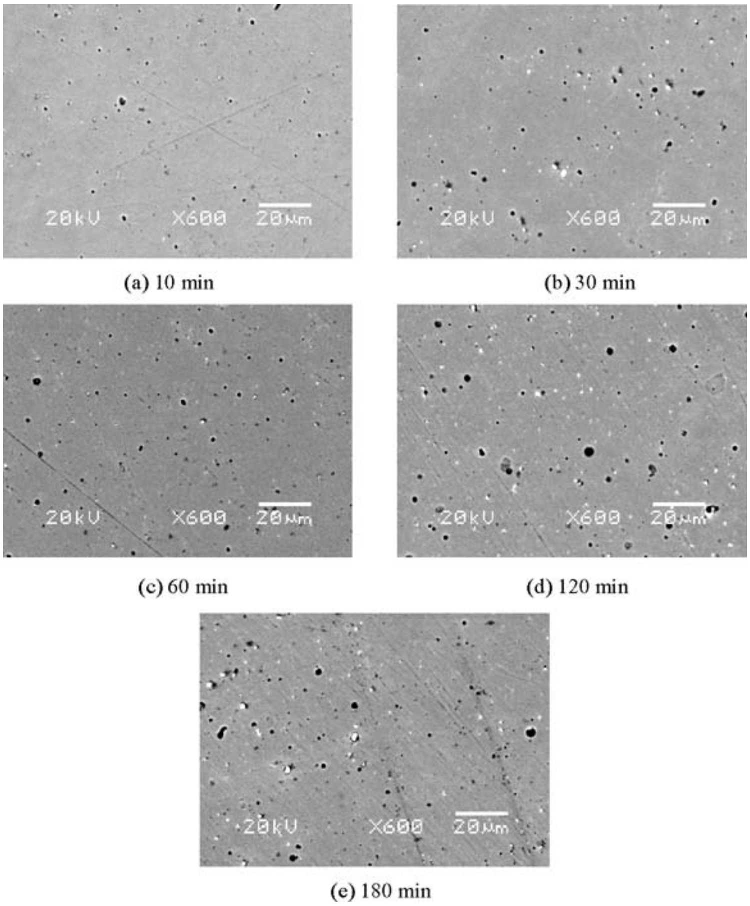



Fig. 8. SEM micrographs of the created microparticles with deposition time.

### 3.2.2. Microhardness

As seen in Fig. 10, the microhardness increases steadily as the deposition time increases, revealing that the increase in the coated layer thickness has strong influence on that in the microhardness(see Fig. 15).

### 3.2.3. Atomic distribution of Ti, N, and Fe elements

Figure 11 shows the variation in the atomic distribution of Ti elements on the coated layer surface with deposition time. The distribution of Ti elements increased sharply with the deposition time for the first 60 min and then remained unchanged. Figure 12 indicates that the atomic distribution of N elements on the coated layer surface varied only slightly with the deposition time. The variation in the atomic distribution of Fe elements on the coated layer surface with deposition time is shown in Fig. 13. The distribution of Fe elements decreased sharply with the deposition time for the first 60 min and then remained unchanged, implying that the surface was covered with the coated layer after 60 min and that the coated layer shielded the Fe-based specimen afterward.

### 3.2.4. Coated layer thickness and adhesion

The coated layer thickness was measured using a SEM technique, as shown in Fig. 14. The coated layer thickness increased by about 1 $\mu$m for every hour after the first 60 min of deposition time, as illustrated in Fig. 15. At least 60 min was required to obtain a 2 $\mu$m thick layer, which has been suggested as the lower boundary of the thickness requirement when coating forming dies. The coated layer thick-ness also had a large influence on the microhardness, which increased steadily with deposition time (see Fig. 10)

Figure 16 shows SEM micrographs of the micro-cracks in the TiN-coated layer after the Rockwell C indentation tests. Figure 16(a) and 16(b) give the micro-crack patterns on specimens after 10 and 30 min of deposition time, respectively. Because of the diffi-culty in identifying the micro-cracks from the figures, the micro-crack patterns after 10 and 30 min of de-position time were evaluated as grade HF6. However, distinct micro-cracks are evident in Fig. 16(c)~16(e) after 60, 120, and 180 min of deposition time, res-pectively. These crack patterns were graded HF2.

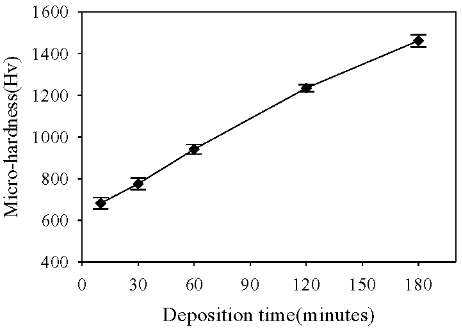



Fig. 10. Variation of the microhardness with deposition time.

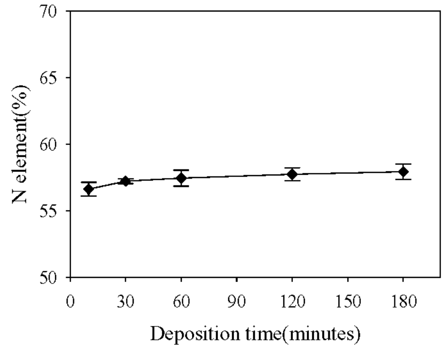



Fig. 12. Variation in the atomic distribution of N elements with deposition time.

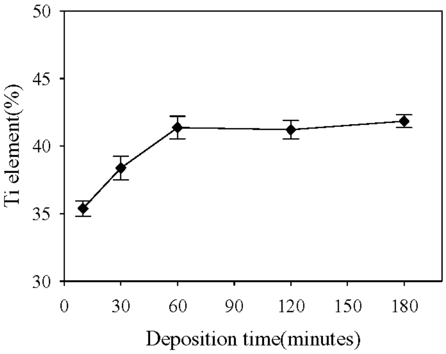



Fig. 11. Variation in the atomic distribution of Ti elements with deposition time.

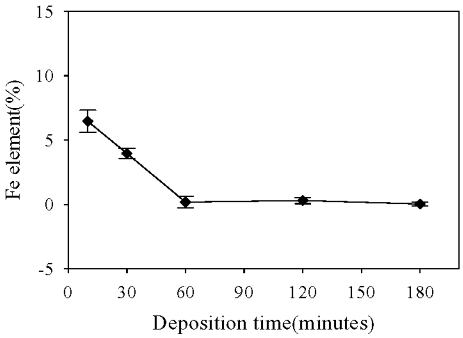
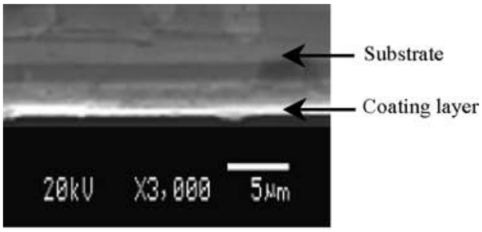
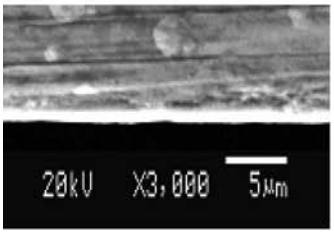


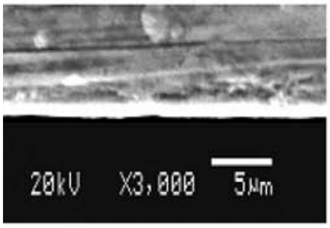

Fig. 13. Variation in the atomic distribution of Fe elements with deposition time.



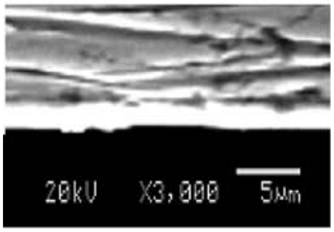

(a) 10 min



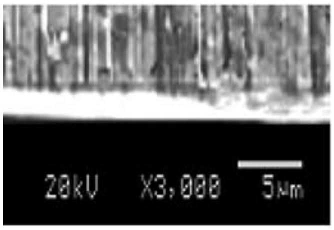

(b) 30 min




(c) 60 min




(d) 120 min




(e) 180 min

Fig. 14. SEM cross-sectional view of the TiN-coated layer.

Thus, grade HF2 adhesion was created when the deposition time exceeded 60 min.

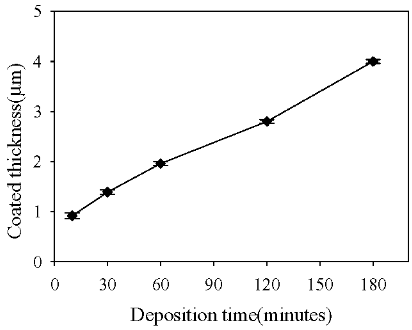
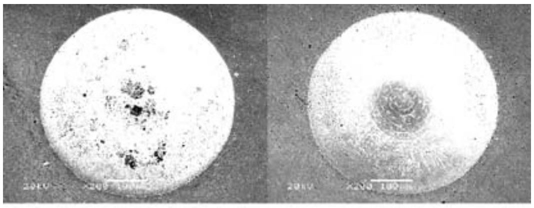
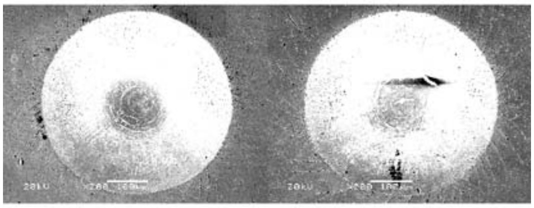


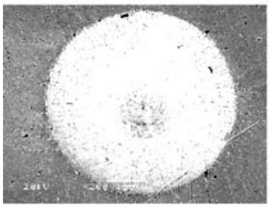

Fig. 15. Variation in the coated thickness with deposition time.



(a) 10 min (b) 30 min



(c) 60 min (d) 120 min



(e) 180 min

Fig. 16. SEM micrographs of micro-cracks in the TiN-coated layer after the Rockwell C indentation tests, indicating adhesion properties.

Figure 17 shows the optical micrographs of the scratch channel after the scratch test. As illustrated in Fig. 18, a failure of the TiN-coated layer after 10 and 30 min of deposition time was observed at approximately 32 and 41 N, respectively. A failure of the TiN-coated layer was observed for critical loads greater than 60 N when the deposition time was more than 60 min.

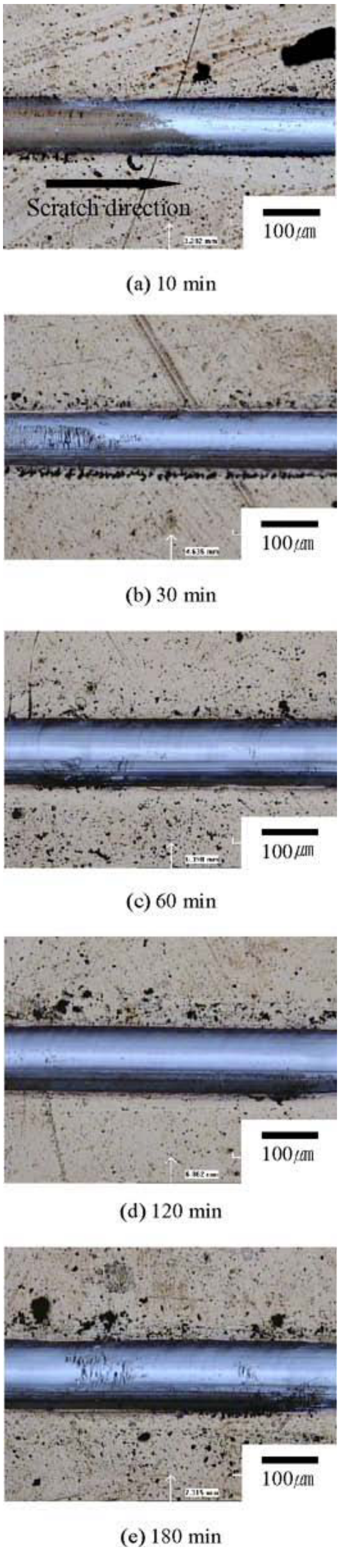



Fig. 17. Optical micrographs of the scratch channel after the scratch test.

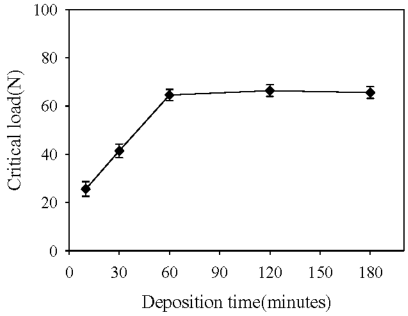



Fig. 18. Variation in the critical scratch load with deposition time.

## 4. Conclusions

In this study, we experimentally determined the effects of the deposition temperature and the deposition time on the surface characteristics of the coated layer, including the microparticles, surface roughness, microhardness, coated layer thickness, chemical composition of the coated surface, and adhesion strength of AISI M2 high-speed steel. The test deposition temperature ranged from 400 to 500 °C and the test deposition time ranged from 10 to 180 min. The results indicated that the deposition temperature only had a slight influence on the surface characteristics of the coated layer while the deposition time had a much larger influence.

As the deposition time increased, the number of microparticles increased and the surface roughness became greater. The microhardness also increased steadily with the deposition time as the coated layer thickness increased. The atomic distribution of Ti elements increased sharply while that of Fe elements decreased by a similar amount for the first 60 min of deposition time, indicating that the material was covered by the coated layer after 60 min.

Thus, 60 min of deposition was optimal in terms of the adhesion strength of the coated layer. The coated layer was 2 ㎛ thick after the first 60 min of deposition, but the accumulation rate was only 1 ㎛ per hour afterward.

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