

# Study on preparation and properties of AlTiN nanostructured multilayer coatings

ERGENG ZHANG\*, TIBO ZHANG

*\*School of Mechanical Engineering, Shanghai Institute of Technology, Shanghai 201418, PR China*

AlTiN nanostructure multilayer coating were deposit adopting alternating Arc source technology, two layers Ti+TiN/AlTiN was deposit alternately. During thick coating experiments and cutting experiments show the comparison within AlTiN monolayer gradient coating & nanostructure multilayer coatings, The AlTiN nanostructure multilayer coatings show better mechanical properties such as adhesion of coating, wear resistance & etc. Experiments show that AlTiN nanostructure multilayer coatings show more excellent wear resistance impact than AlTiN monolayer gradient coating. And AlTiN nanostructure multilayer films relieve the stress concentration inside the coatings, so more layer of thick coating can be deposited if needing; the structure of nanostructure multilayer improves adhesion of coating, and showing better mechanical performance.

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*Keywords:* PVD, Nanostructure multilayer coatings, Monolayer gradient coating, Stress concentration, AlTiN, Mechanical properties

## 1. Introduction

With the rapid development of modern manufacturing industry, the requirement of high-performance cutting tools is ever increasing for more and more difficult to machining hard materials. Because of its properties such as high hardness and good wear resistance, AlTiN monolayer gradient coating has been applied to cutting tools and work-piece in order to increase their lifetime and performance by physical vapor deposition coating technology. L.A.Dobrzański et al. (2008) [1] showed that the coating on the investigated materials causes the 35%-76% increase of micro-hardness value, and contributing most probably in this way in machining to the decrease of the wear intensity of cutting tool's flanks made from cemented carbide, cermet and tool ceramics. (Ti, Al)N coatings by PVD enhance the wear, thermal, and oxidation resistance of a wide variety of tool materials. High hardness, relatively low residual stress, superior oxidation resistance, high hot hardness, and low thermal conductivity make (Ti, Al)N coatings most desirable in dry machining and machining of abrasive alloys at high speed (S.PalDey et al, 2003)[2].

As monolayer gradient coating, AlTiN coating cutting tools on cutting high hardness materials have been commercialized and widely used, such as hardened abrasive tool steel hardness below HRC55, and it has been developing rapidly. However, the cutting hardness meets the requirements, but this kind of coating has the disadvantage of brittleness and poor impact resistance etc. S.PalDey et al (2003)[3] discussed the deposition parameters and the properties of cathodic arc deposited

single layer and gradient (Ti,Al)N coatings using TiAl cathodes. The adhesion strength of (Ti,Al)N coatings deposited on M2 steels is in the range of 44-92 N (Lc1). Therefore, monolayer gradient coating cannot meet the requirement of the improving comprehensive mechanical properties of cutting tool, the coating will tend to diversification and compound; Li Chen (2007)[4] found that the TiN/(Ti, Al)N multilayer coating exhibits higher hardness, lower brittleness and excellent cutting performance, compared with (Ti, Al)N monolayer coating. WEI Yong-qiang et al.(2011)[5] the TiN/TiAlN multilayer coatings were deposited on M2 high speed steel by arc ion plating, the measured hardness of TiN/TiAlN multilayer coatings accords with the mixture principle and the maximum hardness is HV2495. The adhesion strength reached 75 N. Li Chen et al (2007) [6]found the (Cr0.28, Al0.72) N coating shows better flank wear resistance than (Ti0.34, Al0.66) N because of the higher Al content and enhanced formation of protective oxide films. A. Knutsson et al.(2011) [7]showed that the multilayer coated inserts show a decrease of wear with decreased multilayer period, both on the rake and flank face. The wear on the rake face was lower on all the multilayer coated tools compared to the references. A.Rizzo et al (2013) [8]found that nano-multilayer structure improved thermal stability of coatings. Li Chen et al (2011) [9]found multilayer structure results in an increase in adhesion with substrates from 72 N for Ti-Al-N single layer coating to 98 N for TiAlN/TiN nano-multilayer coating.

AlTiN nanostructure multilayer coating is expected to be a promising candidate as super-hard coating because it shows excellent properties, especially for high wear

resistance, high adhesion of coating, low stress concentration inside the coating and meet the need of coating thickness. Therefore, the demand to develop a new wear resistant hard coating with has become crucial to enhance tool life as machining speed increases. Compared to AlTiN monolayer gradient coating, the performance of AlTiN nanostructure multilayer coatings is significantly improved, it eases the stress concentration inside the coating, increases the crack propagation resistance, slows crack growth rate and improves the coating toughness, impact resistance and a coating- substrate adhesion, reduces the coating peeling in the mechanical action, improves wear resistance. S.J.Suresha et al (2006)[10] found the hardness of the multilayer films as measured by nano-indentation (30-33GPa) is similar to those of the monolithic materials and is insensitive to bilayer thickness or to any of the more complex additional periodicities that develop. F.-R.Weber (2004) [11] found that in the multi-layer coatings, there is a transition in the texture from (2 0 0) to (100) with increasing negative bias voltage, beginning at approximately -75 V. E.Vogli et al (2011)[12] that the multilayer with the thickest ceramic layers has the highest hardness and the lowest wear coefficients as well as the lowest compressive residual stress within studied multilayers. Thickest ceramic layers have the highest hardness and the lowest wear coefficients as well as the lowest compressive residual stress within studied multilayers. J.C. Caicedo et al(2012) [13] mechanical and tribological properties exhibited an improvement as a function of increase of bilayer number due to multilayer effect. Therefore, these values represented an increase at hardness (44%, 37%) and elastic modulus (27%, 23%) in relation to monolithic TiN and ternary TiAlN single layer respectively.

This paper describes the properties of AlTiN monolayer gradient coating and AlTiN nanostructure multilayer coatings which deposited by using PVD alternate arc sources technology. The results of tests presented coating-substrate bonding strength, abrasion resistance of AlTiN nanostructure multilayer coatings.

## 2. Experimental

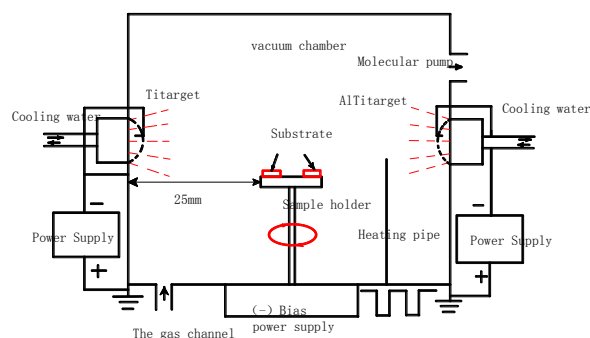
### 2.1 Coating deposition

AlTiN nanostructure multilayer coatings were deposited on carbide samples by means of arc ion plating technique. a Swiss-made machine for the ICS-04 ARC PRO PVD coating equipment was adopted (Fig. 1), which has four targets, including two pure Ti (99.99%) targets, two Al<sub>67</sub>Ti<sub>33</sub>(99.99%) targets. The samples were loaded into the vacuum chamber after they were cleaned, then to set process parameters, such as nitrogen flow, installing furnace capacity and temperature. Before depositing, the samples were heated and plasma-etched using an argon-ion-etching process, the base pressure in the chamber was less than 0.05 mBar. The thickness of

each layer of coating was controlled by two pairs of target material alternating pilot arc, Ti+TiN/AlTiN coating was alternating deposited on carbide samples (Fig. 2, Fig. 3). The number of layers of nanostructure multilayer coatings is 50 layers, and the total thickness of the coating experiments is 2.5~3.0 microns.



(a)



(b)

Fig. 1. Physical vapor deposition coating equipment and Schematic diagram.

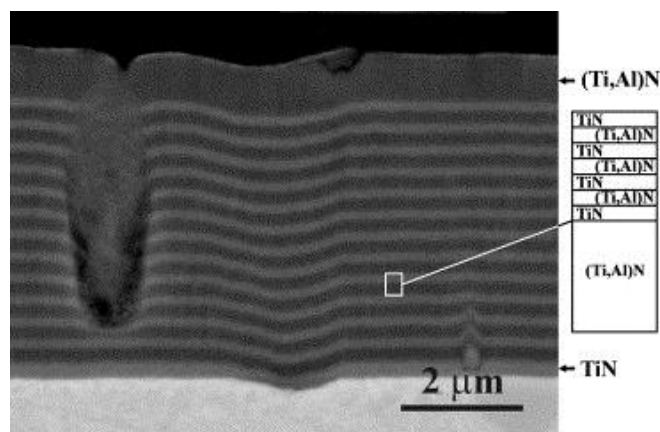


Fig. 2. AlTiN nanostructure multilayer coatings.

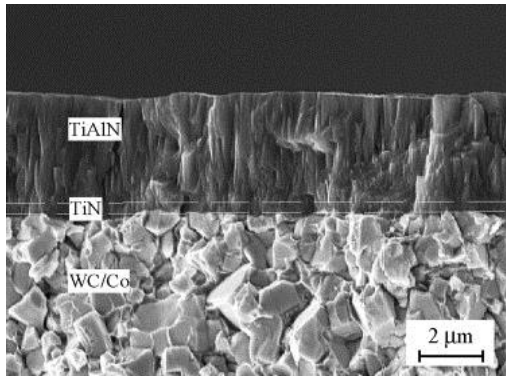


Fig. 3. Microstructure of Ti + TiN /AlTiN layer.

## 2.2 Thick coating's test

In this experiment, AlTiN Monolayer gradient costing and AlTiN nanostructure multilayer coatings were deposited on carbide shank, coating thickness was measured by KaloMax NT ball mill; standard high-speed steels were selected as samples ( $\phi 20 \times 3$ mm), AlTiN monolayer gradient coating and AlTiN nanostructure multilayer coatings were deposited respectively, the indentation experiment was done on HRS-150 Digital Rockwell Hardness Tester, and indentation morphology was observed under a microscope.

## 2.3 Cutting test

The cutting test was performed on Makino S33 milling machine, side milling mode grooves (Fig. 4), milling a certain length repeatedly. The maximum amount of flank wear as a standard to estimate abrasion resistance of coated tool, the two sets of cutting experiment were done.

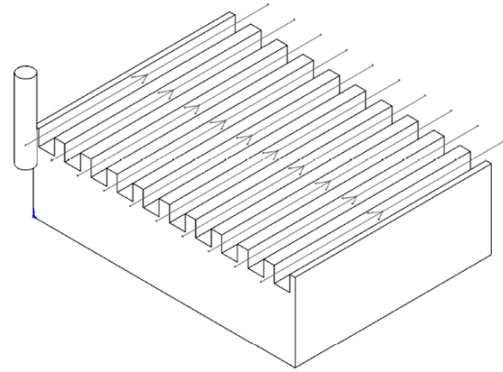


Fig. 4. Schematic of experimental cutting.

In the first set of cutting experiment, AlTiN monolayer gradient coating and AlTiN nanostructure multilayer coatings were deposited on cemented carbide milling cutter respectively. The thickness of coating is 2.5-3 $\mu$ m, the experimental parameters shown in Table 1. The second cutting experiment still selected two kinds of coated tools as in first set of cutting experiment, experimental parameters shown in Table 2.

Table 1. Cutting experiment parameters (cutting distance / layer = 75mm).

| tool parameters                             | f (mm/min) | $f_z$ (mm/tooth) | $a_p$ (mm) | Cooling mode | $v_c$ (m/min) | Materials to be processed |
|---|------------|------------------|------------|--------------|---------------|---------------------------|
| $\phi 9 \times \phi 10 \times 19 \times 69$ | 113        | 0.084            | 9          | oil mist     | 19.085        | SKD61                     |

Table 2. Cutting experiment two parameters (cutting distance / layer = 75mm).

| tool parameters               | f (mm/min) | $f_z$ (mm/tooth) | $a_p$ (mm) | cooling mode   | Speed(rpm) | Materials to be processed |
|-------------------------------|------------|------------------|------------|----------------|------------|---------------------------|
| $\phi 10 \times 25 \times 75$ | 540        | 0.10             | 3          | Compressed air | 4500       | SKD61                     |

### 3. Results and discussion

#### 3.1 Results and analysis of thick coating

Indentations of AlTiN monolayer gradient coating and AlTiN nanostructure multilayer coatings taking by thickness calo test shown in Fig. 5 and Fig. 6, the thickness of coating measured was  $2.83\mu\text{m}$  and  $3.77\mu\text{m}$  respectively. Due to the presence of PVD coating tip accumulation effect, the blade coating thickness should be 2.5~3 times than shank, whereby, the thickness of tool edge measured was  $7.54\mu\text{m}$  and  $11.02\mu\text{m}$ .



Fig. 5. AlTiN monolayer gradient coating (thickness of coating is  $2.83\mu\text{m}$ ).

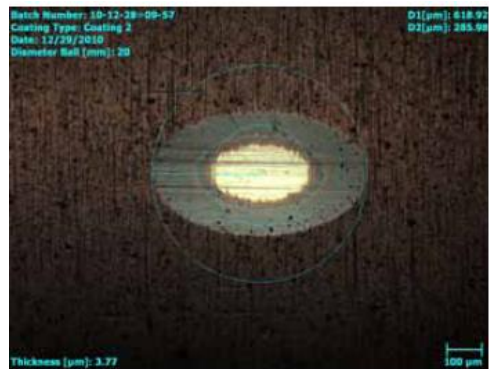


Fig. 6. AlTiN nanostructure multilayer coatings (thickness of coating is  $3.77\mu\text{m}$ ).

The coating condition of cutting edge was observed on AlTiN monolayer gradient coating cutting tool, result shown in Fig. 7 (a). As seen from the figure, the coating has been peeled off. Which means when the tool shank thickness is  $2.83\mu\text{m}$ , the cutting edge thickness is  $7.54\mu\text{m}$ , high stress in the film is likely to cause its auto-off. Through repeated experiment proved that the optimum thickness of AlTiN monolayer gradient coating is no more than  $4\mu\text{m}$ . As seen from Fig. 7 (b), when tool shank thickness of AlTiN nanostructure multilayer coatings is

$3.77\mu\text{m}$ , the tool edge thickness is  $11.02\mu\text{m}$ , it is clear that coating has peeling signs, the experiment also confirmed optimum thickness of AlTiN nanostructured multilayer coatings is no more than  $8\mu\text{m}$ .



(a) 200×



(b) 200×

Fig. 7. (a) AlTiN monolayer gradient coating cutting tool edge status after coating (200×) (b) AlTiN nanostructure multilayer coatings cutting tool edge status after coating(200×).

Fig. 8 shows the indentation microstructure of the coating sample, Fig. 8 (a), (b) for AlTiN nanostructure multilayer coatings, Fig. 8 (c), (d) as AlTiN monolayer gradient coating.

From Fig. 8 can be seen obviously, though the indentation of AlTiN nanostructure multilayer coating is enlarged many times under the microscope, the crack is still extremely small, by contrast, AlTiN monolayer gradient coating has emerged a large area of coating peel-off, it illustrates that the adhesion of AlTiN nanostructure multilayer coatings and the substrate is far better than AlTiN monolayer gradient coating.

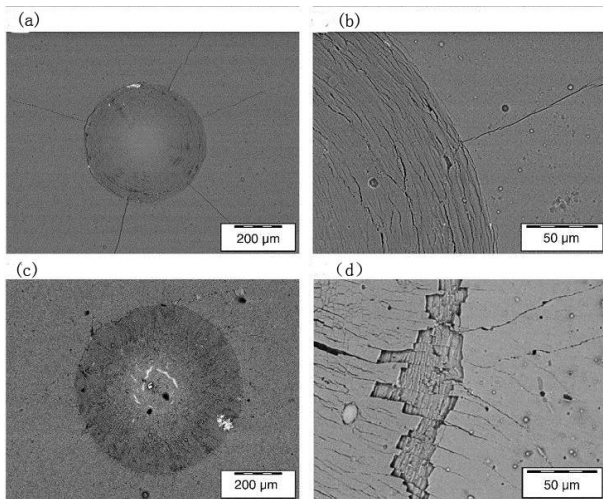


Fig. 8. Coating the indentation microstructure of the sample.

### 3.2 Results and analysis of cutting test

According to the results of first set of cutting experiment (Fig. 9), When cutting distance up to 20 meters, AlTiN monolayer gradient coating has small amount of wear relatively, which is due to the use of a primer layer Ti + TiN, AlTiN as surface layer, so that the coatings have higher overall hardness, wear resistance is also just fine. When the length of cutting was more than 20m, wear resistance of AlTiN nanostructure multilayer coatings are significantly higher than AlTiN monolayer gradient coating, the reason is that although the overall hardness of AlTiN nanostructure multilayer coatings are slightly inferior to AlTiN monolayer gradient coating, its mitigation capacity of stress concentration and external shock is very large, this also means that when cutting is repeated thermal shocks repeated constantly, so the coating thermal shock increases, corresponding thermal stress is large. AlTiN nanostructure multilayer coatings consist of alternating Ti + TiN/AlTiN, this kind of soft layer and hard layer structure, similar to the spring-like, can reduce external shocks, making the film hard to fall off, and then wear life is even higher.

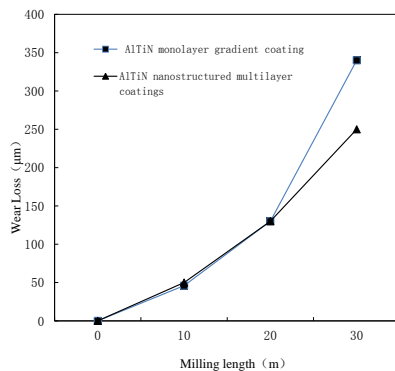


Fig. 9. Results of cutting experiment.

Fig. 10 and Fig. 11 are wear images of cutting edge of AlTiN monolayer gradient coating and AlTiN nanostructured multilayer coatings. Comparison of Figs. 10 and 11 it can be seen, although the overall hardness of AlTiN monolayer gradient coating is higher than AlTiN nanostructure multilayer coatings, the high impact stresses and premature loss of its interior, the loss of tool edge protection, resulting in excessive wear of the tool, however, there is a good adhesion between AlTiN nanostructure multilayer coatings and substrate, good impact resistance, so good for the tool edge protective effect, and the amount of wear of the tool also reduced accordingly.

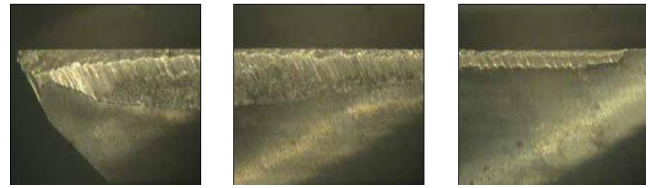


Fig. 10. AlTiN monolayer gradient coating tool edge wear morphology.

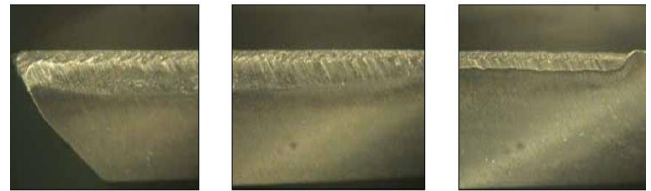


Fig. 11. AlTiN nanostructure multilayer coatings tool edge wear morphology.

In the second set of cutting experiment, the researchers regularly choose certain cutting length in the process, and then measured the rear face's wear breadth to judge wear resistance of coated tool. Under the microscope, the amount of wear and wear the appropriate milling cutter distance after a certain distance in the milling was observed and measured, the results are shown in Table 3, Table 4. By the analysis of the data in the table, with the aggravation of tool wear, cutting force increasing, on the whole, the wear of AlTiN nanostructure multilayer coatings cutter are significantly less than the AlTiN monolayer gradient coating cutter. The wear amount of AlTiN monolayer gradient coating tool wear divided by that of AlTiN nanostructure multilayer coatings cutter can be obtained, the wear life of AlTiN nanostructure multilayer coatings cutter is nearly 1 time than AlTiN monolayer gradient coating cutter.

Table 3. Wear of AlTiN nanostructure multilayer coatings.

| cutting distance (m) | 0.23  | 0.45  | 0.68  | 0.90  | 1.35  | 2.1   | 2.85  | 3.3   |
|----------------------|-------|-------|-------|-------|-------|-------|-------|-------|
| Wear (mm)            | 0.010 | 0.017 | 0.023 | 0.029 | 0.046 | 0.099 | 0.054 | 0.057 |
| Load (%)             | 10.00 | 10.10 | 10.20 | 10.40 | 11.50 | 10.60 | 12.60 | 12.90 |

Table 4. Wear of AlTiN monolayer gradient coating.

| cutting distance (m) | 0.23  | 0.45  | 0.68  | 0.90  | 1.35  | 2.1   | 2.85  | 3.3   |
|----------------------|-------|-------|-------|-------|-------|-------|-------|-------|
| Wear (mm)            | 0.016 | 0.026 | 0.032 | 0.033 | 0.038 | 0.044 | 0.058 | 0.064 |
| Load (%)             | 10.80 | 10.90 | 11.00 | 11.00 | 11.00 | 11.04 | 13.8  | 13.9  |

Fig. 12 is the curve of wear of coated cutting tool, it can be seen from the figure, with increasing cutting length, two kinds of coating tool flank maximum wear width are increased. The wear of AlTiN nanostructure multilayer coatings tool is less than AlTiN monolayer gradient coating cutter. In part of the curve, wear amount of AlTiN monolayer gradient coating cutter is less than AlTiN nanostructure multilayer coatings, this may be associated with non-uniformity of hardness, the cooling of the work-piece and the measurement error etc. With increasing wear of coated cutting tools, the cutting force increasing correspondingly, load of the corresponding machine also increasing. As can be seen from Fig.13, the wear of coated cutter caused machining add additional load, both the increased load compared with AlTiN monolayer gradient coating close to 1 times, it shows that AlTiN nanostructure multilayer coatings can save energy nearly one times than AlTiN monolayer gradient coating in the cutting process.

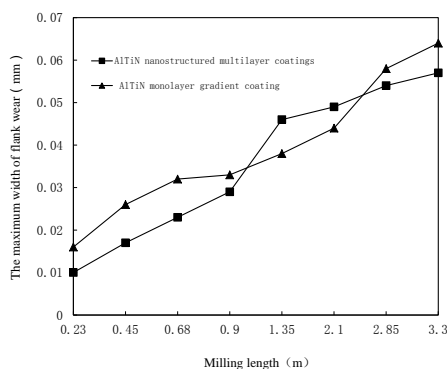


Fig. 12. Relationship of coated cutting tool length and wear volume.

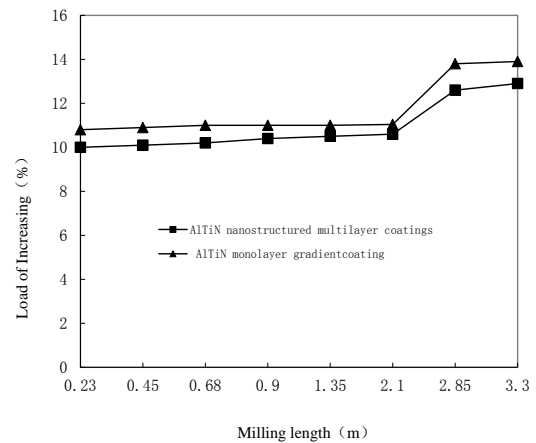


Fig. 13. Relationship of milling length and load of increasing.

#### 4. Conclusions

In this study, AlTiN nanostructure multilayer coatings were deposited, and compared it with the performance of conventional AlTiN monolayer gradient coating. The conclusions were summarized as:

(1) AlTiN nanostructure multilayer coatings can be made thick deposited, whose thickness is double times than the corresponding AlTiN monolayer gradient coating, and showing the excellent adhesion of the coating.

(2) Under the same experimental conditions of milling, abrasion of AlTiN nanostructure multilayer coatings is better than AlTiN monolayer gradient coating, wear resistance is increased about 1 times; the machine reduces energy consumption is nearly doubled.

(3) AlTiN nanostructure multilayer coatings have good wear resistance, under the same conditions, the load caused by the machine tool wear is small, so not only saving energy, but also having low noise cutting and small cutting forces.

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### References

- [1] L. A. Dobrzański, L. W. Żukowska, J. Mikula, K. Golombek, *Journal of Materials Processing Technology*. **201**, 310 (2008).
- [2] S. PalDey, S. C. Deevi, *Material Science and Engineering*. **A342**, 58 (2003).
- [3] S. PalDey, S. C. Deevi, *Materials Science and Engineering*. **A361**, 1 (2003).
- [4] Li Chen, Yong Du, Fei Yin, Jia Li, *International Journal of Refractory Metals & Hard Materials*. **25**, 72 (2007).
- [5] Wei Yong-qiang, Li Chun-wei, Gong Chun-zhi, Tian Xiu-bo, Yang Shi-qin, *Trans. Nonferrous Met. Soc. China*. **21**, 1068 (2011).
- [6] Li Chen, Yong Du, S. Q. Wang, Jia Li, *International Journal of Refractory Metals & Hard Material* **25**, 400 (2007).
- [7] A. Knutsson, M. P. Johansson, L. Karlsson, M. Odén, *Surface & Coatings Technology*. **205**, 4005 (2011).
- [8] A. Rizzo, L. Mirengi, M. Massaro, U. Galiotti, L. Capodieci, R. Terzi, L. Tapfer, D. Valerini, *Surface & Coatings Technology*. **235**, 475 (2013).
- [9] Li Chen, Yong Du, Xiang Xiong, Ke K. Chang, Ming J. Wu, *Int. Journal of Refractory Metals and Hard Materials*. **29**, 681 (2011).
- [10] S. J. Suresha, R. Bhide, V. Jayaram, S. K. Biswas, *Materials Science and Engineering*. **A429**, 252 (2006).
- [11] F.-R. Weber, F. Fontaine, M. Scheib, W. Bock, *Surface and Coatings Technology*. **177-178**, 227 (2004).
- [12] E. Vogli, W. Tillmann, U. Selvadural-Lassl, G. Fischer, J. Herper, *Applied Surface Science*. **257**, 8550 (2011).
- [13] J. C. Caicedo, G. Cabrera, H. H. Caicedo, C. Amaya, W. Aperador, *Thin Solid*, **520**, 4350 (2012).

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\*Corresponding author: zhangeg@yeah.net