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Transactions of Nonferrous Metals Society of China

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# Trans. Nonferrous Met. Soc. China 25(2015) 2581-2586

# Microstructure and superhardness effect of VC/TiC superlattice films

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Received 22 September 2014; accepted 23 April 2015

**Abstract:** Vanadium carbide/titanium carbide (VC/TiC) superlattice films were synthesized by magnetron sputtering method. The effects of modulation period on the microstructure evolution and mechanical properties were investigated by EDXA, XRD, HRTEM and nano-indentation. The results reveal that the VC/TiC superlattice films form an epitaxial structure when their modulation period is less than a critical value, accompanied with a remarkable increase in hardness. Further increasing the modulation period, the hardness of superlattices decreases slowly to the rule-of-mixture value due to the destruction of epitaxial structures. The XRD results reveal that three-directional strains are generated in superlattices when the epitaxial structure is formed, which may change the modulus of constituent layers. This may explain the remarkable hardness enhancement of VC/TiC superlattices. **Key words:** superlattice films; carbide films; microstructure evolution; superhardness effect; epitaxial growth

# **1** Introduction

Hard films have been successfully applied for the protection of materials and particularly to increase the efficiency and extend the lifetime of cutting tools since the 1970s [1]. With the development of modern high-speed cuttings, it is demanded for protective hard films that both hardness and wear resistance should be continuously increased. It was found in 1987 that superlattices deposited alternately by two kinds of materials at nanometer-scale thickness, i.e., superlattice films, could exhibit an anomalous increase of hardness as compared with the monolayer films [2]. Therefore, to synthesize superlattice films has become an important approach to obtain the films with high hardness and excellent wear resistance.

For superlattice films, their constituent layers can be metal layers, nitrides, carbides or oxides of different materials or a combination of one layer made of nitride, carbide, or oxide of one metal and the second layer made of another metal [3]. However, less attention has been paid to carbide superlattice films compared with those superlattices consisting of nitrides, oxides or metals [4–8]. Generally, the carbide films have the same

crystal structure as nitride films and even higher hardness, lower friction coefficient and superior wear resistance performance. This supplied an alternative kind of material to obtain the superlattice films with excellent properties. Although some carbide superlattice films, e.g., VC/TiC [9,10], TiC/NbC [11] and VC/SiC [12], have been reported, further studies on the relationship between the microstructure and mechanical properties of these superlattices are still required. For instance, there exist different results regarding whether the superhardness effect can be obtained in the VC/TiC superlattices [9,10], indicating that the strengthening mechanism also needs to be further investigated.

This study intends to clarify the relationship between the microstructure and the superhardness effect of VC/TiC superlattices. A series of VC/TiC superlattice films with different modulation periods were prepared by magnetron sputtering method. Then, the microstructure and mechanical properties of these superlattice films were investigated. TiC/VC superlattices were chosen in this study for two reasons. First, both TiC and VC have already been applied practically due to their excellent properties. Second, they both exhibit the same structure (NaCl-type) and have smaller lattice mismatch ( $a_{VC}$ =0.4160 nm,  $a_{TiC}$ =0.4328 nm), which is believed to

Foundation item: Project (51201187) supported by the National Natural Science Foundation of China

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be favorable to obtaining high hardness increment by forming epitaxial structures.

# 2 Experimental

VC/TiC superlattices and TiC, VC monolayer films were prepared by SPC-350 multi-target magnetron sputtering system. VC (99.8% in purity) and TiC (99.8% in purity) targets were placed on two radio frequency cathodes, while the metallic Ti target (99.99%) was placed on the DC cathode. Mirror polished stainless steel substrates were ultrasonically cleaned in acetone and absolute alcohol before being mounted on a rotatable substrate holder in the vacuum chamber and the distance between target and substrate was about 5 cm. The base pressure was pumped down to  $2 \times 10^{-4}$  Pa before deposition. Ar (99.99% in purity), whose pressure was kept at 0.6 Pa, was introduced into the chamber. The VC/TiC superlattice films were prepared at 300 °C by alternating deposition of TiC and VC layers. In order to improve the adhesion. Ti transition layer with 100 nm in thickness was deposited before depositing superlattices. A series of superlattices with different modulation periods but the modulation ratio of about 1:1 were prepared by controlling the target power and the resident time that substrates were exposed to the target. And the VC and TiC monolayers were deposited under the same conditions as that of VC/TiC superlattices. The total thickness of each specimen was about 2 µm.

The composition of the films was determined by energy dispersive X-ray spectrometry analysis (EDXA). The microstructures of all the films were characterized by Rigaku D/max-2550/PC X-ray diffraction (XRD) with Cu K<sub>a</sub> excitation radiation, and high-resolution transmission electron microscopy (HRTEM) using a Philips CM200-FEG. The hardness (HV) and elastic modulus (*E*) of all the samples were measured by a Fischerscope H100VP nanoindenter equipped with a Vicker's indenter and the values for specimen were evaluated by an average of at least 20 measurements.

## **3 Results and discussion**

## 3.1 Microstructure of VC/TiC superlattice films

EDXA results show that the C/Ti mole ratio of the TiC monolayer films is 52.8:47.2, and the C/V mole ratio of the VC monolayers is 47.6:52.4. This indicates that both TiC and VC monolayers in this work are near stoichiometric.

The low-angle XRD patterns of the VC/TiC superlattice films with different modulation periods ( $\Lambda$ ) are shown in Fig. 1. The low-angle diffraction peaks, which clearly appear to be systematically distributed in the diffraction patterns, can be attributed to the

modulation period  $\Lambda$  of the superlattices. This indicates an obvious layered structure for all superlattices. Correspondingly, the modulation period of the superlattices can be figured out from [13]

$$\Lambda = \frac{\lambda}{2(\sin\theta_n - \sin\theta_{n-1})} \tag{1}$$

where  $\lambda$  is the X-ray wavelength,  $\theta_n$  and  $\theta_{n-1}$  are the diffraction angles, *n* is diffraction series. Fitting of the  $2\theta$  values for the diffraction peaks into Eq. (1) resulted in the  $\Lambda$  of superlattices, which are shown in Fig. 1.



**Fig. 1** Low-angle XRD patterns of VC/TiC superlattices with modulation ratio of 1:1

Figure 2 displays the cross-sectional HRTEM image and selected area electron diffraction (SAED) pattern of the VC/TiC ( $\Lambda$ =5.2 nm) superlattice film. The SAED pattern on the top-left corner is indexed as a NaCl-type structure, and the low-magnification image on the bottom-left corner of Fig. 2 clearly shows that this superlattice specimen possesses a well-defined composition modulation structure consisting of VC (dark layers) and TiC (light layers). The high-magnification image on the right side shows that the lattice fringes continuously go through several modulation layers and interfaces. This indicates that the two constituent layers,



Fig. 2 Selected area election diffraction (SAED) pattern, low-magnified and high-magnified cross-sectional HRTEM images of VC/TiC superlattice with  $\Lambda$ =5.2 nm

VC and TiC, grow coherently each other and the epitaxial structure is therefore formed in this superlattice film.

Figure 3 shows the high-angle XRD patterns of the VC/TiC superlattices along with VC and TiC monolayers. As can be seen, NaCl-type VC and TiC monolayer films present relatively broad (111) diffraction peaks. By contrast, the VC/TiC superlattices with modulation period ranging from 1.9 to 12 nm only present one fundamental peak, which is located between TiC (111) and VC (111) peaks and thus can be regarded as VC/TiC superlattice (111) peak. Additionally, when  $\Lambda$ is less than 6.8 nm, the VC/TiC superlattice (111) peaks are much higher and sharper than those of VC or TiC monolayers. With the increase of  $\Lambda$ , the superlattice (111) peaks gradually decrease in the intensity and also broaden. After  $\Lambda$  exceeds 12 nm, the superlattice (111) peaks become quite diffusive and are divided into two peaks, as shown in the local amplification figure on the left side of Fig. 3.

Combined with the HRTEM results in Fig. 2, the higher (111) peak intensity of VC/TiC superlattices with  $\Lambda$  less than 6.8 nm compared with that of VC or TiC monolayers, as shown in Fig. 3, indicates that these superlattices form an epitaxial structure and their crystal integrity is greatly improved. Similar phenomena have also been observed in some other superlattice systems such as ZrO<sub>2</sub>/TiN [14], VC/AlN [15], HfN/Si<sub>3</sub>N<sub>4</sub>[16] and TiAlN/AlON [17], and the corresponding reasons have also been explained from both thermodynamics and kinetics factors of film growth in these studies [14–17].

Furthermore, the VC/TiC superlattice (111) peaks are located between the VC (111) and TiC (111) peaks when  $\Lambda$  is lower than 12 nm, which can be attributed to the fact that the two (111) peaks from VC and TiC sublayers in superlattices approach each other and then overlap when the epitaxial structure is formed. Correspondingly, the (111) interplanar spacings ( $d_{111}$ ) of TiC and VC sublayers also approach each other. That is,  $d_{111}$  of VC layers increases along the direction perpendicular to the superlattice interface (out-of-plane), while  $d_{111}$  of TiC decreases along the same direction. Therefore, it can be inferred that the uniaxial tensile strains in VC and the uniaxial compressive strains in TiC sublayers along the out-of-plane direction are induced from epitaxial structure. When  $\Lambda$  increases to 25.1 and 26.9 nm, the superlattice (111) peaks are divided into two parts, i.e., VC (111) and TiC (111) peaks, suggesting that the strains start to be relaxed in superlattices due to the destruction of epitaxial structure.

On the other hand, in order to grow epitaxially on the VC sublayers ( $a_{VC}$ =0.4160 nm) in superlattices, the TiC sublayers with bigger lattice constant ( $a_{TiC}$ = 0.4328 nm) will be subjected to the biaxial compressive strains in the (111) plane (i.e., parallel to the superlattice interface, in-plane). In contrast, the VC sublayers will be under the biaxial tensile in-plane strains. Combined with the above discussion for the out-of-plane strains in superlattices, the strain states of VC/TiC superlattices with epitaxial structure are shown in Fig. 4. It reveals that when the epitaxial structure is formed in superlattices, three directional strains are generated in the different sublayers, for example, the TiC sublayers are under the biaxial (in-plane) and uniaxial (out-of-plane) compressive strains, while the VC sublayers are under biaxial and uniaxial tensile strains, respectively.

# 3.2 Hardness and elastic modulus of VC/TiC superlattice films

Figure 5 demonstrates the nanoindentation hardness (HV) and elastic modulus (*E*) of VC/TiC superlattices as a function of modulation period. The hardness and modulus of VC and TiC monolayers are also shown in Fig. 5. As can be seen, the hardness and modulus of VC/TiC superlattices can be significantly increased compared with VC and TiC monolayers. With the increase of modulation period (1.9 nm  $\leq \Lambda \leq 5.2$  nm), the HV and *E* increase rapidly and reach peak values of



Fig. 3 High-angle XRD patterns of VC, TiC monolayer and VC/TiC superlattice films



Fig. 4 Strain states in VC /TiC superlattices with epitaxial structure



**Fig. 5** Dependence of hardness and elastic modulus of VC/TiC superlattices on modulation period

41.9 GPa and 330 GPa when  $\Lambda$  is 5.2 nm. However, after  $\Lambda$  exceeds 5.2 nm, the HV and *E* begin to decrease by further increasing  $\Lambda$  and finally tend to be near the rule-of-mixture value.

#### **3.3 Strengthening of superlattice films**

So far, there exist two main theories, the Koehler's modulus-difference strengthening theory [18] and the alternating-stress strengthening theory [19], which are always used to explain the superhardness effects of superlattices. Both of these theories are based on the model of dislocations being blocked at interfaces.

According to the Koehler's strengthening theory, the modulus difference between two superlattice constituents is the key factor to restrict the dislocations from gliding across the interfaces and thus determine whether the superhardness effect could be obtained in superlattices. The hardness increment ( $\Delta H_{max}$ ) of superlattices compared with the constituent material with lower hardness can be calculated by this theory as

$$\Delta H_{\rm max} = \frac{3RG_{\rm A}\sin\theta}{8\pi m} \tag{2}$$

where  $\theta$  is the smallest angle between the interface and

the dislocation slip plane of constituent material with smaller elastic modulus; *m* is the Taylor factor, calculated as 0.3 for TiC and VC [20];  $R=(G_B-G_A)/(G_B+G_A)$ , where  $G_A$  and  $G_B$  are shear moduli of two constituents, respectively, and  $G_B>G_A$ , G=E/[2(1+v)] (*E* is the elastic modulus and *v* is the Poisson ratio). Fitting of the elastic modulus of TiC and VC ( $E_{TiC}=255$  GPa,  $E_{VC}=245$  GPa), and *v* of 0.25 for TiC and VC [20], and  $\theta$  of 45° for NaCl-type VC/TiC superlattices into Eq. (1), yields a  $\Delta H_{max}$  of 0.6 GPa compared with VC monolayer. This value is much lower than the measured  $\Delta H_{max}$  of about 12.6 GPa in this study, indicating that it is not enough for the Koehler's strengthening theory to directly explain the hardness enhancement of VC/TiC superlattices.

In reference to the alternating-stress theory [19], it is known that when the dislocations go across the interfaces in superlattices, they are resisted not only by the potential barriers coming from the change of line energy of dislocations due to the two different modulus layers (Koehler's strengthening theory), but also by that from the alternating-stress field. The maximum hardness increase from the alternating-stress field can be estimated as

$$\Delta H_{\rm max} \cong 10\tau_{\rm max} \tag{3}$$

where  $\tau_{\text{max}} = (2/\pi^2)A\eta E$  [21] is the maximum shear stress at the interface; *A* is the modulation amplifying factor that is closely correlated to structural parameters of superlattices such as modulation period, modulation ratio, roughness and width of interface; *E* is the weighted average elastic modulus of two constituents of superlattices; and  $\eta$  is the lattice mismatch between two layers of superlattices.

Based on the studies from MIRKARIMI et al [22] and SHINN and BARNETT [23], *A* takes the value of 0.5 for calculation in this study. Using the data of *E* of TiC and VC, 250 GPa, and  $\eta$  of 0.04 ( $a_{VC}$ =0.4160 nm,  $a_{TiC}$ =0.4328 nm), we can roughly estimate  $\Delta H_{max}$  as ~10.1 GPa. However, this estimation is not enough to explain the actual hardness enhancement. It should be noted that even the sum of the hardness increase from the alternating-stress field model and modulus-difference model, 10.7 GPa, is still lower than the measured value (12.6 GPa).

Actually, the discrepancy between the estimated value from these two strengthening theories and the actual result is due to approximation made when not considering any changes in the modulus of constituent layers in the superlattices under epitaxial strains. It is noted that the modulus changes have an important influence on the hardness increment based on these two theories. Some studies [24,25] have demonstrated that an applied elastic strain can significantly change the in-plane biaxial modulus of thin films, that is, the biaxial modulus increases with compressive strains, and reduces with tensile strains. As shown in Fig. 5, the remarkable increase of elastic modulus of superlattices is closely related to the epitaxial strains when the modulation period is less than a critical thickness. Thus, the biaxial modulus of TiC sublayers with relatively high modulus in the presence of VC/TiC superlattices should increase, while the corresponding biaxial modulus of VC sublayers with lower modulus should decrease. This leads to the fact that the modulus difference, R, between TiC and VC sublayers in superlattices would be increased under the epitaxial strains. Therefore, the hardness enhancement in VC/TiC superlattices should increase in reference to the Koehler's strengthening theory as well as the alternating-stress field theory, when taking into account the modulus change of constituent layers coming from the epitaxial strains.

According to the (111) peak positions of TiC, VC monolayers and VC/TiC superlattices in Fig. 3, it can be calculated that the out-of-plane compressive strain in TiC sublayers is about 2.5%, and the out-of-plane tensile strain in VC sublayers is about 1.5%. Also, the calculations from Refs. [24,25] revealed that ~2.5% compressive strain could increase the biaxial modulus of materials by about 100%, and about 1.5% tensile strain could lead to 30% decrease in biaxial modulus. It is noted that these calculations agree well with the measured maximum modulus of VC/TiC superlattices, as shown in Fig. 5. If it is presumed that the in-plane strain is equal to the out-of-plane strain in constituent layers, we can accordingly obtain  $\Delta H_{\text{max}}$  of 9.7 GPa by the Koehler's theory, and 13.8 GPa by the alternating-stress field theory in VC/TiC superlattices. Here, it can be found that the sum of hardness increase from the Koehler's theory and alternating-stress field theory, 23.5 GPa, seems to be quite competent to explain the actual experimental results in this work.

From the present VC/TiC superlattices, it can be deduced that in order to synthesize the superlattices with superhardness effect, the epitaxial structure should be formed primarily. On the other hand, the threedirectional strains induced from the epitaxial structures should increase the modulus difference between two constituent layers in superlattices. Otherwise, the superhardness effect could not even be obtained in some superlattice systems with epitaxial structure, for example, our previously reported  $ZrO_2/TiN$  superlattices [14], in which the modulus difference between two constituent layers was found to be decreased by the epitaxial strains.

# **4** Conclusions

1) The VC/TiC superlattices can form an epitaxial structure when the modulation period is less than a critical value (~5.2 nm), resulting in that their hardness and elastic modulus are significantly increased. Further increasing the modulation period, the hardness and elastic modulus of VC/TiC superlattice films gradually decrease to be close to the rule-of-mixture value due to the destruction of epitaxial structure.

2) The hardness enhancement of the VC/TiC superlattices is closely related to the three-directional strains, which are induced from the epitaxial structure and may change the interplanar spacing and thus the modulus of constituent layers when the epitaxial structure is formed.

3) It is found that when taking into account the modulus change of VC and TiC sublayers in superlattices under the three-directional strains, the remarkable hardness enhancement of VC/TiC superlattices could be interpreted by combining the Koehler's theory and the alternating-stress field theory.

# References

- [1] KANG M S, WANG T G, SHIN J H, NOWAK R, KIM K H. Synthesis and properties of Cr–Al–Si–N films deposited by hybrid coating system with high power impulse magnetron sputtering (HIPIMS) and DC pulse sputtering [J]. Transactions of Nonferrous Metals Society of China, 2012, 22(S3): s729–s734.
- [2] HELMERSSON U, TODOROVA S, BARNETT S A. Growth of singlecrystal TiN/VN strainedlayer superlattices with extremely high mechnical hardness [J]. Journal of Applied Physics, 1987, 62(2): 481–485.
- [3] MUSIL J. Hard and superhard nanocomposite coatings [J]. Surface & Coatings Technology, 2000, 125(1–3): 322–330.
- [4] MARULANDA D M, OLAYA J J, PIRATOBA U, MARIÑO A, CAMPS E. The effect of bilayer period and degree of unbalancing on magnetron sputtered Cr/CrN nano-multilayer wear and corrosion [J]. Thin Solid Films, 2011, 519(6): 1886–1893.
- [5] SUN P L, SU C Y, LIOU T P, HSU C H, LIN C K. Mechanical behavior of TiN/CrN nano-multilayer thin film deposited by unbalanced magnetron sputter process [J]. Journal of Alloys and Compounds, 2011, 509(6): 3197–3201.
- [6] LAO J J, SHAO N, LI G Y, MEI F H, GU M Y. Mutual promotion effect of crystal growth in TiN/SiC nanomultilayers[J]. Appl Phys Lett, 2005, 86(1): 011902.

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- [7] KALIARAJ G S, RAMADOSS A, SUNDARAM M, BALASUBRAMANIAN S, MUTHIRULANDI J. Studies of calcium-precipitating oral bacterial adhesion on TiN, TiO<sub>2</sub> single layer, and TiN/TiO<sub>2</sub> multilayer-coated 316L SS [J]. Journal of Materials Science, 2014, 49(20): 7172–7180.
- [8] LI W, LIU P, ZHAO Y S, MA F C, LIU X K, CHEN X H, HE D H. Crystallization of amorphous SiC and superhardness effect in CrAIN/SiC nanomultilayered films [J]. Surface & Coatings Technology, 2013, 214: 168–172.
- [9] HÖGBERG J, BIRCH J, JOHANSSON M P, HUTMAN L, JANSSON U. Deposition of epitaxial transition metal carbide films and superlattices by simultaneous direct current metal magnetron sputtering and C<sub>60</sub> evaporation [J]. Journal of Materials Research, 2001, 16(3): 1301–1310.
- [10] BELGER A, REIBOLD M, PAUFLER P. Modulus and hardness change of silicon and sapphire substrates by TiC/VC multilayer coatings [J]. Materials Sciences and Applications, 2012, 3: 185–194.
- [11] SEKKAL W, ZAOUI A, SCHMAUDER S. Nanoindentation study of the superlattice hardening effect at TiC(110)/NbC(110) interfaces [J]. Applied Physics Letters, 2005, 86(16): 163108.
- [12] LI G Y, LI G Q, YANG D. Li. Microstructure evolution and mechanical properties in VC/SiC nanomultilayers [J]. Applied Surface Science, 2012, 258(24): 9856–9858.
- [13] KIM C, OADRI S B, SCANLON M R, CAMMARATA R C. Low-dimension structural properties and microindentation studies of ion-beam-sputtered multilayers of Ag/Al films [J]. Thin Solid Films, 1994, 240(1-2): 52–55.
- [14] YUE J L, LIU Y, ZHAO W J, LI G Y. Template-induced coherent growth and mechanical properties of ZrO<sub>2</sub>/TiN nano-multilayers [J]. Scripta Materialia, 2006, 60(4): 240–243.
- [15] LI G Q, LI Y G, LI G Y. Crystallization of amorphous AlN and

superhardness effect in VC/AIN nanomultilayers [J]. Thin Solid Films, 2012, 520(6): 2032–2035.

- [16] LI G Q, LI Y G, LI G Y. Crystallization of amorphous Si<sub>3</sub>N<sub>4</sub> and superhardness effect in HfC/Si<sub>3</sub>N<sub>4</sub> nanomultilayers [J]. Applied Surface Science, 2011, 257(13): 5799–5802.
- [17] LI W, LIU P, WANG J T, MA F C, LIU X K, CHEN X D, HE D H. Investigation on microstructure and mechanical properties of reactively synthesized TiAlN/AION nanomultilayers [J]. Vacuum, 2012, 86(9): 1300–1304.
- [18] KOEHLER J S. Attempt to design a strong solid [J]. Physical Review B, 1970, 2(2): 547–551.
- [19] KATO M, MORI T, SCHWARTZ L H. Hardening by spinodal modulated structure [J]. Acta Metall, 1980, 28: 285–291.
- [20] CHU X, BARNETT S A. Model of superlattice yield stress and hardness enhancements [J]. Journal of Applied Physics, 1994, 77(9): 4403–4407.
- [21] XU J H. Studies on microstructure and superhardness effects of ceramic nano-multilayer films [D]. Shanghai: Shanghai Jiao Tong University, 2000: 79–100. (in Chinese)
- [22] MIRKARIMI P B, BARNETT S A, HUBBARD K M, JERVIS T R, HULTMAN L. Structure and mechanical properties of epitaxial TiN/V0.3Nb0.7(100) superlattices [J]. Mater Res, 1994, 9: 1456–1463.
- [23] SHINN M, BARNETT S A. Effect of superlattice layer elastic moduli on hardness [J]. Appl Phys Lett, 1994, 64: 61–63.
- [24] JANKOWSKI A F, TSAKALAKOS T. The effect of strain on the elastic constants of noble metals [J]. J Phys F: Met Phys, 1985, 15: 1279–1292.
- [25] CAMMARATA R C, SIERADZKI K. Effects of surface stress on the elastic moduli of thin films and superlattices [J]. Physical Reviews Letters, 1989, 62(17): 2005–2008.

# VC/TiC 超晶格薄膜的微结构及超硬效应

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摘 要:采用磁控溅射工艺制备 VC/TiC 超晶格薄膜,并采用 EDXA 、XRD、HRTEM 和纳米力学探针研究调制 周期对超晶格薄膜的微结构和力学性能的影响。研究结果表明,当超晶格薄膜的调制周期低于临界厚度时,制备 的 VC/TiC 超晶格薄膜能够形成共格生长结构,并获得硬度显著提高的超硬效应。然而,随着调制周期的进一步 增大,超晶格薄膜的共格结构遭到破坏,导致薄膜的硬度逐渐降低,并最终趋于其组元的混合平均值。XRD 结果 表明,当形成共格生长结构时,超晶格薄膜内部将产生共格协调应变,从而改变不同调制层的弹性模量,这也是 VC/TiC 超晶格薄膜能够获得超硬效应的重要原因。

关键词:超晶格薄膜;碳化物薄膜;微结构演变;超硬效应;外延生长

(Edited by Xiang-qun LI)

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