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Improved metal cutting performance with bias-modulated textured $\text{Ti}_{0.50}\text{Al}_{0.50}\text{N}$ multilayers

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Abstract

In this work we present the cutting performance of $\text{Ti}_{0.50}\text{Al}_{0.50}\text{N}$ coatings which have been deposited with both a fixed and an alternating bias of -35 V and -70 V together with a $\text{Ti}_{0.33}\text{Al}_{0.67}\text{N}$ reference coating grown at -35 V. During deposition of the bias-layered coatings, the bias was instantaneously changed with four different bias-layer periods ranging from approximately 250 to 1500 nm. For the layers deposited with a fixed bias, a transition from a (100) to a (111) preferred orientation was observed with the change in bias from -35 V to -70 V. The coatings grown with an alternating bias, however, show a (111) preferred orientation with an intensity that slightly depends on bias-layer period. Metal cutting tests were performed in 100Cr6 steel with a cutting speed of 200 m/min, feed of 0.2 mm/rev and a depth of cut of 2 mm. The wear resistance was quantified in terms of crater and flank wear resistance showing an improvement for all bias-layered coatings. This is attributed to a (111) oriented refined grain structure in combination with low residual stresses in the coating.

1. Introduction

$\text{Ti}_{1-x}\text{Al}_x\text{N}$ [1,2] is well known as a hard coating in metal cutting applications due to its good oxidation resistance and its ability to age harden [3,4] at the elevated temperatures [5-7] present during metal machining. A large number of previous publications have established the age hardening to originate from the isostructural spinodal decomposition [3,4,8-11] of the as-deposited cubic (B1) structured c- $\text{Ti}_{1-x}\text{Al}_x\text{N}$ into nanometer sized coherent c-TiN and c-AlN rich domains. These domains have a difference in elastic properties [12] and exhibit coherency strains from the lattice mismatch, thus act as excellent obstacles for dislocation movements. With increasing time or temperature the domains grow in size [13-15] after which the c-AlN rich domains eventually transforms into the thermodynamically stable hexagonal (B4) structure, h-AlN. The presence of incoherent precipitates of h-AlN reduces the mechanical properties and severely degrades wear resistance and hence the life time of the cutting tool [16]. Means to alter the thermal stability of $\text{Ti}_{1-x}\text{Al}_x\text{N}$ includes alloying with additional elements, e.g. Cr, Hf, Ta, Zr or Y [17-26].

Commonly, $\text{Ti}_{1-x}\text{Al}_x\text{N}$ coatings deposited by physical vapor deposition exhibit a fiber texture with a preferred orientation that may be controlled by, e.g., the bias or the film thickness [27-32]. This preferred orientation is one of the factors determining the cutting behavior due to the anisotropic elastic properties of $\text{Ti}_{1-x}\text{Al}_x\text{N}$ [12] and we have earlier shown [33] a strong alignment of the spinodally decomposed domains along the elastically softer $\langle 100 \rangle$ directions. The alignment was shown to scale with the Al content due to its strong effect on the anisotropy in the system and this was discussed to contribute to the better flank wear resistance with increasing Al content, up to $x=0.67$, shown by Hörning *et al* [16]. However, the resistance against crater wear is often not coupled with the flank wear resistance and there is in general a tradeoff between crater and flank wear resistance. For example, Khatibi *et al* [34] showed an improvement of crater wear resistance for some arc-evaporated oxides over $\text{Ti}_{1-x}\text{Al}_x\text{N}$ coatings but at the expense of the flank wear resistance. In contrast, Knutsson *et al* [35] showed that an improvement of both crater and flank wear resistance for a $\text{TiN}/\text{Ti}_{0.34}\text{Al}_{0.66}\text{N}$ multilayered structure compared to $\text{Ti}_{0.34}\text{Al}_{0.66}\text{N}$ monoliths can actually be achieved when the thermal stability is improved [36]. The improved thermal stability was achieved by an alternation of the decomposition process of the $\text{Ti}_{0.34}\text{Al}_{0.66}\text{N}$ layers caused by the presence of the TiN-layers

through promotion of the beneficial spinodal decomposition and suppressing the detrimental the c-AlN to h-AlN transformation. However, the influence of the grain size, residual stress, and texture on the high temperature wear resistance of $\text{Ti}_{1-x}\text{Al}_x\text{N}$ coatings remains less understood despite their known influence on the metal cutting behavior.

In this work, we investigate the crater and flank wear resistance of $\text{Ti}_{0.50}\text{Al}_{0.50}\text{N}$ coatings deposited such that a multilayer structure was achieved without alternating chemical composition of the different sub-layers. Instead variations in residual stress and grain size were achieved by an alternating bias voltage. The bias voltage was alternated instantly between -35 and -70 V with four different bias-layer periods. As a reference, monolithic fixed bias coatings were deposited using -35 and -70 V separately. The crater and flank wear resistance during metal cutting is shown to be improved for all bias-layered coatings compared to the bias monoliths. In addition, the preferred orientation and residual stress of the coatings were determined with x-ray diffraction (XRD) using an Euler cradle. Microstructural information was obtained using transmission electron microscopy (TEM) in as-deposited coatings and after annealing.

2. Experimental details

Reactive cathodic arc evaporation was used for deposition of the coatings in a commercial system (Sulzer Metaplas MZR323). Polished and ultrasonically cleaned WC-Co (WC 93.5, Co 6, (Ta,Nb)C 0.5 [wt%]) blanks (ISO SNUN120408) and cutting inserts (ISO TPUN160308) were mounted on a one-axis rotating substrate holder facing a total of six cathodes on two side walls. The composition of the compound Ti/Al-cathodes was 50/50 (or 33/67 for the reference coating). Prior to deposition, the substrates were Ar ion etched. Coatings were deposited at 4.5 Pa N_2 atmosphere, a cathode current of 60 A, and a substrate temperature of about 400 °C. The deposition time was set to one hour yielding a total coating thickness between 4 and 5 μm . For the bias monoliths, the substrate bias voltage was held constant at -35 V and -70 V, respectively. A periodic and incremental alternating substrate bias voltage between -35 and -70 V was applied during growth of the bias-layered coatings. The thickness of each sub-layer, grown with either a -35 V or a -70 V bias, was controlled through the period time of the applied bias voltage. The first sub-layer in all multilayers was always grown with a -35 V bias. Multilayers containing 6,

10, 20 and 36 layers with corresponding bias-layer periods of about 250, 450, 900 and 1500 nm, respectively, were deposited.

Heat treatments were performed on the coatings, cut in smaller dimensions ($1.8 \times 0.5 \times 0.5$ mm³), in a low vacuum furnace (base pressure $\sim 10^{-1}$ Pa). The heating rate was 20 °C/min followed by an isothermal step of 10 min at 1000 °C after which the samples were cooled down with a rate of ~ 50 °C/min.

θ -2 θ XRD measurements were employed in a PANalytical X'Pert PRO whereas a PANalytical Empyrean was used for residual stress and pole figure measurements. The residual stress was measured with the $\sin^2 \psi$ method by assuming a bi-axial stress state and using the 111 or the 200 peak depending on the preferred orientation. The range of $\pm\psi$ was chosen to give $\sin^2 \psi$ values between 0 and 0.8 with a step of 0.1. The elastic constants, E_{hkl} and ν_{hkl} , were taken from *ab initio* calculations [12] ($E_{111}=489$ GPa, $\nu_{hkl}=0.18$, $E_{200}=386$ GPa, $\nu_{200}=0.25$). Measurements from high indices reflections were not possible due to their low intensity, but the linear regressions from the low indices reflections were however satisfactory as is reflected in the relative errors. To verify the fiber texture of the coatings, pole figures were measured using the 2 θ -values of 111, 200, and 220 reflections with a resolution of three degrees for ψ and ω .

The cutting tests were performed in 100Cr6 steel with a cutting speed of $v_c = 200$ m/min, feed of $f_z = 0.2$ mm/rev and a depth of cut of $a_p = 2$ mm. A maximum cutting time of 15 min was used with intermediate cutting times of 1.5, 4.0 and 7.5 min. Scanning electron microscopy (SEM) using a Leo 1550 Gemini together with light optical microscopy was employed to evaluate the crater and flank wear behavior of the coatings. The crater wear was evaluated by measuring the area of the exposed substrate whereas the average flank wear land width (VB_B) was measured according to the ISO standard 3685 [37].

Mechanical grinding and polishing of samples for cross sectional transmission electron microscopy (TEM) was performed before thinning to electron transparency using a Gatan Precision Ion Polishing System. The TEM samples were characterized using a Fei Tecnai G² TF 20 UT analytical TEM operated at an acceleration voltage of 200 kV.

3. Results

Figure 1 shows normalized pole figures of $\text{Ti}_{0.50}\text{Al}_{0.50}\text{N}$, (a) -35 V bias monolith, (b) -70 V bias monolith and (c) bias-layered, λ_{900} . For the -35 V coating the 111, 200, and 220 pole figures have their maximum intensities at ψ values around 45, 10 and 30 degrees, respectively, thus indicating a preferred orientation close to the (100) orientation. The results of the -70 V coating shows a preferred orientation closer to the (111) orientation, i.e. the maximum intensity of (111) planes is closer to $\psi=0$ degrees. There is a clear difference between the two bias voltages where the preferred orientation changes from close to (100) at -35 V to close to (111) at -70 V. The pole figures for the bias-layered coating indicate a (111) preferred orientation which is slightly weaker compared to the -70 V coating which can be seen from the overall increase at all ψ -values >5 degrees. Similar results are found for all bias-layered coatings (not shown).

Figure 1. Pole figures showing the fiber texture of $\text{Ti}_{0.50}\text{Al}_{0.50}\text{N}$: (a) -35 V bias, (b) -70 V bias and (c) λ_{900} bias-layered

Figure 2 (a-c) shows bright field TEM (BF-TEM) micrographs of three (λ_{250} , λ_{450} and λ_{1500}) bias-layered coatings in their as-deposited state. All bias-layered coatings reveal a dense and fine-grained microstructure with a strong contrast variation between layers grown with different substrate bias. Here, the slightly brighter contrast belongs to the layers deposited with a bias of -35 V.

Figure 2. Overview TEM micrographs of bias-layered coatings for $\text{Ti}_{0.50}\text{Al}_{0.50}\text{N}$ at different bias-layer periods: (a) 200 nm, (b) 450 nm and (c) 1500 nm.

To clarify the origin of the contrast variations, a higher magnification micrograph of the λ_{450} coating is seen in Figure 3. In the figure, the different bias layers are marked with dashed lines between.

Figure 3. Bias-layered $\text{Ti}_{0.50}\text{Al}_{0.50}\text{N}$ (λ_{450}) in more detail. Approximate dashed lines separate the individual bias periods.

The layers deposited with the higher bias of -70 V reveal a blurry contrast indicating a defect rich lattice whereas the defect concentration in the -35 V layer is less pronounced. The dark crystallite seen in the -35 V layer continues from and to the -70 V layers but the diffraction contrast is clearly reduced in the -70 V layers, implying a rotation of the crystallite during growth.

Figure 4 (a-c) shows θ - 2θ diffractograms, normalized with the (100) WC peak, for all coating configurations, (a) the bias monoliths, (b) the bias-layered coatings and (c) magnified (111) peak of the bias-layered coatings.

Figure 4. θ - 2θ diffractograms of (a) fixed bias coatings, (b) bias-layered, and (c) detailed bias-layered. The stars correspond to substrate peaks or sample holder.

In Figure 4 (a), a larger shift in the -70 V coatings towards lower 2θ angles is seen compared to the -35 V coating, indicating a larger compressive stress. Results from the residual stress measurements are presented in Table 1. A clear trend of increasing compressive stress, from -3.3 GPa to -6.4 GPa, with the change in bias from -35 V to -70 V is seen. All bias-layered coatings show residual stresses slightly higher than the -35 V bias monolith. The bias-layered $\text{Ti}_{0.50}\text{Al}_{0.50}\text{N}$ coatings in Figure 4 (b) show an increased (111) preferred orientation with a reduced layer period and peak positions close to the -35 V monolith with a slight asymmetry towards lower angles. The asymmetry of the peak is revealed in Figure 4 (c) with vertical lines showing the (111) peak positions of the bias monoliths.

Table 1 Residual stress and thickness for the different coating configurations

Sample	Residual stress	Thickness
	[GPa]	[μm]
$\text{Ti}_{0.50}\text{Al}_{0.50}\text{N}$ (-35 V)	-3.3 $\pm$ 0.2	4.7
$\text{Ti}_{0.50}\text{Al}_{0.50}\text{N}$ (-70 V)	-6.4 $\pm$ 0.4	5.2
$\text{Ti}_{0.33}\text{Al}_{0.67}\text{N}$ (-35 V)	-2.4 $\pm$ 0.1	5.0
$\text{Ti}_{0.50}\text{Al}_{0.50}\text{N}$ (λ_{1500})	-3.8 $\pm$ 0.2	4.5
$\text{Ti}_{0.50}\text{Al}_{0.50}\text{N}$ (λ_{900})	-3.4 $\pm$ 0.1	4.5
$\text{Ti}_{0.50}\text{Al}_{0.50}\text{N}$ (λ_{450})	-3.6 $\pm$ 0.1	4.4
$\text{Ti}_{0.50}\text{Al}_{0.50}\text{N}$ (λ_{250})	-3.8 $\pm$ 0.1	4.3

Overview BF-TEM micrographs after 10 min annealing at 1000 °C are shown in Figure 5 of the two bias monoliths (a-b) and one of the bias-layered (λ_{450}) coatings (c). The -35 V bias monolith in Figure 5 (a) shows a distinct columnar microstructure with larger grains, as compared to Figure 6 (b-c), throughout the coating from substrate to top. In Figure 6 (b), the microstructure of the -70 V bias monolith is instead shown to be fine-grained and feather-like. This should be compared to the -70 V bias monolith in Figure 5 (b) which shows a fine-grained feather like-microstructure. A fine-grained microstructure is apparent in the bias-layered coating in Figure 3 (b) as well, although closer to columnar and not feather-like. By comparing the as-

deposited bias-layered coating in Figure 3 (b) with the annealed coating in Figure 5 (c) the contrast variations from the layering are reduced but still distinguishable.

Figure 5. Overview TEM micrographs of heat treated coatings for 10 minutes at 1000 °C: (a) -35 V bias, (b) -70 V bias and (c) λ_{450} bias-layered

Figure 6 shows the crater wear (a) and flank wear (b) as a function of cutting time for all coatings and one additional reference $\text{Ti}_{0.33}\text{Al}_{0.67}\text{N}$ monolithic coating deposited with -35 V. The insets show the measured crater area and average flank land width. All $\text{Ti}_{0.50}\text{Al}_{0.50}\text{N}$ coating variants exhibit a better crater wear resistance compared with the $\text{Ti}_{0.33}\text{Al}_{0.67}\text{N}$ coating, which has a crater wear around 0.9 mm^2 after 15 min of cutting. $\text{Ti}_{0.50}\text{Al}_{0.50}\text{N}$ deposited with a fixed bias of -35 V bias shows wear score of about 0.8 mm^2 and the -70 V monolith 0.6 mm^2 .

Figure 6. (a) Crater and (b) flank wear evolution with cutting time for all coatings including a -35 V biased $\text{Ti}_{0.33}\text{Al}_{0.67}\text{N}$ reference. The error bar in the lower right corner indicates a typical error from previous measurements.

Furthermore, all bias-layered coatings perform better compared to the bias monoliths coatings, where the best crater wear (below 0.4 mm^2) is seen for λ_{450} . With the exception of λ_{250} , there is also an inverse relationship with crater wear and bias-layer period. As the crater wear is affected by coating thickness, these are presented Table 1 where all coatings are shown to have a thickness of roughly $4.5 \pm 0.5 \text{ }\mu\text{m}$. Considering flank wear, the -35 V monolith shows a flank wear of 0.18 mm after 15 min whereas the -70 V monolith have a maximum flank wear of 0.16 mm. The flank wear for the $\text{Ti}_{0.33}\text{Al}_{0.67}\text{N}$ reference coating is improved compared to the $\text{Ti}_{0.50}\text{Al}_{0.50}\text{N}$ bias monoliths with a maximum flank wear of 0.13 m. As with the crater wear resistance, all the bias-layered coatings show an improved flank wear resistance compared with the bias monoliths where λ_{450} shows the best performance with 0.11 mm.

4. Discussion

4.1 Preferred orientation

All coatings exhibit a crystallographic fiber texture along the growth direction (GD). The results for the -35 V monolith show a preferred orientation consisting of slightly tilted grains grown along $\langle 100 \rangle$. These results are similar to the findings of e.g. Falub *et al* [27] and Karimi *et al* [28] which both show a preferred orientation of the high indices lattice planes (013, 113 and 115). Based on the ψ -angles where we have the maximum intensity in the (111), (200) and (220) pole figures these results imply a preferred (015) orientation. A transition to a preferred orientation of (111) occurs when the bias is changed to -70 V. For this coating the (111) oriented grains show less tilt in the GD, i.e. the orientation is a more distinctly (111) compared to the more spread (200) orientation seen for the -35 V monolith. The transition towards a (111) preferred orientation with increasing bias was discussed by Falub *et al* [27] who correlated the change in preferred orientation during growth to the apparent residual stress. The authors stated that the higher residual stress due to a higher substrate bias favors (111) growth. They however also mention the changed surface mobility of incoming and adsorbed atoms during growth as a factor determining the preferred orientation. A direct correlation between the residual stress and the preferred orientation is not found in this study as is seen in Figure 1 and Table 1. The modified surface mobility was also discussed by Alling *et al* [38] where the authors compared the ad-atom mobility on $\text{Ti}_{0.50}\text{Al}_{0.50}\text{N}(100)$ and $\text{TiN}(100)$ surfaces. Upon alloying TiN with AlN,

there is a gradual change from a (111) to a (100) preferred orientation [16]. According to Alling *et al*, the addition of Al surface atoms on TiN causes high energy barriers for surface diffusion that reduces the mobility of ad-atoms on (100) surfaces. This restrains the growth on the (100) surfaces and thus promotes the (100) growths at a low bias. However, the change in bias in this paper from -35 V to -70 V increases the kinetic energy of the incoming ions. This increases the probability of surface diffusion out of the (100) surfaces which would explain the orientation difference between the monolithic coatings.

Despite a first bias-layer deposited with -35 V in the bias-layered coatings, the competitive growth between (111) and (100) grains are dominated by (111) grains suggesting that a switch from (111) to (100) growth is a slower process than vice versa for the growth conditions studied. That is, for the investigated multilayers the periods with -35 V are not sufficiently long to cause a reversal to (100) growth and instead the (111) growth set during the -70 V bias periods dominates.

4.2 Microstructure and cutting properties

The periodic contrast changes seen in Figure 2 are generated by the bias variation and scale with the expected bias-layer thickness. However, the direct cause of the contrast variation is not caused by actual growth direction variation between the individual layers as discussed above and such variation would have been seen in the pole figures in Figure 1 (c). In Figure 3, the layers with the blurry contrast stem from the layers deposited with a bias of -70 V. This corresponds well with the increased energy of the arriving ions caused by the higher bias, as the probability of inducing lattice point defects in the layer increases with a blurred contrast as a consequence. Since a larger amount of lattice defects also increases the in-plane compressive stress [39], we would expect different stress states in the individual layers and thus a change in the out-of-plane lattice parameter. This is supported by the XRD diffractograms in Figure 4 (c) where the asymmetric 111 peak is convoluted by two peaks at positions identical to the two different positions observed for the two coatings grown with a fixed bias. Further support is seen from the residual stress measurements showing an intermediate stress state for the bias-layered coatings compared to the bias monoliths. The trend with a smaller grain size with an increased bias is also

an effect of the higher energy of the incoming ions as the probability for re-nucleation increases [40]. Hence, the intermediate grain size observed for the bias-layered coatings is expected.

During metal cutting, the position of the maximum temperature and the maximum shear stress is located at around half of the cutting length at the chip-rake interface [6,7,41]. Due to the high loads and high temperature, the crater originates at this position and eventually results in substrate material exposure. The crater wear is often referred to as a combination of chemical wear due to diffusion [42], abrasive wear from the chip sliding [43], and plastic deformation of the coating [44,45], and it depends on work material and cutting parameters. Due to the nature of the crater wear it is also influenced by the coating thickness but in this study the coating thicknesses for the bias-layered coatings are all smaller than the bias monoliths (Table 1).

Apart from the reference coating, all coatings have identical compositions and the differences in crater wear resistance observed in this work must thus be related to the differences in microstructure, preferred orientation or the residual stress.

We attribute the improved crater wear resistance in the -70 V monolith and bias-layered coatings over the -35 V monolith to a combination of the decreased grain size and the (111) preferred orientation. The smaller grain size increases the resistance against crack propagation and hence the toughness of the coating as opposed to the larger columns in the -35 V monolith. The introduction of layers with different stress states in the bias-layered coatings further increases the crack resistance in combination with the small grain size. We also note the change in preferred orientation in the -70 V monolith and the bias-layered coatings, where the (111) planes are close packed in the B1 structure, which may lead to a limited heat dissipation in the growth direction and instead to a larger extent through the chip. The crater wear resistance is also likely affected by the detrimental transformation of c-AlN to h-AlN which explains the improvement in the $\text{Ti}_{0.50}\text{Al}_{0.50}\text{N}$ monoliths over the $\text{Ti}_{0.33}\text{Al}_{0.67}\text{N}$ reference coating as the high Al content exhibits an earlier onset of the h-AlN transformation [4]. Furthermore, the combination of higher residual stress and normal stress acting on the rake face has in previous publications [46-48] been shown to promote spinodal decomposition and stabilize c-AlN over h-AlN. Thus, the higher residual stress in the -70 V monolith compared to the -35 V monolith is beneficial in terms of suppressing the detrimental h-AlN transformation although some stress relaxation is expected to occur during annealing. However, too large residual stresses increase the risk of

cohesive failure [49] which explains the further improved crater wear resistance in the bias-layered coatings. Here, the overall residual stress is lower, but the layers grown with -70 V benefit from their individual higher stress state.

The progression of the flank wear is mainly attributed to abrasive wear due to the sliding between the finished work material and the tool [50]. One of the parameters determining the flank wear is hence the hardness of the coatings at the elevated temperatures. Due to the lower temperature at the flank face compared to the rake face [6], the flank wear resistance is less prone to depend on the h-AlN transformation and instead depends on the age hardening behavior during spinodal decomposition. This explains the ranking of the bias monoliths due to the more pronounced age hardening of hardness of $\text{Ti}_{0.33}\text{Al}_{0.67}\text{N}$ compared to $\text{Ti}_{0.50}\text{Al}_{0.50}\text{N}$ [33]. However, a hardness ranking of the bias-layered coatings is not feasible since the penetration depth of 300-400 nm during nanoindentation is not sufficient to penetrate the different bias-layers. But we note that the flank wear resistance is improved for all bias-layered coatings when compared to the monolithic $\text{Ti}_{0.50}\text{Al}_{0.50}\text{N}$ coatings. The bias-layered λ_{1500} , λ_{900} and λ_{450} also show a similar or improved flank wear resistance when compared the $\text{Ti}_{0.33}\text{Al}_{0.67}\text{N}$ reference coating. Combining the overall wear resistance, all bias-layered coatings outperform the bias monoliths, including the $\text{Ti}_{0.33}\text{Al}_{0.67}\text{N}$ reference.

5. Conclusions

$\text{Ti}_{0.50}\text{Al}_{0.50}\text{N}$ coatings have been grown using a fixed or alternating bias of -35 V and -70 V. A clear transition from a (100) to a (111) preferred orientation in the bias monoliths is clearly seen with the change in bias from -35 V to -70 V. This is not apparent in the bias-layered coatings where the growth of (111) grains dominates during deposition of $\text{Ti}_{0.50}\text{Al}_{0.50}\text{N}$ with a slight increase at the decreased bias-layer periods. The microstructural differences observed in TEM between the individual bias-layers originate from a degraded lattice quality in the -70 V layers due to the higher energy of the incoming ions. The layered microstructure restrains the residual stress in the bias-layered coatings as compared to the -70 V bias monoliths. There are also small microstructural differences between the heat treated bias-layered coatings where $\text{Ti}_{0.50}\text{Al}_{0.50}\text{N}$ show a tendency towards a columnar structure with retained bias-layers whereas the -35 V monolith show an columnar microstructure with relatively large grains and the -70 V

monolith a notable decrease in grain size. The combination of the layered microstructure, with small grains in a (111) preferred orientation and a small residual stress results in an improvement of both crater and flank wear resistance for all bias-layered coatings compared with the bias monoliths.

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