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Study of Structural And Mechanical Properties of Tungsten Carbides, Coatings With Different Cutting Performance of Multilayer Diamond Coated Silicon Nitride Inserts in Machining Aluminum−Silicon Alloy

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ABSTRACT

Hard coatings of tungsten carbides have been obtained by the deposition of tungsten thin layers, on steel substrates (containing 0.5% to 0.75 wt.carbon), according to the cathodic magnetron sputtering held at temperature of 650°C. It is established by X-rays diffraction that, in thetemperature range 650-900°C, no formation of tungsten carbides was observed. However, the annealing at a temperature greater than orequal to1000°C promotes the reaction between the constituents of the samples (W, Fe, C) and hence the formation of W2C carbide. No othercompounds were detected. The micro-hardness measured by Vickers tests, increases with the rise in temperature, particularly from 1000°C.The morphology of the surface samples depends on the temperature and duration of thermal annealing.Aluminum−silicon (Al−Si) alloy is very difficult to machine and diamond tools are considered by far the best choice for the machining of these materials. Experimental results in the machining of the Al−Si alloy with diamond coated inserts are presented.Considering the fact that high adhesive strength and fine surface morphology play an importance role in the applications of chemical vapour deposition (CVD) diamond films, multilayer technique combining the hot filament CVD (HFCVD) method is proposed, by which multilayer diamond-coating on silicon nitride inserts is obtained, microcrystalline diamond (MCD)/ nanocrystalline diamond (NCD) film. Also, the conventional monolayer NCD and MCD coated inserts are produced for comparison. The as-deposited diamond films are characterized by field emission scanning electron microscopy (FE-SEM) and Raman spectrum. All the CVD diamond coated inserts and uncoated insert endure the aluminum−silicon alloy turning to estimate their cutting performances. Among all the tested inserts, the MCD/NCD coated insert exhibits the perfect behaviour as tool wear due to its very low flank wear and no diamond peeling.

Keywords: Thin films, RF magnetron sputtering, Coating, tungsten carbides, aluminum−silicon alloy , multilayer diamond films; silicon nitride; cutting performance .

I. INTRODUCTION

Diamond coated materials possess excellent mechanical and tribological properties, such as high hardness, high elastic modulus, good wear resistance,good corrosion resistance, low friction coefficient and thermal conductivity. Currently, the utilization of diamond films deposited by chemical vapor deposition (CVD) method has been increased in the important market of machining nonferrous materials [1]. It is worth noting that silicon nitride ceramic is considered an attractive substrate for depositing diamond films due to its low thermal expansion mismatch to diamond [2], which is useful for the improvement of adhesive strength between diamond film and substrate, and thus the prolonged lifetime of CVD diamond coated cutting inserts [3,4]. Therefore, CVD diamond coated silicon nitride inserts have gained considerable interest and are increasingly used for turning abrasive and hard materials, such as graphite, tungsten carbide, aluminum–silicon alloys and aluminum matrix composites [5−8]. Many types of diamond coated silicon nitride substrates are designed to examine their mechanical properties, including cutting performance. ALMEIDA et al [9] employed the hot filament CVD to deposit microcrystalline and nanocrystalline diamond coated inserts. The experimental results indicate that thesmoother nanocrystalline coatings can obtain a better workpiece surface roughness than the microcrystallineones, in turning the electrical discharge machining (EDM) graphite. It was also reported by HU et al [10,11] thatnanostructured diamond films have a better wear resistance than the micro-structured one. Therefore,nanostructured diamond film had been considered an outstanding candidate for the machining of nonferrousmetals [12]. However, the brale tip indentation testing suggests that microcrystalline diamond (MCD) gradeexhibits the best behavior of adhesion strength due to its crystallinity and superior hardness. In contrast,nanocrystalline diamond (NCD) coatings show the less effective chemical bonding to the ceramic substrate dueto the higher degree of sp2 content [13]. Therefore, to achieve the combination of strong adhesive strength andrefined surface morphology, many investigations on depositing nano-microcrystalline diamond films on theCo-cemented tungsten carbide (WC−Co) substrate using hot filament CVD (HFCVD) method were reported inRefs. [14,15]. Such diamond films already prove their adequacy in metal product industry. Compared with thecemented carbide drawing die, the working lifetime of the diamond-coated drawing die increases by a factor ofabove 15, and the quality and smoothness of drawn products can be greatly improved [16]. However, thecutting performance of the nano/microcrystalline diamond coated silicon carbide inserts has less been investigated, and the interface adhesion and wear endurance against the hardmetal parts are not clear. On the other hand, there is also lack of literature discussing the performance of multilayer diamond films, althoughtheir extraordinary wear resistance has been predicted [7].In this work, multilayer diamond films with two layers and distinct grades of nanocrystalline and micrometric grain sizes are fabricated by the HFCVD technique on silicon nitride inserts. The corresponding monolayer MCD and NCD films are also grown for comparative purpose. The as-deposited diamond films are characterized by field emission scanning electron microscopy (FE-SEM) and Raman spectrum. Their cutting performance is evaluated by turning aluminum−silicon alloy workpieces. the high temperature springs in equidistance and parallel manner. The substrates were put below the filaments, and the distance between the filaments and substrates was fixed at about 12 mm. A DC bias was applied betweenstrates so as to enhance thediamond nucleation density. The detailed depositionparameters are listed in Table 1.In this work, nano-microcrystalline diamond films,namely MCD/NCD coating, were deposited to evaluatetheir cutting performances. The monolayer MCD andNCD films were also produced for comparison underconditions A and B, as shown in Table 1, respectively.For depositing MCD/NCD film, a layer of fine-grainedMCD film was deposited on the silicon nitride substratefirstly under the condition A. Thereafter, the surface ofas-deposited MCD film was polished with diamond grits(50 μm) and subsequently a layer of NCD film was deposited on the polished surface under the condition B.Field emission scanning electron microscope (FE-SEM) was employed to characterize the surface morphology of coatings. The identification of diamond and graphitic phases was done by Raman spectroscopy at room temperature. Carbides, particularly of the transition metals, have a number of valuable properties, which make them the most promising materials for use in various new fields of technology [1]. They are widely used in cutting tools, tools resistant to wear, brasive and hard coatings [2]. They are also used for catalytic applications, (similar to the noblemetals) [3]. Tungsten carbide is one of these carbides throughout these years. The coatings of pure tungsten carbide, or alloyed with cobalt or iron tungsten carbide, exhibit high wear resistance and low friction [4,5].Furthermore, their hardness at high temperatures is out standing [6]. Tungsten carbide is also highly corrosionresistant in acidic media. Owing to its high-temperature stability, chemical inertness and good electrical conductivity, tungsten carbide is a promising thin film diffusion barrier material for the microelectronic devices designed to function at sustained elevated temperature and in hostile environments [7].The investigation of thin layers for hard coatings or electrical applications requires the preparation of a homogeneous material. However, tungsten carbide exists indifferent phases, most important are WC and W2C [8].Although the W2C phase is unstable below 1300°C [8],normally a mixture of both WC and W2C was found by most of the thin layer techniques like sputtering [9,10] and reactive sputtering [11,12], chemical vapor deposition (CVD) [13], solid-phase reaction [14] and ion beamsynthesis [15].In the present work, we have formed thin hard coatings of tungsten carbides. The samples are thin layers of tungsten deposited by RF magnetron sputtering on steel substrate. The samples were submitted to thermal annealing in vacuum, at various temperatures (650-1100°C). The formation of tungsten carbides, the evolution of the microstructure and the morphology of the surface of samples were followed by X-ray diffraction (XRD) and scanning electron microscopy (SEM). The measurements of micro-hardness were carried out by Vickers tests.

II. EXPERIMENTAL DETAILS

One series of samples (thin layer of tungsten / steel substrate XC70) are prepared. The thin layers of tungsten(6 μm) are deposited by RF magnetron sputtering in a vacuum 10-7 mbar 650°C.After the deposition process, samples (W layer &substrate) were submitted to thermal annealing in vacuum,at various temperatures (650-1100°C) and during different times.

2.1 Multilayer Diamond Coated Silicon Nitride Inserts

The plane silicon nitride inserts with square geometry produced by the reaction-sintered processing route were used as the substrates for fabricating the multilayer diamond films. Prior to deposition, all substrates were submitted to the three-step pretreatment to strengthen the adhesive strength between the diamond films and the substrate: 1) ultrasonically cleaning in distilled water for 30 min to remove binder phases from the surface; 2) scratching by hard cloth for 30 min in the suspension with 50 μm diamond power; 3)dipping the substrates in high-purity acetone ultrasonic vessel for 20 min to eliminate impurities adhered on the surface.The diamond films were fabricated by HFCVD technique in a home-made bias-enhance apparatus. Six pair-twisted tantalum wires were used as the hot filaments, and all of them were dragged to be straight by the high temperature springs in equidistance and parallel manner. The substrates were put below the filaments, and the distance between the filaments and substrates was fixed at about 12 mm. A DC bias was applied between the filaments and substrates so as to enhance the diamond nucleation density. The detailed deposition parameters are listed in Table 1. In this work, nano-microcrystalline diamond films, namely MCD/NCD coating, were deposited to evaluate their cutting performances. The monolayer MCD and NCD films were also produced for comparison under conditions A and B, as shown in Table 1, respectively.For depositing MCD/NCD film, a layer of fine-grainedMCD film was deposited on the silicon nitride substrate firstly under the condition A. Thereafter, the surface ofas-deposited MCD film was polished with diamond grits (50 μm) and subsequently a layer of NCD film wasdeposited on the polished surface under the condition B.Field emission scanning electron microscope(FE-SEM) was employed to characterize the surface morphology of coatings. The identification of diamondand graphitic phases was done by Raman spectroscopy at room temperature.

2.2 Machining test

Dry machining tests were performed in an industry facility, INDEX G200 turning machine, which is a highperforming CNC multi operations millturn lathe with 10 controlled axes (including *Y*- and *B*-axis) and counterspindle, bar capacity 60 mm and 28 tool stations (14 in each revolver) with every station equipped for bothrotating and rigid tools. All of the inserts were mounted on PSSNR2020K12 tool holder. Aluminum−silicon (Al−Si) alloy with about 14% silicon was used as the work piece material because the trend to substitute thesteel and cast iron vehicle components by Al−Si light alloy is part of the effort to allow improved fuel economy and lower the emissions of pollutants. On the other hand, such material is also suitable for machining studies because it has a tendency to form considerable dust during machining due to the presence of hard Si-rich phase particles in the alloy matrix [7]. Experimental results indicate that the cutting performance of Al−Si alloy is widely influenced by the silicon content. Increasing amount of Si results in harder, stiffer and more wear resistant alloy. However, Si contents above 12% result in extremely abrasive materials withdetrimental effects on their workability and on the wear of the tools used to shape them [17]. Thus, Al−Si alloy is very difficult to machine, and many machining experiments suggested that diamond tools are considered the best choice for the machining of these materials. In our following tests, the cutting parameters were set as follows: cutting speed of 350 m/min; cutting depth of 0.4 mm; feed rate of 0.1 mm/r.Tool wear was conducted to evaluate cutting performance of inserts. The criterion used for the tool wear measurement was the maximum width of the flankwear width because the tool was not regularly worn and chipping was the major reason for the failure [7]. Thelifetime of the examined insert is evaluated by the flank wear where one insert will be thought as failure once its

tool wear surpasses 0.3 mm or the diamond coating peels off from the substrate. All measurements were taken near the nose radius. Prior to the wear measurement, the hydrofluoric acid was used to etch off the workpiecematerial that was adhering to the edge of the tool. The tool wear of the insert was evaluated in an analysis of theimages of the flank wear using a tool microscope. All of values were measured after each turning 375 m of cutting length by a digital image processing system. Then the terminal flank wear was examined by measuring scars that appeared on the cutting edge with FE−SEM.

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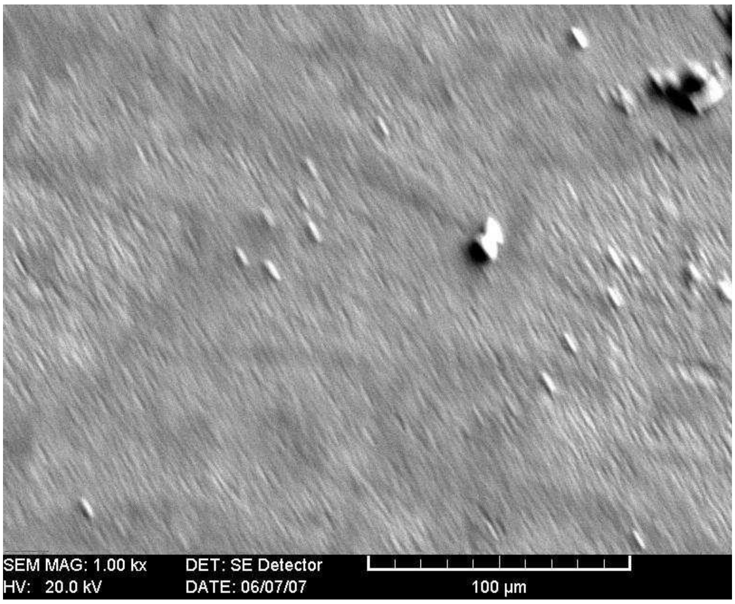
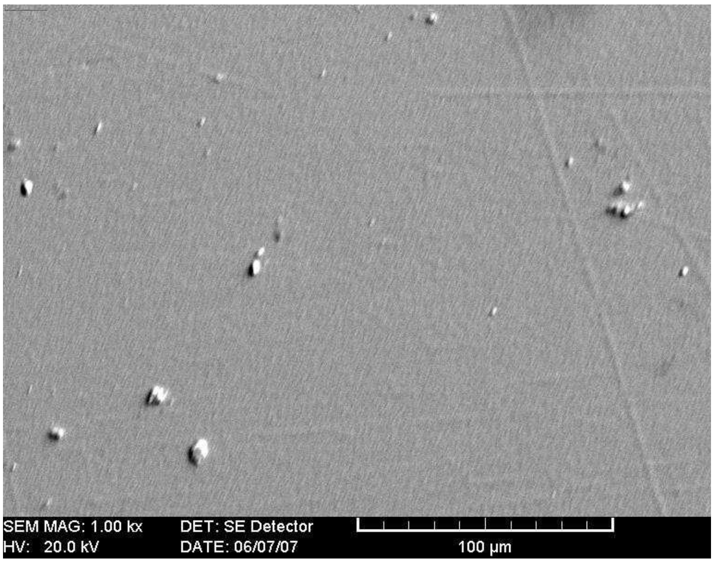
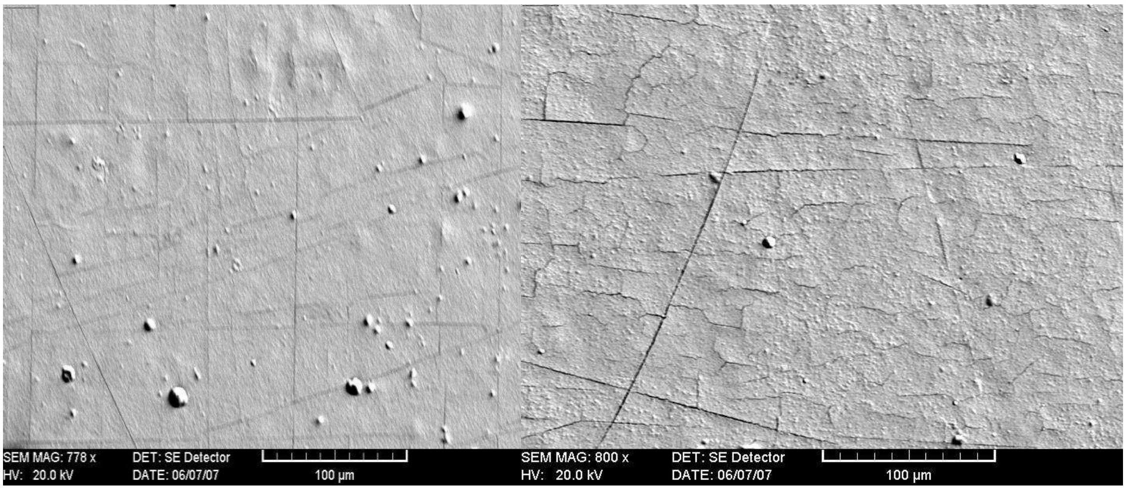
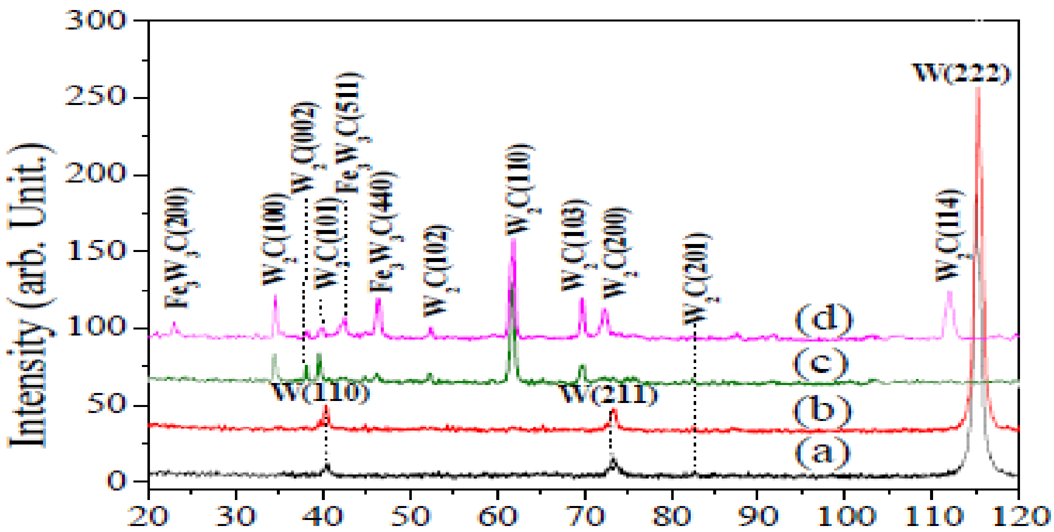
Deposition parameters of as- deposited multilayer diamond films

condition	Volume fraction of acetone /H2/Ar	Pressure /kPa	Deposition temperature /ºc	Filament temperature /ºc	Bias current /A	Duration/h
Condition A (for MCD films)	1.6/97.5/0	4-5	800-850	2200±100	3	6
Condition B (for MCD films)	3/66/30	1-3	850-900	2200±100	4	5









SEM surface images of the samples [W (8µm)/XC85]: before (a)and after annealed during 30 min at 800°C (b), 1000°C (c) and 1100°C (d).

III. RESULTS AND DISCUSSION

3.1 Analysis by X-rays diffraction (XRD)

Figure 1 shows the XRD patterns for the samples. The spectrum of the not-annealed samples shows the existence of only one phase of W, represented by 3 peaks with 40.6°,73.6° and 115.16°, corresponding to the plans (110), (211)and (222) respectively. On the other hand the annealing during 30min at 700°C samples does not make any structural modification compared to the state "not annealed", where we noted the existence of the three peaks of W with a texture of the layer according to the direction (222) Fig. 1: XRD spectra of the samples [W (8µm)/XC85]: before (a) and afterannealed during 30 min at 800°C (b), 1000°C (c) and 1100°C (d).However, the annealing of 30 min at 1100°C, allows the observation of new peaks with the disappearance of twopeaks of tungsten W(110) and W(211). These new peaks indicate the formation of two new phases: the binary phase W2C and ternary Fe3W3C.The annealing of 30 min at a higher temperature, 1100°C,does not change anything in the composition of samples ,except that it supports the growth of the phases formed previously (W2C and Fe3W3C).

3.2. Study of Surface Morphology

The study of the morphology of the samples by SEM also shows that the surface morphology of samples angleswith the time and the temperature of annealing. The figure 2 illustrates the images obtained with the SEM.In the case not-annealed and annealing at 700°C during 30 min (fig. 2.a & b) the surface of the samples is relatively smooth, but it presents small white particles. After an annealing with 900°C during 30 min (fig. 2.c), the surface morphology changes with the appearance of the cracks on the surface of the samples W(8μm)/XC85.The annealing at 1100 ° C for 30 min caused a remarkable increase of the cracks (fig. 2.d). This may be related to the reaction between the substrate and the thin layers of W

3.3. Micro-Hardness Measurements

Figure 3 shows the variation of microhardness (Hn) of the samples as a function of annealing temperature. It is clear that micro-hardness increases slightly with annealing temperature. The value of micro-hardness of not-annealed sample equals 312 kg/mm2 (value comparable to that of solid tungsten) [8]. However, after annealing the values of microhardness are distinct: for example the samples annealed at 900°C for 30 minutes increased the microhardness up 392 kg/mm2. At 1000°C/30min microhardness was maximum and takes the value 442 kg/mm2.This increase is due, probably, to the formation and growth of W2C carbide. On also note that the values of microhardness throughout the annealing temperatures are lower than those of massive carbides WC (~2000kg/mm2)

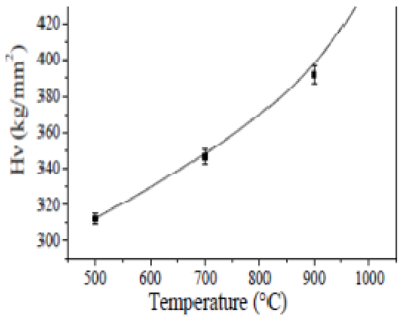


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Fig. 3: Variation of the micro-hardness in function of the temperature of annealing.

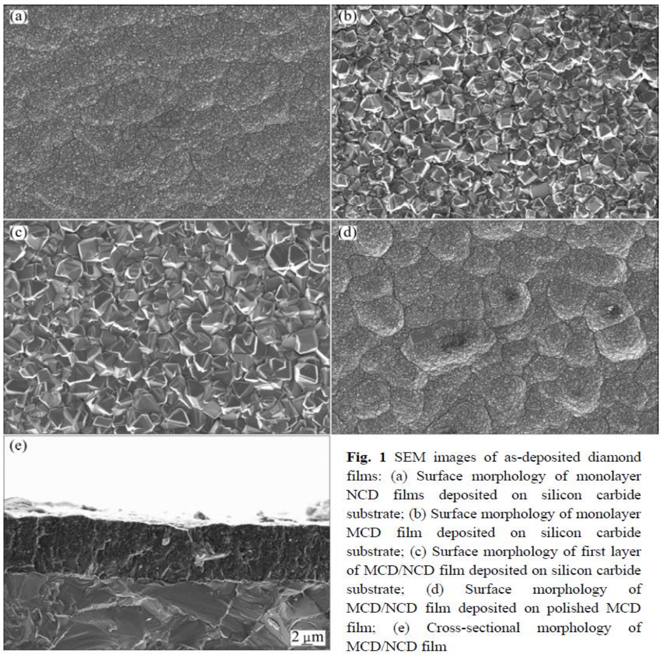

Fig. 1 SEM images of as-deposited diamond films: (a) Surface morphology of monolayer NCD films deposited on silicon carbide substrate; (b) Surface morphology of monolayer MCD film deposited on silicon carbide substrate; (c) Surface morphology of first layer of MCD/NCD film deposited on silicon carbide substrate; (d) Surface morphology of MCD/NCD film deposited on polished MCD film; (e) Cross-sectional morphology of MCD/NCD film

3.1 Characterization of as-Deposited Diamond Films

Figure 1 shows the surface morphologies of as-deposited diamond films. For the monolayer NCD film, the E−SEM image of its surface (Fig. 1(a)) shows that the substrate is covered by a layer of NCD filmswith diamond grains in nanometric level. Figure 1(b) shows the surface morphology of the monolayer MCDfilm, which indicates the typical features of conventional microcrystalline diamond coating with highly facetedgrains and the columnar structures. For MCD/NCD film,firstly, a layer of MCD film is deposited on the siliconcarbide insert. This layer of MCD film can facilitate adhesion strength enhancement of deposited multilayerdiamond films on silicon carbide substrate due to the mechanical interactions between diamond and SiC grainsof substrate [15]. The surface morphology suggests that the MCD film has a columnar structure, originating from large diamond grains at the free surface (Fig. 1(c)). These are responsible for the high values of roughness.Therefore, the as-deposited MCD film was polished with diamond grits in order to produce a flat surface. Subsequently, a layer of NCD film was deposited on this polished MCD film. Then, the surface morphology ofas-deposited MCD/NCD film can be seen in Fig. 1(d), which shows that the micron-sized diamond grains are

completely covered by many nano-sized diamond crystallites. It is clear that MCD and NCD films have avery distinct kind of structure as grain growth is suppressed by decreasing the atomic H density with theincrement of partial substitution by Ar, thus increasing the renucleation of diamond [6]. The cross-sectionalSEM image (Fig. 1(e)) indicates a thickness of 12–14 μm for the deposited MCD/NCD film.The quality of as-deposited coatings was examined by their physical properties by the corresponding Raman signatures. The typical NCD features present in the Raman spectrum of as-deposited NCD and MCD/NCD films, as presented in Fig. 2. A significantly broadening peak near 1332 cm−1 can be observed due to the decreasein grain size and phase purity, which is indicative of sp3diamond. Another peak located at approximately 1560cm−1, which can be assigned as the G band (1550−1580cm−1) [18,19], is responsible for the contribution fromthe sp2 component. The presence of scattering intensityat 1560 cm−1 is due to increasing graphite-like sp2bonded components at the grain boundaries in films. Onthe other hand, the Raman spectrum for the monolayerMCD film shows a sharp peak located in 1335 cm−1 andno graphitic peaks, which reveals the high purity andexcellent quality of the film. The shift between the sharppeak and the diamond peak (1332 cm−1) is associatedwith the residual stress of the MCD film [20].

3.2 Cutting Performance of as-deposited Multilayer

diamond films

Figure 3 shows the flank wear for all the as-deposited diamond coated inserts and uncoated insert as a function of cutting length. The worn surfaces of uncoated insert, NCD, MCD and MCD/NCD coated inserts are shown in Figs. 4−7. The corresponding surface morphologies of their cutting edges are also examined by FE−SEM (Fig. 8). The flank wear of the uncoated insert surpasses 0.3 mm after 1875 m of cutting length. An important feature of the NCD coated insert is the coating peeling during the initial 375 m turning process, as shown in Fig. 5 and Fig. 8(b). Such phenomena are a result of the higher degree of sp2 content in NCD films because the higher the graphite content at the interface, the lower the bonding strength to the substrate [21]. It is worth noting that the MCD crystalline purity is higher compared with NCD grade. In fact, the MCD sample has the smallest non-diamond phase content and thus its adhesive strength is strengthened. As a result, the MCD film relative to NCDfilm exhibits a significant enhancement in its cutting performance. Its flank wear shows that MCD coatedinsert can have a life of more than 1500 m of cutting length. However, the failure still occurs after 2250 m ofcutting length because the diamond coating peels off from the substrate, as shown in Fig. 6(e) and Fig. 8(c).Comparatively, the MCD/NCD coated insert shows a slow increasing tendency in flank wear during wholeturning process, which suggests that CVD diamond film can protect insert from suffering severe wear due to itsextremely high hardness and unique wear resistance, and plays a decisive role in prolonging the lifetime of theinserts. The results show that the MCD/NCD coated insert exhibits very lower flank wear than MCD coatedinsert and uncoated insert, which may be attributed to the nanocrystalline diamond coating with smooth surfaceand high wear resistance [6,12]. No coating peeling is NCD film compared with NCD film. On the other hand,the smoother surface of MCD/NCD film can lead to potential lower friction between the insert and workpiecematerial, which is beneficial to the chip evacuation and reducing the cutting force. Therefore, the tool wear ofMCD/NCD coated insert is very low. However, the MCD film has a high surface toughness and thus increasescutting force. This results in the fact that the MCD coated insert exhibits the coating peeling only after 2250m of cutting length

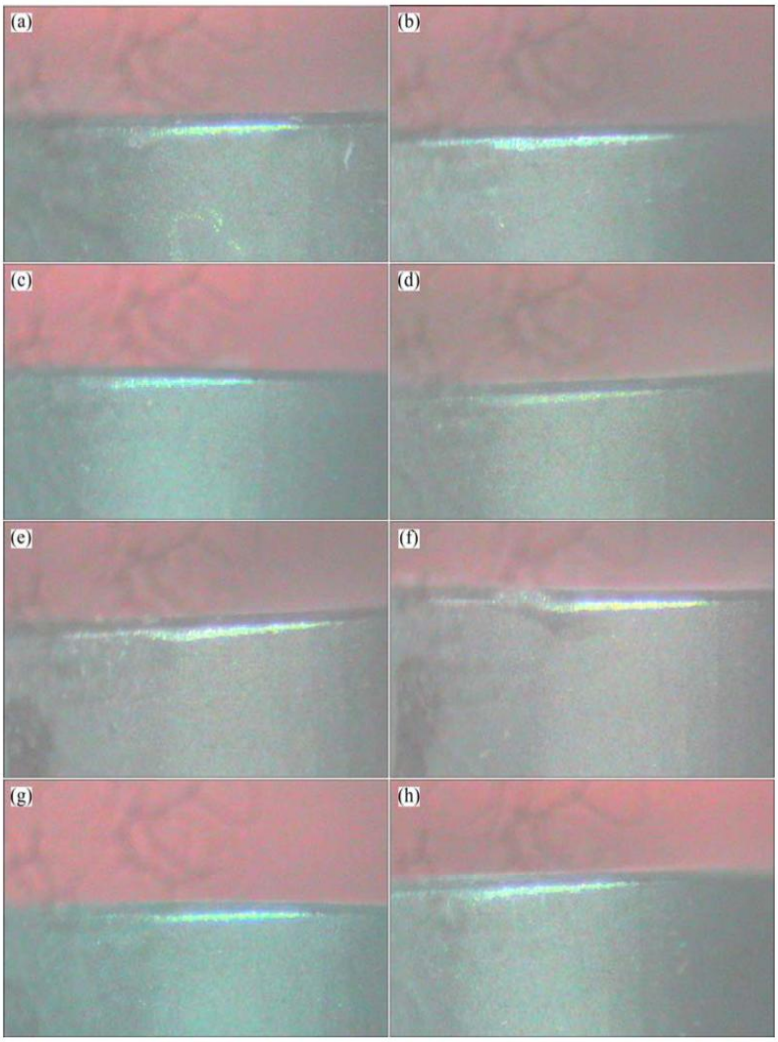

Fig. 7 Images of flank wear of MCD/MCD coated insert after turning 375 m (a), 750 m (b), 1125 m (c), 1500 m (d), 1875 m (e), 2250 m (f), 2650 m (g) and 3000 m (h) of cutting length

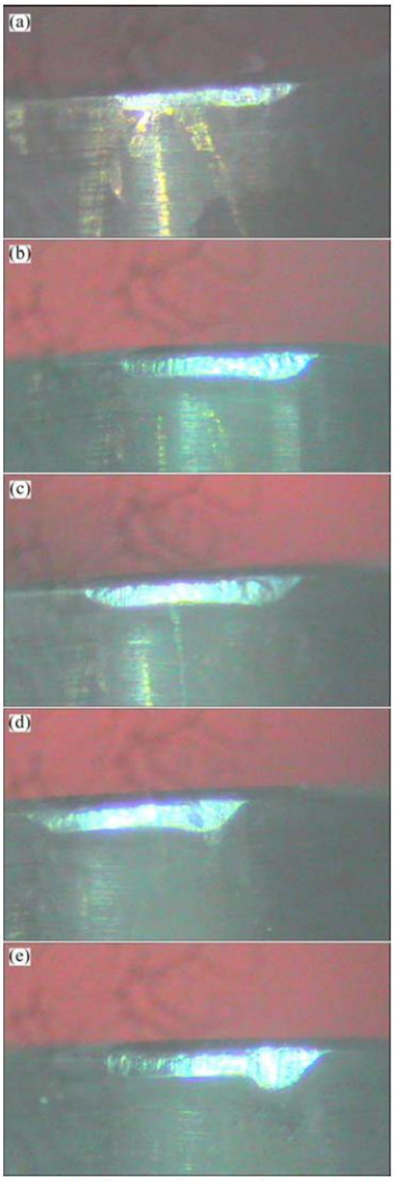

Fig. 4 Images of flank wear of uncoated insert after turning 375 m (a), 750 m (b), 1125 m (c), 1500 m (d) and 1875 m (e) of cutting length

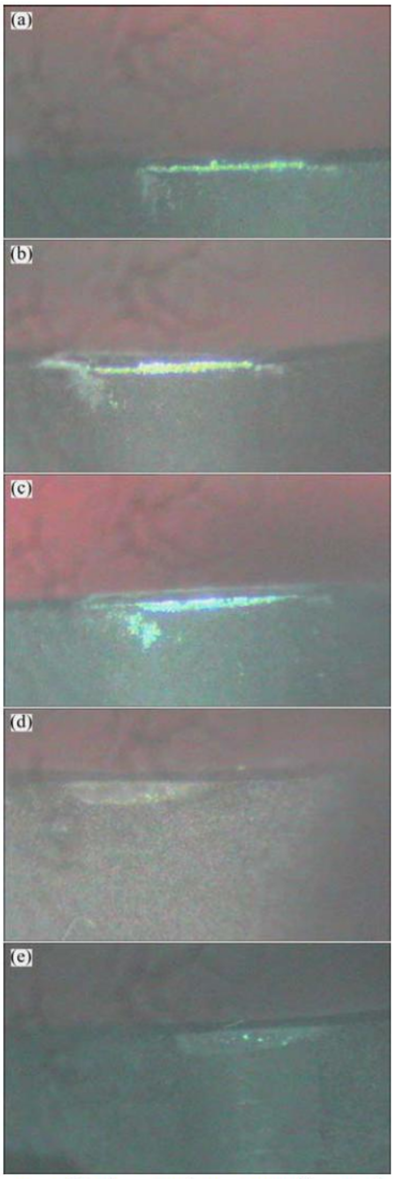

Fig. 6 Images of flank wear of MCD coated insert after turning 375 m (a), 750 m (b), 1125 m (c), 1500 m (d) and 1875 m (e) of cutting length

IV. CONCLUSION

According to the results obtained in this study, we can conclude that it is possible to work out hard tungstencarbide coatings by indirect method consists the coating of a steel substrate, rich in carbon by a layer of tungsten, then the annealing of together to support the diffusion of carbon and the formation of carbides with tungsten. The thermal annealing of the samples leads to the formation of the carbide W2C but with a lower growth rate for the thin layer of thickness 8 µm. The morphology of surface and the micro hardness also depends on the thicknesses of the thin layers of tungsten. 1) Multilayer diamond films are deposited onsilicon nitride inserts with a mixture of acetone,hydrogen and argon as the reactant gas by the HFCVDmethod. Double-layer structure (MCD/NCD) is fabricated initially by the deposition of the rough

microcrystalline diamond layers and then the smooth fine-grained nanocrystalline diamond layers. Suchmultilayer diamond coatings not only display good adhesion and wear resistant properties, but also havehigh surface smoothness.2) For comparative purpose, the uncoated insert and monolayer NCD and MCD coated inserts are also adopted in this work. The respective coating structure results in dissimilar cutting performance in dry turning of Al−Si alloys at 350 m/min of cutting speed, 0.4 mm of cutting depth and 0.1 mm/r of feed. The MCD/NCD coated insert exhibits the prefect behavior as tool wear.

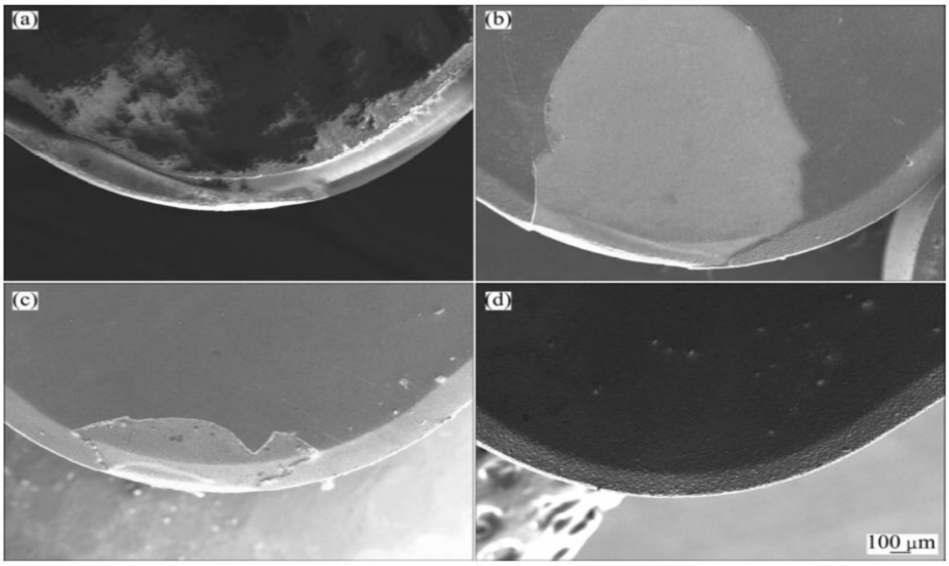

Fig. 8 FE−SEM images of surface morphology of inserts after turning Al−Si alloys: (a) Uncoated insert; (b) NCD coated insert; (c) MCD coated inserts; (d) MCD/NCD coated inserts

For this coating, the flank wear is very low after turning 3000 m of cutting length and no diamond peeling occurs. The performance of the monolayer MCD coated insert is slightly inferior due to the high hardness. Theconventional monolayer NCD film, featured by a cauliflower-like morphology, suffers the coating peelingfrom its lower adhesion between diamond coating and substrate. 3) Compared with the uncoated inserts, themultilayer diamond film can protect inserts from suffering severe wear due to its extremely high hardnessand unique wear resistance, and plays a decisive role in prolonging the lifetime of the inserts.

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