



# Carbon Nanotubes Coating for Micropunch

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**Abstract:** The multi-walled carbon nanotubes (MWCNTs) were coated on micropunch homemade equipment with the waste alcohol as a resource. The correlated characteristics were evaluated by scanning electron microscopy (SEM), transmission electron microscope (TEM), and Raman spectroscopy. Moreover, the synthesized MWCNTs were grown on some micropunches to confirm the relevant, beneficial effect on the service life of micropunches compellingly and convincingly. The results indicate that MWCNTs coated on micropunch can enhance its service life up to 35% of that without MWCNTs. Due to the lubrication of MWCNTs coating between the micropunch and the specimen, the wear of the micropunch coated with MWCNTs distinctively decreases, even in the severe wear period. As a result, the correlated wear loss is also less than that of the micropunch without MWCNTs coating, ascribed to the graphitic nature of MWCNTs. Meanwhile, because of the usage of the waste alcohol, the technique of the relevant synthesized MWCNTs is green to the environment, which is promising for practical applications.

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## 1. INTRODUCTION

Carbon nanotubes (CNTs) have attracted intense attention since their discovery [1, 2]. Due to the fascinating property of CNTs, they have been playing crucial but irreplaceable roles in many different kinds of applications in modern electronics, field emission sources, photodetectors, nonvolatile memory, ultrasensitive chemical and biosensors, and transparent conductive membranes. Nowadays, CNTs are integrated into many aspects of our everyday life, from rechargeable batteries, mobile screens, conductive additives, sporting goods, and water filters [3, 4].

Because of their unique structure, excellent electrical conductivity, mechanical property, high tensile and flexural strengths, high elastic modulus, and high aspect ratio, CNTs have been regarded as an attractive contender in tribological applications [5-10]. Interesting results were reported with a significant reduction in friction and wear rates in nanotube-polymers, ceramics, and metal composites. Theoretically speaking, the friction coefficient between the walls of MWCNTs can be extremely low [11-13].

It is well known that advances in telecommunication, transportation, and medical fields lead to challenges in microfabrication, miniaturization, and multi-functional

technologies in manufacturing. Feature sizes are in the range of a few microns up to hundreds of microns. The ability to fabricate micro holes in large quantities has potential applications in medical implants, diagnostic and remediation devices, micro-scale batteries and fuel cells, fluidic microchemical reactors requiring micro-scale pumps, valves and mixing devices, micro-fluidic systems, cooling holes in jet turbine blades, fibre optics, micro-nozzles for higher energy efficiency and fewer pollutant emissions to the environment, micro-molds and deep X-ray lithography masks, optical lenses, and micro components in daily life products such as compact disc players, airbags and inkjet printers. Currently, with the ever increasing demand for smaller, higher-quality, and lower-priced products from almost all fields of industry, household equipment, and entertainment electronics, the development of manufacturing methods that are tailor-made for the microsystems technique with higher precision, lower cost, larger quantities, more eco-friendly to the environment are extremely urgent [14-16].

Punching is the process of forcing a punch through the material and into a die to create a hole in the workpiece. It is often an economical way of creating shaped holes in mass production. Being a mechanical process that does not involve the use of chemicals, punching is friendly to the environment. Therefore, micropunching is an economical and environment-friendly way of fabricating shaped micro holes in mass production.

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It is well known that in the punching process, tool wear is an important issue urgent to be solved. Even with punches made of hard and tough materials like tungsten carbide (WC), the quality of the punched holes declines rapidly under repeated punching [17, 18].

The higher quality of micropunches is a key to the successful application of micro-punching. The punching number exceeds

1000, and the quality of the micropunch decreases obviously due to the serious wear of the micropunch. During the micropunching, the friction between micropunch and substrate deteriorates micropunch. In addition, the price of WC/Co micropunches is costly [18].

Because of the unique property of CNTs, the friction between micropunch and substrate can be decreased by a CNTs

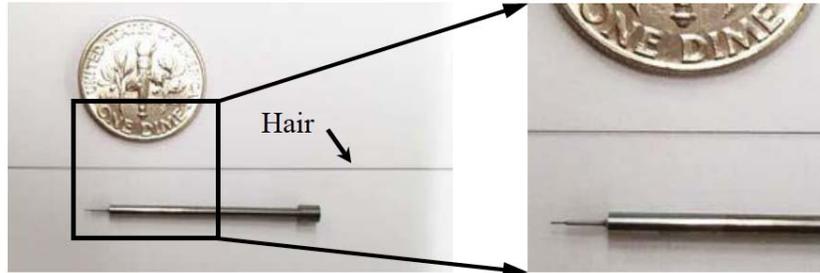
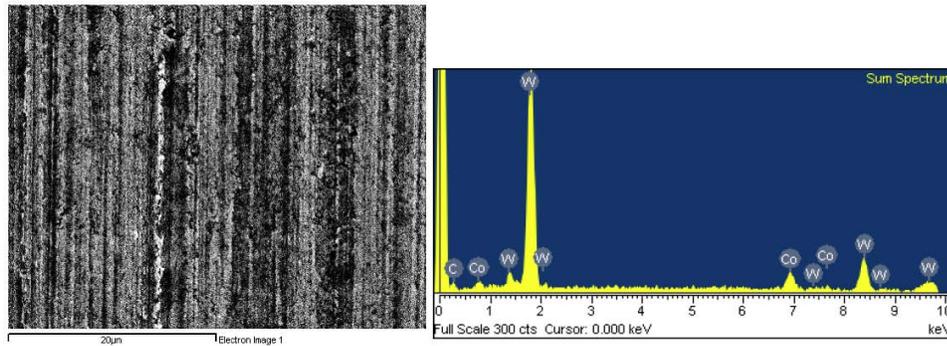


Figure 1: Profile of Ø150 µm micro punch.



Carbon Ka1\_2

Cobalt Ka1

Tungsten Ma1

Figure 2: Surface texture of WC/Co micropunch.

coating on the surface of micropunches to increase the wear property of micropunches and improve the tool life in the end. Therefore, aim to form an effective CNTs coating on WC/Co micropunches, the multi-walled carbon nanotubes (MWCNTs) were synthesized by homemade equipment with the waste alcohol, which is green/eco-friendly to the environment. Meanwhile, the effect of synthesized MWCNTs coating on the tool life of WC/Co micropunches has been fully investigated by confocal laser, SEM, TEM, digital balance, etc.

**2. EXPERIMENTAL MATERIALS AND PROCEDURES**

**2.1. Experimental Materials**

Micropunch with 75 % volume fraction WC particle and 25 % volume fraction Co particle of 50 μm mean size, 150 μm in diameter, is shown in Figure 1. Specimens for punching experiments were pure titanium sheets 200 μm in thickness.

Figure 2 shows the surface texture of micropunch and its elements' (Carbon, Tungsten, Cobalt) distribution. Plows induced by the ultraprecise machining can be obviously detected, and some debris distributed on the surface of micropunch. Moreover, the distribution of tungsten elements is denser than that of both carbon elements and cobalt elements, which indicates the hardness of the surface of micropunch is hard and suitable for the micro-punching process.

**2.2. Experimental Procedures**

**2.2.1. CNTs Growth**

Micropunches were cleaned with acetone and pure ethyl alcohol for contaminant removal prior to CNTs growth/deposition.

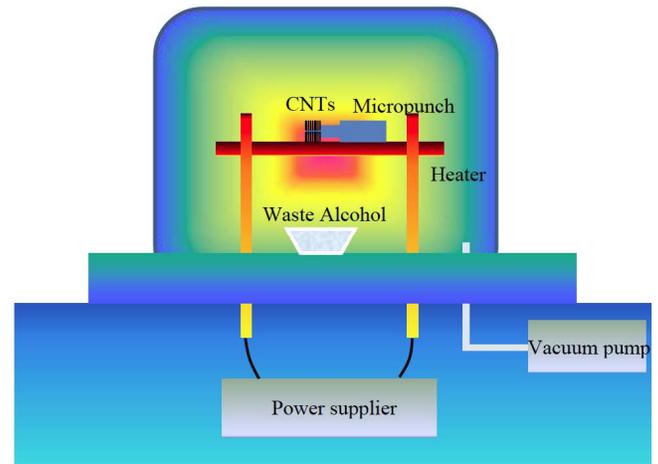
**Table 1: ECR Processing Parameters for Fe Deposition**

Substrate	WC/Co micropunch
Catalyst	Fe
Irradiation time	60 s
Accelerated voltage	2500 V
Ion current density	12.0 mA/cm <sup>2</sup>
Gas	Ar
Gas flow rate	0.6 SCCM
Vacuum	1.5×10 <sup>-4</sup> Pa

In this research, Fe was taken as the catalyst for CNTs forming. In the initial, the catalyst was coated on the required area of micropunch by electron cyclotron resonance (ECR). The relevant processing parameters are listed in Table 1. It is well known that the distribution of cobalt elements in micropunch is also a catalyst for CNTs forming; CNTs will be consequently synthesized during the CNTs' growth. Therefore, in order to protect the other areas from

synthesizing CNTs, aluminum foil is adopted as the cover for the remaining areas of micropunches [19, 20].

For the Fe coating process, micropunches were set in a vacuum chamber for CNTs forming. Figure 3 shows the relevant schematic diagram of homemade CNTs forming equipment. The micropunch was set on the heating area for CNTs growth. The dimension of the vacuum chamber was Ø100 mm × 150 mm. The waste alcohol, which was directly taken from the alcohol after the specimens' cleaning, met with the GB-HW06 standard, was put into a ceramic container under the heater to provide a carbon source for chemical vapor deposition (CVD). Conditions for CNT growth are summarized in Table 2.



**Figure 3: Setup for CNTs growth.**

**Table 2: Parameters for CNTs Growth**

Heating rate	3 ~4 A/s
Cooling rate	5 A/s
Reaction direct current	38 ~ 40 A
Reaction time	10 s
Vacuum	1×10 <sup>-2</sup> Pa

**2.2.2. Micropunching**

The acetone and pure ethyl alcohol were taken as a cleaner to make substrate-pure titanium sheet clean contaminants. After that, the substrate is carefully put into the micro-die. When all is set properly, the micropunching is in the process by the correlated micro-processing machine, where the federate of micro-punching was 2 mm together with 20 pulses of one minute.

The characteristics of the microholes punched by WC/Co micropunch in various periods were investigated by LEXT confocal laser-OLS3000, scanning electron microscopy (SEM), and energy dispersive X-ray analysis (EDX), transmission electron microscope (TEM), etc.

### 3. RESULTS AND DISCUSSION

#### 3.1. Micropunch with MWCNTs Coating

An image of a CNTs coated micropunch is shown in Figure 4, and its higher magnification image is expressed in Figure 5. It illustrates that the synthesized CNTs coat the surface of micropunch successfully, and the height of CNTs is about 15  $\mu\text{m}$ . Meanwhile, CNTs vertically align with high density. Moreover, CNTs appear wiggly due to the tightly packed during growth. TEM image of CNTs is shown in Figure 6. It indicates that CNTs are multi-walled nanotubes (MWCNTs), and their diameter is about 3-5 nm.

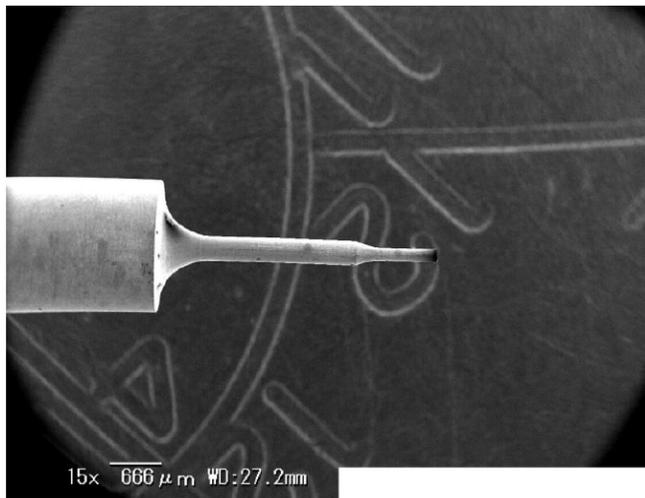


Figure 4: A micropunch with MWCNTs coating.

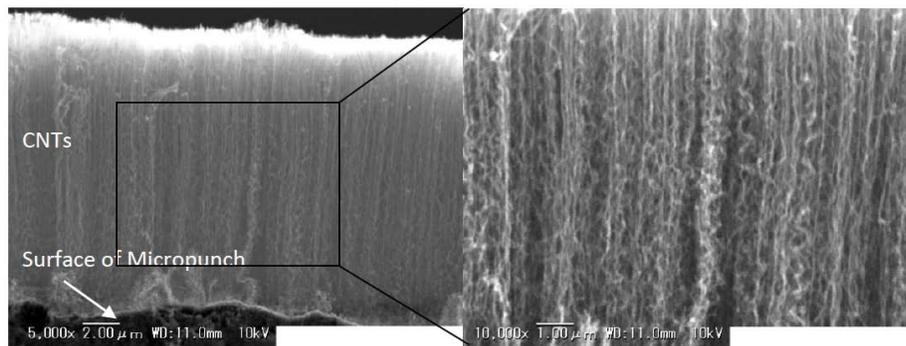


Figure 5: MWCNTs coating on the surface of micropunch.

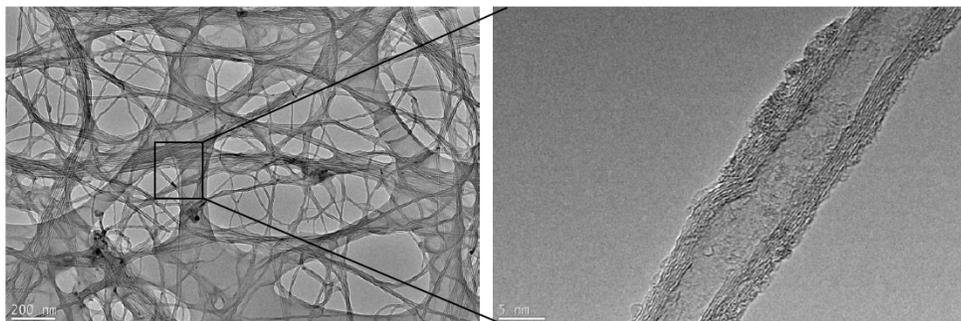


Figure 6: TEM image of synthesized MWCNTs.

#### 3.2. Raman Spectroscopy Analysis

Raman spectroscopy analysis is taken for testing the property of the synthesized vertically aligned MWCNTs. The parameters of Raman spectroscopy analysis are listed in Table 3. It is well known that the G-band, the D-band, and RBM are included in the typical Raman shifts ( $\text{cm}^{-1}$ ), and the corresponding peak of the G-band is at about  $1590 \text{ cm}^{-1}$ , and the D-band is  $1350 \text{ cm}^{-1}$ . The characteristic of the synthesized MWCNTs is evaluated according to the ratio between the G-band and the D-band.

Table 3: Conditions for Raman Spectroscopy Analysis

Laser wavelength	532 nm
Laser power	30 mW $\times$ 0.5%
Irradiation spot	5 $\mu\text{m}$
Integration times	30

Figure 7 expresses the result of Raman spectroscopy analysis of the synthesized vertically-aligned MWCNTs. It shows the ratio of IG/ID is about 0.75. Meanwhile, the peak of the D-band is higher than that of the G-band. It reveals that the synthesized vertically-aligned MWCNTs are highly disordered. As the media in tribology, the disordered MWCNTs will be more inclined to show their attractive lubricant characteristics more easily. Therefore, the micropunch with such MWCNTs must be more effective for practical applications than that of the micropunch without MWCNTs coating.

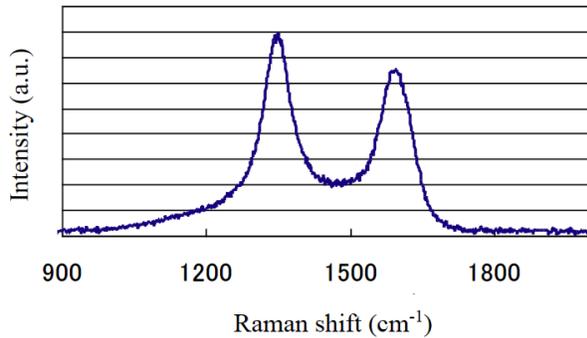


Figure 7: Raman spectra of synthesized MWCNTs.

### 3.3. Wear Loss of Micropunches

#### 3.3.1. Uncoated Micropunch

During the micropunching, micropunches vary during the various periods due to the wear of the micropunch along with the correlated weight changes. The variation of the micropunches was measured by the intermittent weight calibration by digital balance during punching. Figure 8 shows the relationship between the weight loss of micropunches and the punching number in the range of 1600 shots at the condition of taken and non-taken MWCNTs as micropunches underwent. The testing measurements were repeated 5 times, and the final results are based on the averaged results.

Figure 8 clearly shows that there are three stages during the micropunching process they are initial stage, quasi-stable stage, and severe wear stage. The initial stage is also known as the run-in stage. In this stage, for the micropunch without CNTs coating (cycle no. <500: labeled in black with the vertical dash line), the wear loss of the micropunch is

definitely obvious and relatively high. With the effect of friction between the uncoated micropunch and the substrate, the uncoated micropunch wears, and the surface is smoothed. At the same time, the crests, as shown in Figure 2 on the surface of the uncoated micropunch, are lowered; the correlated image is shown in Figure 9.

When the punching number is in the range of 500~1200, the wear characteristic of the uncoated micropunch is quasi-stable. In this stage, the wear loss is very little, and the variation of the surface texture of the uncoated micropunch is also hardly detected, as shown in Figure 10, in which WC particles can be observed on the surface and are rather uniformly distributed.

However, when the punching number exceeds 1200 (labeled as a vertical dash line), due to the severe cobalt loss, which is the bonding material in the micropunch, tungsten carbide (WC) is more easily peeled off seriously, resulting in the distinctive weight loss. Moreover, the uncoated micropunch is more inclined to break in this stage, as shown in Figure 11. It shows that large amounts of WC particles are distributed on the surface of the uncoated micropunch because of serious wear loss of Co when the punching number is over 1525.

#### 3.3.2. Coated Micropunch

In the meantime, Figure 8 also expresses that the wear characteristics of the micropunches with CNTs coating are nearly the same as that without CNTs coating. The attractive result of the micropunches with CNTs coating is that the quasi-stable stage forwards and begins at 450 (label in red with the solid line). At the same time, the quasi-stable stage delays and ends at 1400. Moreover, the wear loss of uncoated and coated micropunches in the quasistable period is definitely attractive, especially for punching numbers from

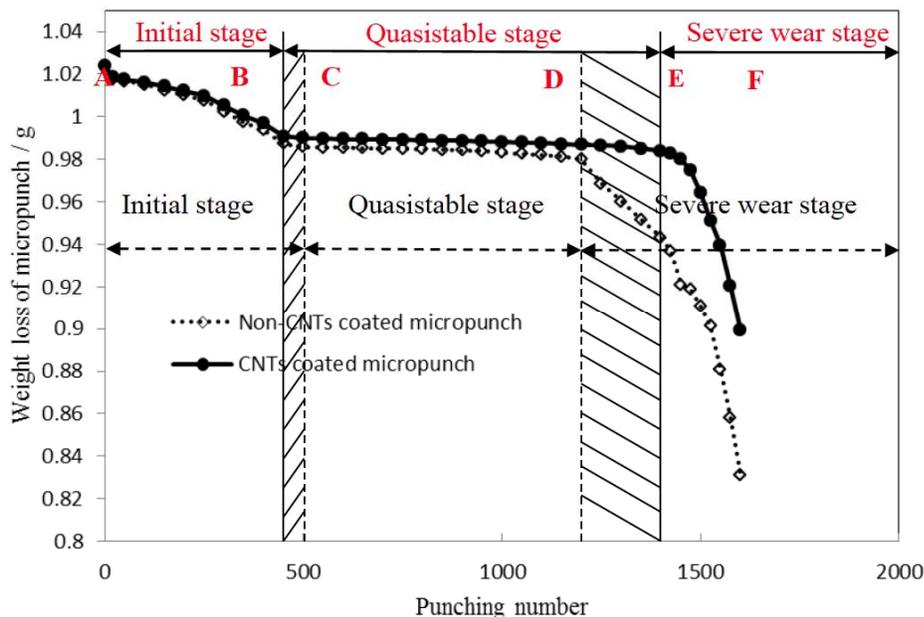
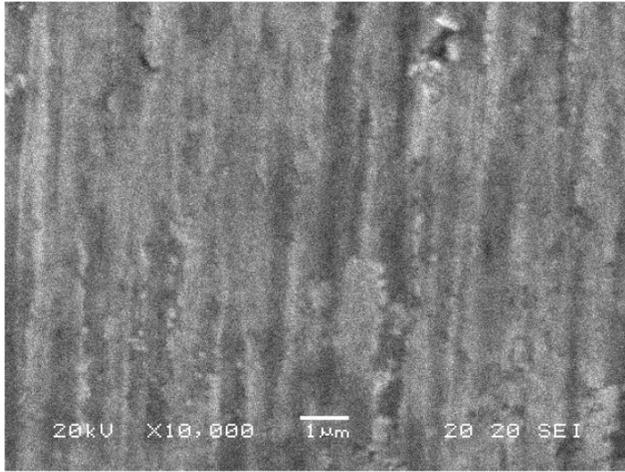
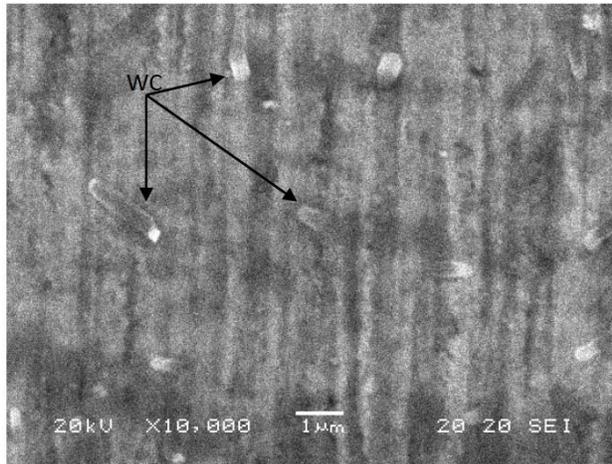


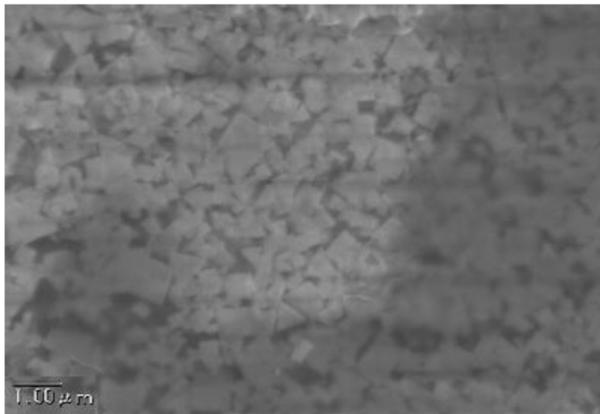
Figure 8: Weight loss of micropunches during punching.



**Figure 9:** Surface texture of the uncoated micropunch in the initial punching period (Punching number < 500).



**Figure 10:** Surface texture of the uncoated micropunch in the quasistable condition (Punching number: 500 - 1200).



**Figure 11:** Surface texture of the uncoated micropunch in the severe wear condition (Punching number over 1525).

500 to 1200 for uncoated micropunches and from 450 to 1400 for MWCNTs coated micropunches. In this period, the effect of MWCNTs on the wear loss of micropunch is obviously sound. In addition, the comparison between the micropunch with and without CNTs coating is affirmatively

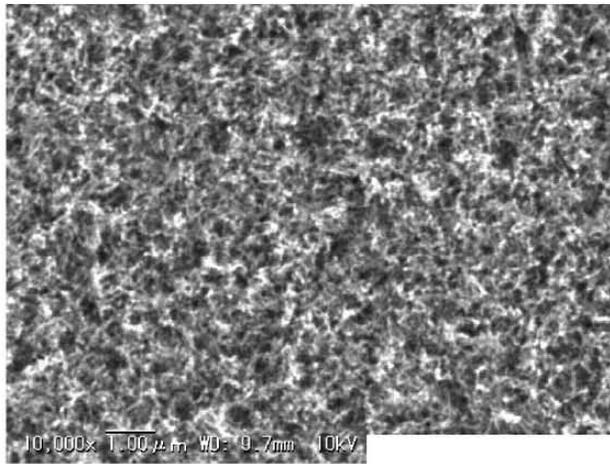
distinctive. For the severe wear stage, the wear loss of the micropunches (both with CNTs coating and without CNTs coating) is certainly obvious. When the punching number exceeds 1200 for the micropunches without CNTs coating, and the punching number exceeds 1400 for the micropunches with CNTs coating, the weight loss of the micropunches under both conditions are all serious, and the beneficial effect of the synthesized MWCNTs on the wear loss disappears, especially with the punching number increasing further as shown in Figure 8 labeled as 'F' area.

It should be noted that with the favorable effect of MWCNTs synthesized on the surface of micropunches, the start of the quasistable period is pushed forward, and the end of the quasistable period is postponed. It elucidates the effective quasistable period of the micropunches with CNTs coating is longer than that without CNTs coating on the surface of micropunches. For MWCNTs coated micropunches, it is  $1400 - 450 = 950$ . By comparison, for uncoated micropunches, it is  $1200 - 500 = 700$ . Furthermore, the total wear loss of MWCNTs coated micropunches is less than that of uncoated micropunches. It demonstrates that the life of a micropunch is improved or prolonged evidently.

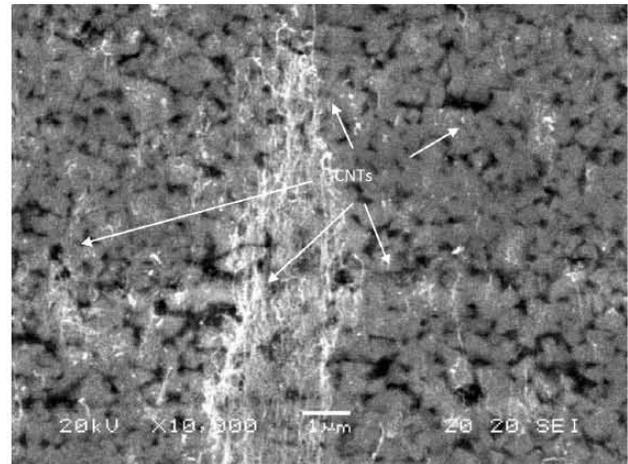
Figure 12 shows the surface textures of MWCNTs coated micropunches during various punching periods. Figure 12a shows the initial surface texture of MWCNTs coated micropunch (see point A in Figure 8). As seen from the top of the synthesized MWCNTs forest, MWCNTs densely populate, mutually tangle, and distribute uniformly on the surface of micropunch.

Figure 12b shows MWCNTs distribution in the run-in period of the micropunching (Point B in Figure 8). It can be seen that MWCNTs distribute non-uniformly on the surface of the micropunch, and under the rubbing effect of interaction between micropunch and substrate, a bundle or bulk of MWCNTs attaches on the micropunch surface. The microtextures of the micropunch surface in the quasistable period are shown in Figures 12c and 12d (Points C and D as shown in Figure 8). It shows that the uniformly distributed MWCNTs attached on the surface of micropunch resulted in forming a favorable transfer film as a lubricant layer between micropunch and substrate in the micropunching procedure. Moreover, owing to this transfer film formation at the interaction area under the effect of CNTs forest stripping, MWCNTs or the formed debris adheres to the surface of micropunch (or the mating surfaces), averting the direct contact between micropunch and substrate in the micropunching process and taking as the lubricant to the interface by virtue of their graphitic nature. The results shown in Figure 12 agree well with the results shown in Figure 8 and provide a promising prospect for future prolonging the life of micropunches in the following practical applications.

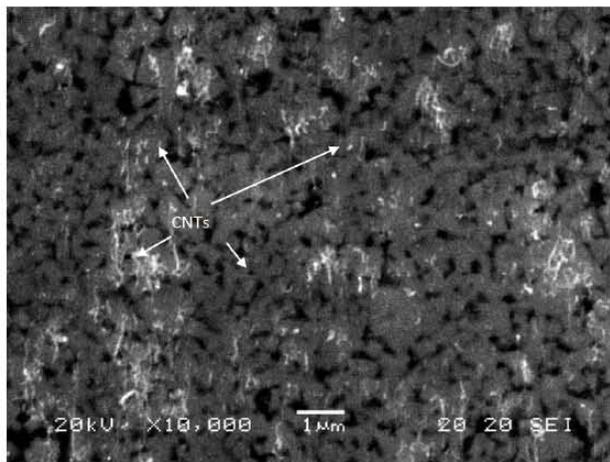
Figures 12e and 12f show the microtexture of the surface of micropunch (Points E and F as shown in Figure 8) when the punching number increases further. It illustrates that



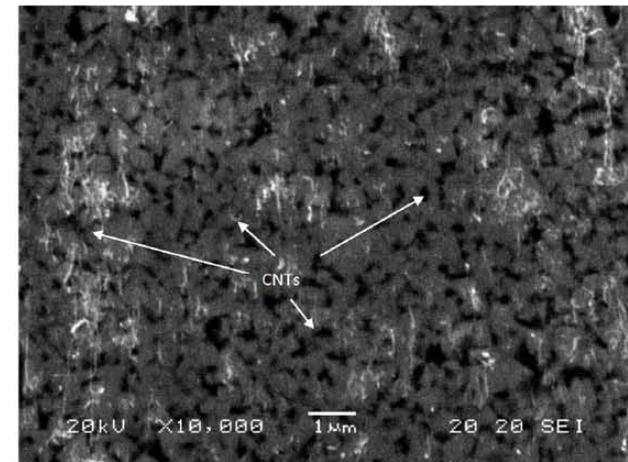
(a) Initial microtexture of MWCNTs coated on the surface of micropunch



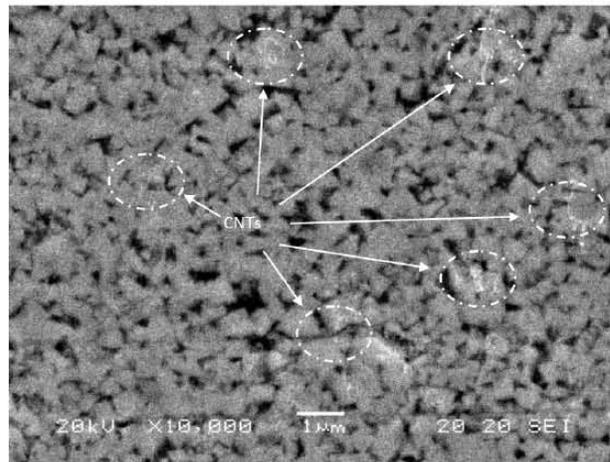
(b) Microtexture of MWCNTs coated on the surface of micropunch in the run-in stage



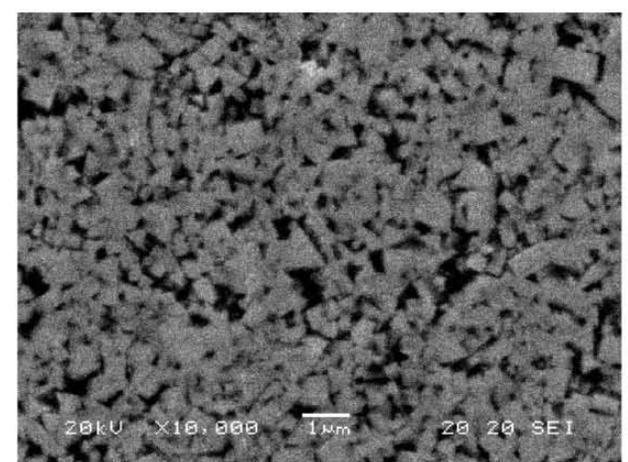
(c) Microtexture of MWCNTs coated on the surface of micropunch at the beginning of the quasistable stage



(d) Microtexture of MWCNTs coated on the surface of micropunch in the quasistable stage



(e) Microtexture of MWCNTs coated on the surface of micropunch in the severe wear stage



(f) Microtexture of MWCNTs coated on the surface of micropunch in the last severe wear stage

**Figure 12:** Microtexture of MWCNTs coated on micropunch in various conditions.

MWCNTs sparsely distribute and disappear (Figure 12f) at the last stage. It reveals that the beneficial effect of MWCNTs distributed initially on the surface of micropunch is lost. As a result, the severe wear of the micropunch appears.

### 3.4. Benefit of Micropunching

Microtechnology encompasses the technological approach directed to the miniaturization of components and systems, down to the micrometer scale. Major goals are integration

and increase in functionality of systems and devices while keeping the dimensions small. Microtechnological components, such as distributed holes, bear the potential to provide further functionality, for instance, in sensor technologies, and in biomedical applications. One prominent example is the lab-on-chip technology, where the analytical functionality of the chip is given by specific functionalized surfaces. Microsystems are going to play an important and critical role in application domains. The development of highly integrated microsystems necessitates the advancement of compatible assembly and joining techniques. Existing macro-level techniques are adapted for downsizing the assembly of hybrid microsystems. The ever-increasing demands for smaller, higher-quality, and lower-priced products from almost all fields of industry, household equipment, and entertainment electronics require the optimization of existing techniques and the development of new methods for the customized manufacture of microsystems with higher precision [21].

However, currently, microdevices are mainly made of silicone or glass and are fabricated using photo-resist techniques and/ or micro-machining and micro-molding techniques. It is well known that with these conventional technologies, the mechanical properties of substrate will be affected seriously. During the processing, when the source energy is input into the substrate, the substrate suffers heating, melting, vaporizing, and solidifying. Consequently, the properties of the substrate will be changed obviously, which cannot meet the requirements of application afterward. Meanwhile, chemicals taken in microfabrication are harmful to the environment. From the eco-friendly viewpoint, minimizing potential environmental and human health risks associated with the manufacture is crucial and urgent. Exploring new methods to lower the cost and produce in large quantities that are more environmentally friendly is the highlighted key issue. As an attractive promising technique, micropunch overcomes the above-mentioned drawbacks of the traditional technologies successfully. HAZ (heat-affected zone) is avoided, and chemicals are not employed. Meanwhile, the efficiency of microhole forming is improved remarkably. Moreover, the debris formed in the process of micropunching can be recycled. According to principles of green engineering, micropunching with CNTs is not only beneficial to microfabrication, such as ensuring that all material and energy inputs and outputs are as inherently safe and benign as possible; minimizing depletion of natural resources; striving to prevent waste; creating engineering solutions beyond current or dominant technologies; improve, innovate, and invent (technologies) to achieve sustainability, but also to encourage the replacement of existing technologies with new methods that are more environmentally friendly throughout their life cycles [22].

## CONCLUSION

1. A dense layer of packed and tangled MWCNTs was grown by the ecofriendly technique on the working

regions of WC/Co micropunches with the waste alcohol, and the height of the correlated synthesized MWCNTs is about 15  $\mu\text{m}$  with the diameter of about 3-5 nm.

2. In the micropunching process, the weight loss of MWCNTs coated micropunches could be divided into three periods: (i) the initial run-in stage, (ii) the quasi-stable stage, and (iii) the severe wear stage, just like the case of micropunches without MWCNTs coating. The micropunch was smoothed during the initial run-in stage. The presence of CNTs had little influence on the run-in. The weighting profile of the micropunch with and without CNTs essentially overlapped. Pieces of CNT were seen attached to the micropunch during the run-in stage and the quasi-stable stage.
3. At the end of the run-in stage and after the micropunch had been sufficiently smoothed, CNTs formed a protective layer and helped to extend the quasi-stable stage by about 250 shots (or 35% of the original length without CNT coating).
4. The wear of the micropunch coated with MWCNTs decreases obviously, even in the severe wear period. Moreover, the wear loss is definitely less than that of the micropunch without MWCNTs coating.

The promising results indicates that owing to the formation of a transfer film at the contact region by rubbing of the MWCNTs forest, MWCNTs adhere to the surface of micropunch, avoiding direct contact with the substrate and providing lubricant properties to the interface by virtue of their graphitic nature.

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