Friction and Wear Behaviors of Carbon Nanotube Reinforced Silica and Alumina Matrix Composites Fabricated by Catalyst Sol-Gel and CVD Process

Kuo-Jung Lee*1, Yu-Ting Yeh*2, Huy-Zu Cheng and Hsun-Yu Lin*1
Department of Materials Science and Engineering, I-SHOU University, Kaohsiung 84001, Taiwan

Fe(NO3)3/TEOS (tetrachlorosilane, Si(OCH3)4) and Fe(NO3)3/boehmite catalyst sol-gels combined with different chemical vapor deposition (CVD) growing/calcining processes were individually adopted to fabricate different carbon nanotube (CNT) composite powders and make different CNT/silica and CNT/alumina composites. Experimental results indicate that the Fe(NO3)3/TEOS and Fe(NO3)3/boehmite catalyst sol-gels with different CVD growing/calcining processes generate different products. Among those different products, CNTs could improve the tribological performance, stabilize the friction coefficient and reduce weight losses of specimens. Furthermore, ceramic products such as cristobalite, α-Al2O3 transformed from the Fe(NO3)3/TEOS and Fe(NO3)3/boehmite catalyst sol-gels at higher calcining temperatures could increase hardness and friction coefficient of specimens, however, they also result in more fluctuant friction coefficient curves and higher weight losses. The final products from different catalyst sol-gels via the two-step growing/calcining process could provide better mechanical and tribological properties of the specimens. The weight losses of CNT contained specimens with stable phase (α-Al2O3 or cristobalite) from different catalyst sol-gels via the two-step growing/calcining process are 1/4 to 1/2 of CNT free specimens and CNT contained specimens with metastable phase (γ-Al2O3 and amorphous SiO2) via the one-step growing/calcining process, respectively.

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1. Introduction

Due to the remarkable chemical and physical properties of carbon nanotubes (CNTs), they have been considered as potential reinforcing materials for composites1.2. CNTs could improve mechanical and tribological properties when they were added to a metallic3–6, polymeric7,8 or ceramic9–12 matrix. In comparison with other matrix based CNTs composites, relatively few studies of CNT/ceramic matrix based composites were reported, probably because of the difficulty in fabrication. However, the alumina–CNT composite was better studied among those CNTs/ceramic matrices based composites whose friction and wear properties were reported. The CNTs were concluded to act as the lubricant resulting in the improvement in tribological performance of the nanocomposites. The friction coefficient was found to decrease as the adding percentage of CNTs increases but to increase as the adding percentage exceeds a certain amount. This phenomenon clearly states that the lubrication resulting from the adding of CNTs is not the only contribution to the improvement in tribological performance of the alumina–CNT composites. The agglomeration of CNTs and the weak interface bonding between CNTs and the alumina matrix also needs to be considered for mechanical properties of composites weakening.

There have been a variety of techniques developed for CNT synthesis. The chemical vapor deposition (CVD) process is one very popular and low cost method for growing CNTs, adopts transition metal particles as the reaction catalyst to reduce the growth temperature. The transition metal catalysts were often found to be incorporated into the silica or alumina framework substrate to synthesize CNTx16–19.

Silica and alumina are known to be the most widely used ceramic materials and have unique property in lubrication and tribology, such as anti-wear, reducing friction, and high load capacity20–22. Many methods are available for silica and alumina synthesis. Among these developed methods, the sol-gel method provides a relatively lower temperatures synthesis process compared to other conventional methods23–25. For sol-gel methods, boehmite (AlOOH) and tetrachlorosilane (TEOS, Si(OCH3)4) are often used as precursors for alumina and silica, respectively.

In authors’ part work26, Fe(NO3)3/TEOS/boehmite catalyst sol-gel had been adopted to fabricate CNT/alumino-silicate powders through different CVD growing/calcining processes. In the past study, we used one catalyst sol-gel and focused on studying the effect of different CVD growing/calcining processes on products. In this study, in order to further investigate the influence of catalyst sol-gel, we prepared the Fe(NO3)3/TEOS and Fe(NO3)3/boehmite catalyst sol-gels which are different from the one in our previous study to make CNT/silica and CNT/alumina composites. The performances of these composites in present study and Ref. 26 were also compared and investigated.

2. Experimental Procedures

2.1 Catalyst sol-gel preparation

Two different catalyst sol-gels: Fe(NO3)3/TEOS and Fe(NO3)3/boehmite had been prepared individually in this study. The Fe(NO3)3/TEOS catalyst sol-gel was prepared by employing two procedures. In procedure 1, 2 M aqueous solution of Fe(NO3)3, was mixed with TEOS and absolute ethanol corresponding to the constant volume ratios 1.24:1:1 and stirred for 45 min. Few drops of concentrated HF solution were added to the mixture and stirred for another 45 min, to achieve slow gelation. The Fe(NO3)3/boehmite catalyst sol-gel was prepared by mixing 2 M aqueous solution of Fe(NO3)3, with boehmite and absolute ethanol corre-
sponding to the constant volume ratios 1.24:1:1 and stirred for 45 min.

2.2 CNT composite powder growth

Prior to powder growth, every catalyst sol-gel mixture was air dried at room temperature for 168 hours and dried stepwise in the oven at 70°C for 24 hours to remove excess solvent. After then, these dried mixtures were ground into catalyst powders.

CNT composite powder growth was conducted in a simple CVD setup made of a tube furnace and gas flow control units. Each catalyst powder was placed in a graphite boat before sending into a horizontal alumina tube reactor. The catalyst powder was first calcined in the vacuum atmosphere at 450°C for 2 hours. After then the catalyst powder was chemically reduced at 550°C with a H2/N2 (10/100 sccm) atmosphere for 2 hours. Two different CVD growing/calcining processes were designed specifically to fabricate given CNT composite powders. For the one-step reaction (continuous) process, the catalyst powder was finally calcined at 750°C (or 1300°C) for 30 min by introducing a mixture of C2H2/N2 into the reactor at a flow ratio of 30/70 sccm. Whereas, the two-step reaction process, the catalyst powder was firstly calcined at 750°C for 30 min by introducing a mixture of C2H2/N2 into the reactor at flow ratio of 30/70 sccm, then the temperature was increased to 1300°C and kept at 1300°C for 30 min by introducing nitrogen at 100 sccm. After the growing/calcining process, the nitrogen was fed into the reactor to purge the acetylene from the tube and prevent back flow of air into the tube. The furnace was then allowed to cool to room temperature before exposing the CNT composite powders to the air.

2.3 Sample preparation

This work first applied the chemical vapor deposition (CVD) with different parameters to grow different CNT composite powders. The as-grown CNT composite powders were ground and mixed with phenolic resin powders with ratio 80/20 in weight percentage before being press-molded. The press-molded procedures were conducted with a heating rate of 3°C/min to 180°C under a unidirectional pressure of 100 MPa for 30 min to make round disc-shaped specimens of 25.4 mm in diameter and 10 mm in thickness. As shown in Table 1, the specimens made by CNT composite powders prepared through different catalyst sol-gels: Fe(NO3)3/TEOS (designated “T”) and Fe(NO3)3/boehmite (designated “B”) and different CVD growing/calcining processes: one-step reaction process at 750°C (designated “1”) or 1300°C (designated “2”), and two-step reaction process at 750°C to 1300°C (designated “3”) were designated as T-1, T-2, T-3, B-1, B-2 and B-3 individually.

2.4 Characterization of different CNT composite powders

X-ray diffractometer (Scintag, X-4000, USA) with Ni-filtered Cu-Kα radiation was operated at 30 kV and 20 mA.

<table>
<thead>
<tr>
<th>Designation</th>
<th>Process parameters</th>
<th>Catalyst sol-gel powders</th>
<th>CVD growing/calcining processes</th>
</tr>
</thead>
<tbody>
<tr>
<td>T-1</td>
<td>One-step reaction process, the catalyst powder was calcined at 750°C</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T-2</td>
<td>One-step reaction process, the catalyst powder was calcined at 1300°C</td>
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<tr>
<td>T-3</td>
<td>Two-step reaction process, the catalyst powder was firstly calcined at 750°C and finally calcined at 1300°C</td>
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<tr>
<td>B-1</td>
<td>One-step reaction process, the catalyst powder was calcined at 750°C</td>
<td></td>
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</tr>
<tr>
<td>B-2</td>
<td>One-step reaction process, the catalyst powder was calcined at 1300°C</td>
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<tr>
<td>B-3</td>
<td>Two-step reaction process, the catalyst powder was firstly calcined at 750°C and finally calcined at 1300°C</td>
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</table>

![Fig. 1](image-url) Device for friction and wear tests.
Data were collected between 10 and 90° (2θ) in a scanning speed of 2°/min to identify the various phases of CNT composite powders prepared through Fe(NO₃)₃/TEOS and Fe(NO₃)₃/boehmite catalyst sol-gel adopted by different CVD growing/calcining processes. Those various phases were identified by matching each characteristic XRD peak with that compiled in JCPDS files.

The morphologies and microstructure of different CNT composite powders were observed using the Hitachi S-4700 (Tokyo, Japan) field emission scanning electron microscopy (FE-SEM) and electron dispersive spectroscopy (EDS). Some CNT composite products were also examined by FEI transmission electron spectroscopy (TEM, Tecnai G² 20 S-Twin, Holland). The TEM specimens were prepared by ultrasonically dispersing grinded the CNT composite powders in alcohol and then making several droplets on TEM copper grids. The samples were observed at various TEM power up to 200 kV.

### 2.5 Hardness Test

Hardness values of all specimens were determined at HRR (Rockwell, R scale) levels by a hardness test machine (Akashi, ATK-600, Japan). The used indenter was a 12.7 mm spherical steel ball, the applying load was 60 kgf and the testing position was along the radial direction of the specimen. At least, five positions were tested for each set of samples and the average values of hardness were measured.

#### Table 2 Comprehensive comparison of process parameters, products and properties in present study and Ref. 26).

<table>
<thead>
<tr>
<th>Composites</th>
<th>Present results</th>
<th>Ref. 26</th>
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</thead>
<tbody>
<tr>
<td><strong>Fe(NO₃)₃</strong></td>
<td>T-1 T-2 T-3 B-1 B-2 B-3 FTB-1 FTB-2 FTB-3</td>
<td></td>
</tr>
<tr>
<td><strong>TEOS</strong></td>
<td>× × × × × × × × ×</td>
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<tr>
<td><strong>Boehmite</strong></td>
<td>× × × × × × × × ×</td>
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<tr>
<td><strong>Catalyst Sol-gel Compositions</strong></td>
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<tr>
<td><strong>Calcination (°C)</strong></td>
<td>750 1300 750 + 1300 750 1300 750 + 1300 750 1300 750 + 1300</td>
<td></td>
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<tr>
<td><strong>Products</strong></td>
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<tr>
<td><strong>CNT (SEM)</strong></td>
<td>× × × × × × × × ×</td>
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<tr>
<td><strong>CNT (TEM)</strong></td>
<td>× × × × × × × × ×</td>
<td></td>
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<tr>
<td><strong>Fe₃C (XRD)</strong></td>
<td>× × × × × × × × ×</td>
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<tr>
<td><strong>Fe₃O₄ (XRD)</strong></td>
<td>× × × × × × × × ×</td>
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<tr>
<td><strong>Amorphous SiO₂ (TEM)</strong></td>
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<tr>
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<tr>
<td><strong>Al₂O₃ (TEM)</strong></td>
<td>× × × × × × × × ×</td>
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<tr>
<td><strong>α-Al₂O₃ (XRD)</strong></td>
<td>× × × × × × × × ×</td>
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<tr>
<td><strong>γ-Al₂O₃ (XRD)</strong></td>
<td>× × × × × × × × ×</td>
<td></td>
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<tr>
<td><strong>Fe₂AlO₄ (XRD)</strong></td>
<td>× × × × × × × × ×</td>
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<tr>
<td><strong>Mullite</strong></td>
<td>× × × × × × × × ×</td>
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<tr>
<td><strong>Properties</strong></td>
<td>HARD HARD HARD HARD HARD HARD HARD HARD</td>
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<tr>
<td><strong>Hardness (HRR)</strong></td>
<td>97.4 ± 5.9 113.5 ± 1.5 114.5 ± 1.0 112.0 ± 1.8 115.5 ± 0.4 117.4 ± 0.7 117.4 ± 0.7 122.0 ± 0.7 122.5 ± 0.7</td>
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<tr>
<td><strong>Coefficient of Friction (COF)</strong></td>
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<tr>
<td><strong>COF Curves</strong></td>
<td>S F I S F I S F I</td>
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<tr>
<td><strong>Average COF</strong></td>
<td>0.135 0.269 0.229 0.242 0.261 0.235 0.210 0.216 0.186</td>
<td></td>
</tr>
<tr>
<td><strong>Max. COF</strong></td>
<td>0.146 0.371 0.286 0.291 0.309 0.274 0.248 0.242 0.241</td>
<td></td>
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<tr>
<td><strong>Min. COF</strong></td>
<td>0.104 0.168 0.182 0.198 0.223 0.157 0.177 0.148 0.151</td>
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<tr>
<td><strong>Weight Loss (mg)</strong></td>
<td>13.5 ± 5.5 20.3 ± 4.7 9.5 ± 4.3 8.3 ± 4.3 18.5 ± 9.8 4.2 ± 2.1 17.8 ± 7.1 5.2 ± 0.8 3.7 ± 1.4</td>
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<tr>
<td><strong>Containing amount:</strong></td>
<td>○--high ×--medium △--low ×--none</td>
<td></td>
</tr>
<tr>
<td><strong>COF Curves:</strong></td>
<td>S--stable I--intermediate F--fluctuant</td>
<td></td>
</tr>
</tbody>
</table>

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2.6 Friction and wear tests

As shown in Fig. 1, friction and wear tests were conducted using a homemade disc-on-disc sliding wear tester. The square-shaped (32 mm × 32 mm) counterface material is SAE-G2500 gray cast iron (the rotor material). Prior to testing, the wear test specimens (included cast iron disc and CNT composites) were polished through a level of #1200 grit paper, followed by ultrasonic cleaning and drying. A fixed load of 1.2 MPa, constant rotor speed of 1000 rpm (linear speed is 0.67 m/s) and testing time of 300 sec (sliding distance is 200 m) were used in every wear test. A strain gage-equipped LRK-100K load cell (NTS Technology, Nara, Japan) was used to determine the friction coefficient. The weight loss was measured and averaged from the CNT composites for each run.

3. Results and Discussion

In order to precisely compare the relationships among process parameters, products and associated properties in present study and Ref. 26, we summarized comprehensive experimental data in Table 2.

3.1 XRD analysis

Figure 2 show the XRD patterns of different CNT composite powders prepared through different catalyst sol-gels and CVD growing/calcining processes. For the T-1 specimen, the spectrum gives weak and broad bands of Fe3C, Fe2SiO4, and Fe3O4. The XRD patterns of T-2 specimen show better degree of crystallization of SiO2, Fe3C, and Fe3O4. It is noted that SiO2 show higher peak intensities among all of products’ patterns. The SiO2, Fe3C, and Fe3O4 peaks were also existed in the XRD patterns of T-3 specimen. The major difference in XRD patterns of T-2 and T-3 specimens were peak intensities of SiO2 and Fe3C. The T-2 specimen shows higher SiO2 but lower Fe3C peak intensities, whereas the T-3 specimen shows higher Fe3C but lower SiO2 peak intensities. The above result reflected that more SiO2 products and less CNT were observed in T-2 specimen, and the converse results were observed in T-3 specimen in follow-up microstructure observation.

The XRD pattern of B-1 specimen show nearly amorphous broad bands of Fe3C, Fe2AlO4, Fe3O4 and minor indication of transitional γ-Al2O3 phases. As the growing/calcining temperature increases to 1300°C, all products’ peak intensities of B-2 specimen were enhanced and the stable α-Al2O3 crystalline phases was detected instead of the γ-Al2O3 phase. The XRD patterns of B-2 and B-3 specimens are approximately similar, except the α-Al2O3 and Fe3C peak intensities of B-2 and B-3 specimens. The B-2 specimen shows higher α-Al2O3 but lower Fe3C peak intensities, whereas the B-2 specimen shows higher Fe3C but lower α-Al2O3 peak intensities. These results reflected that more α-Al2O3 and less CNT products were observed in B-2 specimen, and the converse results were observed in B-3 specimen in follow-up microstructure observation.

3.2 Microstructure

The microstructures of CNT composite powders prepared through different catalyst sol-gels and CVD growing/calcining processes are shown in Figs. 3–6. In T-1 specimen, many entangled CNTs were found (Fig. 3(a)–(b)). These CNTs were identified to be multiwall nanotubes by TEM. The inner diameter of the CNTs is about 13 nm and outer diameter is about 33 nm (Fig. 5(b)). The selected-area diffraction (SAD) patterns revealed that the basal planes of the CNTs were highly aligned to the tube axis. Some amorphous SiO2 particles were also observed by TEM.

For T-2 specimen, only some cristobalite and amorphous silica nanowires were observed in T-2 specimen (Fig. 3(c)–(d)). The diameter of these silica nanowires were about 25 nm (Fig. 5(e)). It should be noted that CNTs were seldom observed in the T-2 specimen, which is consistent with the results from XRD pattern. It is possible that the reaction temperature at 1300°C for one-step reaction (continuous) process is too high to synthesize CNTs. At 1300°C, the elemental carbon decomposed from C2H2 is hard to precipitate from Fe3C to form CNTs because most of the precursor has been transform into liquid iron instead of Fe3C. On the contrary, the iron atom became as the catalyst for silica nanowires synthesis. A more reasonable explanation for higher XRD intensity of SiO2 detected by XRD analysis (Fig. 2) is that the cristobalite, a high-temperature polymorph of silica, was produced from the silica sources at 1300°C (Fig. 5(f)).

It is different from T-2 specimen, CNTs can be observed in the T-3 specimen. (Fig. 3 (f) and Fig. 5(g)). During the two-step reaction process, CNTs were synthesized firstly at the first step (750°C), and silica components in Fe(NO3)3/TEOS catalyst sol-gel were transformed into silica nanowires.
ires and cristobalite at the second step. It seems that the two-step reaction process is beneficial to synthesize CNTs, silica nanowires and cristobalite at the same time for T-3 specimen.

To compare with the T-1 specimen, better quality and quantity of CNTs were found in B-1 specimen (Fig. 4(a)–(b)). According to an earlier study, better quality and quantity of multiwall carbon nanotubes could be produced when alumina instead of silica supported metal catalysts was used. TEM showed that the inner diameter of the multiwall CNTs is about 11 nm and outer diameter is about 22 nm (Fig. 6(b)). Some $\gamma$-Al$_2$O$_3$ particles were produced from the alumina sources at 750°C were also observed by TEM (Fig. 6(c)).

In common with the T-2 specimen, CNTs were also seldom observed in the B-2 specimen (Fig. 4(c)–(d)). The $\alpha$-Al$_2$O$_3$, the most stable phase of alumina induced by heat treatment of boehmite at 1300°C could be observed (Fig. 6(d)–(e)). These observations justified that higher $\alpha$-Al$_2$O$_3$ but lower Fe$_3$C peak intensities in XRD analysis (Fig. 2).

CNTs and $\alpha$-Al$_2$O$_3$ can be observed simultaneously in the B-3 specimen (Fig. 4 (e)–(f) and Fig. 6(f)–(h)). Based on the observation of XRD and microstructure results, we make rational conjectures that CNTs and $\gamma$-Al$_2$O$_3$ were synthesized firstly at 750°C, and $\gamma$-Al$_2$O$_3$ would be further transformed into $\alpha$-Al$_2$O$_3$ at 1300°C for Fe(NO$_3$)$_3$/TEOS specimen calcined by the two-step reaction process.

### 3.3 Hardness

The average hardness of each specimen with different CNT composite powders was shown in Fig. 7. It appeared that the series of Fe(NO$_3$)$_3$/boehmite specimens had higher hardness than that of Fe(NO$_3$)$_3$/TEOS specimens. This phenomenon could be realized that the reinforced effect of alumina phases derived from boehmite is stronger than that of silica phases derived from TEOS. As shown in Table 2, in comparison with the hardness of amorphous SiO$_2$ (T-1), specimens contained $\alpha$-Al$_2$O$_3$ or cristobalite SiO$_2$ synthesized at higher growing/calcining temperatures apparently seemed to be much tougher. This measured result can be attributed to high crystalline alumina or silica phases synthesized at higher growing/calcining temperatures (Figs. 5–6). These crystalline phases supposedly provide more resistant to applied load than amorphous phases. It is noteworthy that
the T-3 and B-3 specimens with CNT composite powders prepared through the two-step reaction process shows the highest hardness among its series. This result may thus be attributed to the high crystalline phases of products as identified by XRD and TEM such as CNTs, cristobalite, \( \alpha\)-Al\(_2\)O\(_3\) which were derived from the Fe(NO\(_3\))\(_3\)/TEOS and Fe(NO\(_3\))\(_3\)/boehmite catalyst sol-gel through the two-step reaction process.

3.4 Friction and wear

Typical friction coefficient curves and weight losses of specimens with different CNT composite powders through different CVD growing/calcining processes are shown in Figs. 8–9. Among those specimens during the wear test, T-2 and B-2 specimens shows higher and more fluctuant friction coefficient curves. It also shows that the weight losses of T-2 and B-2 specimens are roughly related to their friction coefficient curves. As shown in Fig. 9, weight losses of T-2 and B-2 specimens are more fluctuant and much higher than all of specimens. A possible explanation for these results is that only the ceramic products such as cristobalite or \( \alpha\)-Al\(_2\)O\(_3\) were observed but CNTs were seldom observed in SEM and TEM microstructures of T-2 and B-2 specimens. Although these hard ceramic products could increase friction coefficient of specimen, they were too hard to be formed lubricating films on the worn surface and accompanied with fluctuant friction coefficient curves during the wear test.

Different from T-2 and B-2 specimens, the friction coefficient curves of the T-1 and B-1 specimens were more stable. The T-1 specimen with the lowest hardness had the lowest and the most stable friction coefficient curve. According to the reported literature\(^{27}\), this phenomenon could be attributed to the formation of CNT-derived transferred film made up of amorphous carbon between the sliding couples. The CNT-derived amorphous carbon lubricating film acts as a solid lubricant and improves the tribological performance and stabilizes the friction coefficient of specimens. Since \( \gamma\)-Al\(_2\)O\(_3\) particles and CNTs had been synthesized together, the B-1 specimen showed an intermediate but still stable friction coefficient curve.

T-3 and B-3 specimens prepared through the two-step reaction process also show stable and intermediate friction coefficient curves. Significantly, even though the friction coefficient curves of T-3 and B-3 specimens were above the
Fig. 5 TEM microstructures and diffraction (SAD) patterns of different CNT composite powders prepared through different CVD growing/calcining (a)–(c) T-1, (d)–(f) T-2, (g)–(i) T-3.
middle high, weight losses of these two specimens were lower. The B-3 specimen even had the lowest weight losses. The coexistence of CNTs, cristobalite, $\alpha$-Al$_2$O$_3$ in T-3 and B-3 specimens as identified by XRD and TEM apparently provided the best explanation for low weight losses and stable friction coefficient curves. CNTs could provide lubricating effect and cristobalite, $\alpha$-Al$_2$O$_3$ increase friction coefficient of these specimens individually.

Fig. 6  TEM microstructures and diffraction (SAD) patterns of different CNT composite powders prepared through different CVD growing/calcining processes (a)–(c) B-1, (d)–(e) B-2, (f)–(h) B-3.
As shown in Table 2, we can find the weight losses of CNT contained specimens (B-3 & T-3) are 1/4 and 1/2 of CNT free specimens (B-2 & T-2), respectively. More noteworthy is the weight losses of CNT contained specimens (B-3 and T-3) with stable phase (\(\alpha\)-Al\(_2\)O\(_3\) and cristobalite) prepared via the two-step growing/calcining process are also 1/2 and 1/3 smaller than that of other CNT contained specimens (B-1 and T-1) with metastable phase (\(\gamma\)-Al\(_2\)O\(_3\) and amorphous SiO\(_2\)) prepared via the one-step growing/calcining process, respectively.

As shown in Table 2, except for the T1 specimen, the hardness of other specimens is very close (HRR 112–117). Therefore, we can’t find the precise relationship among hardness, friction coefficient and weight losses. It seems that products prepared via different catalyst sol-gels and growing/calcining processes have significant effects on the friction coefficient and weight losses. Especially, the presence of CNTs has a significant effect on the stability of friction coefficient curves and the reduction of weight losses of the specimens. CNTs are able to improve the tribological performance, stabilize the friction coefficient and reduce weight losses of specimens. Furthermore, those ceramic products such as cristobalite, \(\alpha\)-Al\(_2\)O\(_3\) transformed from the Fe(NO\(_3\))\(_3\)/TEOS and Fe(NO\(_3\))\(_3\)/boehmite catalyst sol-gels at higher calcining temperatures allow the increases in hardness and friction coefficient of specimens, however, they also result in more fluctuant friction coefficient curves and higher weight losses.

The presence of CNTs has the same effect in authors’ part work\(^{26}\). The stable friction curves of FTB specimens were also observed in Ref. 26. As shown in Table 2, the B-3 specimen obtained as well as FTB-2 and FTB-3 specimens in Ref. 26) show to have high hardness and low weight loss. However, the friction coefficient of B-3 specimen (0.24) is higher than those (0.17, 0.19) of FTB-2 and FTB-3 specimens. The B-3 specimen with high hardness, high friction coefficient and low weight loss has potential to be utilized for sheet transfer roll.

4. Conclusion

(1) Different CNT composite powders were synthesized through Fe(NO\(_3\))\(_3\)/TEOS and Fe(NO\(_3\))\(_3\)/boehmite catalyst sol-gels with different CVD growing/calcining processes. For the Fe(NO\(_3\))\(_3\)/TEOS catalyst sol-gel, the products synthesized by adopting the one-step reaction process at 750°C were CNTs and amorphous SiO\(_2\). By adopting the one-step reaction process at 1300°C, the products were silica nanowires, cristobalite. In addition, the two-step reaction process could synthesize CNTs, silica nanowires and cristobalite.

(2) For the Fe(NO\(_3\))\(_3\)/boehmite catalyst sol-gel, by adopting the one-step reaction process at 750°C, the products of were CNTs and \(\gamma\)-Al\(_2\)O\(_3\). By adopting the one-step reaction process at 1300°C, the products were \(\alpha\)-Al\(_2\)O\(_3\). Furthermore, the two-step reaction process could synthesize CNTs, \(\alpha\)-Al\(_2\)O\(_3\).

(3) Results from friction coefficient measurements shows that the specimen of CNT reinforced amorphous SiO\(_2\) (T-1) is found to have the lowest and the most stable friction coefficient curve. Although the hardness of amorphous SiO\(_2\) contained specimen (T-1) is not particularly high, either \(\alpha\)-Al\(_2\)O\(_3\) or cristobalite SiO\(_2\) synthesized at higher growing/calcining temperatures apparently contribute the improvement of the hardness value.

(4) Among all of products, CNTs could improve the tribological performance, stabilize the friction coefficient and reduce weight losses of specimens. Ceramic products
such as cristobalite, α-Al₂O₃ transformed from different catalyst sol-gels at higher calcining temperatures could increase hardness and friction coefficient of specimens, but also result in more fluctuant friction coefficient curves and higher weight losses.

(5) The final products from different catalyst sol-gels via the two-step growing/calcining process could provide better mechanical and tribological properties of the specimens. The weight losses of CNT contained specimens with stable phase (α-Al₂O₃ or cristobalite) from different catalyst sol-gels via the two-step growing/calcining process are 1/4 and 1/2 of CNT free specimens and the other CNT contained specimens with metastable phase (γ-Al₂O₃ and amorphous SiO₂) via the one-step growing/calcining process, respectively.

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