Formation Process of Ti Coatings in Mechanical Coating with Different Rotation Speed

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\textbf{Abstract}: Ti coatings on alumina (Al\textsubscript{2}O\textsubscript{3}) balls had been prepared by mechanical coating technique (MCT), and the influence of the rotation speed on the coating formation process were investigated. The crystal structure and microstructure of the Ti coatings were investigated by X-ray diffraction (XRD) and scanning electron microscopy (SEM). The formation process of Ti coatings was examined and discussed. The results showed that the rotation speed had great influence on the coating formation process. With increasing the rotation speed, the speed of coating formation had been accelerated and the surface became to be uneven. It was considered that the formation of metal coatings consists of 4 stages: nucleation, formation and coalescence of the discrete islands, formation of the continuous coating, and exfoliation.

\textbf{Introduction}

Ball milling is widely used to fabricate the materials with a variety of equilibrium and non-equilibrium phases that are difficult or impossible to be obtained by traditional techniques [1]. Mechanical coating technique (MCT) as a novel coating formation process was proposed and developed based on mechanical frictional wear and impacts among Al\textsubscript{2}O\textsubscript{3} balls, columns, disks and particles of metal powders in the bowl of planetary ball mill or pot mill as shown in Fig. 1 [2]. Metal coatings of Ti, Cu, Ni, Fe and Zn on Al\textsubscript{2}O\textsubscript{3} balls could be easier prepared by MCT, compared with traditional techniques [3]. MCT is a simple, low cost and useful coating formation process and can form metal and composite coatings on ceramic balls as the substrates. MCT has been applied to prepare TiO\textsubscript{2} composite photocatalyst coatings [3-5].

In this work, the formation process of Ti coatings by MCT was investigated experimentally. The crystal structure and microstructure of the coatings were investigated. The relationship between the rotation speed and the coating formation process was discussed.
Experimental

Ti powder (purity of 99.1%, average diameter of 30 μm) was used as the coating material. Al₂O₃ balls (purity of 93%, average diameter of 1 mm) were used as the substrates. 40 g Ti powder and 60 g Al₂O₃ balls were charged to an alumina bowl with dimensions of ∅75 mm × 70 mm and a volume of 250 ml. A planetary ball mill (Type: P6, Fritsch, Germany) was employed to perform the mechanical coating operation. The rotation speed of the planetary ball mill was set from 200 to 480 rpm for a series of milling time, shown in Table 1, with a 10-min milling operation and a following 2-min cooling interval to prevent the bowl from overheating.

The prepared samples were labeled as follows. "TAx-yh" is the sample fabricated with Ti powder by MCT at x rpm for y h. XRD analyzer (JDX-3530, JEOL, Japan) with Cu-Kα radiation at 30 kV and 20 mA was used to determine the compositions and crystal structures. The surface morphologies and cross-sectional microstructures of the samples were observed by SEM (JSM-5300, JEOL, Japan).

Table 1 Experimental conditions for fabricating Ti coatings by MCT.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Rotation speed, x (rpm)</th>
<th>MCT time, y (h)</th>
<th>Collision power, (×10⁹ J·m⁻²·s⁻¹)</th>
<th>φ (Hz)</th>
<th>Collision strength, (×10⁹ N·m⁻²·s⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TA200-yh</td>
<td>200</td>
<td>12, 26, 32, 40, 60</td>
<td>13.59</td>
<td>14.09</td>
<td>61.57</td>
</tr>
<tr>
<td>TA300-yh</td>
<td>300</td>
<td>4, 8, 12, 16, 20, 26, 32, 40</td>
<td>23.91</td>
<td>21.15</td>
<td>108.50</td>
</tr>
<tr>
<td>TA400-yh</td>
<td>400</td>
<td>4, 8, 12, 16</td>
<td>35.83</td>
<td>28.21</td>
<td>162.49</td>
</tr>
<tr>
<td>TA480-yh</td>
<td>480</td>
<td>4, 8, 12, 16</td>
<td>46.33</td>
<td>33.84</td>
<td>210.15</td>
</tr>
</tbody>
</table>

Fig. 1 Schematic diagram of fabricating metal films by MCT.

Fig. 2 Photographs of Ti/Al₂O₃ balls.

Fig. 3 XRD patterns of samples.
Results and discussion

The samples of TAx-ylene after MCT show metallic color, shown in Fig. 2. At relatively lower rotation speed, the surface is smooth and the speed of coating formation is slow. With increasing the rotation speed, the speed of coating formation had been accelerated and the surface became to be uneven, which had been caused by higher collision power, frequency and strength, shown in Table 1 [7].

Fig. 3 shows the XRD patterns of the samples. At 200 rpm, the diffraction peaks of Ti are clearly detected after 26 h, while at 480 rpm, the peaks of Ti could be detected only after 4 h. The influence of rotation speed on coating formation is match to the appearance of samples.

The SEM images of TAx-ylene samples are shown in Fig. 4. With increasing the MCT time, the coatings had grown at each rotation speed, which hints that the required time for preparing Ti coatings is shortened, under the

Fig. 4 SEM images of the cross-sections for the samples.

(a) TA200-12h  (b) TA200-26h
200μm  50μm
(c) TA200-60h

(d) TA300-8h  (e) TA300-12h  (f) TA300-20h

(g) TA400-4h  (h) TA400-8h  (i) TA400-20h

(j) TA480-4h  (k) TA480-12h  (l) TA480-16h

Fig. 5 SEM images of the surfaces for the samples.

(a) TA200-12h  (b) TA200-26h  (c) TA200-60h
200μm

(d) TA300-8h  (e) TA300-12h  (f) TA300-20h

(g) TA400-4h  (h) TA400-8h  (i) TA400-20h

(j) TA480-4h  (k) TA480-12h  (l) TA480-16h

Fig. 6 Schematic diagram of formation process and SEM images of surfaces for the Ti coatings by MCT.
higher rotation speed by MCT. While at relatively higher speed, the surface of coatings becomes to be uneven, and Ti coatings finally exfoliated from Al₂O₃ balls, when the rotation speed up to 480 rpm with 16 h, shown as Fig. 4(i).

Compared with the cross-sections of samples under different rotation speed, shown as Fig. 5, the metal coatings become to be uneven, thicken and formed crack, even with shorter time under relatively higher rotation speed. According to the above results, the evolution model of coating formation process had been proposed, which consists of 4 stages: nucleation, formation and coalescence of the discrete island, formation of the continuous coating, and exfoliation, shown as Fig. 6 [8].

Fig. 7 shows the area ratio during the 4 stages of coating formation process by MCT. At the stage of nucleation (I-stage), the surface coverage hardly changes as the nuclei are too small to be detected. With increasing the MCT time, the discrete islands of metal particles are formed, then larger metal particles coat on the surface and hence the coverage greatly increases (II-stage). When the continuous metal coatings are prepared, and the coverage reaches to 100% (III-stage). During III-stage, the thickness of coatings is quickly increased. Finally, the coverage will be decreased due to the exfoliation of metal coatings(IV-stage).

Conclusions

The Ti coatings had been successfully fabricated on Al₂O₃ balls by MCT. It was found that the rotation speed had great influence on the coating formation process. With increasing the rotation speed, the speed of coating formation had been significantly accelerated and the surface became to be uneven. The formation process of metal coatings was considered to consist of 4 stages: nucleation, formation and coalescence of discrete island, formation of continuous coating, and exfoliation. With the higher rotation speed, the required time for preparing metal coatings would be shortened.

References