Evaluating Adhesion of Ceramic Coatings by Scratch Testing

Kunming Li\textsuperscript{1, a}, Yiwang Bao\textsuperscript{1, b}, Detian Wan\textsuperscript{1, c}, Yanli Huo\textsuperscript{2, d}, Jinyi Wu\textsuperscript{3, e}

\textsuperscript{1}Key State Lab of Green Building Materials, China Building Materials Academy, Beijing 100024, China
\textsuperscript{2}Ceramic Research Institute, China Building Materials Academy, Beijing 100024, China
\textsuperscript{3}Materials and Chemical Engineering College, Hainan University, Haikou 570228, China
\textsuperscript{a}inonelife@yahoo.com.cn, \textsuperscript{b}byw@ctc.ac.cn, \textsuperscript{c}dtwan@ctc.ac.cn, \textsuperscript{d}huoyanli@cbmamail.com.cn, \textsuperscript{e}wujinyi1976@yahoo.com.cn

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Abstract. Adhesion is one of the most important mechanical properties of ceramic coatings. Scratch testing is considered as a simple and effective method to evaluate adhesion of ceramic coatings. In this paper, the critical normal forces and scratch morphologies for different coating-substrate systems were studied by scratch testing. It is shown that the critical normal force obtained by acoustic emission (AE) signals decreases from 12 N to 7 N when the applied normal force rate increases from 20 N/min to 100 N/min for deposited SiC coating on C by CVD, and the failure area of this scratched sample increases with increasing maximum normal force. Based on scratch morphologies, spallation or delamination can be observed for hard-brittle coatings on glass or metal, while discontinuous or continuous ductile perforation can be observed for ductile coatings on metal. The critical normal force for hard-brittle coatings can be effectively obtained by AE signals.

Introduction

There has been increased interest in the use of ceramic coatings on aerospace instrument, cutting tools and electronic devices. These coatings are mainly to increase substrate performance and mechanical properties, such as hardness, strength and wear resistance. However, it is indicated that in many cases the coating-substrate interface represents the weakest part of the coated component, which constrains their applications [1, 2]. Therefore, it is necessary to evaluate the adhesion between ceramic coating and substrate. There are a number of adhesion test methods, such as pull-off test [3], adhesive tape test [4], interfacial indentation test [5] and scratch test [6-8]. The scratch test is a simple and effective method to assess adhesion of ceramic coatings, which is also used in this paper.

The scratch test method consists of drawing a stylus of defined shape (usually a diamond with Rockwell C geometry) over the surface of the coating-substrate system to be tested, either under a constant or progressive normal force [8]. In order to detect the failure event, resultant scratches are then observed by an optical or scanning electron microscope and sometimes by using acoustic emission (AE) measurement. The adhesion of the coating is characterized by the critical normal force, \( L_c \), which is the minimal normal force at which failure occurs [6].

The critical normal forces at which the failure events appear depend not only on the adhesion of the coating but also on other parameters, such as rate of increase of normal force and traverse speed [8]. The aim of this paper is to investigate the effect of rate of increase of normal force on microscopic scratches and on the critical normal force which can be obtained by acoustic emission signals, to find the differences of acoustic emission signals and microscopic scratches for different coating-substrate system under the same test condition, and to detect for which kind of ceramic coating the critical normal force can be attained by acoustic emission signals.
Experimental

In order to fix the specimen on the sample stage of scratch tester, firstly coating-substrate system should be cut to the size of 20×20×20 mm$^3$ using cutting machine if the coating-substrate system is larger than this size. The minimal size of the coating-substrate system was 10×10×10 mm$^3$. The cross section of coated sample should be polished by polishing machine if needed to observe it. The scratch tests on the samples were performed by a scratch tester (WS-2006, Lanzhou Zhongkekeaihua technology development Co., Ltd., China) at room temperature. The relative humidity should be between 40% and 60%. A Rockwell C diamond stylus with radius 0.2 mm and cone angle 120 degree was used to carry out the scratch tests. The normal force was progressively varied from 0 to 100 N over a scratch length of 4 mm with speed 4 mm/min and normal force rate from 20 N/min to 100 N/min. Each scratch test is finished with 1 min. The AE signals can be collected by the scratch tester automatically. Then, the scratch morphologies can be observed by an optical digital microscope (VHX-600E, KEYENCE Co., Ltd., Japan).

![Silicon carbide coating](image1)

Fig. 1 Cross section of CVD SiC coating on C

![Typical AE curve](image2)

Fig. 2 Typical AE curve, $L_c$: 11 N

Table 1 Critical normal force for CVD SiC on C under different rates of increase of normal force and corresponding maximum normal forces

<table>
<thead>
<tr>
<th>Rate of increase of normal force (N/min)</th>
<th>20</th>
<th>40</th>
<th>60</th>
<th>80</th>
<th>100</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximum normal force (N)</td>
<td>20</td>
<td>40</td>
<td>60</td>
<td>80</td>
<td>100</td>
</tr>
<tr>
<td>Critical normal force, $L_c$, (N)</td>
<td>12</td>
<td>11</td>
<td>10</td>
<td>8</td>
<td>7</td>
</tr>
</tbody>
</table>

Results and Discussion

To investigate the effect of rate of increase of normal force on the critical normal force and on microscopic scratches, SiC coating on Carbon (C) by chemical vapor deposition (CVD) was chosen as the specimen (which is short for CVD SiC on C) because it is a typical coating-substrate system. Fig. 1 shows a cross section of CVD SiC on C. The SiC coating thickness is about 65 µm. As shown in Fig. 1, there are many holes in the carbon substrate, but SiC coating is very compact and the coating-substrate interface is clear, indicating the coating-substrate bond is very strong.

As for CVD SiC on C, the normal force rate is chosen from 20 N/min to 100 N/min (20, 40, 60, 80 and 100 N/min, respectively) and corresponding maximum normal force is from 20 N to 100 N (20, 40, 60, 80 and 100 N, respectively). Fig. 2 shows a typical AE curve for CVD SiC on C. The rate of increase of normal force is 40 N/min and the maximum normal force is 40 N. As it is shown in this curve, the AE signals abruptly strengthen when the normal force is about 11 N, indicating that first failure event occurs on the surface at this time. The normal force at this time can be called critical normal force ($L_c$). Table 1 shows critical normal force for CVD SiC on C under different rates of increase of normal force. As the table presents, when the rate of normal force increase from 20 N/min to 100 N/min, the corresponding critical normal force decreases from 12 N to 7 N, respectively. It is
indicated that the rate of increase of normal force is an important effect parameters on the critical normal force. Therefore, during each scratch experiment, the rate of increase of normal force should be clarified. Fig. 3 shows scratch morphologies for CVD SiC on C under different rates of increase of normal force and corresponding maximum normal forces. The rate of increase of normal force from (a) to (e) is 20 N/min to 100 N/min, respectively. Except for some surface defect, it is shown that the failure area of this scratched specimen increases with increasing maximum normal force. There is not any spillage or delamination on these surfaces, indicating that the adhesion of the coating is very high.

![Scratch morphologies for CVD SiC on C under different rates of increase of normal force and corresponding maximum normal forces](image)

Table 2 Critical normal force for different coating-substrate systems (normal force rate: 100 N/min)

<table>
<thead>
<tr>
<th>Coating-substrate system</th>
<th>PVD TiAlSiN on high speed steel</th>
<th>PVD AZO on glass</th>
<th>PVD ITO on glass</th>
<th>PVD FTO on glass</th>
<th>TS ZrO$_2$ on Al</th>
<th>TS ZrO$_2$ on steel</th>
<th>MAO TiO$_2$ on Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>Critical normal force (N)</td>
<td>12</td>
<td>10</td>
<td>12</td>
<td>22</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

On the other hand, the difference of AE signals and the difference of scratch morphologies for different coating-substrate systems were investigated under the test condition of normal force rate 100 N/min and corresponding maximum normal force 100 N. Fig. 4 shows scratch acoustic emission curves and scratch morphologies for different coating-substrate systems. As for PVD (physical vapor deposition) TiAlSiN on high speed steel, the critical normal force is 12 N (Fig. 4(a)), but there is not any AE signals when the applied normal force exceeds 60 N, which could be attributed to the detachment between TiAlSiN coating and high speed steel. Meanwhile, spallation phenomenon can be found from the scratch morphology (Fig. 4(a)). As for PVD AZO (Al-doped ZnO), ITO (Indium tin oxide) and FTO (SnO$_2$ doped fluorine) on glass, on the basis of AE curves (Fig. 4(b1-d1)), the corresponding critical normal forces are 10 N, 12 N, and 22 N, respectively. When the applied normal force reaches near to 100 N, delamination phenomenon can be seen from the scratch morphology (Fig. 4(b2-d2)). As for TS (thermal spraying) ZrO$_2$ on Al and steel, there are only several small AE signals or almost no AE signals, so the corresponding critical normal force can not be obtained (Fig. 4(e1-f1)). It is the same for MAO (micro arc oxidation) TiO$_2$ on Ti (Fig. 4(g1)). According to the scratch morphologies, discontinuous ductile perforation can be observed for TS (thermal spraying) ZrO$_2$ on Al and steel (Fig. 4(e2-f2)); and continuous ductile perforation can be observed for MAO (micro arc oxidation) TiO$_2$ on Ti (Fig. 4(g2)). Based on the AE curves of the scratch testing, Table 2 lists the critical normal force for the different coating-substrate systems. The critical normal force for the
former four coating-substrate systems can be obtained. These systems are hard-brittle ceramic coatings on metal or glass, while for the latter three coating-substrate systems are ductile coatings on metal. Therefore, it is indicated that the critical normal force for hard-brittle coating can be effectively obtained by AE signals.
Fig. 4 Scratch acoustic emission curves and scratch morphologies for different coating-substrate systems under the same test condition (normal force rate: 100 N/min, maximum normal force: 100 N). (a) PVD (Physical vapor deposition) TiAlSiN on high speed steel; (b) PVD AZO (Al doped ZnO) on glass; (c) PVD ITO (Indium tin oxide) on glass; (d) PVD FTO (SnO$_2$ doped fluorine) on glass; (e) TS (thermal spraying) ZrO$_2$ on Al; (f) TS ZrO$_2$ on steel; (g) MAO (micro arc oxidation) TiO$_2$ on Ti.

Summary

1) As for deposited SiC coating on C by CVD, the critical normal force obtained by acoustic emission (AE) signals decreases from 12 N to 7 N when the applied normal force rate increases from 20 N/min to 100 N/min, and the failure area of this scratched specimen increases with increasing maximum normal force.

2) Based on scratch morphologies for different coating-substrate systems, it is shown that spallation or delamination can be observed for hard-brittle coatings on glass or metal, while discontinuous or continuous ductile perforation can be observed for ductile coatings on metal.

3) According to AE curves of scratch testing for different samples, it is indicated that the critical normal force for hard-brittle coatings can be effectively obtained by AE signals.
Acknowledgments

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References

High-Performance Ceramics VII
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