The Influence of Parameters on the Adhesion of γ-Al₂O₃ Layers Deposited on FeCrAl Metallic Supports

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Abstract. Using the three-step method (pre-oxidation, primer deposition and coating deposition), a well-adhered γ-Al₂O₃ coating on FeCrAl metallic support was attained. The research researched the primer deposition including the influence of the acidity ([H⁺]/[AlOOH]) in starting dispersion, aging time and temperature.

Introduction

Metallic supports with high-temperature resistance appeared, solving the durability problem but bringing another difficulty. As we know, the metallic support and ceramic washcoat have different thermal expansion coefficients. It is necessary to deposit a porous material (e.g. Al₂O₃) with high surface area on the metallic substrates. A widely used method to form ceramics coatings on metallic supports is the dip-coating method [1-6]. This method is more economical and simple to be used in practice, besides it allows to obtain uniform coating on complex-structured substrates [7]. But to compensate the intrinsic disadvantage, some pre-treatments have been presented. Ferrandon [8] developed a technique to grow a number of textured alumina whiskers on the surface of the metal support before dip coating, which greatly improved the combination ability between the alumina washcoat and the support. Jingsheng Jia et al. [9] carried out a three-step method to study the effects of the main preparative parameters on the coating adherence of FeCrAl metallic supports. The work presents the effects of the major parameters of the primer deposition.

The Effect of Acidity ([H⁺]/[AlOOH]) in Starting Dispersion

We have investigated a range of [H⁺]/[AlOOH] ratios in the slurry between 0.10 and 0.15 at constant solid content aluminum sol of 5.5%, aging time and temperature being 85° and 8h, respectively. The resulting slurries were used to coat the pre-oxidized FeCrAl alloy.
Fig. 1 and Fig. 2 show the influence of solid content on coating load and weight loss of sol layer and corresponding SEM photographs of transition coating. The load of deposited coating was determined by weighing. As illustrated in Fig. 1, the plot of the deposited coating load per unit surface area versus the HNO₃ concentration in the slurry and weight loss continued to increase ranged from 0.10 to 0.15. The deposited layers with an acid/alumina ratio of over 0.14 were poorly adherent. And that can also be evidenced by Fig. 2, in which the
coating appeared obvious cracks when the $[\text{H}^+]/[\text{AlOOH}]$ reached 0.15. It can be explained that as the acid increased, the viscosity increased and the coating became thicker. So in the course of drying that led to more stress which induced cracks thus the adhesion decreased. The optimal acidity was confirmed between 0.12-0.14. But the result was different with the literature reported by Liying Yang [10]. He found the sol solution of smaller particle size (average size $\approx 19.3$nm), narrower particle size distribution (4.1-53nm) and better stability was obtained when solid content and $[\text{H}^+]/[\text{AlOOH}]$ were 5%-5.5% and 0.08-0.1, respectively. But this research found when $[\text{H}^+]/[\text{AlOOH}]$ was less 0.1, the sol could not be formed. As other preparation conditions were same, maybe this different results resulted from the component of pseudo-boehmite (PB).

### The Effect of Aging Time and Temperature

<table>
<thead>
<tr>
<th>Aging temperature</th>
<th>pH</th>
<th>Stability of sol</th>
</tr>
</thead>
<tbody>
<tr>
<td>60$^\circ$C</td>
<td>3.56</td>
<td>little deposit</td>
</tr>
<tr>
<td>70$^\circ$C</td>
<td>3.68</td>
<td>little deposit</td>
</tr>
<tr>
<td>80$^\circ$C</td>
<td>3.75</td>
<td>stable sol</td>
</tr>
<tr>
<td>85$^\circ$C</td>
<td>3.73</td>
<td>stable sol</td>
</tr>
<tr>
<td>90$^\circ$C</td>
<td>3.77</td>
<td>stable sol</td>
</tr>
</tbody>
</table>

In Table 1, the pH and stability of aluminum sol as a function of the aging time are reported when solid content, acid content and aging time were 5.5%, 0.13, and 8h, respectively. It can be observed that pH was increasing as the aging temperature was raised. When the aging temperature increased into 80°C and higher, blue and transparent sol with a good fluidity was formed, and there was no gel formed after 10d. Hydrolysis-sol course is of absorption of heat, so as the temperature increases the reaction rate accelerates and consumes more H$^+$. This can explain the pH increased on increasing aging temperature. Besides, the correlative literature [11] reported that in the aging process the pseudo-boehmite (PB) transforms into aluminum trihydroxide whose particles can’t be peptized, so the stable sol was hard to be obtained. Otherwise, there was no phase transition at higher temperatures. But then water evaporated rapidly, and the concentration would change. So the optimal aging temperature is confirmed 85°C.

The below curves coating load and weight loss of sol layer at different aging time are plotted in Fig.3 with a solid content aluminum sol of 5.5%, acid content aluminum sol of 0.13, and aging temperature of 85%. It illustrates the coating load increased quickly in first aging hours. After 8h the tendency reached plateau. The value was observed around $3.45 \times 10^{-4}$g/cm². The weight loss was steady at 10%-15%. The behavior was due to the reaction equilibrium after 8h. So the appropriate aging time can be confirmed 8h.
Figure 3. Influence of aging time to coating weight and weight loss of sol layer.

Fig.4 shows the cross-sectional SEM image of metal substrate after the transition coating in the optimal process parameter above and calcinated at 800°C for 2h two times. It is clear that the white transition layer is homogeneous with a thickness of 5-6μm. It was reported in the literature[12] that the appropriate thickness of transition coating was about 3-10μm. Thicker would induce cracks, but thinner can have enough combination and also fall off. So the coating in this experiment is cohesive.

Figure 4. Cross-sectional SEM image of the metal substrate after the transition coating.

Summary
According to the research, we found covering the sample with 0.13 [H⁺]/[AlOOH], aging temperature and time of 85°C and 8h, respectively, and calcination at 800°C for 2h, the
premier coating thickness of 5-6μm was optimal. A well-adhered coating on a metallic support to hold the catalysts was attained.

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References