Preparation and characterization of nanomaterial of LaAl\(_{11}\)O\(_{18}\) produced by chemical co-precipitation method

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Abstract: In this paper the methods of chemical co-precipitation method were used to produce nanomaterial of LaAl\(_{11}\)O\(_{18}\). In order to find the effects of the preparation parameters on its microscopical structure and the influences of calcinated temperature, keeping warm time and pH value on organizational structure and generate efficiency. And use the X-ray powder diffraction (XRD) and the scanning electron microscopy (SEM) to find the impacts of calcinated temperature, keeping warm time and pH value on the composition and microscopical structure.

1 Preface

LaAl\(_{11}\)O\(_{18}\) composite oxide, as a new inorganic functional material, has been widely used in many areas of research since 70s. For instance, it can be used as laser material and the crystal matrix of superconducting and fluorescent materia. As the catalytic materials of high temperature catalytic combustion reaction, particularly, it has been wildly researched\(^{[1-3]}\). LaAl\(_{11}\)O\(_{18}\), a new thermal barrier coating materials, is a good alternatives of the zirconia which is partially stable to Yttrium oxide. LaAl\(_{11}\)O\(_{18}\) is magnetic lead iron ore structure which is a mixed structure of cube and hexagonal body. In this structure, One layer of LaO\(_3\) which is found showing crystalline mirror next to four layers of aluminum spinel layer which is divided by symmetrical mirror. In the layer of LaO\(_3\), Highly loaded La\(^{3+}\) is located at the oxygen position in the hexagonal tight structure of the oxygen ions. La\(^{3+}\) diffusion is strongly suppressed so that the growth rate of the layered structure compound in the C-axis direction perpendicular to the mirror surface is relatively slow, which makes it has good thermal stability. Compared to the oxygen ion conductor of zirconia, the applicable temperature of LaAl\(_{11}\)O\(_{18}\) is allowed at 1300 °C for the thermal stability of LaAl\(_{11}\)O\(_{18}\). In addition, this arrangement makes the holes in the structure very little, which can effectively inhibit the diffusion of oxygen atoms, reduce the bonding layer and the substrate oxidation rate, reduce the amount of oxide, thereby improving the coating reliability and service life\(^{[3-6]}\).

2 Preparation of test materials

In the coprecipitation method, Al (NO\(_3\)) 3 reacts with ammonia to produce Al (OH) 3, La (NO\(_3\)) 3 and ammonia react to produce La (OH) 3. And, it is most important that the following reaction occurs during the heating process\(^{[7]}\).

\[
\text{2Al(OH)}_3 \rightarrow \text{Al}_2\text{O}_3 + 3\text{H}_2\text{O} \quad (1)
\]

\[
\text{2La(OH)}_3 \rightarrow \text{La}_2\text{O}_3 + 3\text{H}_2\text{O} \quad (2)
\]

\[
\text{La}_2\text{O}_3 + \text{Al}_2\text{O}_3 \rightarrow 2\text{LaAlO}_3 \quad (3)
\]

\[
\text{LaAlO}_3 + 5\text{Al}_2\text{O}_3 \rightarrow \text{LaAl}_{11}\text{O}_{18} \quad (4)
\]

Specific ingredients shown in Table 1.
### 3. Results and discussion

#### 3.1 Influence of calcinated temperature

As shown in Figure 1 a), when the calcination temperature is 1250 °C, the peak intensity of the...
diffraction powder is relatively small and the peak intensity is more balanced and only a small amount of lanthanum aluminate and a small amount of lanthanum hexaerythrite produced, the content is very small. At this point the powder is mostly alumina, lanthanum oxide, aluminum hydroxide, lanthanum hydroxide and some other impurities. And the strongest peak is lanthanum aluminate, which presumably at 1250 °C may be just the reaction temperature. And Figure 1 b) significantly increased the strong peak whose intensity was significantly enhanced and less peak. And from the XRD diffraction diagram, at this time, the resulting substances are mostly LaAlO₃ and LaAl₁₁O₁₈, indicating that the reaction between the oxides has begun to strengthen. By the above X-ray diffraction analysis, it can be seen that the actual onset temperature of the LaAl₁₁O₁₈ phase is between 1200 and 1250 °C. When the holding temperature is higher than this temperature, the generation efficiency of the LaAl₁₁O₁₈ phase increases with the temperature at different temperatures.

![X-ray diffraction pattern of powder at different heat treatment temperatures](image)

Fig.1 X-ray diffraction pattern of powder at different heat treatment temperatures

3.2 Influence of pH value

Figure 2 a), b), c) three XRD test results corresponding to the samples are roasting to 1400 °C, holding 4 hours under the conditions of the obtained, Wherein the pH of a) is 8, the pH of b) is 9, and the pH of c) is 10. The diffraction peaks of the three samples were all obvious, and the peaks were also less and the main substances are LaAlO₃ and LaAl₁₁O₁₈. This confirms the higher the sintering temperature, the longer the holding time, the more complete the reaction of this conclusion. The difference between the three is pH. In the X-ray powder diffraction pattern, the strongest peak in Fig. 2 (b) corresponds to LaAlO₃ and LaAl₁₁O₁₈. And Figure 2 a) and Figure 2 c) correspond only to a product of LaAl₁₁O₁₈. This indicates that pH 9 is relatively easy to produce LaAl₁₁O₁₈ phase. And then from the intensity of the diffraction peak, The intensity of the diffraction peak in Fig. 2b) is significantly lower than that in Fig. 2 (a) and Fig. 2 (c). It can also be seen from Figure 3 that the sample in Figure 3 b) has the highest transparency. This indicates that when the pH is 9, the lanthanum aluminate phase is further converted to the LaAl₁₁O₁₈ phase. It can be seen from Fig. 2 (b) that the LaAl₁₁O₁₈ phase occupies the dominant position. This indicates that the choice of pH is
very important when other conditions are the same.

Figure 2 X-ray diffraction pattern of powders with different pH

a) 1400 °C holding 4 h  pH8  
b) 1400 °C holding 4 h  pH9  
c) 1400 °C holding 4 h  pH10
3.3 Analysis of SEM

In Figure 4, most of the SEM images are lamellar, with only a few small, irregularly granular, which is enough to conclude that the structure of lanthanum hexaaluminate is lamellar. And the scanning electron microscopy in the granular, clumps of things may be the La$_2$O$_3$, Al$_2$O$_3$ and so on which did not react completely. The front has been analyzed by XRD and transmission electron microscopy. From Fig. 4 (a), it can be seen that the thickness of the lamellae is 40 nm and the thickest is 120 nm.

4 Conclusion

This article mainly discusses the influence of calcinated temperature, keeping warm time and pH value on micro structure and character during the production of the LaAl$_{11}$O$_{18}$. Conclusion as below.

(1) The key of the production of the LaAl$_{11}$O$_{18}$ is the calcinated temperature, keeping warm time. The actual generate temperature of the LaAl$_{11}$O$_{18}$ is between 1200 and 1250 degrees. When the temperature higher than this point, the production amount of the LaAl$_{11}$O$_{18}$ will increase with the calcinated temperature and keeping warm time increasing. In different temperatures, the production effect of the LaAl$_{11}$O$_{18}$ will increase the calcinated temperature increasing.

(2) During the process of the LaAl$_{11}$O$_{18}$ produced by chemical co-precipitation method, PH has the effect on LaAl$_{11}$O$_{18}$ LaAl11O18 production efficiency. It is the highest production efficiency and the more completely reaction of LaAl11O18 when the other experimental conditions are the same.

(3) This paper use the scanning electron microscopy (SEM) method found the Morphology of LaAl$_{11}$O$_{18}$ is lamellar and the thickness of the film is 40 nm.
5 References


