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Preparation of a novel magnesium oxide nanofilm of honeycomb-like structure and investigation of its properties

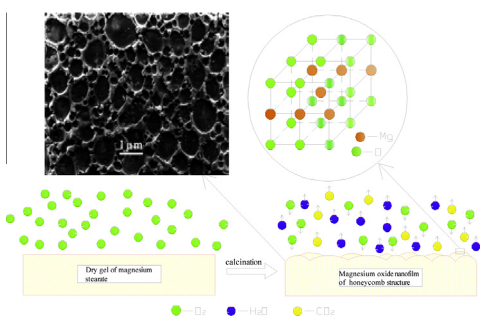
Lifeng Chen [a,b,*], Peng Bai [b], Wenxiu Li [a]

[a] Shenyang University of Chemical Technology, Shenyang 110142, China
[b] Pharmaceutical Engineering Department, School of Chemical Engineering and Technology, Tianjin University, Tianjin 300072, China

HIGHLIGHTS

- Novel magnesium oxide nanofilm of honeycomb-like structure was prepared for the first time.
- Formation process and mechanism of the nanofilm was derived.
- The nanofilm has antimicrobial property.
- The nanofilm has property of high temperature resistance.

GRAPHICAL ABSTRACT



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ABSTRACT

A novel magnesium oxide nanofilm of honeycomb-like structure was prepared for the first time. The nanofilm was fabricated via sol-gel method and was characterized by Fourier infrared absorption spectra, scanning electron microscope, ellipsometer, X-ray diffraction, energy dispersive spectrometer, atomic absorption spectrum, surface area analyser and membrane density detector. The obtained nanofilm sample is single-phase, cubic in crystal structure, atomic ratio of O to Mg is approximate 3:1, the multipoint BET specific surface area is 5.2 m^2/g and the density is 3.16 g/cm^3. The thickness of the nanofilm is about 65 nm. In view of the above, we derived the formation process and mechanism of the nanofilm, and proposed that the nanofilm would be made from cuboid units. The cuboid units should be connected one another by adsorption force. Moreover, the antimicrobial property and high temperature resistance of the nanofilm were investigated. The results showed that the nanofilm has well antibacterial effect on *Staphylococcus aureus* and could withstand high temperature at nearly 2000 °C. This would provide a reference to improve the existing antibacterial and refractory materials.

1. Introduction

The research of nano-materials [1–5] is an emerging field. Magnesium oxide thin film belongs to inorganic nano-materials. Magnesium oxide thin film is a special material, which has characteristics of high melting point, wear-resisting, low erosion rate and high secondary electron emission coefficients [6,7]. Therefore, magnesium oxide thin films as protective layers are widely applied to ceramics, aviation, foundry and electronics. Magnesium oxide thin films have been prepared via several methods, such as sol-gel method [8,9], electron beam evaporation [10,11], laser pulse deposition sputtering [1,12,13], metal organic chemical vapor deposition [14,15], ion beam assisted deposition [16,17] and spray pyrolysis method [18,19]. However, the public's focus is mainly on the preparation method [20–22] of magnesium oxide

* Corresponding author.
E-mail address: ttclf@126.com (L. Chen).

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thin film until now. And there is not any report about the micro structure, formation process and mechanism of magnesium oxide thin film.

For the above reasons, in this paper, we studied formation process and mechanism of a novel magnesium oxide nanofilm of honeycomb-like structure, which was prepared via sol-gel method. The change of morphology of the nanofilm would be presented, and the basic micro structure of the nanofilm would be proposed in the meantime.

In addition, it has not been reported on antibacterial property of magnesium oxide nanofilm and other inorganic oxide monomer nanofilm until now. Under this condition, we investigated the antimicrobial property of the novel nanofilm, based on the studies of MgO nanoparticles [23–25] and organic cellulose dry films [26] in this aspect. Moreover, in order to broaden the applicability of the novel nanofilm, we also did some high temperature resistance tests on the nanofilm.

2. Experimental

2.1. Preparation of magnesium oxide nanofilm of honeycomb-like structure

The preparation of magnesium oxide nanofilm was based on sol-gel method [21]. In the reaction process, the reaction volume was 1 L, in which the $Mg(NO_3)_2$ (analytically pure, Sinopharm Chemical Reagent) was 0.5 mol/L and the stearic acid ($C_{17}H_{35}COOH$, analytically pure, Sinopharm Chemical Reagent) was 2 mol/L. The pH value of the mixture was adjusted to 4 by dropping trace amounts of HNO_3. The mixture was stirred at 2000 rpm and heated in 90 °C. Colorless transparent sol was formed in 20 min. Then a lifting machine was used to lift coating on aluminum alloy substrate with the sol at a speed of 2 cm/min. Next, the aluminum alloy substrate coated with sol was dried in vacuum oven at 90 °C for 0.5 h to form dry gel. Finally, it was calcinated at 400 °C for 1 h. On the surface of aluminum alloy substrate, a layer of magnesium oxide nanofilm could be obtained. Multi-layer magnesium oxide nanofilm could be obtained by repeating the operation.

In this work, the nanofilms with one to four layers coating were selected, and the change of the formation process and morphology of magnesium oxide nanofilms was investigated. When the nanofilm was coated with four layers, a novel magnesium oxide nanofilm of honeycomb-like structure was formed. The formation mechanism of the novel nanofilm was also studied.

2.2. Antibacterial experiments

The antibacterial test of the novel nanofilm was done via Baird-Parker method [27]. The antibacterial property of the nanofilm was checked by the appearance and quantity of the plaque. The *Staphylococcus aureus* reference strain (ATCC 25923) was used. The bacteria strain was cultured in a broth of 7.5% (mass fraction) NaCl at 37 °C for 7 h. Subsequently, the above culture was vaccinated to four Baird-Parker plates respectively. Two of the four plates were covered with test papers (HTX, 3 M), and they were cultured at 37 °C for 24 or 48 h severally. Similarly, the other two plates were covered with the novel nanofilm samples, and they were also cultured at 37 °C for 24 or 48 h severally. Finally, the nanofilm samples were compared with the test papers after the cultivating, and the antimicrobial property of the nanofilm was determined.

2.3. Refractory and high temperature resistance tests

The refractory and high temperature resistance tests of the novel nanofilm were done by using oxy-acetylene flame. Here, the oxy-acetylene flame was chosen as the heat source of maximum temperature, which we could get under our existing condition. Meanwhile, it could be adjusted and used expediently, and the maximum temperature of oxy-acetylene flame could be close to the melting point of magnesium oxide (2852 °C). In the test, we made the oxy-acetylene flame burn the magnesium oxide nanofilm vertically, without burning Al alloy substrate directly. The temperature of oxy-acetylene flame was controlled around 2000 °C, and it was measured by a handheld infrared thermometer (CIT-G, Sciample). In this case, the nanofilm was heated for 1 or 2 h. Subsequently, the change of the film was investigated.

2.4. Analysis and characterization

The fourier infrared absorption spectra (FT-IR, Agilent 5500) was used to analyze the composition of dry gel, under the condition of KBr discs and the temperature of testing room is 40 °C. The images of films and dry gel were obtained via scanning electron microscope (SEM, JSM-6390LV) with wide-angle lens of 3.6 mm and the voltage of 30 kV. Meanwhile, the qualitative and quantitative detections were carried out via EDS (falcon), which was combined with SEM. The phase composition and crystalline form of samples were analyzed via X-ray diffraction (D8 Advance), under the condition of Cu Kα radiation (λ = 0.15046 nm, 40 kV, 50 mA). Surface area analyzer (ASAP2020) was used under the condition of N_2 atmosphere (0-10^5 Pa, absolute pressure). In the test, the BET (Brunauer-Emmett-Teller) method was applied to measure the parameters of specific surface area of the samples.

3. Results and discussion

3.1. Characterization of magnesium oxide nanofilm of honeycomb-like structure

Fig. 1 is the Fourier infrared absorption spectra (FT-IR, Agilent 5500) of dry gel. The peak at 1469 cm^{-1} caused by C═O key stretching vibration symmetrically is the characteristic peak of carboxylate. The peak at 1271 cm^{-1} is caused by C—O— vibration. The peak at 941 cm^{-1} of carboxylic acid dimers is characteristic peak of carboxylic acid, which was caused by vibration. The peak at 716 cm^{-1} is methylene (CH_2) plane swing vibration absorption peak. The peak at 565 cm^{-1} is the characteristic peak of the groups of $-(CH_2)_n-$. Based on the above analysis, combined with research of Gairong Chen [28], we could deduce that $Mg(NO_3)_2$

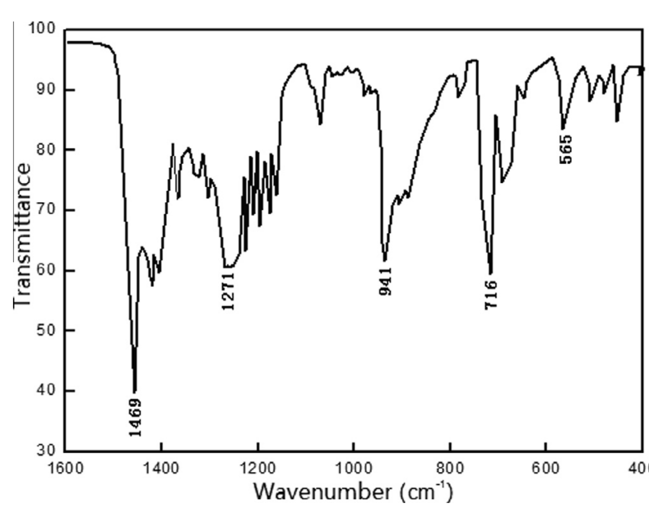

Fig. 1. Infrared spectrum of dry gel.