Synthesis of nanoporous acicular-mullite ceramic and electroless platinum coating for particulate matter entrapment and catalytic combustion

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Abstract. Crystal-structured acicular-mullite ceramic with high porosity and interpenetrated networks was fabricated for diesel particulate filter (DPF), followed by electroless deposition of platinum (Pt) catalysis throughout ceramic surfaces from an aqueous Pt (IV) solution. The integrated material was characterized and compared with commercial cordierite particulate matter (PM) filter, exhibiting many excellent properties in surface area, porous ratio, and loading capacity for both soot and catalyst. The resulting surface density of Pt nanoparticles attained as high as $8.0 \times 10^9$/cm² due to the 3-dimensional architecture, while maintaining a pore ratio, 63.72%, relative to 52.60% in its counterpart. Deposited Pt nanoparticles artfully serve as catalysis for combustion of carbon soot, potentially implemented for diesel engine filter, reducing the particulate matter emission for environmental protection. The catalytic performance was evaluated by a regular thermal gravity analysis (TGA) measurement, showing that the significant drop of soot conversion temperature, and soot convert rate could be further improved by co-precipitating CeO₂ catalyst additive. This material, thereby, could readily adapt itself into large-scale production.

1. Introduction

Diesel engines powered global transportation with a great fuel economy for off-road and on-road vehicles. However, current concerns over the health issues and climate changes associated with particle matter (PM) produced by diesel engines attracted considerable attention of legislation and set a challenging bar on vehicular exhaust soot and gas emission [1]. For example, the crucial mass concentration of PM$_{2.5}$ in the atmosphere must be lower than 10 mg/m$^3$ according to the current Europe Standard [2, 3]. Undisputedly, health and climate risks promote a primary impetus of the lower carbon emissions by seeking alternative energy sources or treatment that can drastically reduce PM concentration before exhaustion. On latter dimension, many after-treatments and enabling technologies were established on current exhaust systems, e.g., diesel particulate filter (DPF), diesel oxidation catalyst, selective catalytic reduction, selective non-catalytic reduction [4-6], among which, the DPF is the most pervasive and popular device implemented in off-road vehicles, complying with these increasingly stringent regulations.
Materials used for DPF should have sufficient porosity, high mechanical integrity, chemical durability, and stability at high temperatures. To date, many porous ceramic materials have been developed [7], with each exhibiting its own advantages and disadvantages [8, 9]. In particular, acicular-mullite constituted by the interpenetrated nanocrystal networks exhibits many uncompromised characteristics about effective particle removal. Thus, it has been widely used for manufacturing DPF [10]. Firstly, it produced enormous specific surface areas due to anisotropic features, consequently increasing loading capacity for embarked catalysts and soot. Secondly, the unique “needle-like” micro-crystals forms a special interconnected texture, attaining 63 ~ 65% porosity [11, 12], higher than conventional cordierite and silicon carbide (45 ~ 55%) [13-15], intriguingly reducing the backpressure, even under the circumstance of high soot loads. Additionally, the protrusion of crystals provides substantial contacts between soot and catalyst. Particularly, in “recycling” oxidation mode, disoriented crystal needles developed enhanced axial velocity components in a flowing stream, allowing soot deposition on additional dimensions. Finally, the unique geometry effectively prevents the formation of soot cake during filtration and combustion.

Here, an upgraded protocol of ceramic synthesis was employed, attaining the exquisite crystal structure and interpenetrated network, which can entrap the microscale particles. Combined with suitable catalysts, the filter can chemically convert the C and N compounds in unburned soot into harmless CO₂ and N₂, which can be released to the atmosphere without special caution [16]. Thus, coinage metal has been used in automotive exhaust purification because of its high catalytic activity, chemical inertness and thermal resistance. Thus, coating catalyst on microporous structures becomes a challenge for efficient DPF because of reaction in enclosed micropores and overall cost for large-scale production. Incipient-wetness [17, 18], impregnation [19], sol-gel [18, 20], and gas bubbling-assisted membrane reduction (GBMR) [21] methods were widely used to incorporate catalytic layer onto filter. However, catalytic particles often exhibited poor dispersion, uneven loading, and degradation under the harsh thermal conditions. The electroless deposition approach, on the other hand, exhibits many promising advantages [22]: 1) It does not require electrical contact, regardless of chemical property of substrate; 2) It is an aqueous solution reaction and fits nicely to large-scale production without extra facilities; 3) With appropriate treatment, it generates exquisite nanostructure, lowering the surface energy of catalytic metals, such as Pt and Pd.

In this work, we used the modified in situ reaction sintering to get a high-quality acicular mullite with easily obtainable materials, including kaolin clay and Al₂O₃. The parameters of surface density, pore ratio, and amount of Pt loadings were characterized with appropriate facilities and were quantitatively compared with a conventional porous substrate. The catalytic performance on the resulting surfaces was attested on a mimic system, which was comprised of the mixture of Pt-coated mullite powders and 25 nm in diameter carbon soot. Moreover, the doped catalytic system, CeO₂/Pt, was co-participated on mullite surfaces, which benefited the catalytic reactions by providing additional dimension for oxygen migration and storage [23]. Our work shows that modified acicular-mullite can effectively entrap particulate matter and catalytically convert carbon soot to gaseous compounds.

2. Materials and methods

2.1. Materials and chemicals

Acicular-mullite was synthesized by in situ sintering reaction with Al(OH)₃, Kaolin, and AlF₃ as raw materials, and the detailed procedures have been reported elsewhere [24]. Briefly, Al(OH)₃ was calcined at 420°C for 60min to obtain γ-type Al₂O₃ with a specific surface morphology. Kaolin powder and a mixture containing γ-Al₂O₃ and AlF₃ were blended with the stoichiometric ratio of 3Al₂O₃·2SiO₂·AlF₃ and V₂O₅ were added into the ingredient as the additional material with approximate 3% of the total weight. Then the mixture was put into a ball-milling to get homogeneous mixing. After granulation the samples were placed into a covered corundum crucible, gradually increasing temperature to 950 °C with a step of 5 °C/min and sintering at 1400 °C for 150 min in a high-temperature sintering furnace. Porous cordierite (Φ127×118 mm and 200 pores per inch) was ordered...
from Prince Ceramics Company, Nanchang, Jiangxi, China. Kaolin earth, V$_2$O$_5$, Al(OH)$_3$, and AlF$_3$ were purchased from Longyan Kaolin Limited Company, China. Carbon black was purchased from JCC Chemical trading Company. Tin chloride (SnCl$_2$), trifluoroacetic acid (C$_2$HF$_3$O$_2$), AgNO$_3$ (99.99 %), potassium hexa-chloroplatinate (IV) (K$_2$PtCl$_6$, 98.0 %), dodecylbenzene sulfonic acid sodium salt (C$_{18}$H$_{29}$NaO$_3$S), ammonium hydroxide (NH$_3$·H$_2$O, 28 % NH$_3$), hydrazine hydrate (H$_4$N$_2$, 80.0 %) and nitric acid (HNO$_3$) were purchased from Sigma-Aldrich Company.

2.2. Catalysis synthesis
A two-step method for electroless deposition of Pt nanoparticles (NPs) was reported by Zhou et al. (2015) [25]. CeO$_2$ additive doping was following the wet impregnation protocol. Briefly, cerous nitrate (Ce(NO$_3$)$_3$·6H$_2$O) was dissolved in DI water to give a solution concentration of 1.0 M. Then the prepared mullite and cordierite fragments were impregnated with the solutions and dried at 120 °C for 4 hours, after which the samples were calcined under air at 500 °C for 1.5 hours.

2.3. Materials characterization
The SEM micrographs were acquired on an inspect F50 (FEI, USA) scanning electron microscope equipped with both a field-free line detector (for large area imaging) and a high-resolution backscatter detector (for <50 μm diameter regions). Pore size distribution and specific surface areas were gauged by mercury intrusion porosimetry (MIP) using the PoreMaster 33 (Quantachrome Corporation) with pore size distribution measurement range of 6×10$^{-3}$~1080 μm. For both mullite and cordierite samples, the low pressure was around 7.19×10$^{-3}$~0.27 MPa and the high pressure was 0.15~103.42 MPa. X-ray diffraction (XRD) spectra were analyzed using an X-ray diffractometer (PANalyticalX'Pert Power) with Cu-κα radiation and a scan rate of 0.013 °/second in the 2θ range of 10°~80° operating at 4 KW and 40 mA.

2.4. Catalytic performance
The activity of the catalyst for diesel soot combustion reaction was measured in a thermal gravity analysis (TGA) apparatus (TGA/DSC1, Mettler Toledo). Carbon black (Printex-U, Degussa) was used to mimic diesel soot in gauging catalytic activity. The primary particle size was 25 nm-in-diameter with a specific surface area of approximately 100 m$^2$/g [26]. The weight changes were recorded from 30 to 700 °C with a heating step of 10 °C/min, used for analyzing the catalytic process of Pt NPs on different substrates. Since the way soot and catalyst contact will significantly impact upon the results of catalytic combustion reaction [27], in this study, tight contact was used for catalytic performance, that was, soot and catalyst were intimately mixed in an agate mortar.

3. Results and discussions

3.1. Crystal structure of acicular mullite
In the present study, the in situ sintering method was employed for acicular mullite synthesis under air conditions. SEM micrograph (Figure 1) shows the as-synthesized acicular mullite crystal, on which a three-dimensional network was formed with a plethora of nano-pores. A previous study showed that acicular mullite crystal was the hexagonal crystal structure, exhibiting the anisotropic growth characteristic [28]. Relative to conventional cordierite or SiC substrate, it rendered many uncompromised characteristics, massive specific surface area, and high porosity. The former gave rise to greater loading capacity for both catalyst and soot. The latter was responsible for lower backpressure, even under heavy loads of soot or catalyst. According to the recycling mechanism, oxidase agents, such as NO$_x$, traveled multiple times between catalysts and soot with the presence of these acicular crystal protrusions, which in return magnified the catalytic reactions. In addition, these interpenetrated crystals can prevent the throat block from overloaded soot disc and shorten the travel distance between catalyst and soot. The XRD of the synthesized mullite sample exhibited in Figure 2 indicates that the material was single-phase mullite (JCPDF Card No. 79-1455).
Figure 1. SEM micrographs of cordierite and acicular-mullite: (a) Virgin microporous cordierite; (b) Unmodified mullite structure; (c) Cordierite with Pt nanoparticles; (d) Mullite surface after electroless Pt deposition following the same protocol.

Figure 2. Power X-ray diffractive profile of synthesized mullite.

The porosity of acicular mullite in this study is 63.7 ± 2.7%, higher than 27.0 ~ 55.6% reported by Chen et al. with a similar synthesis method [29]. The higher porosity can be attributed to the addition of V₂O₅, which can act as a mineralizer to facilitate the development of the crystal nucleus. During mullite formation, the octahedral coordination of vanadium could enter the mullite lattice and replace Al³⁺, which would result in lattice expansion, crystal defect and distortion in local symmetry and then accelerated formation of mullite. In addition, a covered crucible was employed during the high-temperature sintering to prevent the leaking of vapor, which increases porosity [29], resulting in the comparable number reported by Hsiung et al. where the calcined samples were exposed to SiF₄ [30]. Accordingly, high porosity would alter mechanical properties of as-synthesized materials, such as strength, elastic modulus and toughness. The complicated relationship was described by the Gibson-Ashby foam model [11]. The ceramic became fragile and less mechanical durability if its porosity was greater than 68%. Therefore, this protocol produced the finest porous material, well-balanced mechanic strength and permeability.
3.2. Morphology and structure of the acicular-mullite structure
The aspect ratio is an important index for evaluating mechanical strength and porosity. The average aspect ratio of mullite made from the sintering reaction was as high as 9.13, with a grain length of 3.48 μm and needle diameter of 383.33 nm. Earlier research has shown that the acicular microstructure was beneficial for the stiffness and strength of mullite, and increased interconnectivity led to increased stiffness if other variables remained unchanged [11]. Cordierite were used as a control (Table 1). The specific surface area of acicular mullite was 1.96 m²/g which was an order magnitude higher than the commercial cordierite (0.18 m²/g). Pore diameter of cordierite was bigger than mullite (Figure 3), suggesting lower reactivity on the cordierite.

Table 1. Comparison of commercial cordierite and as-prepared acicular-mullite.

|                     | Density (g/mL) | Porosity (%) | Specific Surface Area (m²/g) | Pt NP density (×10⁹/cm²) | Average Diameter of Pt NPs (nm) |
|---------------------|---------------|--------------|-----------------------------|--------------------------|--------------------------------|
| commercial cordierite | 1.19±0.02     | 52.6±0.5     | 0.1832                      | 3.2±0.2                  | 57.0                           |
| acicular-mullite    | 1.18±0.02     | 63.7±0.6     | 1.9627                      | 8.0±0.2                  | 83.3                           |

Figure 3. Pore size distribution on cordierite and mullite materials, gauged by mercury intrusion data.

3.3. Eletroless growth of Pt NPs
Electroless deposition is well recognized as a facial approach for planting metal on any given substrate, regardless of its conductivity, but very few publications implemented this technology on catalytic DPF [31]. The size of produced Pt nanoparticles hinged on ceramic crystals, ranging from 40 nm to 100 nm (Figure 1(d)). To verify catalyst loading capacity, we compared specific surface area and density of Pt NPs on mullite against cordierite (Table 1), suggesting that mullite was an excellent substrate that was suitable to the electroless deposition because of both large specific surface area and intrinsic nanoscale roughness. The Pt particle size on acicular-mullite was larger than on cordierite, indicating higher reactivity of acicular-mullite. The fact of higher porosity and larger specific surface area of acicular-mullite with respect to cordierite did not affect the Pt particle size due to that the particle size of Pt was...
much smaller than that of the pores on both acicular-mullite and cordierite (Table 1). Averagely, mullite structure developed 10 times the greater specific surface area per unit weight than the regular cordierite materials.

Furthermore, EDX analysis of the semi-quantified Pt concentrations showed that higher Pt was found on mullite than commercial cordierite (Figure 4). The former managed a good texture to accelerate deposition rate because electroless deposition exhibited preference on coarse and irregular surfaces [32-34]. Moreover, on the rugged surface, normal direction tended to expedite reaction relative to lateral along needle crystal due to recycling of flow. Consequently, discrete grains were formed on crystal facets and within micropores, rather than only top surface in other deposition methods. By this method, more catalytic-active sites were available for soot combustion.

![Figure 4. EDX spectrums: (a) Virgin Mullite; (b) Mullite with Pt nanoparticles; (c) Cordierite with Pt nanoparticles.](image)

### 3.4. Catalytic performance

The primary goal of this study is to design an effective DPF system with lower back pressure and higher catalytic combustion rate. Two mechanisms could explain the high catalytic efficiency for soot combustion of Pt, one is the spill-over of activated oxygen and the other is NO-assisted soot oxidation [35]. Theoretically, the catalytic oxidation occurs at the three-phase interfaces, namely, a solid catalyst, a solid reactant and gaseous oxidizing agents, such as O₂ or NOₓ, which is a typical heterogeneous catalytic reaction [21]. Thus, the supporting substrate and catalyst's microscopic structure would significantly impact backpressure and soot removal efficiency. In this study, a mixture of carbon soot and Pt-coated ceramic rubbles was utilized to mimic the reactions inside the DPF. The catalytic activity was evaluated by a special TGA equipped with a gas chamber, allowing the reaction to be carried out under varied simulated atmospheric conditions. Here, T_{10%} and T_{50%} were used to assess the catalytic performance. The definition of above characteristics and an associated equation, by which, conversion temperature was computed, and results were described in Supporting Information. The results of the TGA experiments under different catalyst/soot combinations in tight contact were shown in Figure 5. The T_{50%} for non-catalytic soot combustion in the air was 605 °C, close to 613 °C reported by Bueno-Lopez [36]. With nanostructured Pt catalysts on acicular mullite, the conversion temperature dropped to 505 °C, implying that the noble metal nanoparticles facilitated the catalytic reaction. Intriguingly, needle-like mullite crystals could benefit “recycling” oxidative agents by intensifying the flow rate in axial direction, supported by weaker catalytic performance on alternative porous cordierite.
(Figure 5). Under the same condition sets, the T\textsubscript{50\%} on Pt-coated cordierite was 595 °C. The difference suggested soot oxidation complied with the gas phase catalytic mechanism [36-38]. These unstable intermediates traveled a short distance between the catalyst and trapped soot. Enhanced axial airflow in porous mullite increased the recycling rate of oxygen free radicals, in which each oxidative agent was used multiple times. Recently, the spill-over mechanism was proposed by Baumgarten and Schuck [39], showing that adsorbed O\textsubscript{2} over Pt surfaces splits to oxygen radical and diffuses to carbon materials. The entire process was recorded with isotope-labeled oxygen, demonstrating that the catalytic activity virtually relied on the degree of physical contact between soot and catalyst [39]. Reduced conversion temperature may also be because of the larger loading capacity of Pt catalysts caused by the greater specific surface area of the acicular mullite. Besides catalyst load and microscopic morphology of supporting materials, the addition of additive agents enhanced oxygen migration and storage and improved catalytic performance and conversion efficiency by recycling oxygen radicals [40]. Ceria has been used as a fuel additive to lower the ignition temperature of soot and a catalyst in Three-Way Catalysts (TWC). Because of the ability of cerium to switch between the Ce\textsuperscript{4+} and Ce\textsuperscript{3+} oxidation states and adsorption oxygen into the crystal structure, ceria can store and release oxygen which makes ceria exceptionally effective in soot catalytic oxidation [27]. In order to further improved the catalytic efficiency, CeO\textsubscript{2} was used as an additive agent and was thermally calcined on acicular mullite after the wet impregnation. The evolution of black carbon conversion with a course of temperature in the presence of CeO\textsubscript{2}/Pt catalyst was shown in Figure 5. As indicated in Figure 5, the T\textsubscript{50\%} for soot combustion of CeO\textsubscript{2}/Pt catalyst on acicular mullite was down to 434 °C from 505 °C of Pt NPs on mullite with a difference of 71 °C, which was caused by the oxygen storing and release caused more O radicals to react with the trapped soot [41]. Compared with cordierite that equipped only serpent porous structure, CeO\textsubscript{2}/Pt on mullite substrate exhibited more reactivity and lower T\textsubscript{50\%} (490 °C vs. 421 °C) (Figure 5). The three-dimensional network structure of acicular mullite might be beneficial for the contact efficiency between Pt NPs and soot in diesel exhaust and the synergistic effect of Pt NPs and CeO\textsubscript{2} increase the amount of active oxygen species delivered to soot. To this sense, acicular microscopic structure in DPF was of great importance to facilitate the surface catalytic reactions. Notably, many factors strongly impacted the catalytic performance for soot removal, such as catalyst load, nanoscopic feature of catalyst, microstructure of supporting ceramic and additives. To achieve high soot removal efficiency and optimized work conditions, systematic design of DPF is mandatory, which can finely modulate catalytic performance and maintain the acceptable backpressure.

![Figure 5. Comparison of the catalytic performance on mullite and cordierite substrate: The T\textsubscript{50\%} for pure soot is 605 °C; The T\textsubscript{50\%} for Pt-coated mullite and Pt-coated cordierite are 505 °C and 595 °C; The T\textsubscript{50\%} for Pt/CeO\textsubscript{2}-coated mullite and Pt/CeO\textsubscript{2}-cordierite are 434 °C and 501 °C, respectively.](image-url)
Acicular mullite ceramic with high porosity was synthesized by a burning-out additive method from the cost-effective starting materials. The as-synthesized acicular mullite consisted of anisotropic acicular crystal with an aspect ratio of 9.13, exhibited a highly developed interconnected microstructure. The specific surface area of acicular mullite was an order magnitude higher than the commercial cordierite, and the porosity of the acicular mullite is 11.1% higher than its counter with a similar density. Because of the unique architectural feature, more Pt NPs were loaded on acicular mullite than cordierite using an electroless deposition approach. The catalytic performance of Pt-coated mullite for soot combustion was higher than the commercial cordierite. The catalytic efficiency could be further improved by doping rare-earth compounds (CeO$_2$), due to the enhanced oxygen migration and storage. In a word, acicular mullite will be the perfect candidate for supporter materials of DPF. Overall, easily obtainable starting materials and facile synthesis protocols were readily adapted to large-scale manufacture, emphatically compromising the technical and financial ends.

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