Novel Al/Al$_2$O$_3$ composite foams by direct oxidation conversion

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Abstract. In this paper we combined an investment casting technique and a direct oxidation conversion (DOC) method to fabricate novel Al/Al$_2$O$_3$ composite foams. We treated open cellular Al foams with dense struts in oxidizing atmosphere and obtained novel Al/Al$_2$O$_3$ composite foams. These novel-type foams had dense struts, and owned a ternary-layered structure within struts, which consisted of the inner layer of Al, the middle layer of $\alpha$-Al$_2$O$_3$ oxidation scale and the outer layer of $\alpha$-Al$_2$O$_3$ ceramic coating. The resulting foams were characterized with respect to micro and macro structure, phase composition and compression behaviour. Comparing with conventional reticulated porous ceramics (RPCs), the novel composite foams demonstrated a new deformation mode in compression, behaving in accumulative damages instead of catastrophic collapse, with more deformation capacity and better ductility. The DOC approach might inspire a new way to manufacture composite foams, and such foams might be alternatives to other cellular solids for specific applications.

1 Introduction
Ceramic foams have been utilized in a wide range of fields [1]. In the family of ceramic foams, the class of RPCs plays as a unique member. The geometrically reticulated structure gives them great permeability, high interconnectivity, high specific surface area, low thermal conductivity and low density. The nature of ceramic materials offers them high wear resistance and high resistance to chemical corrosion [2, 3]. They have found applications in metal melt filters [4], porous medium burners [5], catalytic substrates [6, 7] and volumetric solar absorbers [8, 9], etc.

The predominant processing route to RPCs is the technique developed by Schwartzwalder and Sommer in the early 1960s [10]. In this method, a polymeric sponge is usually used as a template to be coated with a ceramic slurry. After the burn-out of the sponges and high-temperature sintering, reticulated ceramic bodies remain, owning a similar structure as the sponge does. However, the pyrolysis of polymeric templates naturally leaves triangular voids within struts, tending to cause stress concentration, especially at the corners [3]. In addition, due to thermal expansion of sponges during the pyrolysis, longitudinal cracks may occur within the struts [11]. Both flaws aforementioned substantially impact the mechanical performances of RPCs, resulting in a limitation of their applications. By the nature of brittleness in ceramics, RPCs usually deformed in the mode of catastrophic collapse under extra loading, just allowing a very limited deformation, which indicates their low mechanical stability and reliability [12, 13].
Up to now, a great variety of approaches has been proposed to improve mechanical properties of RPCs. They can be totally classified into three categories according to their foci. The first strategy was focusing on pretreating the polymeric templates in order to modify the wetting behavior [14, 15]. The second one focused on improving ceramic coating slurries, either by optimizing its rheological properties [16, 17] or by adding reinforcements, like fibers or whiskers [18-20]. The third route was to post-treat the as-received foams, such as recoating or spraying the green bodies or pre-sintered foams to repair surface cracks [21-28], infiltrating the pre-sintered foams to fill the hollow struts [29-35], etc. Some of these approaches [30, 32-34] are suitable to enhance the compressive strength of RPCs substantially with only slight reduction in porosity. However, all these methods mainly focused on improving the mechanical strength of RPCs, the deformation mode of catastrophic collapse in RPCs did not change, and still needs to be addressed.

Here we propose a novel strategy that attempts to ameliorate the deformation behavior of RPCs and enhance the ductility for them. Based on replica technique, we used investment-cast metallic aluminum foams as original templates instead of polymeric ones. Unlike polymeric templates, the aluminum ones did not decompose to induce hollow struts, instead they would be partly oxidized during high-temperature sintering and stay in resulting struts. The struts obtained a ternary-layered structure, in which the unoxidized Al foams acted as the core, the surrounding oxidized products as the second layer, the ceramic coating as the outside layer. For the Al foams, the processing of high-temperature sintering can be called direct oxidation conversion (DOC), which was initially developed by Lanxide Corporation to fabricate bulky Al/Al2O3 composite materials [36]. As with that, our prepared foams were designated as Al/Al2O3 composite foams. They were characterized with respect to micro- and macro-structure, phase composition as well as mechanical behavior.

2 Experimental details

2.1 Materials preparation

The first step was to prepare ceramic slurries. An ethanolammonium citrate based deflocculant (Dolapix CE 64, Zschimmer and Schwarz Chemie GmbH, Lahnstein, Germany) was dissolved in deionized water, and then Al2O3 powder (CT 3000 SG, d50 = 0.5 µm, Almatis GmbH, Ludwigshafen, Germany) was added to the solution. The solution was mixed with a planetary centrifugal mixer (THINKY Mixer ARE-250, THINKY Corp., Tokyo, Japan) at 1500 rpm for 15 min. Afterwards, a polyvinylalcohol-based binder (Optapix PA 4G, Zschimmer and Schwarz Chemie, GmbH, Lahnstein, Germany) and a nonionic alkyl polyalkylene glycolether-based anti-foaming agent (Contraspum K1012, Zschimmer and Schwarz Chemie GmbH, Lahnstein, Germany) were added and the mixing proceeded with the mixer at 1500 rpm for another 15 min. The solid load of the ceramic slurry was adjusted to 70 wt.%, in which the weight ratios of dispersant, binder, and anti-foaming agents to the mass of Al2O3 powder were 1.0 wt.%, 1.5 wt.% and 0.1 wt.%, respectively.

Subsequently, investment-cast aluminum foams (15*20*20 mm³, 10 PPI (pores per inch), 99.7 wt.% Al, MAYSER, Lindenberg, Germany) were totally immersed in the as-prepared slurry. Afterwards, the soaked foams were pulled out slowly with a pair of tweezers for the consecutive drying process. Within the first two minutes, the contact face to the drying plate were switched every 15 seconds to offset the influence of gravity on the flowing of ceramic coating and to guarantee uniform coating. The following drying step lasted for 24 hours at ambient conditions. The process described above referred to the first coating cycle. To obtain varied coating thicknesses, this process was repeated for two, three and four cycles, respectively. To investigate the influence of the sintering temperature, dried green bodies were sintered by varying the temperature from 1200 °C to 1600 °C with 100-K-steps, and the dwelling time was fixed for 6 hours. To explore the influence of the dwelling time, another two sample series, fired at 1600 °C, were treated for 3 hours and 12 hours, respectively. All the heat treatments were carried out in an air furnace (KU 40/04/A, THERMCONCEPT Dr. Fischer GmbH, Bremen, Germany), with heating and cooling rates of 1 K/min and 3 K/min, respectively. The labels of specimens were named after a...
combination of the processing parameters, e.g. “3-1600-6h”; “3” stands for the number of coating cycles, “1600” refers to the sintering/oxidation temperature and “6h” refers to the dwelling time, respectively.

2.2 Characterization
The geometrical density of Al/Al$_2$O$_3$ composite foams were the ratio of total mass to geometrical volume. The strut density was determined by the Archimedes principle. The total porosity was deduced from the geometrical density and the strut density. The composite foams were characterized by micro-computed tomography (µ-CT; GE Sensing and Inspection Technologies GmbH, Wunstorf, Germany). Their 3D structures were reconstructed with the software Phoenix Datos|X 2.0 (Phoenix/GE Sensing & Inspection) and visualized with myVGL V2.1 (Volume Graphics GmbH, Heidelberg, Germany). The as-prepared foams were mechanically milled in a steel can with ZrO$_2$ balls as grinding media to prepare powder for XRD powder diffraction analysis (PANalytical GmbH, Kassel, Germany) with Cu-Kα radiation. The microstructure of selected foams was imaged with scanning electron microscopy (SEM; XL30 ESEM-FEG, FEI/Philips, Hillsboro, OR, USA), and uniaxial compression tests were performed with a uniaxial testing machine (TIRAtest 2825, Tira-GmbH, Schalkau, Germany), with a loading rate of 1 mm/min for ten specimens for each test. For the compressive strain-stress curves, the strain and stress corresponding to the maximum stress point were extracted as peak strain and compression strength, respectively. The Weibull modulus in compression strength for selected specimens was determined by linear regression.

3 Results and discussion

3.1 Macrostructure characterization

![Figure 1](https://example.com/f1.png)

Figure 1. Optical images of the Al/Al$_2$O$_3$ composite foams with three coating cycles sintered at 1200-1600℃ for 6 hours.

Figure 1 shows the appearance of Al/Al$_2$O$_3$ composite foams sintered at varied temperatures for 6 hours. It is obvious that the composite foams survived severe high temperature sintering and retained their open-cellular structure. The outer ceramic coating tightly adhered to the Al substrates, and no spalling of them was found. Even though the clogging of cells took place occasionally within the resulting foams, they still showed high permeability, implying by the see-through of dark background. It also was observed that the as-received foams did not shrink significantly. This can be explained by the opposite evolution between Al substrates and Al$_2$O$_3$ coating. The oxidation of aluminum induced a volume expansion, because the volume of oxide was about 2.3 times as that of the aluminum consumed in the oxidation reaction [37]. By contrast, based on the study by Athena et al. [38], Al$_2$O$_3$ coating was estimated to have a volume shrinkage of around 30%.

The µ-CT 2D reconstructions in Figure 2 represent the cross sections of the resulting foams; the irregular green patterns represent the foam struts. Unlike conventional RPCs, novel Al/Al$_2$O$_3$ composite foams owned solid struts, having no triangular voids. This means that to replace the polymeric templates
with metallic investment-cast Al foams was an effective approach to generate dense struts, which may also be achieved by infiltration techniques [30-34]. Under the sintering, metallic Al foams melted in liquid state with active mobility, but solid struts indicate that most of the molten aluminum remained within the Al₂O₃ ceramic shell, instead of splitting the ceramic coating to flow out. The flowing out of aluminum only occurred locally, generating voids in the struts. On the µ-CT slices, the voids in the struts are implied by the black close pores within the green bodies. Besides strut voids, surface cracks were also produced during direct oxidation conversion (DOC) processing, implied by the black lines within the green bodies. As shown in the images, the amount of surface cracks tend to increase with an increase in temperature, specifically in the specimens oxidized at 1500 °C and 1600 °C, in which cracks occurred massively. The occurrence of surface cracks can be attributed to thermal mismatch between the molten aluminum (coefficient of thermal expansion, CTEₐₐ = 9.8 × 10⁻⁶ K⁻¹) [39] and Al₂O₃ (CTEₐ₂o₃ = 7.5 × 10⁻⁶ K⁻¹) [40] coating. The volume expansion of molten aluminum imposed stress to the shrinking ceramic shell, creating cracks on the outer ceramic coating.

![Figure 2. µ-CT 2D reconstructions of Al/Al₂O₃ composite foams with three coating cycles sintered at 1200 °C to 1600 °C for 6 hours: (a) 1200 °C, (b) 1300 °C, (c) 1400 °C, (d) 1500 °C, (e) 1600 °C.](image)

The properties data of Al₂O₃ composite foams are collected in Table 1. They were also analysed specifically in following separate figures. As shown in Figure 3(a), the geometrical density of resulting foams stands around 0.5 g/cm³, which is largely higher than that of the original density of Al foams. This data is comparable to that of conventional RPCs [30]. With varying sintering temperatures, the density of resulting foams was not influenced significantly, just with slight fluctuations. This also applied for the porosity of the foams, which all valued around 84% for all foams.
Table 1. Properties of Al/Al₂O₃ composite foams.

| Specimens | Geometrical Density [g/cm³] | Total porosity [%] | Strut density [g/cm³] | Mean strut thickness [mm] | Compression strength [MPa] | Weibull modulus m |
|-----------|-----------------------------|--------------------|-----------------------|---------------------------|---------------------------|------------------|
| Al foam   | 0.10±0.003                  | 96.4               | 2.71±0.01             | 0.40                      | -                         | -                |
| 3-1200-6h | 0.48±0.04                   | 85.5               | 3.31±0.04             | 1.28                      | 0.38±0.05                 | 9.5              |
| 3-1300-6h | 0.52±0.03                   | 84.3               | 3.31±0.02             | 1.16                      | 0.52±0.12                 | 7.1              |
| 3-1400-6h | 0.46±0.02                   | 86.4               | 3.38±0.03             | 1.18                      | 0.26±0.05                 | 5.6              |
| 3-1500-6h | 0.48±0.04                   | 85.5               | 3.32±0.07             | 1.08                      | 0.42±0.11                 | 4.0              |
| 3-1600-6h | 0.50±0.05                   | 84.6               | 3.25±0.06             | 0.94                      | 0.60±0.08                 | 6.5              |
| 3-1600-3h | 0.51±0.03                   | 83.8               | 3.15±0.03             | 0.95                      | 0.46±0.08                 | 6.8              |
| 3-1600-12h| 0.52±0.04                   | 83.8               | 3.20±0.09             | 0.95                      | 0.59±0.14                 | 5.2              |
| 2-1600-6h | 0.49±0.02                   | 85.5               | 3.37±0.03             | 0.76                      | 0.51±0.07                 | 9.0              |
| 4-1600-6h | 0.98±0.06                   | 71.8               | 3.47±0.01             | 1.40                      | 1.85±0.27                 | 8.5              |

Figure 3. The influence of the sintering temperature on (a) density and porosity and (b) mean strut thickness of Al/Al₂O₃ composite foams oxidized for 6 hours.

Comparing with conventional RPCs [3, 32, 34], where the porosities were achieved above 90%, the porosity of Al₂O₃ composite foams was slightly lower, and this was due to the thickening of struts by repeated coating processes. In Figure 3(b), the mean strut thickness of resulting foams witnessed a decrease with an increasing sintering temperature. This is explained by varied densification of ceramic coating with an increasing sintering temperature, which was also observed in Al₂O₃ ceramic processing by slip casting [38]. The ceramic coating tended to be denser as sintering temperature rised, so the strut thickness of the foams showed a tendency to decrease.

To investigate the influence of the oxidation and sintering time on the macrostructure of the Al/Al₂O₃ foams, the sintering time was varied, as shown in Figure 4(a). It was observed, that the impact of sintering time on porosity and strut thickness can be neglected, resulting in overall values of 84% and 0.95 mm, respectively.
In order to create Al/Al$_2$O$_3$ composite foams with different strut thicknesses, repeated coating was employed, as displayed in Figure 4(b). It clearly indicates that repeating the coating process can significantly thicken the struts of the foams, but with respect to the mean strut thickness, the thickening effect demonstrated no proportional relation to the number of coating cycles, which was also found in recoated zirconia toughened alumina (ZTA) foams [30]. That may be attributed to the increasing surface area and the evolution of the surface morphology as the coating process was continually repeated.

![Figure 4](image)

**Figure 4.** The influence of (a) the sintering time on the porosity and mean strut thickness of foams processed with three coating cycles at 1600 °C; and the influence of (b) the number of coating cycles on the mean strut thickness of foams treated at 1600 °C for 6 hours.

3.2 Microstructure characterization

In Figure 5, the microstructure of broken struts of Al/Al$_2$O$_3$ composite foams is displayed resulting from SEM imaging. It clearly indicates that the struts of novel foams owned solid formation without triangular voids, which was consistent with μ-CT results. More importantly, the struts attained a novel ternary-layered structure, as shown in Figure 5(c). By means of EDS (Figure 5(e), (g), (f)) and XRD (Figure 6), the composition of each layer was determined.

The inner layer originated from the plain Al foam, which withstood the high-temperature sintering and oxidation process and retained mostly non-oxidized. The middle layer was comprised of α-Al$_2$O$_3$, closely encompassing the aluminum substrate. This layer resulted from incomplete oxidation of the metallic aluminum substrate, amounting from dozens of micrometers to even one hundred micrometers in thickness. The Al substrate was only partly oxidized even at up to 1600 °C for 6 hours. The reason is that, as the temperature and the dwell time increased, the outer coated Al$_2$O$_3$ ceramic became denser and the already formed oxidation scale became thicker, they blocked the paths through which the oxygen diffused into the inner aluminum [38, 41]. It must be note that the grain size of α-Al$_2$O$_3$ in this layer reached dozens of micrometers, indicating abnormal grain growth. This may result from their growing conditions at elevated temperatures where they were soaked in liquid aluminum: Zhang et al. [42] reported that the introduced Al nanoparticles to Al$_2$O$_3$ ceramic were able to induce elongated growth of the Al$_2$O$_3$ grain. The outer layer was an Al$_2$O$_3$ ceramic coating. As shown in Figure 5(a), severe surface cracks appeared on the struts, the reason for that has been discussed in former sections. The existence of surface cracks allowed the flowing out of molten Al, generating cavities locally within the struts, as displayed in Figure 5(d).

In summary, even though flaws exist, such novel Al/Al$_2$O$_3$ composite foams demonstrate unique ternary-layered struts, bridging and combing metal and ceramic. To the best of our knowledge, no such strut formation in ceramic foams has been reported in literature yet [1-3]. It may turn out that the direct oxidation conversion (DOC) might inspire a new way to fabricate ceramic composite foams.
Figure 5. Typical SEM images for Al/Al₂O₃ foams: (a) 1500 °C for 6 hours, (b) and (c) 1600 °C for 6 hours, (d) 1600 °C for 3 hours; EDS measurements for image (c) in different zones, (e) zone 1, (f) zone 2, (g) zone 3.

The phase composition of the Al/Al₂O₃ foams is shown in Figure 6. The resulting foams consisted of α-Al₂O₃ and metallic aluminum, and only α-Al₂O₃ existed, no other types were detected. Note that the intensities of diffraction peaks for aluminum did not precisely represent the real fraction of aluminum in resulting foams. Because the samples were powdered for XRD by mechanical milling; considering the high degree of ductility of metallic Al, not all the aluminum may have been transferred to the XRD’s sample holder.
3.3 Compression strength behavior

Representative stress-strain curves in compression of Al/Al₂O₃ composite foams are arranged in Figure 7, and that of plain Al foam and ZTA foam [30] were added for comparison. The curve of the ZTA foam represents typical compression behavior of conventional RPCs. It shows that when compressive stress reaches the critical strength of RPCs, damage occurs rapidly and the strength falls to a low level, which actually implies the catastrophic collapse in deformation mode for such foams. Commonly, crack of struts or through-body cracks in foams can be observed after the testing. With respect to the deformation capacity, it allowed limited strain having a level of only 5% or less. The Al foam, it showed higher ductility, accompanied with continual densification in compression. Its compression strength was so much low that it cannot be identified accurately under the described testing conditions.

Comparing with that of Al and ZTA foam, the as-received Al/Al₂O₃ foams behaved entirely different in compression. The compressive process consisted of three stages, as labelled in the figure. In the first stage the course of all Al/Al₂O₃ foams follows Hook’s law until a tipping point is reached. In contrast to brittle foams (e. g. ZTA) no catastrophic failure follows, instead a negative slope of the course is observed (stage II) until a further tipping point is reached followed by a steep positive slope (stage III). Stage II represents an accumulated damage where the struts collapse in a mixed ductile and brittle mode. Stage III represents a densification, also like in stage II with a ductile and a brittle fraction.

In summary, the failure behavior of these DOC foams in compression mode represents their composite design build of aluminum as a ductile and alumina as a brittle component. The increase of stress until the first tipping point is reached is a consequence of an increased densification by sintering with an increasing temperature, starting at 1400 °C.
Figure 7. Typical compressive stress-strain curves of Al/Al₂O₃ composite foams with three coating cycles sintered at different temperatures for 6 hours.

Figure 8. Processing conditions and their influence on compressive strength and corresponding Weibull modulus in Al/Al₂O₃ composite foams: (a) and (b) the influence of the sintering temperature for foams sintered at different temperature for 6 hours; (c) the influence of the compression strength on foams sintered at 1600 °C for different times; (d) the influence of the number of coating cycles for foams sintered at 1600 °C for 6h.
The influence of the sintering conditions on compressive strength of the Al/Al$_2$O$_3$ foams is shown in Figure 8: the compressive strength increases from 1200 °C to 1300 °C, drops to 1400 °C and increases with increasing temperature to 1600 °C. A tentative explanation for this course is the following: increasing sintering temperature facilitated the densification of the outer ceramic coating, which is along with increased strut strength [38]. An increasing firing temperature gave rise to surface crack formation, as we had observed by means of μ-CT, and this reduced the strut strength. With increasing temperature the ongoing sintering process reduces strut porosity leading to an increase in strength.

The Weibull modulus in compressive strength also varied with the sintering temperature. The foams processed at 1200 °C and 1300 °C owned relatively larger Weibull moduli. This may be associated with surface morphology of the struts. Totally, though severe surface cracks existed within resulting foams, the Weibull modulus in our foams is still comparable to other studies [7, 30, 34].

The influence of sintering time on compressive strength of the foams as displayed in Figure 8(c) indicates a slight increase in strength with increasing time, which may have influenced the theoretical density of the alumina phase, being higher after longer sintering times. Figure 8(d) shows how the coating thickness, here given as the number of coating cycles, affects the compressive strength. This is related to the thickening of struts under the loss of porosity, see also Figure 4(b).

4 Conclusions
By combining the investment-cast technique with the direct oxidation conversion (DOC) method, novel Al/Al$_2$O$_3$ composite foams were prepared. Replacement of commonly polymeric templates as used in the replica processing of ceramic foams by Al foams with dense struts leads to novel-type Al/Al$_2$O$_3$ foams with ternary-layered structures in their struts, which consists of an inner layer of Al, a middle layer of α-Al$_2$O$_3$ formed by Al oxidation and an outer layer of α-Al$_2$O$_3$ ceramic coating. The process parameter sintering temperature played a crucial role, leading to moderate compressive strength when being over 1500 °C. Compared to conventional RPCs, these novel-type composite foams possess a new deformation mode in compression: they show accumulative damages instead of catastrophic collapse, with more deformation capacity and better ductility. The approach of DOC might inspire a new way to manufacture composite ceramic foams, and such composite foams might be the alternatives to other cellular solids in some applications.

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