Nanoporous Aluminum by Galvanic Replacement: Dealloying and Inward-Growth Plating

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In aqueous solutions, electro/chemically deposited metals usually grow outward into electrolyte. Here we report that the reduced Al grows inward into the sample, surprisingly, while Mg (in pure Mg and Al2Mg3 alloy) is galvanically replaced with Al in an ionic liquid. The galvanic replacement reaction (GRR) of Al2Mg3 involves a dealloying process that generates a nanoporous Al skeleton, and simultaneously the inward-growth plating of Al that thickens the Al skeleton. The obtained open nanoporous Al might be interesting for many applications including lithium-ion batteries. The anomalous inward-growth of deposited Al is attributed to the low diffusivity of ionic liquid and the negative charge of reduced species. This dealloying/GRR procedure provides a new route to produce hierarchical nanoporous structure in bulk materials.

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Nanoporous (NP) metals, either existing as particles or bulk materials, show a variety of superior mechanical and functional properties compared with their fully-dense counterparts. Galvanic replacement reaction (GRR) is a simple and versatile approach for producing hollow metal nanoparticles. But GRR of a massive single metal is unlikely to generate a bulk NP material because deposition usually grows outward into an electrolyte (partly driven by the gradient of reduced species), ending up with a pile of powder-like remnants. On the other hand, the bulk NP metals can readily be fabricated by dealloying—a electro/chemical corrosion process that selectively dissolves the more-reactive elements from alloy and reorganizes the remaining less-reactive elements into a network-like structure. So far, the NP metals generated by GRR and/or dealloying are limited in a few metals such as Al, Pt, Pt, Ag, Ni, Cu. Most of them are precious metals.

Nanoporous aluminum (NP Al) is a long-expected nanoporous metal that has yet to be successfully synthesized by either GRR or dealloying. The light-weight and low-cost Al foams with mm-scale even larger pore sizes have been widely used in many areas, such as the impact-absorber in the automobile industry and the current collector for batteries. Shrinking the porous structure size to nanoscale may greatly enhance its mechanical performance (i.e., specific strength) and even extend the application to lithium-ion batteries. But electrochemical or chemical synthesis of NP Al in aqueous electrolytes is challenging because Al itself is so reactive that the nanoscale Al liga-
ments may be fully oxidized under this circumstance. Synthesis of an NP Al by dealloying also requires dissolution of even more-reactive elements, which may lead to a surface coverage of oxides and even hydrogen evolution process, which impede the dealloying process or deteriorate the obtained samples. Here we report a new procedure for synthesis of bulk NP Al. It involves both GRR and dealloying, and is performed in an ionic liquid electrolyte so that the oxide formation and hydrogen evolution are prevented. By galvanically replacing Mg with Al, Al2Mg3 and even pure Mg were transformed into pure Al with nanoporous structure. During this procedure, most interestingly, a massive amount of reduced-Al was deposited into the pores and plated uniformly on the ligament surfaces. The high quality NP Al samples developed in this study are readily suitable for many mechanical/functional explorations. We also anticipate that this simple approach can be adopted for synthesis of other hierarchical NP metals in future.

Experimental

Sample preparation.—Parent alloy ingots with nominal composition of Al2Mg3 were prepared by melting high purity Al (≥99.9%) and Mg (≥99.9%) in a resistance furnace under Ar atmosphere. The alloy ingots were sealed in quartz tubes with argon atmosphere, homogenized by annealing at 420 °C for 24 h and then quenched in water. Prior to GRR, the (Al2Mg3 and pure Mg) samples cut from the ingots were polished using abrasive paper and then cleaned using ethanol. GRR was conducted by immersing the Al2Mg3 or pure Mg samples in an [EMIM][AlCl4] ionic liquid, which was prepared by mixing AlCl3 (anhydrous powder, 99.99%, Alfa Aesar) and 1-ethyl-3-methylimidazolium chloride, [EMIM][Cl] (97%, Acros Organics) at room temperature with a molar ratio of 2:1. All experiments that involved ionic liquid were performed in an argon-atmosphere glove box at room temperature, where the oxygen content and moisture were maintained below 0.1 ppm. The obtained NP samples were carefully rinsed in acetone and then dried before taking out of glove box for other measurements.

Materials characterization.—X-ray diffraction (XRD) was performed on the crushed (powder-like) NP samples using a Bruker D8 Advance X-ray diffractometer with Cu Kα radiation. Microstructural morphology was characterized using a scanning electron microscopy (SEM, FEI Nova NanoSEM 430). Cross-sections of nanoporous Al were revealed by manually breaking the sample using tweezers. The fractured surface was also milled using a triple-ion beam slope cutter (Leica EM TIC 3X), which operates at an accelerating voltage of 7 kV and ion beam current of 2.6 mA. Microstructure of NP Al was scanned and reconstructed using a phase contrast 3D X-ray tomography system (ZEISS Xradia 800 Ultra), and then visualized by Avizo fire software. The voxel size is about 32 nm³. A micro-cylinder sample for 3D imaging, ~16 μm in diameter, was machined from a bulk NP Al using dual beam focused ion beam (FIB/SEM FEI Nanolab Helios 650). The Vickers microhardness of pure Al and NP Al samples were measured using an automatic hardness testing system (Qness Q10 A+). The load is 10 g and the duration is 10 s. Pure Al was annealed at 420 °C for 24 h and polished using polishing cloth before hardness test. The hardness of NP Al was measured on a surface that was milled using an ion beam milling system (Leica EM RES101).

Results and Discussion

In this study we used a Lewis acidic AlCl3-based ionic liquid, [EMIM][Cl]/AlCl3 (33.3:66.7 mol%) or [EMIM][AlCl4] as electrolyte for GRR. When the parent alloy, an intermetallic compound of
Figure 1. Nanoporous Al prepared by dealloying/GRR of Al$_2$Mg$_3$ alloy. a) Photographs of a parent alloy before reaction, a nanoporous Al after reaction, and a cell used for GRR. b) XRD patterns of samples before and after GRR. c) Mass changes induced by GRR and resultant relative density of samples with different thickness. $m_i$ and $m_f$ represent the mass before and after reaction. SEM images of d) the outer-surface, e-f) the fractured cross-sectional surfaces, and g-i) the polished cross-sectional surface of NP Al. The inset figure in h) shows a histogram of the ligament size distribution. i) A high magnification view of the dark-line in middle of cross-section (white open box in g), showing that the relative density in this region is lower than the main body of sample.

Al$_2$Mg$_3$, was immersed in this solution, GRR occurred via $^{35-39}$$^{[1]}
3$Mg (s) + 8Al$_2$Cl$_7^-$ → 2Al (s) + 3Mg$_2^+$(solv.) + 14AlCl$_4^-$

or following half-reactions

$^{[2]}$3Mg (s) − 6e → 3Mg$^2+$ (solv.)

$^{[3]}$8Al$_2$Cl$_7^-$ + 6e → 2Al(s) + 14AlCl$_4^-$

Figure 1a shows that the outer dimensions of Al$_2$Mg$_3$ (including thickness) changed very little after GRR. The resultant sample remained intact, unlike the powder-like products generated by GRR in aqueous solutions. XRD patterns shown in Figure 1b indicate that the whole material had been transformed from Al$_2$Mg$_3$ ($\gamma$-phase) to Al. EDS measurements on the fractured surface of a reacted sample detected no trace of Mg, confirming that Mg in the entire sample had been dissolved.

Figure 1c shows that for all samples with different thicknesses, when GRR was complete, the sample weight decreased to $\sim$84.3% of the initial value (weight of the parent alloy), which is approximately twice of the Al concentration ($\sim$42.5 wt%) in the parent alloy. It hints that the half of Al in the resultant NP Al was inherited from the parent alloy during dealloying, and the second half was contributed by Al deposition during GRR. The amount of Al in the sample doubled after reaction, which is consistent with the prediction from Eq. 1. This observation confirms that all reduced Al has been deposited to the sample during reaction.

SEM imaging (see Figure 1d) shows some Al islands deposited on the sample surface after reaction. But their amount is negligibly small compared with the total amount of Al deposition, which is equivalent to the amount of Al in the parent alloy. The SEM observation on a cross-sectional fracture surface (Figures 1e–1f) shows a uniform, open NP structure across the thickness. High magnification image in Figure 1f reveals that the solid phase is very well interconnected. The dangling ligaments, which were often observed in NP Au, were not observed in NP Al. The NP Al is also more dense compared with the previous NP Au prepared by dealloying. $^{4,7}$

SEM observation on the cross-section surface of an NP Al that was polished by ion beam milling reveals more details, as presented in Figure 1h: i) the obtained NP Al is essentially crack-free, while previous NP metals often contained a high density of cracks that is difficult to avoid (if possible), $^{20,42-44}$ ii) the main body of NP Al sample exhibits a uniform structure with ligament size at around 200 nm, although coarser porous structure was occasionally seen near sample surface – its cause is still unknown, iii) A “dark-line” appears in the middle of the cross section, as shown in Figure 1g. The EDS detects only Al in this region, ruling out the possibility of incomplete reaction. A high magnification SEM image (Figure 1i) reveals that along this
“dark line” the structure is more open than the main body. This less dense gap-like region has a thickness of $< 2 \mu m$.

A 3D structure reconstruction and visualization of NP Al (see Figure 2) shows an isotropic porous structure with interpenetrating solid ligaments and pore channels (or the filled liquid phase). The main pore channels are open throughout the sample, although the deposition was involved and the relative density has been increased to as high as $\sim 64\%$.

Selective dissolution of Mg from Al$_2$Mg$_3$ is a dealloying process, which generates a nanoporous structure of more-noble metal, i.e., Al in this case, with relative density of $\sim 32.3\%$ if the changes in sample volume is neglected. The relative density of our NP Al is much denser ($\sim 64\%$), indicating that the reduced Al must have been deposited onto the surfaces of Al nano-ligaments (created by dealloying).

The inward-growth nature of Al deposition is evidenced by a low density gap-like region observed in the middle of sample, where the reaction fronts from two sides met together at last stage of GRR. It clearly shows that the reduced Al deposits on ligaments $\sim 1 \mu m$ behind the reaction front. It also clarifies that the NP structure formed by dealloying of Al$_2$Mg$_3$ is indeed less dense than the obtained np Al (main body). In short, the GRR of Al$_2$Mg$_3$ proceeds by the galvanic dealloying and inward-growth plating (GalDIP) processes.

The inward-growth of deposited Al was even observed when a pure Mg was placed in a same environment for GRR, except that the dealloying was not involved here. As shown in Figure 3a, the initial geometry and volume of Mg were also retained after GRR. Some Al islands can be identified on the sample surface, similar to the Al$_2$Mg$_3$ after GRR. Compared with the NP Al formed by GalDIP of Al$_2$Mg$_3$, the NP Al obtained from GRR of pure Mg is far more fragile and has different structural features. The obtained sample consists of two NP Al plates that loosely bonded together (see Fig. 3b). This illustrates that during GRR of Mg, the deposited Al layers grow inward (from the top and bottom surfaces of the sample) while the Mg surface recedes, and meets in the middle as Mg shrinks and eventually vanishes. The microstructure of both NP Al layers is also anisotropic: the parallel fiber-like Al ligaments (with short branches, see Figs. 3c–3e) align along the reaction direction or normal to the sample surface. A in-plane view of contact surface between two plates (Fig. 3f) shows that these fibers are also transversely interconnected, so that the material can remain intact during GRR.

The dealloying is a decisive process for nanoporosity evolution during GalDIP of Al$_2$Mg$_3$. The dealloyed NP structure, which is isotropic, acts as a framework for the subsequent inward-growth plating of Al. In our case, the dealloyed NP material and the deposited material are identical, resulting in a single phase NP Al. Although the NP structure formed by dealloying was essentially retained in this case, the GalDIP-made structure is denser than that can be achieved by dealloying alone. Relative densities of the dealloyed NP metals are typically well below 50%, because the parting limit for dissolution of the more reactive component from a homogeneous fcc binary...
allyl is usually between 50 and 60 at.%. Therefore, GalDIP provides an opportunity to explore the other end of the spectrum of relative density — increasing relative density is profitable for improving the mechanical stability of NP metals. The Vickers hardness of NP Al presented in this study is $\sim 97 \pm 5$ MPa, which is much higher than that of previous Al foams, and is even $\sim 25\%$ higher than that of a fully-dense pure Al ($78 \pm 4$ MPa).

Furthermore, by using dissimilar metals in dealloyed framework and deposited phase, it is also possible to develop NP metals with core-shell structured ligaments using this approach. For a starting material of Mg-X alloy, if Mg was galvanically replaced with Y, then a NP structure with core (X) – shell (Y) structured ligaments may be obtained.

There are two prerequisites for the uniform inward-growth plating of a deposited species during GRR as observed in this study: i) the deposition of reduced phase occurs at or near the reaction front as it recedes, instead of on the sample surface, ii) the volume of solid phase decreases during GRR, leaving open space for mass and charge transport.

In our case, the second prerequisite is naturally met because the solid volume decreases by $\sim 35.4\%$ when three Mg is replaced by two Al. It is out of our expectation that the first requirement is also fulfilled, i.e., the freshly reduced Al deposits mostly at reaction front, more precisely, $< 1 \mu$m behind the reaction front as illustrated in Fig. 1i. This observation is intriguing because the Al ions need to travel a long distance ($10^{-2} \mu$m) along the narrow channels ($10^{-5}$ nm wide) to reach the reaction front without being reduced and deposited on the pore walls. It should be noted that the pore wall material, Al, is electrically conductive. In the conventional sense, Al would most likely be deposited on the sample surface or in the pores near surface. Furthermore, the concentration of $\text{AlCl}_7^-$ decreases and Mg$^{2+}$ increases with increasing current density during GRR of both Al$_2$Mg$_3$ and pure Mg. Such a concentration gradient may also facilitate the deposition of Al at or near sample surface, rather than at reaction front deep in the material. The “abnormal” inward-growth of deposition observed in this study indicates that GRR may occur through a different mechanism.

One possibility is that Al passesivate by forming a thin oxide layer even in ionic liquid under inert atmosphere, which may thicken with time and inhibit further Al deposition behind the reaction front. During GRR of Al$_2$Mg$_3$, the dealloying continuously generates Al ligaments with fresh surfaces that might act as preferential sites for Al plating. But this scenario cannot explain the inward-growth of Al during GRR of pure Mg, which obviously did not expose any fresh Al from the parent sample. Moreover, the newly deposited Al is as “fresh” as Al exposed by dealloying – from this standpoint, the deposited Al should grow either outward or inward from sample surface, which contradicts our observation. One may argue that the Al surface in the interior of sample is indeed “fresh” because in pore channels the oxygen has been consumed and the oxygen content is lower, which however requires experimental verification.

Another interpretation is relevant to the internal ohmic resistance of the ionic liquid. As distance between Al deposition site (cathode) and Mg surface (anode) increases, the internal resistance of galvanic cell increases. As a consequence, the current and reaction rate decrease when an Al deposition site is farther away from reaction front. This may explain why the Al plating occurs preferentially near reaction front. However, the electrical conductivity of the ionic liquid ($15 \text{ mS/cm}$) used in this study is not much lower than that of normal aqueous solutions ($60\sim80 \text{ mS/cm}$ in Watts bath for Ni plating), in the latter electrolyte the deposited species usually grows outward. Therefore, the internal resistance is unlikely playing a dominant role in our observation.

A more plausible scenario is associated with the slow mass transport rate in ionic liquid originating from its high viscosity. The dynamic viscosity of [EMIM][AlCl$_4$] ($3.36 \text{ mPa s}$) is $\sim 14$ times higher than that of water ($0.89 \text{ mPa s}$). Therefore, the diffusivity is approximately one order of magnitude slower in ionic liquid than in conventional aqueous solutions. As GRR continues, in our case, the dissolution of Mg leads to an accumulation of cations around reaction front, which are then neutralized by an in-flow of anions, i.e., $\text{AlCl}_7^-$ in our case. As a consequence, the concentration of $\text{AlCl}_7^-$ increases rapidly and reaches a maximum near reaction front, as illustrated in Figure 4. It should be noted that in our case, the plating of Al is achieved by reducing negatively charged $\text{AlCl}_7^-$ instead of positively charged metal ions in aqueous solutions. Therefore, the peak concentration of $\text{AlCl}_7^-$ near the reaction front makes this location a kinetically more favorable site for Al reduction. Our observation of a $\sim 2 \mu$m thick less-dense “gap” remaining in the middle of sample after GRR suggests that the $\text{AlCl}_7^-$ concentration peaks at $\sim 1 \mu$m behind the reaction front. From this point of view, the inward-growth deposition may be a universal phenomenon for GRR where the diffusivity of electrolyte is low and reduced species are negatively charged, providing that the volume of solid phase decreases during GRR.

**Conclusions**

Galvanic replacement of Mg with Al in an ionic liquid transforms bulk Al$_2$Mg$_3$ to pure Mg into nanoporous Al samples. The reaction of Al$_2$Mg$_3$ involves a dealloying process that creates a network-like framework of Al, and simultaneously a uniform plating process of Al that thickens the existing ligaments. This leads to a robust crack-free Al with isotropic nanoporous structure, which may be desirable for many applications. Without dealloying, the reaction of pure Mg also gives rise to a pure nanoporous Al, with less interconnected anisotropic nanoporous structure. Both experiments confirmed an inward-growth
nature of Al deposition in this environment (i.e., the Al grows toward the receding Mg surface). We attribute it to the slow mass transport rate of ionic liquid electrolyte and the negative charge of reduced species. By combining dealloying/GRR processes, this paper opens a new route for producing hierarchical hollow nanostructures, in bulk form and in non-precious metals, which might be interesting for many structural and functional applications.

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