NOVEL HOLLOW POWDER POROUS STRUCTURES

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ABSTRACT

Recent finite element calculations [1] indicate that structures constructed from partially compacted hollow spheres exhibit a greater stiffness and strength than many other cellular structures at comparable density. It has been observed that gas atomization of metallic powders often leads to entrainment of the flow field gas [2]. The resulting hollow powders are an unwanted by-product in the sense that they lead to porosity and future sites of defect in solid parts. Here a method is developed to separate the hollow powders according to their size, shape and density. They are then consolidated to a porous structure. Examples of this are given for both a titanium alloy and a nickel-base superalloy. The compressive mechanical properties are measured and compared to those of other porous structures.

INTRODUCTION

Porous structures based upon hollow metallic spheres with thin walls, strong interparticle bonds and a close packed arrangement [3] are a new class of cellular materials whose modulus and strength are predicted to exceed those of many other cellular materials [1]. As load bearing structures, their cell geometry is attractive in the sense that each cell can be of uniform size, shape, wall thickness and ligament with those adjacent. This morphology contrasts sharply with that of many cellular solids where cells are abnormally shaped, cell walls can be curved or broken and excess material aggregates at nodes. A group of cells with high aspect ratios then work collectively to cause large localized deformations which propagate and lead to a significant knockdown in weight specific mechanical properties [4]. The cell shapes of the hollow sphere structures on the other hand, resist bending and buckling, and are responsible for the greater predicted stiffness and strengths compared with many other cellular structures of comparable density. Furthermore, controllable amounts of open and closed porosity can be achieved creating multifunctional opportunities for this class of materials.

Researchers have demonstrated methods for producing hollow metallic shells using double wall capillary apparatus [5]. Variants of the method for the casting of ceramic slurries are under development [6]. However, these double wall capillary methods are limited to comparatively large diameter shells owing to difficulties in the machining of small capillary tubes and maintaining them unobstructed. Furthermore, selection of materials for parts in contact (e.g. capillary tubes, crucible, etc.) with the molten shell material is problematic if reactive metals are involved (e.g. titanium alloys). Cleanliness can also be an issue especially when reducing an oxide slurry to create a metal sphere [6]. Processes currently envisioned may also be costly.

It has been observed that gas filled hollow powders are a common by-product of gas atomization [2]. During the atomization process, high velocity gas jets are used to break apart a thin stream of molten material into many small droplets which then rapidly solidify to a powder. The mechanism(s) by which hollow powders are formed remains controversial. One explanation envisions the molten material encircling, closing off and trapping the gas inside [2]. Consider a spherical liquid droplet in a uniform velocity gas flow. Bag formation occurs when the Weber number, \( We = \rho_\infty V_\infty^2 D_1 / \sigma \), is in the range \( 12 < We \leq 50 \) [7] where \( \rho_\infty \) and \( V_\infty \) are the density and velocity (relative to the droplet) of the gas flow, \( D_1 \) and \( \sigma \) are the diameter and surface
tension of the drop. Suppose \( \sigma = 1480 \text{ dyn/cm} \) and \( \sigma = 1760 \text{ dyn/cm} \) for elemental \( \text{Ti} \) and \( \text{Ni} \) at their melting points [8]. \( \rho_{\infty} = 1.78 \text{ kg/m}^3 \) for \( \text{Ar} \) at sea level [8] and \( D_s = 1 \text{ mm} \). Then bag formation is anticipated when \( 100 < V_{\infty} \leq 204 \text{ m/s} \) for \( \text{Ti} \) and \( 109 < V_{\infty} \leq 222 \text{ m/s} \) for \( \text{Ni} \). It is reasonable that these subsonic velocities could be seen by the drop in an atomization plume. This appears to be an important factor if one seeks to produce large numbers of gas atomized hollow powders rather than prevent them. In this paper we develop a method to separate the hollow powders from other gas atomization products, present a way to consolidate them to a porous structure, measure their compression behavior and compare these properties with other porous materials.

**SEPARATION THEORY**

Sedimentation, elutriation and density gradient methods have all been used for separating powders according to their size, shape and density [9,10]. Such methods might be used to recover hollow metallic powders from other gas atomization products (e.g. solid powder, flake, etc.). Consider the levitation of a sphere in a uniform vertical flow, Fig. 1. The gravitational force, \( F_g \), buoyancy force, \( F_b \), and drag force, \( F_d \), are simply related;

\[
F_g = \pi D_s^3 \rho_s g / 6
\]

\[
F_b = \pi D_s^3 \rho_{\infty} g / 6
\]

\[
F_d = C_D \pi D_s^2 \rho_{\infty} V_{\infty}^2 / 8
\]

where \( D_s \) is the sphere diameter, \( \rho_s \) and \( \rho_{\infty} \) are the sphere and fluid densities, \( g \) is the acceleration of gravity, \( C_D \) is the sphere drag coefficient (a shape sensitive parameter) and \( V_{\infty} \) is the fluid free-stream velocity [11]. The drag coefficient depends on the sphere Reynolds number, \( Re_s = \rho_{\infty} V_{\infty} D_s / \mu_{\infty} \), where \( \mu_{\infty} \) is the fluid viscosity. When \( Re_s \leq 1 \) (i.e. Stokes flow regime), \( C_D \sim 24 / Re_s \), when \( 1 < Re_s \leq 400 \), \( C_D \sim 24 / Re_s^{0.646} \), when \( 400 < Re_s \leq 3 \times 10^5 \), \( C_D \sim 0.5 \) [11]. For the fluid in motion, Equations (1), (2) and (3) find the velocity required to lift the sphere:

\[
V_{\infty} > \sqrt{\frac{4D_s(\rho_s - \rho_{\infty})g}{3\rho_{\infty}C_D}}
\]

To prevent unsteady turbulent flow and avoid excessive mixing, the flow Reynolds number, \( Re_{\infty} = \rho_{\infty} \bar{V}_{\infty} D / \mu_{\infty} \), should be kept low. Here, \( \bar{V}_{\infty} \) is the mean flow velocity and \( D \) is a characteristic length. For a circular pipe of inner diameter \( D \), laminar flow is always expected when \( Re_{\infty} < 2300 \). For higher Reynolds numbers, the amount of viscous damping may not be sufficient to quell disturbances and random flow fluctuations (i.e. turbulence) can appear [11].

**SEPARATION**

To evaluate the separation methodology, two different argon atomized metallic powders were obtained from Crucible Research (Pittsburgh, PA) [12]. One was a Ti-6.2Al-3.9V (wt.%) alloy (Ti-6Al-4V) sieved at -14/+35 mesh (i.e. particle diameters varied from 500 \( \mu \text{m} \) to 1.4 mm). The
other was a Ni-21.3Cr-8.8Mo-3.9Nb-0.13Al-0.19Ti (wt.%) nickel-base superalloy (Ni 625) sieved at -10+/+45 mesh (i.e. 355 μm to 2 mm). Both contained solid, porous and hollow powders along with flake. The powders were generally spherical and often had satellites attached to them.

Flake and powders heavily laden with satellites are undesirable in the sense that they add much weight to a hollow sphere structure but with little mechanical advantage. By noting that spherical powders roll more readily, a collection of predominantly spherical powders was obtained by repeatedly placing powders on an inclined surface and agitating. To extract the hollow powders, two approaches were investigated. Using a static fluid approach, 4.5 g of sodium metatungstate (3Na₂WO₄·9WO₃·H₂O) per 1 ml of distilled H₂O were combined to produce a true fluid having a density of 2.9 g/cm³ [10]. Spherical Ti-6Al-4V powders were placed into the heavy fluid and agitated such that hollow powders having densities less than 2.9 g/cm³ floated to the surface. Using a dynamic fluid approach, spherical Ni 625 powders were sieved at -10+/+20 mesh (i.e 850 μm to 2 mm) to obtain a distribution of large, visible, easily handled powders, Fig. 1. These were placed in a vertical elutriation apparatus, Fig. 1, and the mean flow velocity was varied from 25 cm/s to 50 cm/s in 5 cm/s increments. The elutriation column was 1.00 m high and its inner diameter was 1.27 cm (0.50 in). Excessive turbulence was suppressed by keeping the flow Reynolds number low, 3517 ≤ Reₘ ≤ 7034. The size, shape and density of the recovered powders could be systematically controlled by varying the mean flow velocity, Fig. 2. Powders recovered for Vₘ ≤ 30 cm/s were all hollow and kept for consolidation.

a) Sphere equilibrium

\begin{align*}
F_b & \quad \text{Sphere} \\
F_d & \quad \text{Fluid} \\
F_g & \quad \text{Gravity}
\end{align*}

b) Predominantly spherical

Ni 625 powder (-10+/+20 mesh)

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{sphere_equilibrium.png}
\caption{Sphere equilibrium, predominantly spherical Ni 625 powder, elutriation apparatus.}
\end{figure}
**CONSOLIDATION AND MECHANICAL TESTING**

Hollow Ti-6Al-4V and Ni 625 powders were sealed in evacuated, $P < 5 \times 10^{-7}$ torr, cylindrical quartz canisters and sintered. For Ti-6Al-4V, this occurred at 1000°C for 24 hr while for Ni 625, this occurred at 1300°C for 24 hr. Cooling was in furnace. The Ti-6Al-4V sample was cylindrical with a 6.8 mm diameter, 4.0 mm height and 1.2 g/cm³ density. The two Ni 625 samples were also cylindrical but with 10.0 mm and 9.6 mm diameters, 12.7 mm and 12.8 mm heights and 2.7 g/cm³ and 3.1 g/cm³ densities. One of these (possibly located in a furnace hot spot) had cells which were strongly bonded while the other had cells which were weakly bonded, Fig. 3.
Samples were room temperature compression tested at a constant crosshead displacement rate of 0.001 mm/s using a screw driven electromechanical testing machine equipped with stainless steel compression platens. For the weakly bonded Ti-6Al-4V and Ni 625 samples, cells regularly broke away and fell to the compression platens during testing. Thus, failure through cell ligament shearing led to upper strengths of just 6.2 MPa and 6.4 MPa, Fig. 4. For the strongly bonded Ni 625 sample, the ligaments remained intact and a bi-linear stress-strain behavior was observed but with no well defined upper yield. The unloading modulus was about 5.2 GPa.
CONCLUSIONS

Hollow gas atomized metallic powders have been separated based on their size, shape and density. The elutriation method can be used to obtain tight distributions of very spherical powder, to remove non-spherical powders, those laden with satellites or those containing porosity. Control over hollow powder wall thickness can also be gained. Upon sintering, hollow powder porous structures were created. A strong ligament between cells was essential for good mechanical properties. The relative modulus of the well bonded Ni 625 hollow powder structure was comparable to that of honeycomb in-plane. A well defined upper yield strength was not observed which could lead to many interesting applications. Potential advantages of the gas atomized Ti-6Al-4V and Ni 625 hollow powder structures include small grain sizes, high temperature load carry capability and the ability to be heat treated. Furthermore, complex-shaped components with very high internal porosity are possible. This could occur by packing high gas pressure filled spheres in a desired geometry or mold followed by heating and expansion in a reduced pressure environment. The lower the external pressure, the higher will be the porosity.

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REFERENCES