Novel metallic hollow sphere structures: processing and properties

O. Andersen, U. Waag, L. Schneider, G. Stephani, B. Kieback

Fraunhofer Institute for Manufacturing and Advanced Materials, Bremen Department of Powder Metallurgy and Composite Materials, Dresden

Abstract

A novel P/M processing route has been developed for the production of randomly packed hollow sphere structures (RHS). In this approach, metal powder coated styrofoam spheres were used to make green parts in a special die. The subsequent sintering of the sphere walls and the joining of the spheres is accomplished in one step, thus achieving significant cost savings. In order to demonstrate the versatility of this approach, test specimens were manufactured from different metals and alloys. Compression tests of different RHS from stainless steel (316L) were conducted.

1 Introduction

The obvious advantages of metallic hollow sphere structures have lead to several attempts to realize such structures. Due to the very high cost of earlier solutions, it must be stated that none of these approaches has gained practical significance. Mostly, galvanically produced hollow spheres were used to build such structures by adhesive bonding, brazing, or sintering, sometimes with the help of added metal powders [1-3]. Apart from the materials limitations which are inherent in the galvanizing process, the cited process routes suffer from the large number of required process steps, i.e. manufacturing of hollow spheres, assembling, bonding.

Hurysz et al. [4] take a different approach in which hollow green spheres are produced via coaxial atomization. In order to make RHS, the green spheres which consisted of an appropriate mixture of iron and chromium oxide and a binder, were put in cylindrical crucibles or processed into larger RHS by adding a bond phase. After a suitable heat treatment, metal foams with a composition close to AISI stainless steel 410 or 420 were obtained.

Mechanical testing revealed quite low yield strengths being close to the theoretical values for open cell foams. These results were mostly attributed to the fact that the strength of the cell wall material was supposedly much lower than that of the solid material.

In 1987, Jaeckel [5] invented a powder metallurgical process for the production of metal hollow spheres. Here commercially available styrofoam spheres are coated with a metal powder and binder suspension in a fluidized bed. The resulting green spheres undergo a heat treatment in which the organic contents are removed. Subsequent sintering yields metallic hollow spheres. In principle, this allows for the production of hollow spheres from arbitrary metals and alloys. The IFAM Department of Powder Metallurgy and Composite Materials,

Dresden, has put this process to work and added a method which produces RHS directly from the green spheres.

The feasibility of the powder metallurgical approach was demonstrated with a variety of different materials, including Ti. Figure 1 shows cross sectional cuts of Ti hollow spheres with a diameter of approx. 4 mm and a mean wall thickness of $125 \,\mu m$. The mechanical properties of these samples were not satisfactory due to an oxygen content of $0.7 \,\%$ and a carbon content of $0.3 \,\%$. However, it has to be noted that the processing conditions (atmospheres, binder system) were not optimized for Ti.

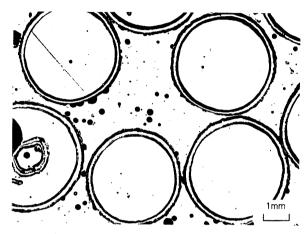


Fig. 1: Cross sectional cuts of Ti hollow spheres

2 Process route and resulting structure

One of the goals of the IFAM approach to RHS was to avoid pure point contacts between the spheres in order to enhance the fatigue behavior. Instead, the establishment of large contact areas between the spheres was achieved by applying a suitable forming process to the green spheres.

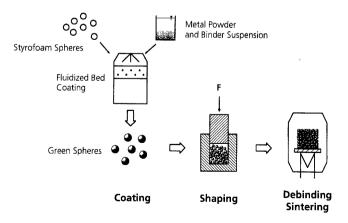


Fig. 2: Process scheme for the production of RHS

In this stage, the elastic styrofoam core helps to prevent buckling. The forming process results in a slightly anisotropic structure. To date, nothing is known whether this actually affects the mechanical behavior in different directions of loading. A process scheme is given in Figure 2.



Fig. 3: Cross sectional cut of contact zone in a 316L RHS

Figure 3 shows the contact zones within a 316L RHS. It can be seen that there is a favorable tendency towards densification in the area of contact which results from the forming process in the green state.

In general, it can be stated that the uniformity of the coating is very good, as is demonstrated by the sintered Ti hollow spheres of Figure 1. Hence, it can be expected that the reproducibility of the mechanical behavior across different samples should be excellent.

3 Mechanical behavior of RHS from 316L

A first set of compression experiments was carried out on RHS made from 316L. Small cylinders were made from different batches of green spheres. In the case of test specimens from the same batch of green spheres (sample 1 to 3 and 4 to 5, respectively), it was found that the variations in the density and dimensions of the samples were very small (Table 1).

Sample No.	Sphere Dia.	Mean Wall Thickness	Sample Dia.	Sample Height	Density
	mm	μm	mm	mm	g/cm ³
1	2.0 - 3.0	250	24.5	23.4	1.43
2	2.0 - 3.0	250	24.5	23.5	1.44
3	2.0 - 3.0	250	24.7	23.4	1.43
4	0.5 - 1.0	40	24.0	25.8	1.05
5	0.5 - 1.0	40	24.0	25.5	1.02

Table 1: Geometrical characteristics of the tested cylindrical RHS

In order to characterize the performance of the RHS, it is reasonable to scale the physical and mechanical properties with the properties of sintered 316L. These can be taken from MPIF standard 35 (issued in 1997) and are given in Table 2. The sintering conditions for the MPIF standard are: 1288 °C in partial vacuum. It should be noted that 316L can be sintered only to about 85 % of the theoretical density even in the pressed and sintered state. In the case of the RHS, commercial gas atomized metal powder (Ampersint < 22 μ m by H.C. Starck) was used. In contrast to the MPIF standard, the RHS were sintered at 1250 °C in hydrogen for 1 hour.

Table 2: Mechanical and physical properties of sintered SS-316L-22 (MPIF standard 35)

Density g/cm ³	Young's Modulus GPa	Compressive Yield Strength 0.1 % MPa
6.9	140	200

The compressive tests were carried out on a Zwick universal testing machine with a speed of testing of 10 mm/min. The tests were stopped at a load of 50 kN. Figure 4 shows test specimens with a sphere size of 2.0 to 3.0 mm before and after compressive deformation.

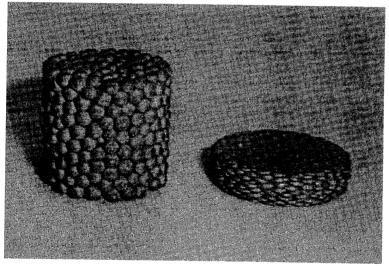


Fig. 4: RHS with a sphere size of 2.0 to 3.0 mm before and after compressive deformation

It was found that the stress-strain curves of comparable samples were almost identical. They show a smooth plateau of plastic deformation. Typical curves are given in Figure 5.

The values for the compressive stress at 25 and 50 % deformation can be taken directly from the measured data. The compressive modulus of elasticity was determined from the slope of a straight line fit to the unloading behavior in the region of 10 to 20 % strain (the unloading data are omitted in Figure 5). Within this range, no depence of the compressive modulus on the amount of strain was observed. The results are given in Table 3 and show rather small deviations across samples of identical sphere size.

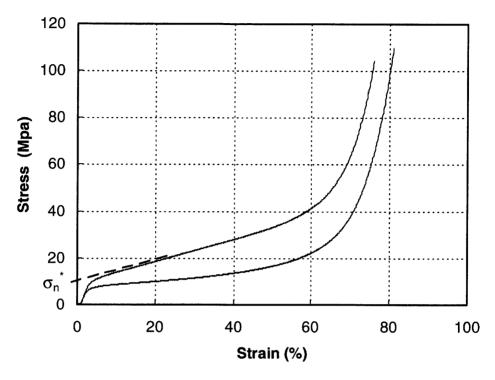


Fig. 5: Typical engineering stress-strain curves for 316L RHS (sample no. 3 and 4). The dashed line is the extrapolation line for obtaining the nominal compressive yield strength σ_n^*

Table 3: Mechanical properties of RHS from 316L and comparison of measured nominal and theoretical compressive yield strength

Sample No.	Compressive Modulus of Elasticity of a Deformed RHS	σ _{25%}	σ _{50%}	σ [*] _n (measured)	σ* (calculated from Eq. (1))	(σ [*] _n / σ [*]) * 100 %
	GPa	MPa	MPa	MPa	MPa	%
1	1.44	22.3	34.9	10.1	14.5	69.5
2	1.42	20.9	33.3	9.0	14.6	61.5
3	1.31	20.9	32.9	9.0	14.5	62.3
4	0.95	10.7	16.5	7.4	10.7	68.8
5	1.01	10.0	15.4	6.7	10.3	64.9

The nominal compressive yield strength σ_n^* was determined by fitting a straight line to the linear portion of the plastic deformation plateau and taking the stress value at the intersection of the linear fit with the ordinate (*Degischer et al.* [6]). In Ref. [4], the following expression for the yield strength σ^* was used as an estimate for the theoretical upper bound of closed cell structures made from randomly packed spheres:

$$\sigma^* = 0.35 \, \sigma_s \frac{\rho^*}{\rho_s} \tag{1}$$

Here, σ_s depicts the yield strength of the solid material, while ρ^* and ρ_s are the densities of the RHS and the solid material, respectively. In Table 3, the theoretical values calculated from Eq. (1) are compared with the measured data. It can be seen that the performance under compression is well above 60 % of the theoretical value which is a very good performance considering that Eq. (1) gives an upper bound.

4 Conclusions

Despite the small dimensions of the test specimens, first results show that the IFAM approach towards RHS yields reproducible structures with regard to dimensional tolerance, density, and mechanical behavior. The compressive stress-strain curves show smooth, linear, and rather long plateaus of plastic deformation which seem to be entirely reproducible. Densification sets in at approx. 50 % strain. When using scaling laws, it is reasonable to apply the values tabulated for sintered solid materials, since they constitute the optimum that can be achieved for the cell walls along the process route presented in this paper.

Future work will have to confirm these results with a larger number of test specimens, as well as to extend these results to lower relative densities and different geometries. An important issue will be the further improvement of the cell wall properties and the establishment of suitable theoretical approaches for the prediction of the mechanical behavior of RHS.

Acknowledgements

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References

- [1] German Patent, A. Roßmann, DE 2737248, 1977 (sintered or infiltrated structures)
- [2] French Patent, A. Rousset et al., FR 8707440, 1987 (ordered agglutinated or brazed structures)
- [3] German Patent, W. Smarsly, W. Track, DE 3902032, 1989 (bonding of spheres by loose powder sintering)
- [4] K.M. Hurysz, J.L. Clark, A.R. Nagel, C.U. Hardwicke, K.J. Lee, J.K. Cochran, T.H. Sanders: Steel and titanium hollow spheres. Mat. Res. Soc. Symp. Proc. Vol. 521, 1998, p. 191
- [5] European Patent, M. Jaeckel and H. Smigilski, DE 3724156, 1988 (coating of polymeric spheres with particles)
- [6] H.P. Degischer, U. Galovsky, R. Gradinger, R. Kretz, F. Simancik: Über mechanische Eigenschaften von Aluminiumschäumen. Proceedings Symposium Metallschäume, 6.-7.3.1997, Ed. J. Banhart, Bremen, MIT-Verlag, 1997