

STATIC COMPRESSION AND RESONANCE VIBRATION TESTS ON CELLULAR MATERIALS GRAVITY SINTERED FROM HOLLOW BRONZE SPHERES

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The cylindrical and rod-shaped specimens were prepared by gravity sintering from Cu-Sn hollow spheres. On these samples, both static compression tests and measurements of resonance frequencies were performed. The compressive stress-strain curves revealed the features characteristic for a closed-cell ductile cellular solid. The removal of in general open porosity among loosely packed closed metallic hollow spheres was recognized as the principal mode of plastic deformation. The approximative effective moduli of elasticity were determined for cellular materials under consideration by means of the measurements of resonance frequencies on rod-shaped specimens.

Key words: *cellular solid, effective modulus of elasticity, static compression test, dynamic resonant method*

Ispitivanja statičke kompresije i rezonantne vibracije celularnih materijala dobivenih gravitacijskim sinterovanjem šupljih brončanih kugli. Cilindrični i šipkasti uzorci su pripremljeni gravitacionim sinterovanjem šupljih Cu-Sn kugli. Na tim uzorcima su provedena ispitivanja statičke kompresije i mjerenja rezonantnih frekvencija. Krivulja naprezanje-rastezanje nam pokazuje svojstva karakteristična za plastične celularne krute materijale sa zatvorenim ćelijama. Uklanjanje uglavnom otvorene povezanosti među labavo povezanim šupljim metalnim kuglama prepoznajemo kao glavni način plastične deformacije. Za celularne materijale koji se razmatraju određeni su približno učinkoviti moduli elastičnosti mjerenjem frekvencija rezonancije na šipkasto oblikovanim uzorcima.

Ključne riječi: *celularni čvrsti materijali, učinkovit model elastičnosti, ispitivanje statičkog sabijanja, dinamičke metode rezonancije*

INTRODUCTION

Metallic foams and cellular solids represent a relatively new class of materials with perspective applications in lightweight structures, in acoustic and thermal management, in energy-absorbing parts, etc. Efficient use of these advanced materials requires detailed information on their mechanical behaviour, even when their primary use is not linked to mechanical loading (e.g., thermal insulation, filters, etc.). The most characteristic feature of the materials mentioned is their low density. The large pore volume minimizes the load-bearing cross section. Consequently, the foams and cellular solids usually cannot withstand high tensile forces. Therefore, mechanical experiments are conducted primarily in compression and flexure loads.

When a foam or cellular solid is compressed, the stress-strain curve exhibits a rather standard behaviour, whatever material the samples are made of [1-3]. There are three distinct regions on these curves:

- 1) quasi-elastic linear increase of stress for small compression,
- 2) plateau-like region characterized by small or even vanishing slope of the stress-strain curve,
- 3) region of densification with rapidly increasing stress. The quasielastic linear increase is controlled by the cell wall bending and cell face stretching.

Plateau-like region is associated with collapse of the cells. There are several modes of cell collapse: elastic buckling, plastic yielding, or brittle crushing of the cell walls, depending on the properties of material the samples are made of. The detailed shape of the plateau-like region depends on the particular mode of cell collapse. For nonlinearly elastic and plastic materials, the stress-strain curve is quite smooth in the plateau-like region and the stress more or less

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risers with the strain. For brittle materials, the stress-strain curve is rather rugged in this region, but the average stress is almost constant, giving a long flat plateau.

The form of the plateau-like part of the stress-strain curve depends also on whether the cells are open or closed. Open-cell foams collapse at almost constant load, providing a long flat plateau. In closed cell foam and cellular solids, the membrane stresses appearing in the cell faces, together with the compression of the gas within the cells, provide the stress-strain curve that increases with the strain. In both cases it can be said that the plateau-like regime is the longer - i. e. one it extends to the higher total strain - the lower the foam densities are.

In the regime of densification, the cell walls touch each other. Consequently, the further strain compresses the solid itself, providing the final region of rapidly increasing stress as typical for solid materials.

However, an observable deflection in the static compression test is practically impossible without plastic deformation of a foam or cellular material also occurring. Therefore, the elastic properties should be investigated by using other techniques. It is generally expected that the dynamic resonant method [4] represents one of the possible procedures. It is based on measurement of resonance frequencies of small flexural vibrations instead of relatively large (to be observable) static deflections. These vibrations can easily be kept within the elastic range even for foams.

In this work, both preliminary static compression tests and measurements of resonance frequencies on samples prepared by sintering from Cu-Sn hollow spheres were performed. As regards the compression tests, the experimentally determined curves show all the above mentioned characteristic features and favour the conclusion that the samples tested are closed-cell ductile cellular solids. The experimentally measured values of resonance frequencies were used for evaluating the effective flexural moduli of the materials being tested. The results concerning the values of the effective flexural modulus for particular materials are at least qualitatively in good agreement with the results of static compression tests.

EXPERIMENTAL

The samples from bronze hollow spheres were prepared as follows [5]: As a starting material, spherical iron powder was produced from iron oxide by reduction in flowing hydrogen for some hrs at 800 °C. This powder was screened, and the fraction between 700 and 500 mm was used. Deposition of copper on the spherical particles was done by cementation, and even complete replacement of Fe by Cu took place. Since Cu is deposited at the surface while Fe is dissolved in the core, hollow particles were obtained. These particles were very weak and difficult to handle, therefore a consolidating heat treatment at 800 °C in H₂ atmosphere

was done. This caused reduction of the oxides and some sintering within the shell, resulting in mechanically reliable particles that could be handled better.

The heat-treated Cu hollow spheres were tin coated in aqueous solution. In a single step process, maximum Sn contents of 5 mass % can be introduced. If more Sn is to be added, an intermediate anneal at 700 °C in H₂ has to be done; afterwards a second deposition run is possible by which a Sn content of 10 mass % can be attained.

The Sn coated Cu hollow spheres were tapped into ceramic moulds and gravity sintered for 3 hrs in hydrogen. The sintering temperature has to be carefully selected in accordance to the Sn content. For the Cu-5% Sn materials the selected temperature was 900 °C, for the Cu-10 % Sn 800 °C.

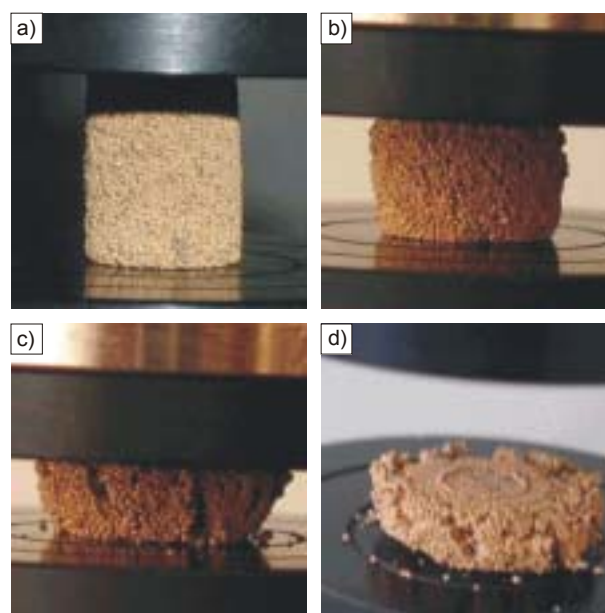


Figure 1. Cylinder gravity sintered from Cu-10%Sn hollow particles at various stages of compressive deformation. Deformation increases from a) to d)

Slika 1. Gravitacijski sinterovani cilindar iz šupljih čestica Cu-10 % Sn pri raznim stanjima deformacije kompresijom. Deformacija se povećava od a) do d)

For a static compression test, an INSTRON universal testing machine was used. Ram speed was 0.2 mm/s. The samples were cylindrical, with the diameter 1.516 cm, height 1.940 cm and density 2.14 g·cm⁻³ for the Cu-5 % Sn material and 1.606 cm, 2.092 cm, 1.73 g·cm⁻³ for the Cu-10 % Sn material.

For the resonance frequency measurements, rod-shaped samples with a trapezoidal cross section were used. The trapezoidal cross section was the consequence of the fact that it was impossible to shape the sintered samples without significant plastic deformation or even destroying the specimen. So the samples had to be used in the shape as sintered.

The frequencies of flexural vibrations were measured at the Institute of Materials Physics, University of Vienna, by means of the equipment developed at this Institute [6].

DISCUSSION

During compressive deformation, originally cylindrical samples (Figure 1.a) gained a characteristic barrel shape (Figure 1.b) due to frictional forces between sample bases and pads that prevent the transverse deformation of the sample bases. In the final stage, radial cracks (as typical for compression test) appeared that spread from the perimeter to the centre of sample (Figure 1.c, d), but the samples still remained in one piece.

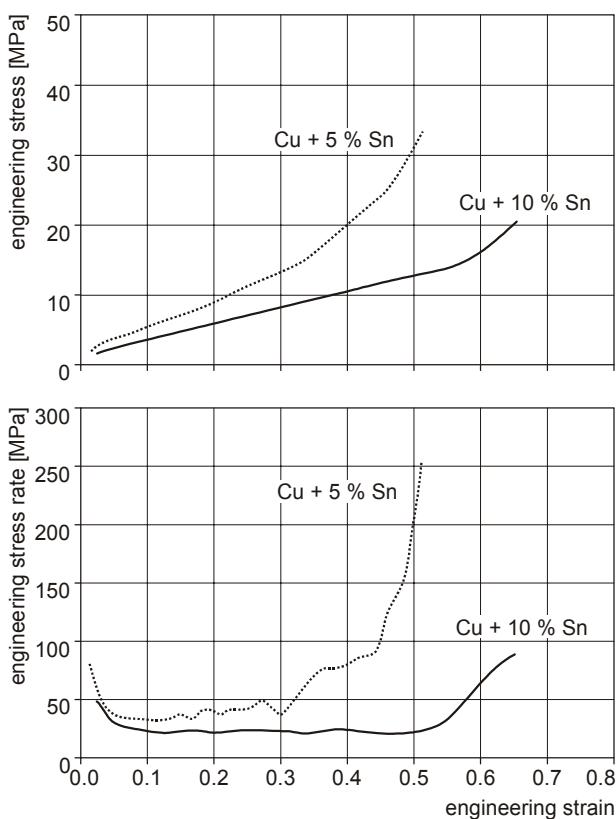


Figure 2. **Compressive stress-strain and stress rate ($d\sigma/d\varepsilon$) - strain curves for cellular samples made of gravity sintered Cu-5%Sn and Cu-10%Sn hollow spheres. The relative densities of the samples were 0.24 for Cu-5%Sn and 0.20 for Cu-10%Sn**

Slika 2. **Krivulje kompresije naprezanje - deformacija i stupanj naprezanja ($d\sigma/d\varepsilon$) - krivulje deformacije celularnih uzoraka napravljenih gravitacijskim sinterovanjem šupljih kugli od Cu-5 % Sn. Relativna gustoća uzoraka je 0.24 za Cu-5 % Sn i 0.20 za Cu-10 % Sn**

The compressive stress-strain curves obtained for these samples are presented in Figure 2.a. It can be seen that the curves are smooth, and there is no horizontal or very flat plateau-like region. Within the plateau-like region the stress increases with strain.

To gain more detailed information about the behaviour of the stress-strain curve, its derivative $d\sigma/d\varepsilon$ was investigated (Figure 2.b), that is, the rate of the change of stress with increasing strain. The curves revealed that the rates are smooth functions of the strain in the first (quasi-elastic) and the third (densification) regions, while in the intermediate (plateau-like) region the rate is a waved function of strain. This means that although our stress vs. strain dependences look quite smooth in the plateau-like region, in reality they are slightly waved functions, though not so rugged as they are for a typical foam.

There are several reasons (or their combinations) for such behaviour of a stress-strain curve (see Introduction): higher relative density of the samples, closed-cell character of the solid, ductile material of the cell walls, etc.

Investigation by means of SEM revealed that the structure of undeformed samples was rather "loose" (Figure 3.a), that is, the hollow spheres were not packed very tightly and there was a lot of free space between them. As the sample is compressed, hollow spheres are pushed close to each other, and the free space among them is reduced (Figure 3.b). Hollow spheres are also slightly deformed, but they are not broken.

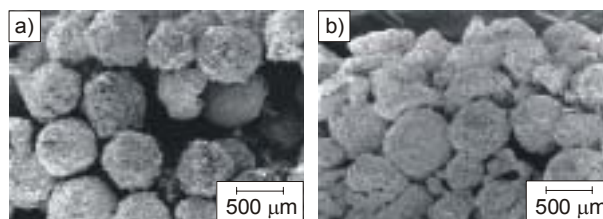


Figure 3. **SEM photographs of the sample made of Cu-10%Sn material before (a) and after (b) deformation**
Slika 3. **SEM fotografije uzoraka napravljene od materijala Cu-10 % Sn prije (a) i poslije (b) deformiranja**

The values of derivative $d\sigma/d\varepsilon$ at the limit of low strains could indicate something about the values of the effective modulus of elasticity. So, from Fig. 2b it follows that the value of the effective modulus for the material Cu-5%Sn could be higher than that for the material Cu-10%Sn. Of course, this conclusion is only approximative as a certain amount of plastic deformation of the sample occurs in the whole range of strains measured. Furthermore, the relative density of Cu-5 % Sn material (0.24) is higher than that of Cu-10 % Sn material (0.20).

It was intended to determine the values of the effective modulus by means of the dynamic resonant method [4]. Unfortunately, although the resonance frequencies of the fundamental flexural mode were measured with sufficient accuracy, the shape nonuniformity of the samples prevented evaluation of more accurate values for the effective flexural modulus.

The rod-shaped specimens possessed the cross sections of the form of an isosceles trapezoid, but the particular

cross-sectional dimensions slightly varied along the rod length. Nevertheless, the mean values of cross-sectional dimensions were used to obtain at least approximative values for the moduli required.

As the rod cross section resembles an isosceles trapezoid, there are in general two different fundamental flexural modes with two different frequencies: vibration with bending in the longitudinal plane of symmetry and vibration with bending in the plane perpendicular to the former. For long slender quasihomogeneous rods the ratio of frequencies of these two modes depends only on the shape and dimensions of the cross section and is independent of the material Young's modulus. So, this frequency ratio was calculated for each our test rod and compared to the ratio of frequencies measured for a given rod. For subsequent evaluating the modulus values only these rods were used for which the both ratios were equal or nearly equal. Consequently, the following values for the effective flexural modulus were obtained: $E = 1.014 \pm 0.009$ GPa for material Cu-5 % Sn (relative density 0.23), and $E = 0.668 \pm 0.103$ GPa for material Cu-10 % Sn (relative density 0.22).

CONCLUSIONS

So, taking into account our experimental data, it can be concluded that the principal mode of plastic deformation of our samples is the removal of in general open porosity among metallic hollow spheres by pushing the

spheres more closely together, which is accompanied by a slight plastic deformation of the spheres themselves, too.

As regards the elastic properties, both static compression test and dynamic resonant method suggest that our Cu-5 % Sn material possesses a higher stiffness than the Cu-10 % Sn material. While in the case of compression tests this difference could be ascribed to the different relative densities of the materials tested, the materials of the test rods used in the dynamic resonant method have nearly the same relative density. So, the stiffness differences are probably caused by the cell-wall material itself.

To understand how the properties are affected by such parameters as relative density, shape and size of cells, composition of cell walls, etc., more detailed measurements on series of samples with varying parameters and more sophisticated evaluating procedures are needed. This will be the goal of further investigations.

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