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Determination of the effective Young‘s modulus of cellular materials from hollow bronze spheres by means of dynamic resonant method

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Abstract Cellular materials from hollow bronze spheres were prepared and investigated. The main aim was to determine the effective Young's modulus of cellular materials by means of the dynamic resonant method, and the next goal was to demonstrate the possibility of using sandwich samples for such determination. For this purpose, two kinds of samples were prepared: quasihomogeneous rods consisting entirely of a cellular material, and sandwich rods containing the cellular material only in a core. Resonant frequencies of the fundamental flexural modes of all these samples were measured, and corresponding flexural rigidities were evaluated. Obtained values of flexural rigidities were subsequently utilized for calculation of the effective Young's modulus of cellular material. A reasonable agreement between the modulus values determined by means of quasihomogeneous and sandwich samples was obtained. This indicates that the sandwich samples could be used for measuring the material properties of particular layers.

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Introduction

Cellular solid is basically an assembly of cells with solid edges and/or faces packed together so that they fill the space [1]. These loose structures are developed, improved and investigated within the search for new lightweight structural materials.

At the present times, cellular construction units are mainly applied in acoustic and thermal management, they are utilized as energy-absorbing parts, filters, etc. Nevertheless, even when nowadays the primary use of cellular components is not mechanical, knowledge of mechanical properties is important for engineering analysis of particular applications. Necessary mechanical parameters are determined primarily by means of methods based on compression and flexure, as the man-made cellular solids cannot usually withstand high tensile forces. This is a consequence of the fact that a large pore volume minimizes load-bearing cross sections of the cellular body.

Elastic properties can be conveniently evaluated by means of a dynamic resonant method [2]. This is based on measurements of resonant frequencies of small vibrations, in contrast to static methods which deal with relatively large deflections. The amplitude of vibrations can be kept within the elastic range even for cellular materials.

In the contribution presented, the effort is focused on determining the values of effective elastic modulus of cellular materials sintered from hollow Cu–Sn spheres (the spheres were produced under another programme aimed at production of bronze filters). For this purpose, flexural vibrations of both quasi-homogeneous and sandwich-like specimens were used.

Quasihomogeneous specimens were made entirely of cellular materials. But serious difficulties appeared to be associated with the measurement of the effective Young's modulus of these samples. First, it was hard or even impossible to shape sintered samples into a suitable form. So it was necessary to use rods in the shape as sintered. In addition, when the quasihomogeneous cellular rods underwent flexural vibration, they possessed quite high damping and weak inertia. This adversely affected the quality factor, Q, of corresponding rods. The value of Q decreases with increasing damping (internal friction) and/or decreasing inertia (mass of the rod per unit length).

The rod with a large value of Q resonates at nearly the natural frequency and the resonance is fairly narrow. As the quality factor decreases, resonant frequency is more and more shifted away from the natural frequency, and the resonance broadening increases [3]. Then it becomes more difficult (or even impossible) to determine accurately the value of natural frequency from the shape of measured resonant curves.

So, to obtain reliable results by means of a dynamic resonant method, rods with a high value of Q are desirable. Therefore, to improve the quality factor, rods with a sandwich structure were also prepared and investigated. Sandwich rod possesses a higher mass per unit length and lower apparent damping as a loose cellular material occurs only in a core layer around the rod centre.

The sandwich geometry provides an additional benefit. When a rod undergoes flexural vibration, maximum compressive/tensile stress values occur near rod surfaces which are perpendicular to the bending plane. Near the rod centre the stress is much smaller [4, 5]. Hence, when the cellular material is situated only near the sandwich rod centre, it is better protected against plastic deformation.

On the other hand, the interpretation of results and evaluation of required material properties are less straightforward for sandwich samples. Usually, more sophisticated mathematical treatment of experimental data is necessary [6, 7]. Moreover, the production of sandwich samples is more laborious than manufacturing the quasihomogeneous ones.

Nevertheless, the values of effective Young's modulus of investigated cellular materials were obtained by means of both quasihomogeneous and sandwich samples. A reasonable agreement between the values was obtained. This indicates the applicability of sandwich-like rods for determining the material properties of particular layers.

Experimental

The cellular materials from hollow bronze spheres were prepared by means of the following procedure [8].

As a precursor, hollow particles consisting of Fe oxides were used. Precursor particles represent a waste that arises during industrial production of wires. The oxidic precursor particles were reduced to metallic iron in flowing hydrogen for some hours at 800 °C. Resultant hollow iron spheres were relatively thick-walled ones. The wall thickness was usually about 1/3 of a sphere diameter or more. Nevertheless, such thick-walled spheres were also used for preparation of cellular samples. Experiments and results for those iron-based cellular materials are presented in paper [9].

Cellular solids are attractive largely due to their very low relative density, that is due to a large pore volume and a small cell-wall thickness. But it turned out to be very complicated to thin the walls of above mentioned iron spheres without destroying the spheres themselves. More suitable way how to obtain thin-walled hollow spheres (and consequently less dense cellular materials) was to prepare new bronze spheres by means of old thick-walled iron ones. This contribution is intent on such bronze-based cellular materials.

Above mentioned iron powder was screened, and the fraction between 500 and 700 μm was further used. The electroless deposition of copper on iron 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. Unfortunately, these particles were very weak and difficult to handle. Therefore a consolidating heat treatment at 800 °C in H_2 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.

To obtain an intended bronze material, the heat treated hollow Cu 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 H_2 has to be done; afterwards a second deposition run is possible by which a Sn content of 10 mass % can be attained.

Quasihomogeneous rod-shaped samples were prepared as follows. The Sn coated Cu hollow spheres were tapped into ceramic moulds and gravity sintered for 3 h 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 ^{degr}C, for the Cu-10%Sn 800 $^{\circ}$C. The morphology of sintered material and its cellular character are shown in Fig. 1.

It was impossible to shape sintered cellular samples without significant plastic deformation or even destroying the specimen. So the quasihomogeneous samples had to be used in the shape as sintered. Consequently rod-shaped samples with a trapezoidal cross section were utilized, with dimensions slightly varying rod by rod. The rod length ranged from 99 to 101 mm, height from 9 to 11 mm, and maximum width from 9.5 to 11.5 mm.

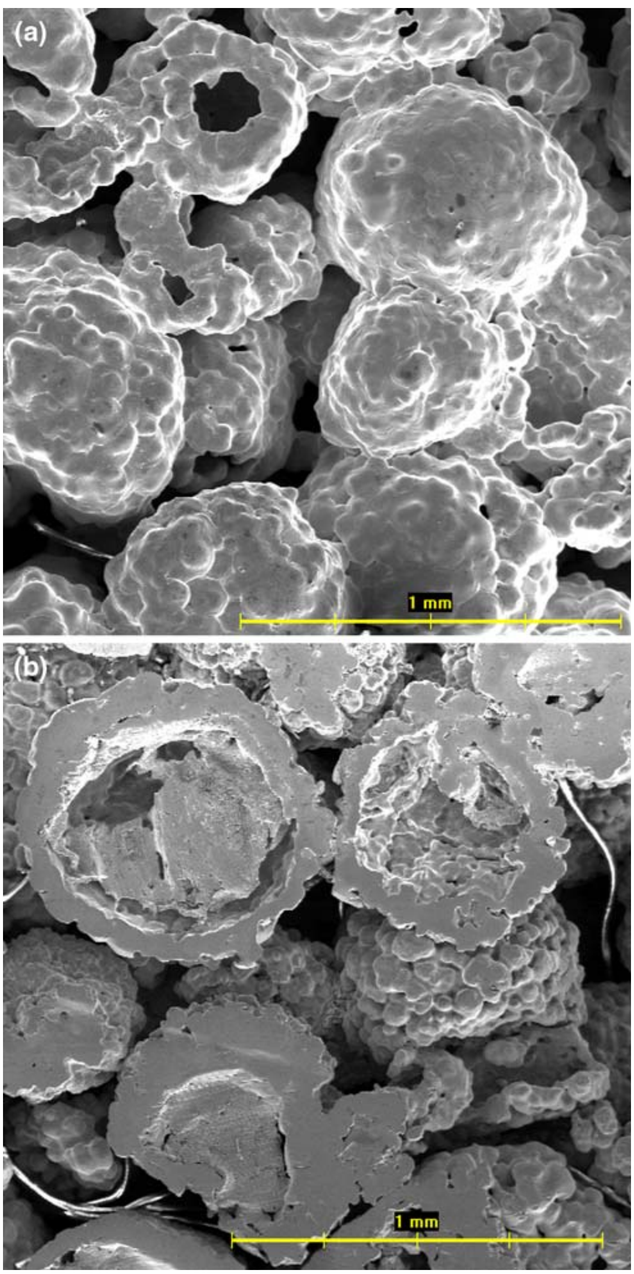

Fig. 1 Morphology of a surface (**a**) and a section (**b**) of the sample sintered from hollow Cu-10%Sn bronze spheres (SEM)

The sandwich bar-shaped specimen was also prepared (Fig. 2). The outer layers—two identical bars made of bronze powder (Eckart 90/10)—were prepared separately. Hollow bronze particles (Cu-10%Sn) were placed between completed outer layers. These three layers were then tapped into ceramic moulds and gravity sintered at the temperature 800 $^{\circ}$ C for 3 h in hydrogen atmosphere. The sintered sample was finally shaped into a uniform rod of rectangular cross section, with the length 100 mm, height 12 mm and width 10 mm.

A slide gauge was used for measuring the length of rods, while cross-sectional dimensions were measured by means of a micrometer screw gauge.

Geometric characteristics of particular layers were determined by means of light micrographs of polished rod cross sections and side faces. The obtained mean thickness of the cellular-material layer was 3 mm.

The dynamic resonant method [2] was used for determining the flexural rigidity of prepared rod specimens. Needed frequencies of flexural vibrations were measured at the Institute of Materials Physics, University of Vienna, by means of the equipment developed at this Institute [10].

Theory

A true material property does not depend on the specimen geometry or the method of testing, whereas most measured properties of sintered samples do. Therefore, to be able to interpret the experimental results correctly and to extract a useful particular information from overall data, it is necessary to possess a mathematical relationship between the apparent material property and microstructure, composition and geometry of the sample being tested.

As for the present the dynamic resonant method employing flexural vibrations is utilized as a testing

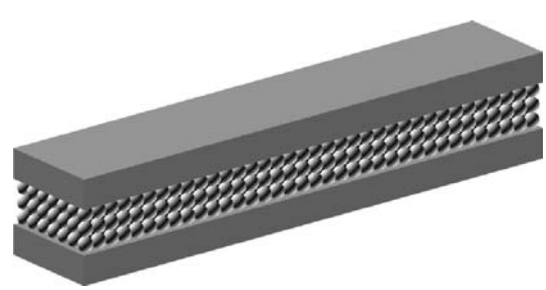

Fig. 2 Schematic sketch of a sandwich beam

method, bending properties of rod-shaped samples and the relations of these properties to the composition, microstructure and internal geometry of samples are just the objects of interest.

To get an overall view of the macroscopic behaviour of cellular samples undergoing flexure and vibrations, a number of model rods with various porosity distributions were investigated numerically (e.g., [11, 12]). With respect to the problem of interest, only a static bending and a few lowest modes of flexural vibrations were considered. It turned out that the mechanical behaviour of ''real'' porous samples was equivalent at the macroscopic level to the mechanical behaviour of a hypothetical rod made of an effective homogeneous material. This equivalence holds even for a quite microheterogeneous distribution of porosity, and is broken only when the characteristic length of fluctuation in material properties becomes comparable to the rod dimensions. That is, only macroscopically heterogeneous rods behave differently from effective homogeneous ones in situations considered [12].

Dimensions of hollow spheres are small compared with rod dimensions, especially with the rod length. So, the only inhomogenity on the scale of rod dimensions is that due to macroscopic layers. Therefore, sandwich rods with a cellular-material core as well as rods made entirely of cellular materials can be assumed as macroscopically equivalent to rods possessing the effective fibrous-like structure. That is, for purposes of theoretical analysis of a macroscopic response, real rods can be replaced by rods with effective material properties constant along the rod length and varying only across the rod.

The representative formula that connects the natural frequency f of a particular mode of flexural vibration with the flexural rigidity B for a rod uniform along its length reads

$$f = \frac{1}{2\pi}\left(\frac{n^4}{L^3 M}B\right)^{1/2}. \qquad (1)$$

Here L and M represent the length and mass of the rod, respectively. n is a proper root of the corresponding characteristic equation. For example, for a rod with free ends the characteristic equation reads $\cos n \cosh n = 1$, and its root for the fundamental mode is $n = 4.7300407$ [13].

The flexural rigidity of a homogeneous rod is proportional to the Young's modulus of material the rod is made of [13]. But the flexural rigidity of sintered rod is an overall ''sample-dependent'' quantity. It depends not only on volume fractions and properties of constituent materials separately, but also on the way in which the materials are combined to constitute corresponding sample.

Mathematical expressions relating the flexural rigidity of fibrous-like rods to their structure and composition, that is the theoretical basis necessary for the analysis of experimental results, were derived previously (e.g., [14]). Here only the basic ideas are briefly sketched and necessary results are presented.

For theoretical analysis, it is convenient to imagine the rod to be a bundle of longitudinal fibres parallel to the longitudinal axis of a rod. The term fibre means a line of effective-material particles. The material properties of each fibre are constant along its length, but the properties of individual fibres may differ from each other. For simplicity, it is supposed that fibres with the same properties are distributed symmetrically with respect to the longitudinal plane of symmetry (LPS). No slippage is assumed between any neighbouring fibres.

Within the slender beam theory, flexural rigidity equals the bending moment needed to produce a unit curvature of a bent rod [4]. So, the stress and displacement fields in a bent rod are to be found to evaluate relevant quantities. For a slender rod, an approximative theory can be used.

The approximative theory of stress–strain state within the bent slender rod is based on two hypotheses: hypothesis of planar cross sections (cross sections, which are plane and are perpendicular to the material fibres of the undeformed rod, remain plane and remain perpendicular to the material fibres of the bent rod), and Kirchhoff's static hypothesis (in a bent thin rod only internal stresses normal to the rod cross section are large).

Using the above mentioned hypotheses, and allowing for the nonuniform effective material properties throughout the cross section, the flexural rigidity of a fibrous-like rod was found to be (a slight modification of results from [14])

$$B_{hh} = \iint E(h,w)h^2 \mathrm{d}h\mathrm{d}w - \frac{\left(\iint E(h,w)h\mathrm{d}h\mathrm{d}w\right)^2}{\iint E(h,w)\mathrm{d}h\mathrm{d}w} \qquad (2)$$

for bending in the longitudinal plane of symmetry (LPS), and

$$B_{ww} = \iint E(h,w)w^2 \mathrm{d}h\mathrm{d}w - \frac{\left(\iint E(h,w)w\mathrm{d}h\mathrm{d}w\right)^2}{\iint E(h,w)\mathrm{d}h\mathrm{d}w} \qquad (3)$$

for bending in the plane perpendicular to LPS. h-axis is parallel to the height of the rod cross section and at the same time parallel to LPS, w-axis is parallel to the width of the cross section. The origin of coordinates can be positioned arbitrarily (Fig. 3), as the expressions are invariant under the displacement of origin of coordinates. $E(h,w)$ represents the effective material Young's modulus that can vary only along the height and width of the rod, but not along the length of the rod. Integration runs over the area of the cross section.

''Geometric'' properties of the cross section are characterized by means of the area moment of inertia about the proper centroidal principal axis of inertia of the cross section, J_{hh} and J_{ww}. Corresponding expressions for J_{hh} and J_{ww} are

$$J_{hh} = \iint h^2 \mathrm{d}h\mathrm{d}w - \frac{\left(\iint h\mathrm{d}h\mathrm{d}w\right)^2}{\iint \mathrm{d}h\mathrm{d}w}, \tag{4}$$

$$J_{ww} = \iint w^2 \mathrm{d}h\mathrm{d}w - \frac{\left(\iint w\mathrm{d}h\mathrm{d}w\right)^2}{\iint \mathrm{d}h\mathrm{d}w}. \tag{5}$$

Quantity B_{hh}/J_{hh} (and B_{ww}/J_{ww}) is often termed as ''the effective flexural modulus''. For a homogeneous rod $B_{hh}/J_{hh} = B_{ww}/J_{ww} = E$, where E is the Young's modulus of the material of the rod. But for a quasilayered rod, B_{hh}/J_{hh} and B_{ww}/J_{ww} differ from each other. In general, the difference in ''effective flexural moduli'' indicates the inhomogenity of the rod being tested.

For a symmetric rectangular three-layer rod, expressions (2)–(5) provide the relations [6]

$$\frac{B_\perp}{J_\perp} = E_\mathrm{s} + (E_\mathrm{c} - E_\mathrm{s})\left(\frac{V_\mathrm{c}}{V}\right)^3 \tag{6}$$

$$\frac{B_{||}}{J_{||}} = E_\mathrm{s} + (E_\mathrm{c} - E_\mathrm{s})\frac{V_\mathrm{c}}{V}, \tag{7}$$

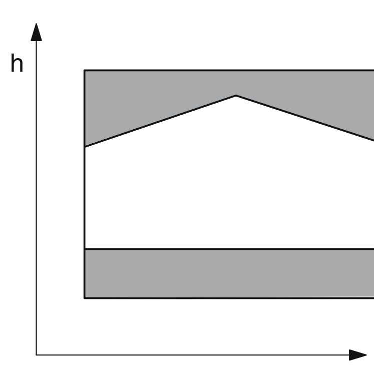

Fig. 3 Schematic sketch of a cross section of a fibrous-like rod, and orientation of coordinate axes

Symbol $\perp$ is for bending in the plane perpendicular to layers, $\|$ for bending in the plane parallel to layers (Fig. 4). E_s and E_c are values of Young's modulus of material of outer (surface) and central layers, respectively. V_c represents the volume of central layer, V is the volume of the entire rod.

So, when the quantities $B_\perp / J_\perp \equiv E_\perp$ and $B_{||}/J_{||} \equiv E_{||}$ are experimentally determined out, Eqs. 6 and 7 can be inverted. Then the Young's modulus of central-layer material can be expressed as

$$E_\mathrm{c} = E_{||} + \frac{E_{||} - E_\perp}{v_\mathrm{c}(1 + v_\mathrm{c})}, \tag{8}$$

when the volume fraction $v_\mathrm{c} \equiv V_\mathrm{c}/V$ of central layer is known, or as

$$E_\mathrm{c} = E_\mathrm{s} + \mathrm{sign}\left(E_{||} - E_\perp\right)\frac{E_{||} - E_\mathrm{s}}{E_\perp - E_\mathrm{s}}\sqrt{(E_{||} - E_\mathrm{s})(E_\perp - E_\mathrm{s})}, \tag{9}$$

when the Young's modulus of surface layer is known.

Accordingly, practical application of the dynamic resonant method consisted of the following steps. First, natural frequencies f of corresponding fundamental flexural modes of test rods were measured. Really two modes were relevant for both quasihomogeneous and sandwich samples: flexural vibrations parallel and perpendicular to the longitudinal plane of symmetry. After the measurement of natural frequencies had been completed, the overall mass M, length L and cross-sectional dimensions of the rod considered were determined. Then the flexural rigidity B was calculated

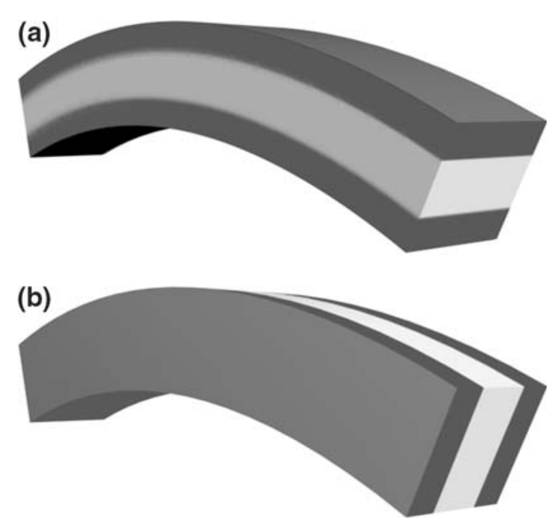

Fig. 4 Shape function of the fundamental flexural mode of a three-layer rod with bending (vibrations) in a plane perpendicular (**a**) and parallel (**b**) to layers (schematic sketch)

by means of Eq. 1 and the area moment of inertia J by means of Eqs. 4 and 5. For a rod of trapezoidal cross section with the height H and bases W_1 and W_2 (Fig. 5a) the relevant moments are $J_{\text{edgewise}} = H^3(W_1^2 + 4W_1 W_2 + W_2^2)/(36W_1 + 36W_2)$ and $J_{\text{flatwise}} = H(W_1 + W_2)(W_1^2 + W_2^2)/48$. For a rod of rectangular cross section of the height H and width W (Fig. 5b) the relevant moments are $J_{\perp} = H^3W/12$ and $J_{||} = HW^3/12$. Consequently, the values of "the effective flexural modulus" B/J were evaluated. For proper samples made entirely of cellular material this represented the final step, as in this case $B/J$is the cellular-material effective Young's modulus. But for sandwich rods effective flexural moduli were further mathematically processed by means of Eq. 8 to determine the effective Young's modulus of central cellular layer.

Results and discussion

As already mentioned, two tasks were undertaken: firstly, to determine the values of effective Young's modulus of bronze cellular materials by means of the dynamic resonant method [2], and secondly, to prove the applicability of a sandwich-like rod for such purpose. Therefore, both quasihomogeneous and sandwich rods with a cellular-material core were prepared and tested.

Prepared quasihomogeneous rod-shaped specimens possessed trapezoidal cross sections. But in spite of the considerable effort for a shape uniformity, particular cross-sectional dimensions slightly varied along the rod length. This shape nonuniformity of quasihomogeneous samples restrained from evaluation of more accurate values for the effective Young's modulus, regardless of the fact that resonant frequencies of the fundamental flexural mode were measured with a sufficient accuracy. Nevertheless, the mean values of cross-sectional dimensions were used to obtain at least approximative values for the moduli required.

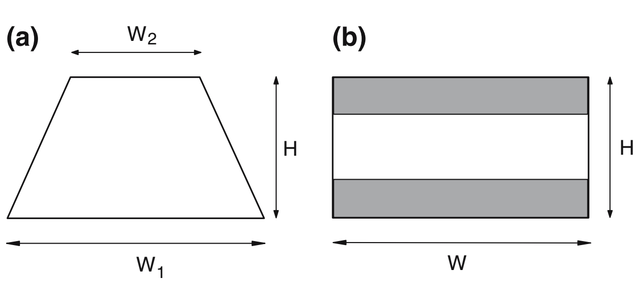

Fig. 5 Schematic sketch of a trapezoidal cross section of quasihomogeneous rods (**a**), and a rectangular cross section of a sandwich rod (**b**)

As the rod cross section resembled a symmetric trapezoid, there were in general two different fundamental flexural modes: vibration with bending in the longitudinal plane of symmetry and vibration with bending in the plane perpendicular to the former. These two modes possessed two different natural frequencies. Under ideal circumstances, when the Eqs. 1–3 hold true, the ratio of frequencies of these two modes reads

$$\frac{f_{\text{edgewise}}}{f_{\text{flatwise}}} = \frac{2H}{W_1 + W_2}\left[1 - \frac{2}{3}\frac{(W_1 - W_2)^2}{W_1^2 + W_2^2}\right]^{1/2}. \qquad (10)$$

Here H is the height, W_1 and W_2 are bases of the trapezoidal cross section (Fig. 5a). The frequency ratio (10) depends only on the shape and size of the cross section, and is independent of the material Young's modulus. So this frequency ratio was calculated for each of our test rods by means of its cross-sectional dimensions, and compared to the ratio of resonant frequencies really measured for a given rod. Only the rods with coinciding or nearly coinciding theoretical and experimental frequency ratios were accepted as sufficiently shape uniform and were subsequently used for evaluating modulus values.

Consequently, the following values of effective Young's modulus were obtained by means of quasihomogeneous samples:

(i) for material Cu-5%Sn with relative density 0.23, the value of effective Young's modulus was $E = 1.014 \pm 0.009$ GPa,
(ii) and for material Cu-10%Sn with relative density 0.22 the effective Young's modulus was $E = 0.668 \pm 0.103$ GPa.

The elastic moduli mentioned above were calculated by means of several independent variables. So, the standard deviations for the effective Young's modulus were evaluated by means of standard deviations for directly measured quantities (such as natural frequencies, mass and overall dimensions of rod, thickness of particular layers, etc.) in accordance with formula that was used for calculation of particular modulus.

As regards the sandwich rod, for technological reasons measurements were carried out only on one sample with the core of Cu-10%Sn material. Measured values of the "effective flexural modulus" $B_{\perp}/J_{\perp}$ were higher than $B_{||}/J_{||}$. This is in accordance with the theory [6] for a three-layer sample with a cellular core and ordinary-material surfaces, that is, for a sample with outer layers stiffer than the core.

Table 1 Type of test sample, corresponding relative density and effective Young's modulus of cellular materials studied

Cellular material	Type of sample	Relative density	Effective Young's modulus [GPa]
Cu-5 mass %Sn	Rod	0.23	1.014 ± 0.009
Cu-10 mass %Sn	Rod	0.22	0.668 ± 0.103
Cu-10 mass %Sn	Core of sandwich rod	0.22	0.738 ± 0.018

Using relation (8), the following value of effective Young's modulus of Cu-10%Sn cellular material was obtained by means of the sandwich sample: $E_c = 0.738 \pm 0.018$ GPa. This value reasonably agrees with those obtained for quasihomogeneous Cu-10%Sn samples. The results for all bronze cellular materials are summarised in Table 1.

The applicability of sandwich rod for determining the modulus of particular layer was demonstrated only for one bronze sample. But this result is in accordance with those obtained previously for iron-based sandwich samples with core layers sintered from hollow ferrous particles. Using a number of such samples with various volume fractions of cellular material, the applicability of a ferrous sandwich for determining the modulus of core was proven in principle [9].

Conclusion

In this contribution, the results of determination of the effective Young's modulus of two kinds of cellular bronze materials are presented. Cellular materials were made of hollow Cu-5%Sn and Cu-10%Sn spheres. Two types of specimens were prepared and tested: quasihomogeneous ones made entirely of cellular materials, and sandwich sample with a cellular material only in a core.

As regards the elastic properties, dynamic resonant method revealed that our Cu-5%Sn material possesses a higher stiffness than the Cu-10%Sn material. As the materials of test rods used in experiments had nearly the same overall relative density, the stiffness differences are most probably caused by properties of the cell-wall material itself.

It was also demonstrated that when the dynamic resonant method is applied, the sandwich-like rod with a cellular-material core between two ordinary-material layers can be used for indirect measuring the effective Young's modulus of cellular material. In comparison with quasihomogeneous cellular samples, the use of sandwich specimen provides some merits: lower damping of vibration, lower internal stresses within the cellular material, better inertial properties and better shaping ability. On the other hand, there are also some disadvantages: preparation of sandwich samples is more laborious, and a more complicated procedure is needed for processing the experimental data. But in situations when the measurement on quasihomogeneous samples is practically impossible, a sandwich sample can represent a useful alternative regardless of the complications mentioned. However, to transform this conception into a practical method, further investigation is necessary, both theoretical and experimental.

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