Influence of nanostructuration on the sound velocity in copper Cu_99.75

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Abstract. The paper proposes is a multidisciplinary study on the influence of nanostructured material obtained by severe plastic deformation, in this case the copper with a purity of 99.75% (Cu_99.75), on the sound velocity. The study of nanomaterials is a branch of material science on the basis of which nanotechnology can be approached. He studies materials with morphological characteristics at the nanoscale, and especially those with special properties resulting from their nanometric dimensions. The nanometer scale is usually defined as less than one-tenth of a micrometer in at least one dimension, although this term is sometimes used for powders. Severe plastic deformation (SPD) is a generic term describing a group of metal and alloy processing techniques involving very high stresses without including significant changes in the overall dimensions of the model or workpiece. Another defining feature of severe plastic deformation techniques is that shape retention is achieved due to special geometries of the mold, involving the free flow of the material and thus producing significant hydrostatic pressure. Because the workpiece dimensions do not change during severe plastic deformation processing, the process can be repeatedly applied to impose very high stresses. The deformation process is discontinuous and comprises deformation processes defining a severe plastic deformation cycle. Samples were obtained by the cyclic closed die forging process, samples that were subjected to 12 deformation cycles. The sample is of a regular quadrangular prism shape with the side square of a = 10 mm and the height of h = 16 mm, so with a dimensional factor h / a = 1.6. The velocity of ultrasound waves propagation was determined by material based on two calibrated dimensions present on the sample, a and h. For each sample, a number of 7 determinations were performed to establish a mean value for the sound velocity. The variation of the sound velocity was plotted against the number of deformation cycles, a variation describing a convex line having a minimum in the area of the deformation cycle number 4. As a result of the microstructural analysis, it is observed that at the deformation cycle 4 the grains have an average size between 250 and 500 nm. On the basis of the above, it can be deduced that the area of passageways 3, 4 and 5 is, in fact, precisely the transition zone between micrometric granulation and mesoscopic (ultrafine) granulation, which is merely an intermediate zone between micrometric granulation and nanometric granulation.
1. Introduction
The paper proposes a multidisciplinary study on the influence of nanostructured material obtained by severe plastic deformation, in this case the copper with a purity of 99.75% (Cu\textsubscript{99.75}), on the sound velocity.

The study of nanomaterials is a branch of material science on the basis of which nanotechnology can be approached. He studies materials with morphological characteristics at the nanoscale, and especially those with special properties resulting from their nanometric dimensions. The nanometer scale is usually defined as less than one-tenth of a micrometer in at least one dimension, although this term is sometimes used for powders, [1-3].

Severe plastic deformation (SPD) is a generic term describing a group of metal and alloy processing techniques involving very high stresses without including significant changes in the overall dimensions of the model or workpiece, [4].

Another defining feature of severe plastic deformation techniques is that shape retention is achieved due to special geometries of the mold, involving the free flow of the material and thus producing significant hydrostatic pressure, [5,6]. Because the workpiece dimensions do not change during severe plastic deformation processing, the process can be repeatedly applied to impose very high stresses, [7-11].

2. Material and method
The deformation process is discontinuous and comprises deformation processes defining a severe plastic deformation cycle.

Figure 1 shows the phases of multiaxial forging at one passage of the parallelepiped blank.

![Diagram of multiaxial forging](image)

Figure 1. Stages of multiaxial forging for a single passage, [12]:

a) blank; b) blank deformation; c) deformed part.

in which: $h_0$ – initial height of the blank, $h_0=20\ mm$;
$L_0$ – initial length of the blank, $L_0=10\ mm$;
$l_0$ – initial width of the blank is kept constant throughout the deformation, $l_0=10\ mm$;
$h_i, h_2, \ldots, h_{n-1}$ – current height of the workpiece deformed values $h_i[mm] \in (10,20), \ i = 1, n-1$;
$L_i, L_2, \ldots, L_{n-1}$ – current length of the workpiece deformed values $L_i[mm] \in (10,20), \ i = 1, n-1$;
$h_n$ – height of the piece at the end of severe plastic deformation process, $h_n=10\ mm$;
$L_n$ – length of the piece at the end of severe plastic deformation process, $L_n=20\ mm$. 

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The samples were obtained by cyclic closed die forging process and they were subjected to 12 cycles of deformation. I used the device designed on the principle of A.K. Ghosh, (figure 2). It was mounted on a 750 kN hydraulic press, equipped with the Material Technology Laboratory of the Department of Materials Engineering and Industrial Safety of the Faculty of Materials Science and Engineering from "Gheorghe Asachi" Technical University of Iasi.

In order to determine the force in the severe plastic deformation process, a dynamic data acquisition system, consisting of a fully equipped master unit, a 1000 kN force transducer and a 0-100 mm displacement transducer was used.

The device for nanostructuring of metallic materials was conceived and realized on the principle of Ghosh, the blank and the piece having the same shapes and dimensions, figure 1.

The device according to figure 3 consists of an active plate (1), placed on a pressure plate (2), the assembly being guided on the inner surface of the support (3).

The blank (4) is centered in the recess of active plate over a counterpunch (5) placed in turn on a tablet pressure (6) positioned in the holder (3).

The deformation of workpiece is achieved by applying a force over the punch (7) moving with the given value of stroke limiter (8).

The deformation force applied is dynamically registered with an acquisition system, not shown, by means of a load cell (9) mounted between the holder (3) and a pressure plate (10). After deformation, the pressure plate (6), counterpunch (5) and limiter (7) are eliminated and the punch is pressed (7) using another stroke limiter until the extraction of the deformed workpiece. The workpiece is rotated by 90° vertical and 90° horizontal, and introduced into the initially described device in order for the next pass and so on until nanostructuring the blank material.
Figure 3. The constructive functional scheme of multiaxial forging device, [4].

The material is 99.75% pure copper, as shown in table 1, the metal composition was determined by the spectrometer GNR Lab 75/80 V.

Table 1. The chemical composition of the copper Cu_99.75.

| Element | Zn   | Pb   | Sn   | P    | Mn   | Fe   | Ni   |
|---------|------|------|------|------|------|------|------|
| Procent | 0.0254 | 0.0114 | 0.0007 | 0.0372 | 0.0000 | 0.0227 | 0.0046 |

| Element | As   | Al   | Co   | S    | Mg   | Si   | Cu   |
|---------|------|------|------|------|------|------|------|
| Procent | 0.1042 | 0.0174 | 0.0105 | 0.0047 | 0.0018 | 0.0036 | 99.754 |

We have used blank of parallelepiped shape with a square base of side $a=10$ mm and a height $h=16$ mm, thus the dimensional factor is $h/a=1.6$.

Figure 4. Sound rate measurement scheme.
The velocity of propagation of ultrasonic waves was determined by material based on two calibrated dimensions present on the sample.

3. Results and discussions
On each sample we performed 7 determinations and in table 2 we present the average values of the sound velocity, where:

- $N$ is the number of passes (deformation), [-];
- $L$ - length of the blank, [mm];
- $l$ - width of the blank, [mm];
- $V_{Cu_{99.75}}$ - the average of the sound velocity for Cu_99.75 samples, [m/s].

| $N$ [-] | $L$ [mm] | $l$ [mm] | $V_{Cu_{99.75}}$ [m/s] |
|---------|----------|----------|------------------------|
| 0       |          |          | 5620                   |
| 1       |          |          | 5350                   |
| 2       |          |          | 5120                   |
| 3       |          |          | 4730                   |
| 4       |          |          | 4530                   |
| 5       |          |          | 4820                   |
| 6       | 16       | 10       | 5030                   |
| 7       |          |          | 5320                   |
| 8       |          |          | 5550                   |
| 9       |          |          | 5680                   |
| 10      |          |          | 5800                   |
| 11      |          |          | 5840                   |
| 12      |          |          | 5850                   |

Based on the values in table 2, we have plotted the variation of the sound velocity according to the number of passes, figure 5.

![Figure 5. Variation of the sound speed according to the number of passes for copper Cu_99.75.](image)
4. Conclusions
The variation of the sound velocity was plotted against the number of deformation cycles, a variation describing a convex line having a minimum in the area of the deformation cycle number 4.

As a result of the microstructural analysis, it is observed that at the deformation cycle 4 the grains have an average size between 250 and 500 nm.

On the basis of the above, it can be deduced that the area of passes 3, 4 and 5 is, in fact, precisely the transition zone between micrometric granulation and mesoscopic (ultrafine) granulation, which is nothing but an intermediate area between micrometric granulation and nanometric granulation.

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