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INFULENCE OF RECRYSTALLIZATION ON DAMPING PROPERTIES OF ALLOY $Fe_{75}Zr_4Ti_3B_{17}Cu_1$

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Abstract:

It is expected that damping in amorphous metals change after its recrystallization. For this purpose study of this phenomena was investigated. An alloy $Fe_{75}Zr_4Ti_3B_{17}Cu_1$ was manufactured in a very specific conditions to obtain amorphous structure. To confirm structure of the manufactured material basic testing was conducted including XRD, AFM, LFM and SEM with EDS attachment. The study showed that the structure in fact is partially amorphous and partially nanocrystalline. To determine expected change of damping sample in form of rod was tested for bounce height in controlled conditions before and after heat treatment that changed materials structure. Obtained results indicate decrease of damping after heat treatment and therefore confirms initial assumption.

Keywords:

experimental mechanics, metallic glass, amorphous alloy, material properties, material science

INTRODUCTION

Internal structure of metal alloys has strong influence on their mechanical properties. It can be observed even during conventional heat treatment of metals [3, 4, 6]. Nowadays advanced technologies combined with specific metal alloys allow us to obtain amorphous structure, under condition cooling rate exceeds the limits for specific alloy [1]. Such structure should poses interesting mechanical properties [5]. Paper is con-

centrated on testing and determination of internal damping of newly obtained material that should be lower than in the case of material possessing typical crystalline structure. Experimental investigation was conducted for alloy $Fe_{75}Zr_4Ti_3B_{17}Cu_1$ possessing partially amorphous and nanocrystal-line structure, what is further discussed in chapter on basic testing. To present change of the properties of the material experiment was conducted two times for the same sample before and after heat treatment.

To determine internal structure of the material after its manufacturing it was necessary to conduct basic testing, only than it was possible to performed further studies of internal damping of the alloy before and after heat treatment.

1. MANUFACTURING

In the study metal rod with diameter of 3 mm was used. It was obtained with rapid overcooling method. In the first step it was necessary to measure required amount of every alloying element, what was very important because every element used in the alloy is influencing, in different matter, final form and properties of the material including its viscosity, strength, or the appearance of internal stresses induced by heat treatment. After weighing of all the components were put in the Arc Melter that allows melting of the elements with use of electric arc.

The method for production of the alloy is based on several melts of all alloying elements in specially prepared cooper form to ensure their full homogenization. The melting of the material takes place in a protective gas atmosphere. In this case argon was used. After alloy was homogenized through several melts it was put in prepared cooper ingot under which there was water cooled cooper form. After that material was again melted and in the moment of transition from solid to liquid state it was sucked to form placed beneath it. High speed at which alloy was sucked into form was used for obtainment of amorphous structure of the material through its rapid overcooling. Cooling speed was about 10^6 K/s. This lead to creation of sample that had shape of a cylindrical rod with diameter of 3 mm and length of 150 mm. In fig. 1 is presented picture of the sample.

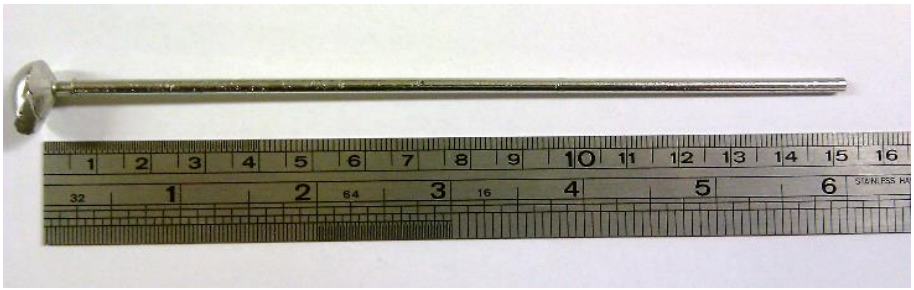


Fig. 1. Picture of the sample used for testing

Source: Own work

After damping testing sample was placed in an oven and heated for 30 minutes in 400°C what induced growth of crystals and therefore small change of internal structure of the material.

2. BASIC TESTING

Knowing the internal structure of the material before testing of internal damping in it was very important. It was necessary to per-form basic testing of the obtained alloy to determine its structure and what is even more important to check if it is amorphous. For this purpose X-ray diffraction (XRD) unit with cobalt lamp was used. Obtained results are presented in Fig. 2.

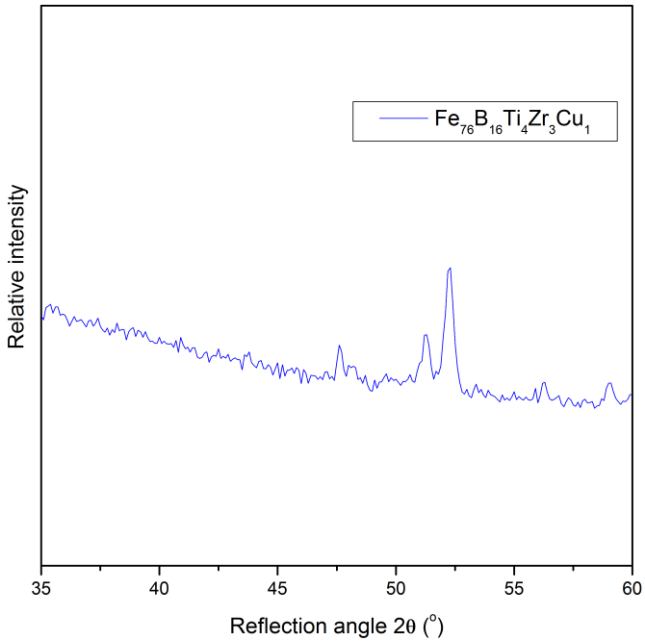


Fig. 2. X-ray diffractogram obtained for the sample

Source: Own work

It can be observe that presented diffractogram differs from typical diffractograms obtained for amorphous material [2]. In case of amorphous materials on the graph there should be no reflexes with distinctively high pick values. In most cases picks on diffractograms occur for relatively high angle range. Results obtained for produced sample indicate that this material have partially crystalline structure.

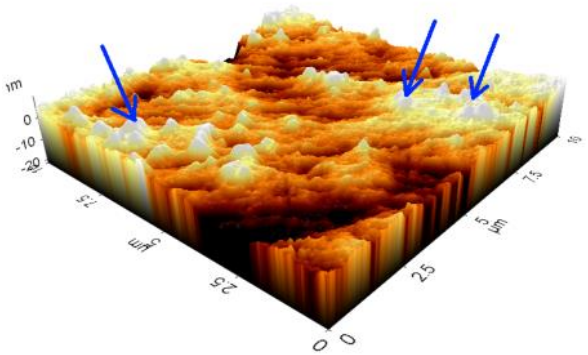


Fig. 3. 3D picture of the sample surface, scanned area had dimension of 10 x 10 µm, arrows point nanocrystaline structures

Source: Own work

To determine size of crystals sample was tested with use of Atomic Force Microscope (AFM). This test is based on scanning of a surface of the sample with use of very small and accurate probe to determine samples surface topography and therefore size of crystals. In Fig. 3 is presented result of sample surface scanning with dimensions of 10 x 10 µm in the contact mode. Arrows present in figure point nanocrystalline structures.

To verify occurrence of nanocrystalline phases in the material scanning using Lateral Force Microscopy was carried out. The principle of Lateral Force Microscopy (LFM) of the XE-series is very similar to that of contact mode of AFM. Whereas in contact mode we measure the deflection of the cantilever in the vertical direction to gather sample surface information, we measure the deflection of the cantilever in the horizontal direction in LFM. The lateral deflection of the cantilever is a result of the force applied to the cantilever when it moves horizontally across the sample surface, and the magnitude of this deflection is determined by the frictional coefficient, the topography of the sample surface, the direction of the cantilever movement, and the cantilever's lateral spring constant. Lateral Force Microscopy of the XE-series is very useful for studying a sample whose surface consists of inhomogeneous compounds. It is also used to enhance contrast at the edge of an abruptly changing slope of a sample surface, or at a boundary between different compounds. Fig. 4 present differences between scans performed with use of AFM and LFM.

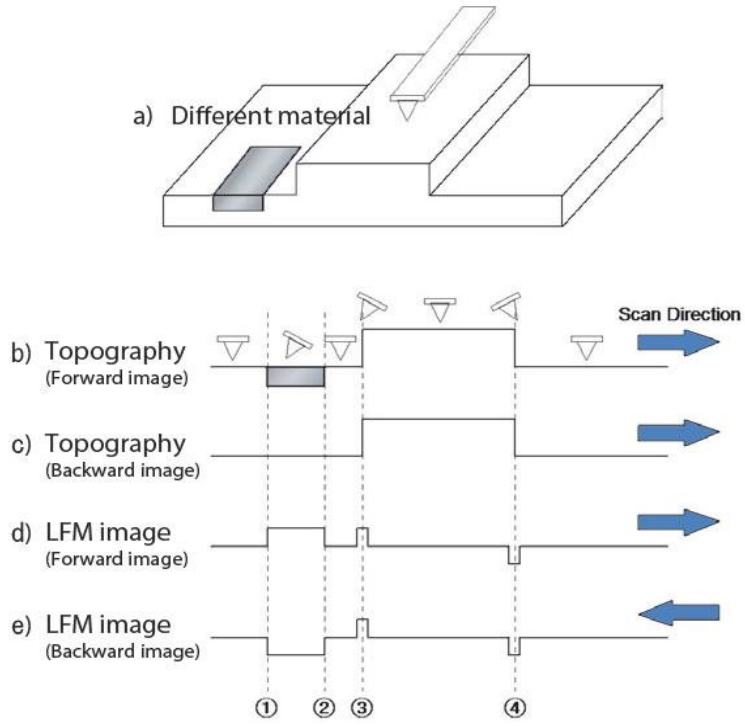


Fig. 4. Comparison of results obtained in case of scans performed with AFM and LFM

Source: Own work

Pictures obtained with LFM for the test sample are shown in Fig. 5. It is clearly seen that, in case of right direction movement of the scanning head, in some areas appeared darker sports pointed by arrows, however in case of left direction of movement

of the head are visible as lighter spots. It gives basis to indicate that in this material there are areas where occurred change of friction caused by emanation of one or more alloying elements or their incomplete homogenization, or the occurrence of nanocrystalline zones.

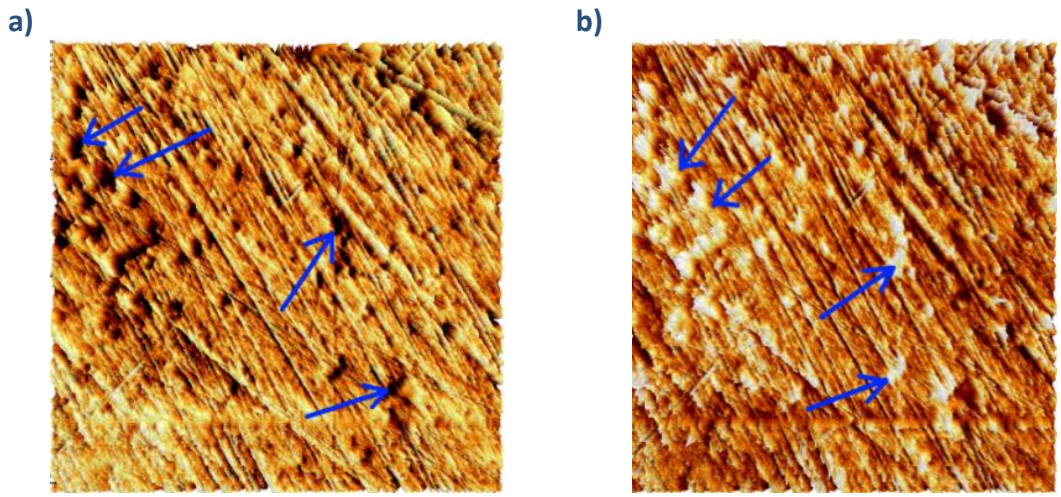


Fig. 5. Picture of the sample surface obtained from Lateral Force Microscopy, where: a) translation of the head right direction, b) translation of the head left direction

Source: Own work

To verify if located zones are nanocrystalline or emanation of alloying elements surface analysis with Scanning Electron Microscope (SEM) with an attachment for Energy dispersive X-ray spectroscopy (EDS) was performed. It allows fast analysis of occurrence and dispersion of elements in a material. Obtained results are presented in Fig. 6.

Pictures from microscope have confirmed appearance of the presence of structures with size in order of tens of nanometers, in the form of dark areas indicated in Fig. 6 with arrows. In addition, analysis of obtained surface scan with use of EDS has shown that alloying elements of the material are distributed uniformly. It indicates full homogenization of the elements during manufacturing process and gives basis to suspect that observed structures are nanocrystals in the structure of the material.

On base of obtained results it is determined that produced material poses structure that is partially amorphous and partially nanocrystalline.

3. DETERMINATION OF DAMPING PROPERTIES

To accurately determine damping properties of a metal rod with relatively small diameter it requires sophisticated measurement test stand. Therefore first it is important to decide if such testing will present any significant results. The simples idea on how to check if there is any change in damping value observer in a sample it to drop it from the same height and check how high it will bounce back. For this purpose a test stand for determination of damping in a rod sample was created. The test stand is presented in Fig. 7 where there was used high speed camera for recording of drop and bounce of the sample, glass tube (inner diameter of 4 mm) with scale and a holder that allowed vertical positioning of the tube. The tube was placed on flat surface that had very high

hardness and was not deflectable to assure the surface was the same during each bounce and for the same reason sample was dropped from approximately the same height.

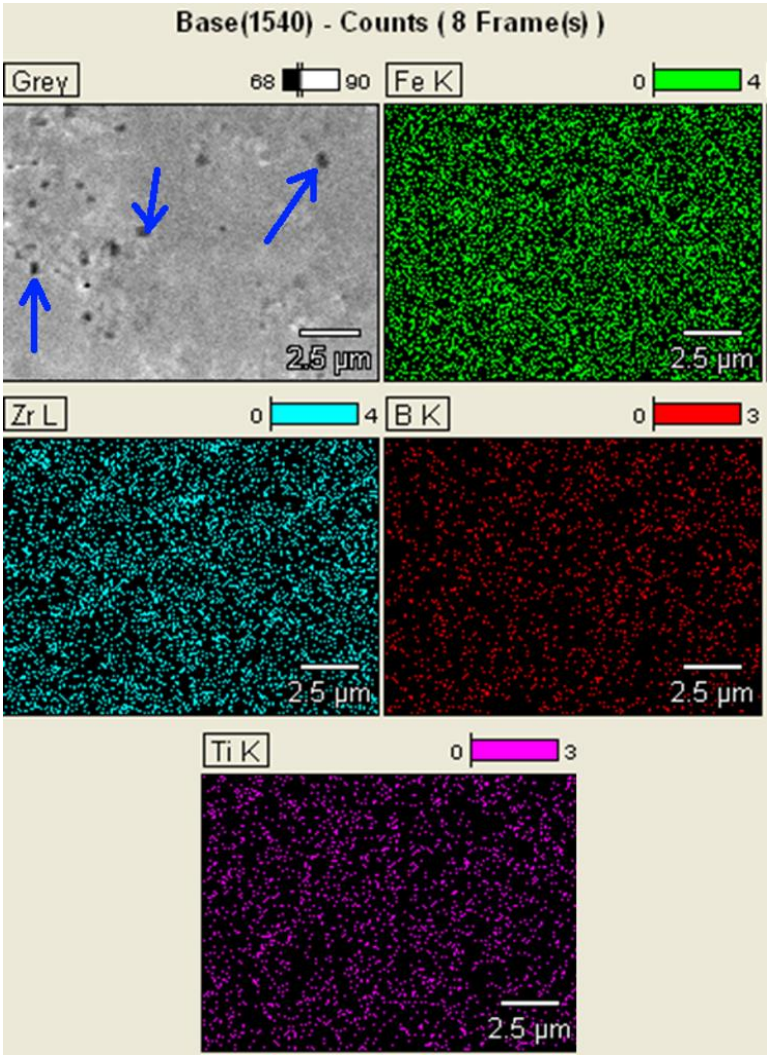


Fig. 6. Picture from Scanning Electron Microscope with magnification x8000 obtained after mapping with use for Energy dispersive X-ray spectroscopy

Source: Own work

The experiment was conducted several times for the sample before and after heat treatment to be able to get mean values and therefore be able to neglect friction between sample and walls of the glass tube. The recording speed of the camera was 300 frames per second what allowed accurate determination of the height of each bounce. In Fig. 8 and 9 are presented exemplary results showing sample in top position after first and second bounce in the tube. Single tests for comparison were chosen on base of their first bounce that was up to almost the same height in the tube, therefore it is possible to easily compare the results.

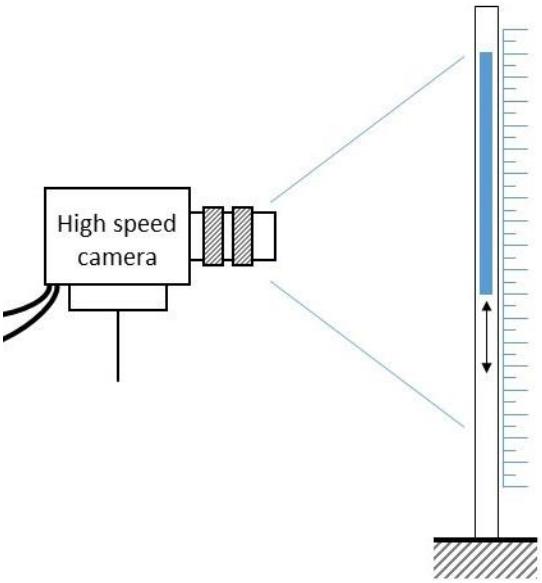
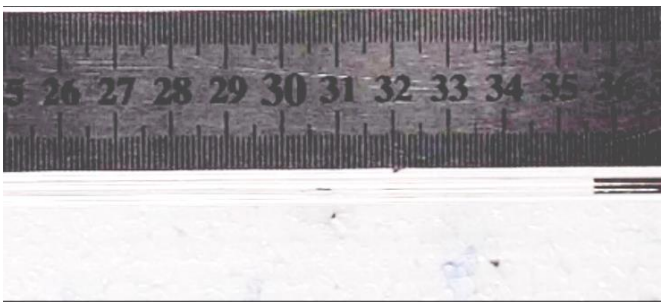


Fig. 7. Scheme of the measurement stand with high speed camera, tube with measuring scale and the sample

Source: Own work

The measurements of the bounce height were done by going frame after frame in the recording of the test and reading the top height from the scale. Resolution of the scale was 1 mm therefore values obtained during this test can be considered only as a reference for further testing.

a)



b)

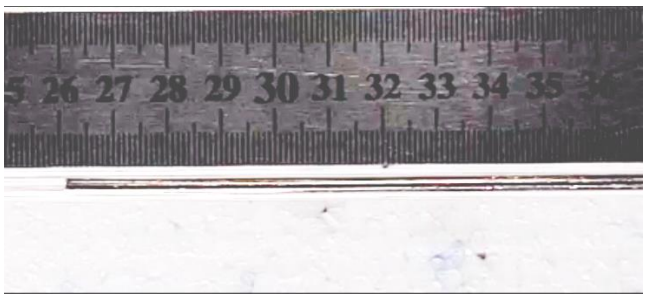
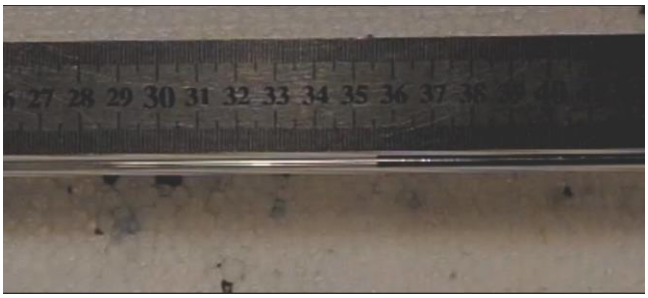


Fig. 8. Pictures of the sample before heat treatment during a) first and b) second bounce on the test stand

Source: Own work

Obtained difference between those drops for the sample before heat treatment was 94 mm what relatively to the height of the first drop gives 19.35%. This value can be considered as the reference of the internal damping in the material but is not reflection of its value. In total there where conducted 20 tests for the sample with amorphous/nanocrystalline structure that gave an average value of damping of 20.9%. All results for this test are presented in Tab. 1.

a)



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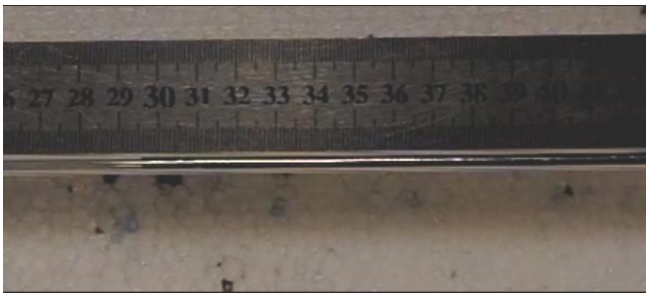

Fig. 8. Pictures of the sample after heat treatment during a) first and b) second bounce on the test stand

Source: Own work

In Fig. 9 are presented pictures presenting analogue situation as in Fig. 8 but for the sample after heat treatment. Obtained difference for presented test was 60.5 mm what gave damping on the level of 12.44% relatively to the height of the first bounce. In total 16 test were conducted for sample after heat treatment and the average value of damping obtained from those test was 18.2% what presents visible decrease of damping properties. All values obtained from tests on sample after heat treatment are present in Tab. 2.

Table 1. Results from the bounce test for partially amorphous sample

No.	1st bounce [mm]	2nd bounce [mm]	Difference [mm]	Relative damping
1	498.8	416.8	82.0	16%
2	466.8	368.0	98.8	21%
3	485.8	391.8	94.0	19%
4	394.8	318.0	76.8	19%

No.	1st bounce [mm]	2nd bounce [mm]	Difference [mm]	Relative damping
5	471.3	390.8	80.5	17%
6	451.8	341.5	110.3	24%
7	447.8	380.8	67.0	15%
8	481.8	383.8	98.0	20%
9	425.8	358.0	67.8	16%
10	479.8	380.8	99.0	21%
11	445.8	341.0	104.8	24%
12	465.3	379.3	86.0	18%
13	456.8	383.8	73.0	16%
14	452.8	358.0	94.8	21%
15	474.3	359.0	115.3	24%
16	461.8	342.5	119.3	26%
17	466.8	347.5	119.3	26%
18	425.8	321.5	104.3	24%
19	457.8	339.0	118.8	26%
20	419.3	324.0	95.3	23%

Source: Own work

Tab. 2. Results from the bounce test for the sample after heat treatment

No.	1st bounce [mm]	2nd bounce [mm]	Difference [mm]	Relative damping
1	458.8	393.0	65.8	14%
2	507.8	391.0	116.8	23%
3	488.8	378.0	110.8	23%
4	486.3	425.8	60.5	12%
5	523.3	447.3	76.0	15%
6	453.3	388.0	65.3	14%
7	501.8	425.8	76.0	15%
8	353.0	275.5	77.5	22%
9	441.8	381.5	60.3	14%
10	444.3	332.5	111.8	25%
11	453.8	392.0	61.8	14%
12	453.8	356.5	97.3	21%
13	446.3	343.5	102.8	23%
14	442.8	372.5	70.3	16%

No.	1st bounce [mm]	2nd bounce [mm]	Difference [mm]	Relative damping
15	465.3	354.0	111.3	24%
16	515.8	435.8	80.0	16%

Source: Own work

Obtained results clearly shows change of damping induced by the change of internal structure of the sample, therefore they justifies conduction of more sophisticated test to determine accurate value of internal damping in alloy $Fe_{75}Zr_4Ti_3B_{17}Cu_1$ with partially amorphous structure and with structure after heat treatment that induces change of the crystal structure of the alloy.

Results can be interpreted as the internal damping is decreasing with growth of the crystals in the structure of the material but this assumption have to be confirmed by further testing.

SUMMARY

Summarizing, for presented study there have been prepared metal rode made out of alloy $Fe_{75}Zr_4Ti_3B_{17}Cu_1$. It was created with use of sophisticated method based on rapid overcooling of the melted metal to create amorphous structure. Basic testing performed for obtained sample indicates that its structure is amorphous with some nanocrystalline structures. To determine this sample was tested with use of XRD, AFM, LFM and SEM with EDS attachment.

Testing of the internal structure was performed in form of a drop test with identical initial conditions. Test stand based on a glass tube with scale and high speed camera was set and used for testing sample before and after heat treatment. For both cases several tests were conducted to eliminate factor related to different values of friction present during tests. For partially amorphous sample damping was identified as 20.9% and after heat treatment this value was 18.2% what shows decrease of damping after structure change.

Recrystallization of the sample should occur at the temperature of 650ºC, therefore the heat treatment applied for the sample was not enough to change its structure. The heat treatment applied for the sample caused only growth of nonocrytals in its structure. Future tests are planned to be conducted on sample recrystallized in 4 temperature levels what will induce recrystallization of the material. Presented test were performed only as initial tests.

Presented results justify continuing of the study with use of more sophisticated methods based on analysis of natural frequency of longitudinal and transverse vibrations induced in the sample.

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NOTA BIOGRAFICZNA

Mirosław BOCIAN - PhD Eng. title obtained in year 2003 on Mechanical Faculty of Wroclaw University of Science and Technology, Assistant Professor at Department of Mechanics, Materials Science and Engineering, Mechanical Faculty, Wroclaw University of Science and Technology; author of 107 works including of 38 journal articles, 3 book chapters and 1 patent, cited 113 times.

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