CAVITATION BEHAVIOUR OF A BIODEGRADABLE ZnCu ALLOY

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The use of zinc in medicine is attributed to its biodegradable qualities and beneficial effects on individual health. Consequently, being part of the category of metallic materials, researchers in materials engineering have expanded studies on zinc alloys for use in orthopedic and cardiovascular surgery. One such alloy, newly developed in the specialized laboratory of the National University of Science and Technology Politehnica Bucharest, is the Zn-Cu alloy, containing approximately 2% Cu. This alloy's application in cardiovascular surgery aims at manufacturing stents and valves that, besides ensuring regular blood circulation, must also withstand the hydrodynamic demands of blood due to pressure fluctuations occurring in various phases of body movement. Since the human body is a network of veins fueled by the heart (the blood system's pump), studies have shown that at certain times, similar to any hydraulic network, the pressure can drop below the vapor pressure, leading to the phenomenon of cavitation, with all its effects. For this reason, the present work, through specific diagrams and macro and microstructural images of the eroded structure, presents the results of the research on the behavior and resistance of the Zn-Cu alloy to cavitation erosion generated by the standard vibratory apparatus within the Specialized Laboratory at Politehnica University of Timişoara. Comparing these results with those of a previously studied ZnCuMg alloy, using reference parameter values recommended by the international ASTM G32-2016 standards, shows that the Zn-Cu alloy has lesser resistance, but considering the lower hydrodynamic intensity of cavitation compared to that of distilled water used in the

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tests, the conclusion is that the alloy can be used in the manufacturing of cardiac devices, such as stents.

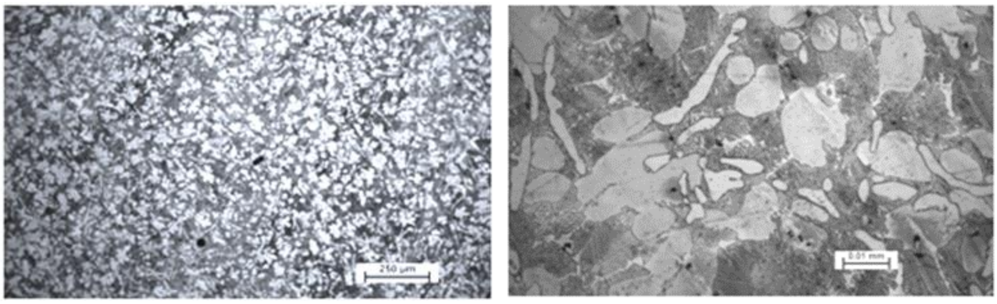
Keywords: Zinc-copper alloy, cavitation erosion resistance, stereomicroscopy, biomedical application, cardiovascular applications

1. Introduction

Engineered or natural materials that are used to supplement the functions of living tissue are known as biomaterials and have been used as implant materials for a long time in the field of medical science [1-4]. Non-biodegradable metal biomaterials possess a number of disadvantages, such as tissue reactions to the release of allergenic metal ions from implants. Thus, the solution of biodegradable metal materials is the most important choice. Among biodegradable biomaterials, zinc, along with magnesium and iron, offers the most interesting applications. Medical scientific research [1-5] confirms the benefits of the presence of zinc and copper in the human body due to their biocompatibility and biodegradability. Recent studies [6-10] focus on the use of these metals in combined alloy structures for interventions in the cardiac surgery system, through stents and devices similar to heart valves. The reason is that the blood circulatory system resembles a hydraulic network [6, 9, 10], in which hydrodynamic phenomena such as cavitation occur, with specific destructive effects of erosion. Consequently, lately in the medical field [2, 9, 10], studies have been developed that analyze the blood circulatory system as akin to the flow in hydraulic machines and installations. The main reason being the occurrence of sudden pressure fluctuations, from vapor pressure to very high values, in various areas of the blood circulatory system, especially at the heart's entrance (compared by hydraulic engineers to a volumetric pump [2, 6]), with harmful effects on the physical life/health of the individual, by causing pinching and even cracking of veins and heart walls [1, 6-8]. Therefore, the introduction of stents and the replacement of heart valves with suitable devices made from biocompatible and biodegradable materials has become a leading field of engineering and medical research in cardiovascular surgery [1, 7-11]. The present work considers a Zn-Cu alloy system, which the literature mentions as being recommended for cardiovascular applications [1-5, 9-32]. This direction of study also includes the research conducted by our team, regarding the behavior and resistance to cavitation of the biodegradable Zn-Cu alloy, created in the Specialized Laboratory of the Center for Expertise in Special Materials at the National University of Science and Technology Politehnica Bucharest. The research conducted in the Cavitation Erosion Research Laboratory at Politehnica University Timișoara, through diagrams and specific parameters of cavitation erosion, demonstrates the behavior and resistance of the alloy to cavitation erosion and confirms its potential for use in manufacturing stents and valves for individuals with cardiac issues, as the hydrodynamic intensity of cavitation in the blood circulatory system is much lower than that produced in hydraulic systems, due to the structure of the blood (which does not adhere to Newton's law of friction and whose speed increases faster than the force causing its disintegration [12].

2. Materials and Experimental Procedures

The alloy investigated was developed in the specialized laboratory of the Center for Expertise in Special Materials at the National University of Science and Technology Politehnica Bucharest. It was melted in a crucible flame furnace at 650°C, using high-purity elements, specifically Zn 99.99% and Cu 99.99%. The casting was done in a stainless steel ingot. The chemical composition of the alloy is as follows: Mg = 0.35%; Fe = 0.39%; P = 0.146%; S = 0.15%; Cu = 2.36%; Si = 0.97%; Al = 4.1%; Ni = 0.019%; Zn = remainder. A characteristic of this alloy is that copper hardens and embrittles zinc due to the formation of hard intermetallic phases (Cu_5Zn_6). The dimensions of the ingots, from which samples were taken for cavitation and mechanical strength and hardness tests, were 20 x 20 x 200 mm. Microstructural investigation to identify the phases and microstructural constituents was conducted using an Olympus metallographic microscope and an Olympus SZX stereomicroscope, equipped with QuickMicroPhoto 2.2 software. The structure of this alloy, presented in Fig.1 (a, b), exhibits dendritic segregation, where dedritic arms are located in the range of 100-180 µm, chaotically arranged in the metallic matrix. At higher magnifications of the microscope (Fig.1b), a complex structure composed of $CuZn_5$ insular compounds and an interstitial solid solution based on zinc is observed.



a-ZnCu (8x) **b-ZnCu (250x)**

Fig. 1. Structural analysis of Zn-Cu alloy specimens etched with 2% nital: a-8, b- detail of image

The values for the mechanical tensile strength and Brinell hardness, determined in the laboratory of the Center for Expertise in Special Materials, are as follows: ultimate tensile strength (Rm) = 80 [MPa], yield strength (Rp0.2) = 67.87 [MPa], hardness = 72 HB.

3. Apparatus and method for cavitation testing

The cavitation testing program was conducted at the Cavitation Erosion Research Laboratory of the Politehnica University of Timișoara [9, 10, 15], using a standard vibratory device with piezoceramic crystals, as shown in Fig.2a, adhering to the international standards of ASTM G32-2016 [16] for the stationary specimen method, Fig.2b. To induce erosion in the structure of the tested specimen, the vibrating probe, mounted in the mechanical vibrating system, was made of STELITE type stainless steel. The distance between the surface of the test specimen (stationary specimen) and the surface of the vibrating probe was 1.00 mm. According to laboratory custom, the total duration of the test is 165 minutes divided into 12 intermediate periods (one of 5 minutes, one of 10 minutes, and ten of 15 minutes each). The methods of washing, drying, measuring mass loss, processing, and evaluating behavior and resistance to cavitation erosion are those used by all laboratories studying material erosion through vibratory cavitation.

The functional parameters of the device are: the power of the electronic ultrasonic generator = 500 W, the double amplitude of vibrations = 50 µm, and the vibration frequency = 20 ± 0.01 kHz. According to ASTM G32-2016 requirements, the diameter of the circular surface of the cylindrical specimen, Fig.2, exposed to cavitation, is d = 15.8 mm. Tests are performed in distilled water at a temperature of t = 22 ± 1°C, as specified by international standards.

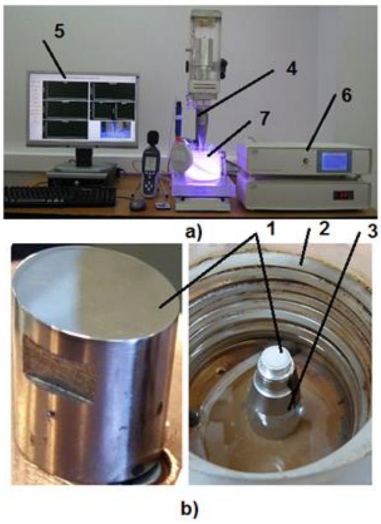


Fig. 2. Experimental setup image and component details
a) Overview image of the vibratory device; b) Details of specimen and clamping device;
Specimen subjected to cavitation; 2. Cooling coil for distilled water; 3. Specimen clamping device;
4. Mechanical vibrating system; 5. Display screen for parameters
monitored during cavitation; 6. Electronic ultrasonic generator; 7. Container with distilled water[9]

The mass eroded by cavitation over the intermediate durations was determined using a KERN ABT 100-5NM digital electronic balance with a precision of 0.01 mg. In accordance with the research requirements set by the laboratory custom [16-21], three specimens were tested, and their findings were algebraically averaged.

Before starting the cavitation tests, the surfaces of the three samples, before being exposed to cavitation attack, according to laboratory custom and the provisions of ASTM G32-2016, were polished to a roughness Ra = 0.8 μm.

4. Results concerning Cavitation Behaviour

To monitor and analyze the behavior of structures under cavitation attacks, based on the mass losses from each intermediate period, the values of the average erosion depths (ΔMDE_i) and the average erosion penetration rates ($MDER_i$) were determined according to the formulas described in [15, 22-24]. Discussion of the cavitation test results is based both on the experimental values of the three specimens and on the evolution of the average erosion depth curves (MDE(t)) and erosion rate curves (MDER(t)), constructed using relations presented in [15], whose forms are included in the legends of the diagrams. The assessment of the structure's resistance at various stages of cavitation, through the deformations and cavities produced, is based on photographic images (Fig. 5) of the eroded surface, taken with a Canon Power Shot A 480 camera, as well as microscopic images (Fig. 6), obtained with a stereomicroscope and a scanning electron microscope (SEM).

Diagrams and Specific Parameters of Behavior and Resistance to Cavitation

Figs. 3 and 4 display diagrams that include the experimentally determined values and the average curves for cumulative erosion depths and average erosion penetration rates in the structure of the surface exposed to cavitation attack.

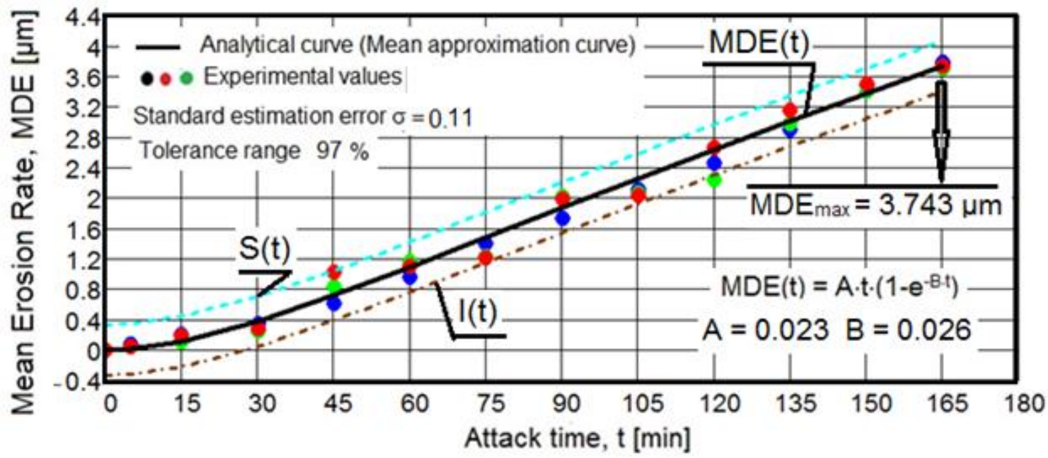


Fig. 3. Variation of the average cumulative depth over the duration of cavitation exposure

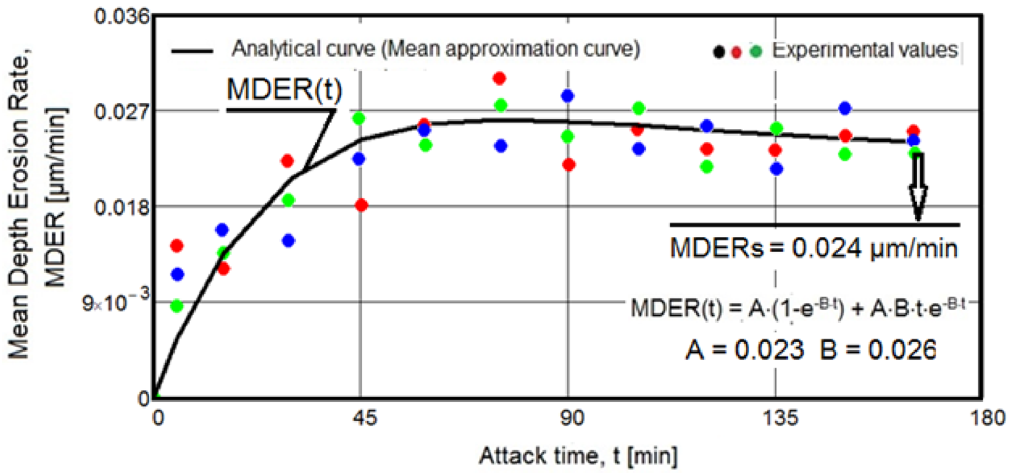


Fig. 4. Variation of the average erosion penetration rate with the duration of cavitation exposure

Data from Fig. 3 shows:

- the average cumulative depth continuously increases throughout the duration of the cavitation attack. After 30 to 45 minutes, the average cumulative depth increases linearly, as indicated by the MDE(t) curves and the evolution of the experimental values;

- the behavior of the three specimens, marked by some differences between experimental values, is determined by each structure's response to the specific mechanism of destroying the peaks of roughness, through creating deformations and generating microcraters, and the response to the impact with pressure forces developed by cavitation microjets, which are influenced by the dendritic microstructures of each, the number and dispersion mode of fragile compounds of the two basic chemical elements (Zn and Cu), as well as by the use of potential pressure energy for deformation, cracking, breaking, and hardening [6, 25-29];

- the dimensions of the dispersion bands, given by the values of the 97% tolerance fields and the average standard deviation, $\sigma = 0.11$, show that the three tested specimens are identical in terms of structural and mechanical property values both within the volume of the specimens and in the cavitated surface area. Additionally, the values of the two parameters (tolerance range and standard estimation error), as well as the dispersion mode of experimental values among themselves and compared to the average curves, confirm the accuracy of the testing program, with strict control of the hydrodynamics of vibratory cavitation through the parameter values of amplitude and frequency of vibrations, power of the electronic ultrasonic generator, the distance between the surfaces of the vibrating and tested specimens, and the temperature of the distilled water.

Data from Fig. 4 confirms the similar behavior of the three specimens, and in addition to the observations made through the analysis of Fig. 3, it is noted the evolution of the MDER(t) curve, which increases with an asymptotic decrease towards the stabilization value $v_s = 0.024$ µm/min, as a result of the absorption of potential energy, created by the impact pressure of the surface with microjets and shock waves. This mode of evolution is beneficial for devices used in the blood circulatory system, as the decreasing tendency of the MDER(t) curve, after reaching its peak value (at 75 minutes), is caused by the hardening of the surface layer, a beneficial effect in terms of increasing the operational lifespan under cavitation conditions [30-32].

Morphology of Structural Degradation

In Fig. 5, photographic images from six durations of the cavitation attack are displayed, taken with the Canon Power Shot A 480 camera. These images show how the increase in cavitation duration leads to an increase in the eroded area, both in depth (the area becomes progressively darker) and in extension across the plane of the attacked surface. The microscopic images in Fig. 6, as well as those in Fig. 5, confirm the assertions made in the analysis based on data from the diagrams in Fig. 3 and 4. Note: The images in Fig. 6 were obtained using a stereomicroscope after sectioning the specimen perpendicular to and along the diameter of the cavitated surface.

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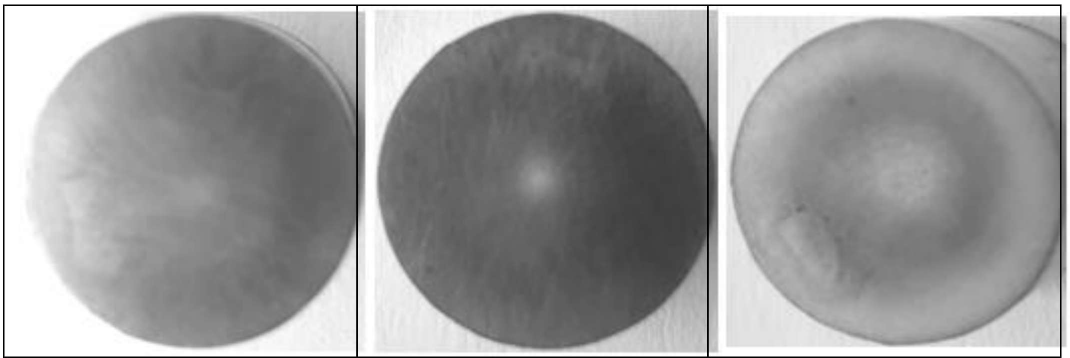
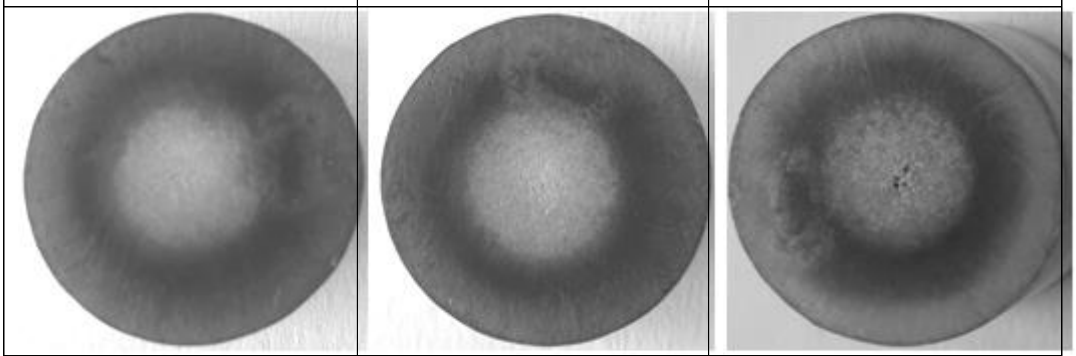
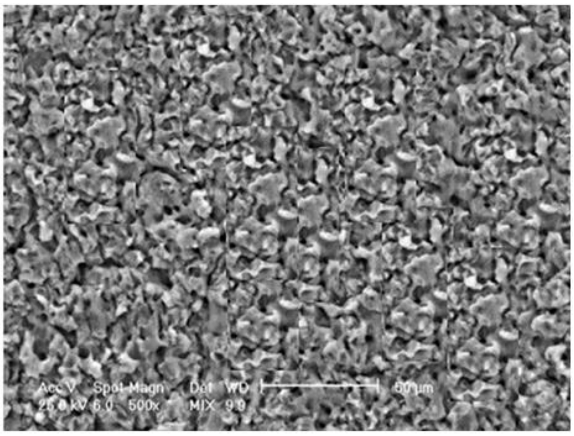


Min 5 ***Min 15*** ***Min 60***

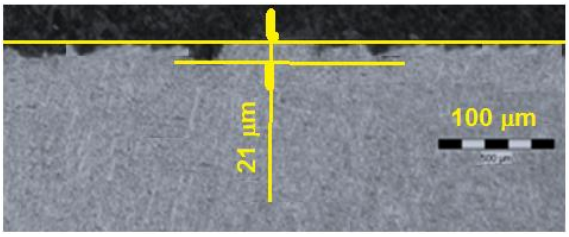


Min 90 ***Min 120*** ***Min 165***

Fig. 5. Macroscopic images (photos) showing the erosion progression in the area of the cavitated surface



a)



b)

Fig. 6. Microstructural appearance and cavities on the eroded surface, after completing the 165-minute test: a- SEM fractographic image, in frontal section, b- stereomicroscope image, in cross-section

The shape of the cavities in these photographic and microscopic images indicates that the sizes of the grains expelled upon impact with cavitation microjets are small, characteristic of structures with high resistance to cavitation stresses [15, 20, 25, 26, 33], which, through biodegradation, can dissolve quickly in the blood circulatory system, thus ensuring a long life for devices used in cardiovascular surgery without endangering life. The aspects captured at 500x magnification (Fig.6a) display a brittle, shiny crystalline appearance, with flattened cavities. However, the shapes of the cavities (see images obtained with the stereomicroscope, Fig.6b) call for caution/medical assistance in cases involving cardiac patients with devices made from these alloys, because from the perspective of hydrodynamics in the blood circulatory system, the use of stents is necessary and beneficial but also disadvantageous for two reasons: 1- because they degrade rapidly, and 2- because these cavities alter the configuration of the hydrodynamic field, generating strong turbulence and pressure variations [6, 34], with effects that can become problematic (even destructive) for the cardiac stability of the patient. The fractographic analysis performed with the scanning electron microscope (Fig. 6a) allows for the evaluation of cavitationally eroded surfaces. Thus, a surface with fine interconnected cavities in a network is noted, with intergranular propagation of the fracture front. The cavities are extremely fine, with an average size of 10-30µm. The general appearance of the cavitationally eroded surface is that of intergranular brittle fracture. The analysis with the stereomicroscope in cross-section allowed the measurement of the instantaneous penetration depth of the cavitation attack, which is 21µm (Fig.6b).

5. Evaluation of resistance to cavitation erosion

The evaluation of the resistance of the Zn-Cu alloy structure is based on the parameters presented in the histogram in Fig.7. This histogram also displays the values of the parameters MDEcav (the depth of the cavity measured in the sectioning plane, Fig.6b) and Rcav = 1/MDERs (resistance to cavitation) as expressions of the effect of the microstructure and its mechanical properties on the surface resistance to cavitation stresses. As a comparative alloy, the ZnCuMg alloy is used, whose chemical composition and mechanical properties are [35]: Mg = 3.66%; Fe = 0.95%; S = 0.14%; Cu = 3.05,09%; Si = 0.36%; Al = 1.05%; P = 0.001%; Ni = 0.02%; Zn = remainder; R_m = 123 [MPa], $R_{p0.2}$ = 84.23 [MPa], 76 HB.

From the perspective of this alloy's effect in operative cardiology, for devices such as stents or valves, the histogram serves as a benchmark for resistance to cavitation, depending on the severity of the cardiac patient and the signals recorded in blood pressure fluctuations.

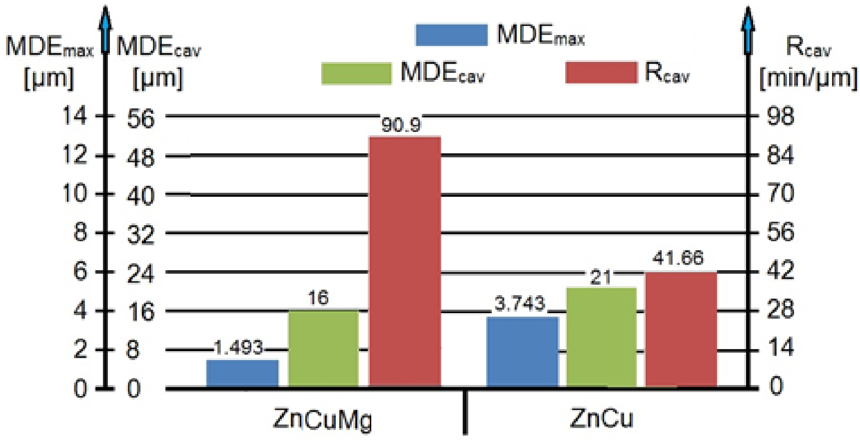


Fig. 7. Histogram comparing cavitation resistance reference parameter values

In terms of the structural resistance to erosion from vibratory cavitation, the data from the histogram shows that the resistance to erosion caused by vibratory cavitation is significantly lower for the ZnCuMg alloy. Quantitatively, this resistance, compared to the ZnCuMg alloy, is:

- about 2.5 times lower according to the MDEmax parameter values;
- about 30% lower according to the MDEcav parameter values;
- about 2.18 times lower according to the Rcav parameter values.

The superior erosion behavior of the ternary alloy ZnCuMg compared to the binary alloy ZnCu is determined by the existence of different intermetallic compounds. Thus, in the binary alloy ZnCu there are only compounds of Cu_5Zn6, while in the ternary alloy ZnMgCu there are two types of compounds Cu_5Zn_6 and Mg_2Zn_{11} that have a synergistic effect of hardening the solid solution. Therefore, the ternary alloy has higher mechanical characteristics, consequently also a superior behavior to cavitational erosion corrosion.

6. Conclusions

The alloy investigated in this study exhibits reduced resistance to cavitation erosion; however, it represents a technological solution for use in manufacturing stent-type devices and valves intended for cardiovascular surgery, due to its biocompatibility and biodegradability, and the fact that the intensity of cavitation, due to the higher viscosity of blood compared to water, produced by pressure fluctuations in the blood circulatory system is significantly reduced compared to the intensity of vibratory cavitation in distilled water.

Macro and microscopic images of the eroded surfaces confirm the dependence of behavior and resistance to cavitation on the type of microstructure and correlation of the values of the main mechanical properties (R_m, $R_{p0.2}$, and HB).

The lower cavitation resistance of the ZnCu alloy, compared to that of the ZnCuMg alloy, is due to the lower level of the three mechanical properties R_m, $R_{p0.2}$, and HB.

Fractographic analysis conducted with the scanning electron microscope allowIing the evaluation of cavitationally eroded surfaces. A surface with fine interconnected cavities in a network was noted, with intergranular propagation of the fracture front. The cavities are extremely fine, with an average size of 10-30 µm. The general appearance of the cavitationally eroded surface is that of intergranular brittle fracture. Analysis with the stereomicroscope in cross-section allowed the measurement of the instantaneous penetration depth of the cavitation attack, which is 21 µm.

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