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INFLUENCE OF THE FABRICATION PROCESS OF COPPER MATRIX COMPOSITES ON CAVITATION EROSION RESISTANCE

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Abstract
Two methods were used to create copper matrix composites enhanced with ZrB2 particles: hot pressing (HP) and laser sintering. Prior to densification procedures, the Cu-Zr-B powder combination was mechanically alloyed. Scanning electron microscopy (SEM) was used to examine changes in the microstructure of treated samples that were collected during the cavitation test. Using vibratory equipment and the usual test procedure for cavitation erosion, the resistance to cavitation erosion was examined. Regardless of the quantity of reinforcements, variations in the mechanical alloying time have a significant impact on the cavitation erosion resistance of Cu–ZrB2 composites. Compared to hot-pressed samples, laser-sintered samples exhibit superior resistance to cavitation erosion.

Keywords: copper-matrix composites; mechanical alloying; hot-pressing; laser- sintering; cavitation erosion; scanning electron microscopy (SEM).

Introduction
The creation of composite materials with the optimal blend of mechanical and physical characteristics has received a lot of attention in recent decades. Composites based on copper are being investigated as possible candidate materials for the electrical, automotive, aerospace, and military industries [1–3]. Even in nuclear technology and the rocket industry, a low concentration of alloying elements in the copper matrix allows for the maximum ratio of mechanical/physical qualities necessary to endure harsh working conditions [4, 5]. Prior research [6–8] shown that adding ZrB2 as a hardening phase to the copper matrix greatly enhanced its mechanical characteristics while preserving high electrical and thermal conductivity. ZrB2 particles may be properly distributed and formed in situ in the copper matrix by combining mechanical alloying (MA) with hot pressing. Due to the avoidance of significant interfacial interaction between the Cu-matrix and coarse ZrB2 particles, this approach has great effect on mechanical and tribological characteristics [3-5]. However, high super saturation that results from laser sintering improves the creation of finer ZrB2 reinforcing particles in the copper matrix. In situ production of ZrB2 in a copper matrix during hot pressing or laser sintering is made possible by the mechanical activation of Zr and B particles during MA [6]. These features allow the Cu-ZrB2 composite to have good mechanical properties. Cavitation erosion occurs often in a variety of technical materials. This finding supports a study of this kind of Cu-ZrB2 composite erosion. The creation, expansion, and disintegration of bubbles in a liquid as a result of local pressure variations is known as cavitation. Microscopic bubbles are created when the liquid's pressure falls below the vapor pressure at a certain temperature [9]. If the liquid is then exposed to a greater hydrostatic pressure, these bubbles may abruptly burst. Collapsing bubbles generate shock waves and microjets that, in a brief amount of time (less than 1 μs), may result in high temperatures (5000˚C) and pressures (103 MPa) on surfaces with tiny dimensions (10-10 m2) [10, 11]. Cavitation erosion may result from fatigue if these collapses occur often near the solid surface. Mass loss against exposure time is often used to characterize the material response to cavitation erosion, and the displayed data should ideally form an S-shaped curve. The incubation phase, accelerating rate period, constant rate period, oscillating rate periods, and, in some situations, a final lower constant rate period are the steps that make up the formation of the S-curve [12]. Inpractice, the shape of this curve depends on material properties and its response tocavitation attack, i.e., cavitation resistance.

Several research studies have been devoted to understanding the phenomenon of cavitation erosion of conventional materials, which are used in the production of hydraulic machinery and other mechanical parts. They proved that the cavitation erosion resistance depends on mechanical parameters (hardness, tensile strength, Young’s modulus, fatigue strength), microstructure (grain size, some material defects, present phases), and also on surface roughness [13-16]. Hence, many studies investigated the cavitation erosion resistance of different materials such as gray cast irons, ductile irons, stainless steels, nonferrous metals and alloys such as aluminum and copper alloys [17- 21].

The present work includes analysis of the influence of mechanical alloying duration on the cavitation erosion resistance of copper matrix composites as well as the influence of different fabrication processes. Cavitation test was performed on hot-pressed samples obtained from powders after 5 and 30 hours of MA, and on laser-sintered samples obtained from powders after 30 hours of MA. The best results of cavitation testing for both processes were compared. Microscopic investigations support discussion of the results.

Experimental work

Materials

The powders used as starting materials were copper (99.5% purity, average particle size 100 µm), zirconium (99.5% purity, average particle size 1 µm) and boron (97% purity, average particle size 0.08 µm). Starting powder mixture with compositions Cu1.1Zr–0.3B (wt.%) and Cu–4.1Zr–1.1B (wt.%) were homogenized in Turbula Type2TC Mixer for 1 hour with stirring speed of 500 rpm. The homogenized powder mixture was mechanically alloyed in Netzsch attritor mill for 5 and 30 hours with stirring speed of 330 rpm. Mechanical alloying was performed in argon atmosphere using stainless steel balls (6 mm in diameter), and ball-to-powder weight ratio was 5:1.

Preparation of hot-pressed and laser-sintered copper matrix composites

Hot pressing of mechanically alloyed mixtures (Cu1.1Zr–0.3B (wt.%) and Cu–4.1Zr–1.1B (wt.%)) was carried out in Astro furnace with graphite mold (10 mm diameter). The heating rate was 15˚C/min, at a temperature of 950 °C and under a pressure of 35 MPa. Retention time was 2.5 hours. Obtained compacts were 5 mm in height. For laser-sintering method, mechanically alloyed powders (Cu–4.1Zr–1.1B (wt%), and after 30 hours of MA) were cold pressed by the applied pressure of 180 MPa, to produce green compacts. Laser-sintering of green compacts was carried out with Nd: YAG pulsed laser radiation in the nitrogen atmosphere. Samples were distinguished by a number of scans from 1 up to 4. Except for the number of scans, parameters of laser sintering for all samples were the same: frequency 3 Hz, pulse duration 10 ms (for the sample with 4 scans 8 ms), pulse energy 22 J (for the sample with 4 scans 18.5 ms). Obtained laser-sintered samples were 300 µm in height. Properties of obtained copper matrix composites are given in Table 1. As was reported in previous studies [6-8] *in situ* forming of ZrB_2 particles was achieved during hot-pressing and laser-sintering process. The density of the samples was determined by Archimedes method, and the rule of mixture determined theoretical density.

Table 1. Properties of hot-pressed and laser-sintered copper matrix composite with corresponding abbreviations.

Sample		Process	Porosity, %	Vickers Hardness**	Abbreviatio n s
Mechanical alloying time / amount of ZrB_2 particles	Pure copper	hot-pressing	0.01	60 ± 0.4	CuHP
	5 h / low (L)		5.38	81 ±4	5hHP-L
	30 h / low (L)		3.12	89±3	30hHP-L
	5 h / high (H)		6.56	101 ±3	5hHP-H
	30 h / high (H)		4.06	155±2	30hHP-H
Number of scans	2	laser-sintering	8.31	140 ± 16	LS2x
	3		2.47	165 ± 12	LS3x
	4		2.16	180 ± 7	LS4x

* Low (L) and high (H) amount of ZrB_2 particles indicates volume fraction of 1% and 7%, respectively. ** For laser-sintering Vickers hardness was measured as HV0.01 and for hot- pressing as HV1. For both measurements dwelling time was 15s.

Cavitation test

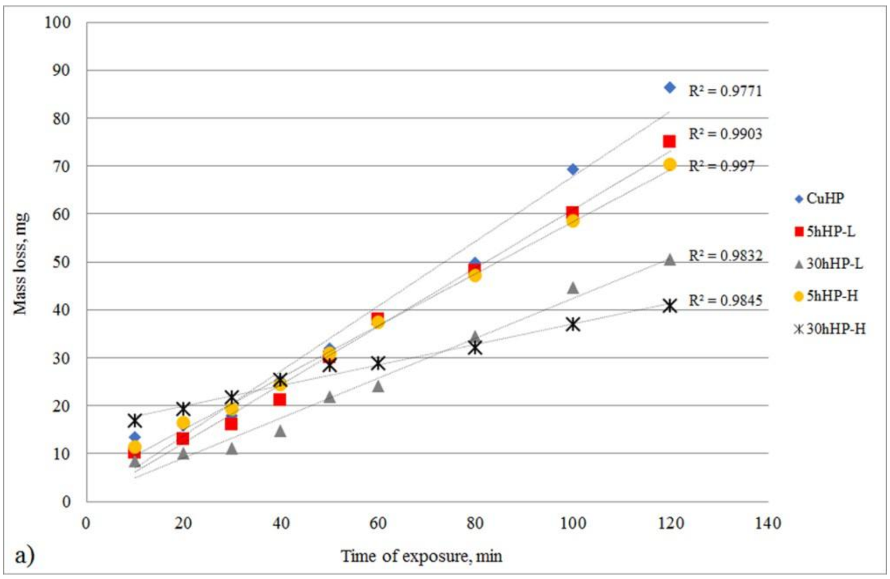
The ultrasonic vibratory cavitation test set up (stationary sample method - according to the ASTM G32 standard) was used for cavitation erosion analysis [22]. The frequency of vibration and peak-to-peak displacement amplitude of the horn were 20 kHz and 50 μm, respectively, with separation of 0.5 mm between the sample and the horn tip. The test liquid was water maintained at 25°C [22-25]. The mass loss measurements were performed after each exposure to the cavitation (every 10 min) for a test period of 120 min for HP compacts and laser-sintered samples every 10 min for a test period of 60 min. Differences in cavitation test duration are due to a thickness of the sample. Before and after each test interval the samples were cleaned and dried with hot air. Mass losses of the tested samples were measured using an analytical balance with an accuracy of ±0.1 mg. Cavitation erosion rate was calculated as a slope of the line obtained after a linear fit of mass loss vs. exposure time curve.

Microscopic examination

The microstructure of samples before and after cavitation test was investigated by JEOL-JSM 5800LV scanning electron microscope (SEM) at an accelerating voltage of 20 kV. Detailed analysis of microstructure before cavitation test was reported in our previous studies [6-8].

Results and discussion

Size and distribution of reinforcements in copper matrix depend on their percentage and mechanical alloying duration or number of scans in case of laser-sintering and show a strong influence on mechanical properties [8, 26]. The highest densities and hardness have samples obtained by hot-pressing from powders mechanically alloyed for 30 h or by 4 scans during laser-sintering. Compared to pure copper sample, the full densification of the Cu-ZrB_2 alloy was not achieved because of hardening effects due to the presence of ZrB_2 particles and agglomerates with varying sizes [6]. Therefore, it was noticed that longer milling time provides more homogeneous distribution of reinforcing particles and better densification which have a substantial influence on the hardness of this composite. In previous studies [7, 8] amount of present reinforcements in the copper matrix was determined as 1% ZrB_2 for system Cu – 1.1 wt.% Zr – 0.3 wt.% B; 7% ZrB_2 for system Cu – 4.1 wt.% Zr –1.1 wt.% B after hot-pressing; around 3.5% for laser- sintered samples of a green compact with Cu – 4.1 wt.% Zr –1.1 wt.% B composition. As was reported [6, 7], the presence of higher amount of submicron reinforcing particles in the copper matrix was observed after 30h of MA comparing to 5h, which leads to higher hardness. In Fig. 1. results of cavitation erosion testing of hot-pressed (Fig.1a) and laser- sintered (Fig.1b) samples are shown. Hot-pressed samples with the same duration of mechanical alloying show similar cavitation resistance despite the various amount of present reinforcements. However, observing the overall behavior of hot-pressed samples, the influence of reinforcement amount on cavitation resistance can be noticed, especially after 30 min of cavitation erosion test. Sample 30hHP-H which shows the highest cavitation erosion resistance also shows the highest mass loss at the beginning of cavitation test, but after 30 min the mass loss rate decreases, i.e., steady-state rate occurs due to surface roughness. The behavior of laser-sintered samples during cavitation erosion test depends on the number of scans. According to the density and hardness values of the laser-sintered samples, it was expected that LS4x shows the highest cavitation erosion resistance and LS2x the lowest.



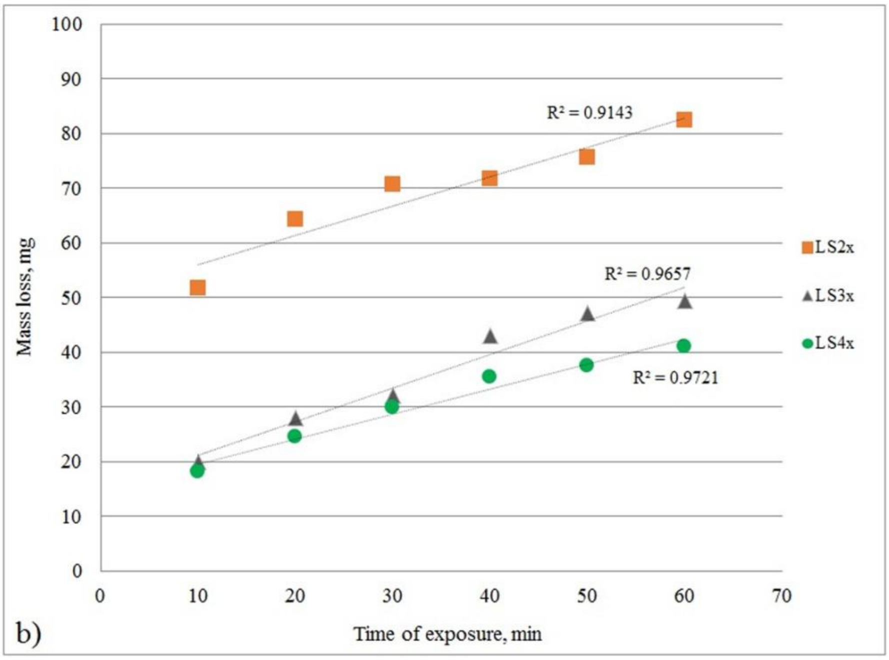
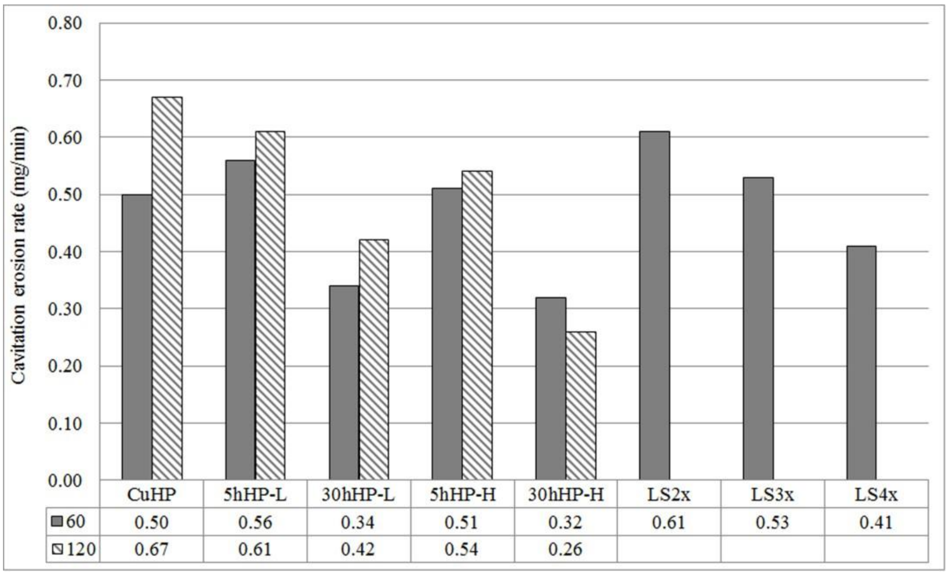


Fig. 1. Mass loss during cavitation erosion testing of a) hot-pressed and b) laser- sintered samples.

All samples do not show any incubation period during cavitation erosion testing, probably due to the presence of agglomerates or coarse ZrB_2 particles which provide increasing of some local peeling of the copper matrix. Also, ZrB_2 particles can sustain higher loads compared to the copper matrix because of their higher hardness and



consequently local strain of the copper matrix is higher. On the other hand, the presence of reinforcements in copper matrix increases the dislocation density and decreases the grain size since the reinforcing particles act as sites of nucleation during solidification. The grain size decreases due to the formation of subgrains with a high dislocation density. Also, it is supposed that in this composite material the subgrains are formed and surrounded by ZrB_2 particles which can act as barriers to the dislocations movement. As can be noticed, samples 30hHP-H and with LS4x show the best results in performed cavitation erosion testing which confirms the correlation between cavitation resistance and mechanical and microstructural parameters. Therefore, comparing these two samples, it can be concluded that laser-sintering technique provides samples with slightly better cavitation erosion resistance than hot-pressed samples. This fact is supported by similar values of cavitation erosion rate after 60 min of exposure (0.32 mg/min for 30hHP-H and 0.41 mg/min for LS4x) despite the significant difference in the sample thickness (5 mm for 30hHP-H and 300 µm for LS4x) as well as the difference in amount of ZrB_2 particles (7%in30hHPHand3.5%inLS4x).Lowervaluesofcavitationratesindicatehighercavitationerosionresistance(Fig .2).

Fig. 2. Cavitation erosion rates of hot-pressed and laser-sintered samples.

The SEM investigation has shown evenly spread corrugated surface of the copper sample and randomly distributed pits in the corrugated surface of other samples. Since material response to cavitation erosion attack differs depending on the production technique, mechanisms of damage also vary. As was reported [21], the final surface damage caused by cavitation erosion is a consequence of two mechanisms: the individual collapse of bubbles and simultaneous collapses of the whole cloud of bubbles close to the solid surface. In ultrasonic vibratory cavitation erosion test set these mechanisms can occur individually or jointly. The progress of cavitation erosion on the surface of hot- pressed samples observed by SEM is shown in Fig. 3. Damaged surfaces of CuHP, 5hHP- L, and 30hHP-H after 120 min of cavitation erosion test are shown in Fig. 3a-c,respectively. These SEM micrographs show mainly corrugated surface. Since copper is a ductile material with a low hardness, it shows a high material loss. It is assumed that the dominant mechanism is a simultaneous collapse of the whole cloud of bubbles. On the other hand, sample (30hHP-H) with the lowest material loss shows the lesser number of shallow pits probably due to the occurrence of mixed ductile/brittle-mode cracks caused by the presence of ZrB_2 particles, which indicates that individual collapse of bubbles is the more dominant mechanism. Eroded surface of CuHP sample shows plastically deformed grains due to the presence of cleavage as dominant fracture mode, but the ductile fracture was also noted (Fig. 3a). Addition of ZrB_2 particles induces mixed fracture (Fig. 3b,c), both ductile and brittle mode cracks occurred, and cavitation attack is primarily on grain boundaries (a few mentioned cracks occurred on the grain boundary were marked with arrows). The corrugated surface is observed in all samples, and distribution and depth of pits are in correlation with the distribution of reinforcing particles. Hot-pressed samples show grain boundary erosion and distribution of pits inside the grains depends on amount and distribution of ZrB_2 particles as well as grain size. Observing the surface of 30hHP-H sample shown in Fig. 3c, it can be noticed the presence of non-damaged areas (marked with arrows) as a onsequence of relatively high hardness due to the presence of submicron ZrB_2 particles and

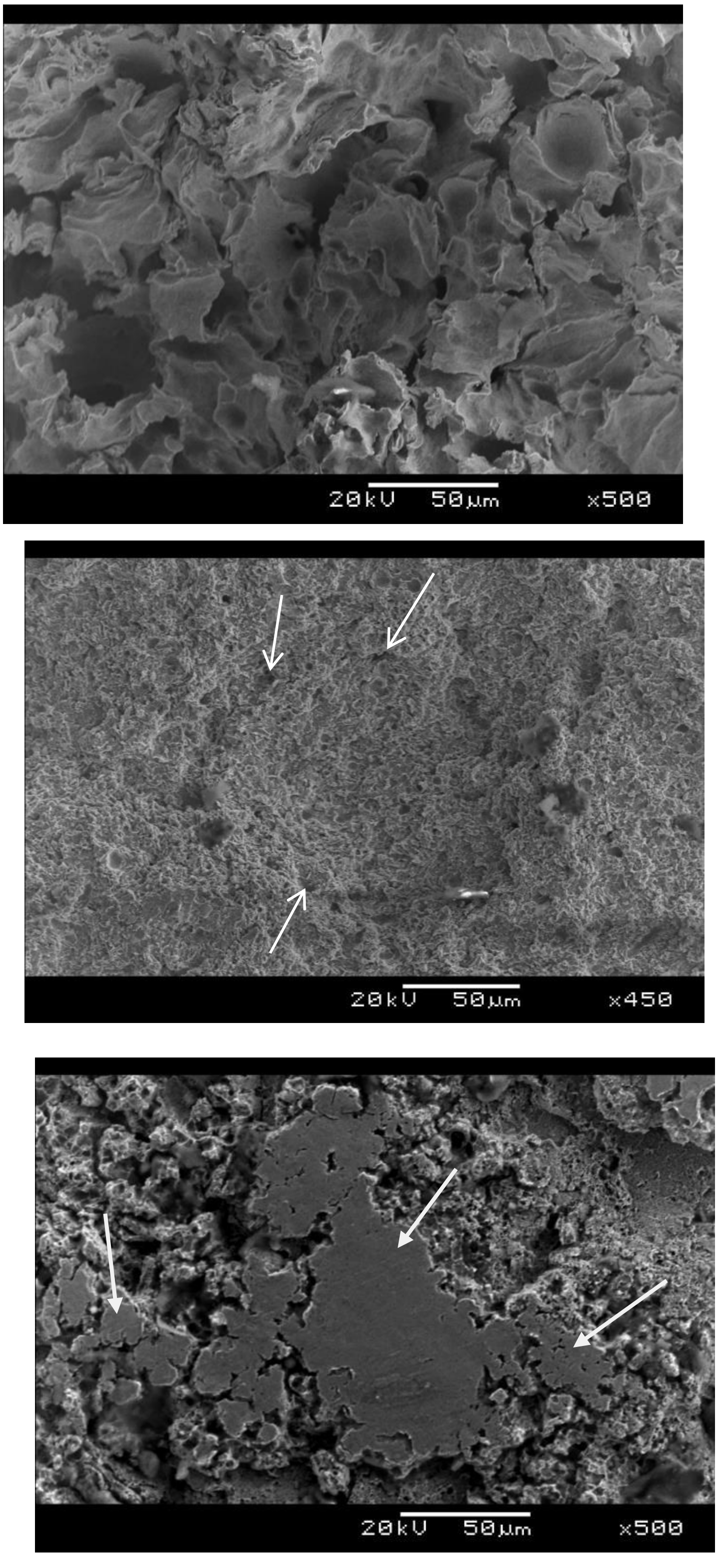
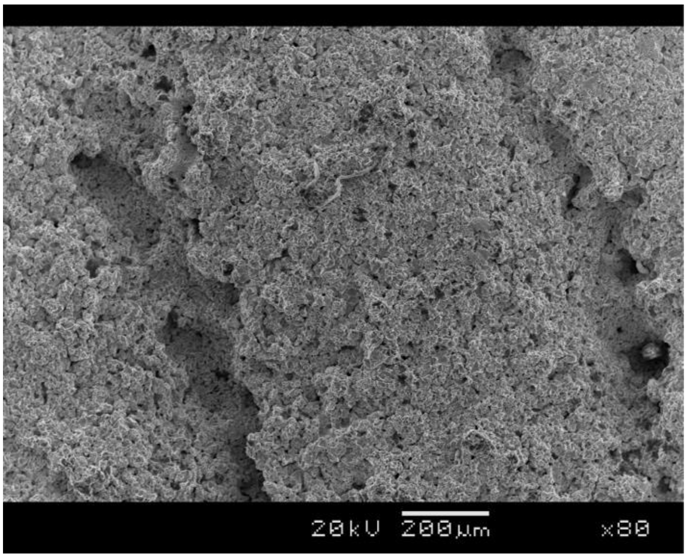


Fig. 3. SEM micrographs of the damaged surface of hot-pressed samples: a) CuHP, a) 5hHP-L, and c) 30hHP-H, after 120 min of cavitation erosion testing.

Laser-sintered samples show similar behavior which is presented in Fig. 4. Sample LS2x (Fig. 4a) shows high material loss between scanning lines after 60 min of cavitation erosion testing due to the presence of pores or unmelted areas. Sample LS3x (not shown in this paper) exhibits the similar behavior as LS2x, i.e., erosion along areas between scanning lines as the dominant damaged surface but with the lower material loss. Observing surface of LS4x sample (Fig. 4b) after 60 min only a few pits were noticed surrounded mainly by non-damaged areas. With increasing the number of scans, material porosity decreases (as well as unmelted regions) and cavitation resistance increases. Also, the presence of ZrB_2 increased the work-hardening rate and combined with all facts mentioned above enables the individual collapse of bubbles as a more dominant mechanism.

a)


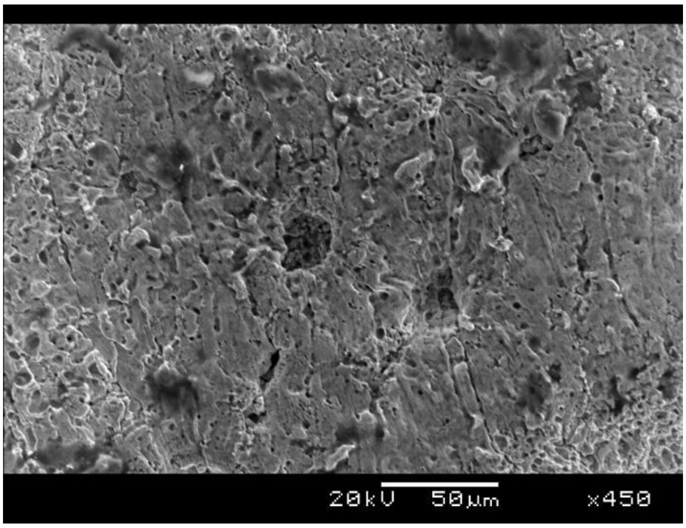
b)


Fig. 4. SEM micrographs of the damaged surface of laser-sintered samples: a) LS2x and b) LS4x, after 60 min of cavitation erosion testing.

It may be concluded that cavitation erosion resistance depends on mechanical alloying duration, as well as production process. Overall, as was expected, samples with higher hardness show better cavitation erosion resistance.

Conclusion

The cavitation erosion resistance of copper matrix composites was measured in this work using mass loss. The copper matrix's resistance to cavitation erosion is enhanced by the addition of reinforcing ZrB2 particles. For samples containing the same quantity of ZrB2 particles, the trend of mass loss is almost the same in the first 30 minutes. After 30 minutes, the trend of mass loss vs. time curve demonstrates the length of mechanical alloying. Considering the substantial variation in sample thickness, it can be inferred that manufacturing methods have an impact on cavitation erosion resistance since laser-sintered samples exhibit somewhat greater cavitation erosion resistance than hot-pressed samples. On the other hand, the copper matrix's capacity to work-harden and absorb cavitation energy is enhanced by the size, distribution, and length of mechanical alloying of ZrB2 particles. After two scans, the Cu-ZrB2 composite with the lowest cavitation erosion resistance displays a laser-sintered sample because of its high porosity and unmelted areas. The cavitation resistance rises as the number of scans increases.

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