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EBSD Strain Analysis of CuZn10 Alloy in As-cast State and After Plastic Working and Annealing During Cavitational Wear

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Abstract

Electron backscatter diffraction (EBSD) system in conjunction with scanning electron microscope (SEM) allows performing full material characterization which include analysis of individual grain orientations, crystal orientation, global and local texture, phase identification and distribution or microstrain mapping. EBSD technique may be also applied for assessing lattice strain introduced during local plastic deformation due to its close relationship with development of dislocation substructure.

The aim of this study was analysis of surface deformation by EBSD method of CuZn10 alloy in as-cast state and after plastic working and annealing, with different grain size at the beginning of the cavitation destruction process. The local changes of plastic deformation inside each grain were expressed by Grain Orientation Spread (GOS) and Kernel Average Misorientation (KAM) parameters.

Keywords: Electron back scattered diffraction (EBSD), Cavitation, Cavitational wear, Strain analysis

1. Introduction

The first observation of a backscattered diffraction pattern upon interaction of electron beam with solid sample was made in 1928 by Shoji Nishikawa and Seishi Kikuchi [1]. The term “electron backscatter diffraction” (EBSD) is often interchangeably used in the literature with “backscatter Kikuchi diffraction” (BKD) or “backscatter electron Kikuchi diffraction” (BEKD).

EBSD analysis is conducted on Scanning Electron Microscope (SEM) equipped with an EBSD detector containing at least a phosphor screen, compact lens and low light CCD camera chip. For an EBSD measurement a polished sample is placed in the SEM chamber at a highly tilted angle (at ~70° to the sample normal direction) towards the diffraction camera. The phosphor screen is located within the specimen chamber of the SEM and is coupled to a compact lens which focuses the image from the phosphor screen onto the CCD camera (Fig. 1).

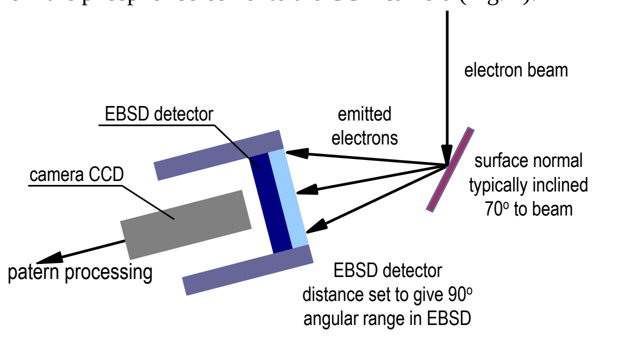

Fig. 1. Typical SEM EBSD set-up

In this configuration, some of the electrons after entering to the sample undergoes backscattering. As these electrons leave the sample, some of them may fulfill the Bragg condition related to the spacing of the atomic lattice planes of the crystalline structure. These diffracted electrons can escape from the sample and collide and excite the phosphor screen causing its fluorescence [2-3].

EBSD patterns are generated on a phosphorous screen by backscatter diffraction of highenergy electrons from a volume of crystal material approximately 20 nm deep in the specimen [4]. The characteristic feature of a backscatter Kikuchi pattern is the regular arrangement of parallel bright bands on a steep continuous background, rather than a regular array of diffraction spots as is generated in the TEM in selected area diffraction from a single crystallite (Fig. 2).

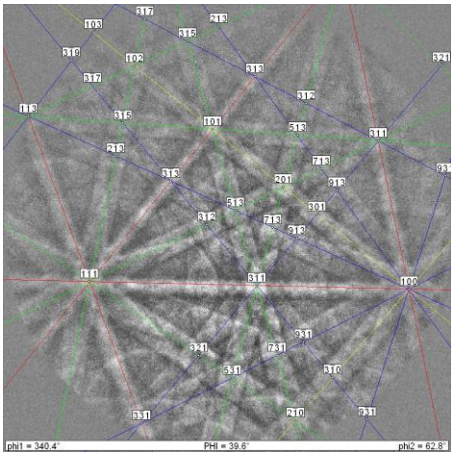

Fig. 2. Backscatter Kikuchi pattern

EBSD technique is currently organized in fully automated setups that contain three main sections: the SEM, the pattern acquisition device (or camera), and the software. Automation of measurement by EBSD allows performing such research as: crystal orientation, misorientation, grain size, global and local texture, recrystallised/deformed fractions, grain boundary characterisation, CSL (coincidence site lattice) boundary distribution, phase identification and distribution. In addition to the listed features, the EBSD method can be used for strain analysis. Thus, EBSD analysis of strain can be effectively applied for assessment of cavitational erosion resistance in the first period of cavitation wear, where there is no mass loss.

The aim of this study was analysis of plastic deformation by EBSD method of CuZn10 alloy in as-cast state and after plastic working and annealing with different grain size at the beginning of the cavitation destruction process.

2. Material and experimental details

The research was carried out on CuZn10 alloy in three various structural states:

- as-cast state;
- fine- and coarse-grained states after plastic working and various recrystallization annealing.

Material has single-phase structure of α solid solution of zinc in copper. As-cast alloy was obtained by melting and casting of pure elements in PIT 10 induction furnace. Microstructure of CuZn10 alloy in as-cast condition is presented in Fig. 3.

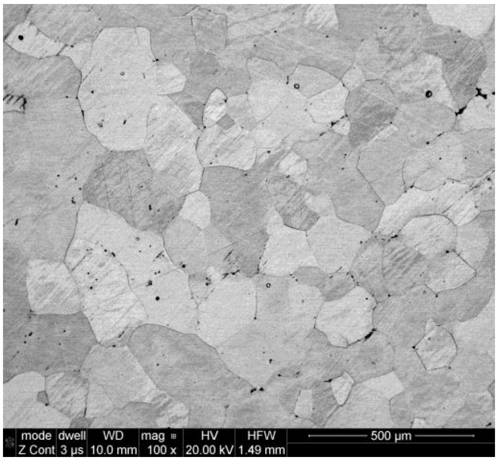

Fig. 3. Microstructure of CuZn10 alloy in as-cast state [7]

CuZn10 brass in as-cast state in the form of plates (L:150 x W:30 x T:12mm) was subjected to two-step thermomechanical treatment in order to obtain samples with different grain size. In the first step, investigated alloy was cold rolled to 50% of thickness reduction. Cold rolling was carried out on sexton-type rolling mill (with Ø85 mm working rolls) at 2 m/min constant velocity and 10% reduction per pass. Cold-rolled alloy was subsequently annealed for 1h at 400ºC or 750°C. As a consequence, two fully-recrystallized material states with different grain size were obtained: fine-grained with ~20µm average grain size (Fig. 4) and coarse-grained with ~200µm average grain size (Fig. 5).

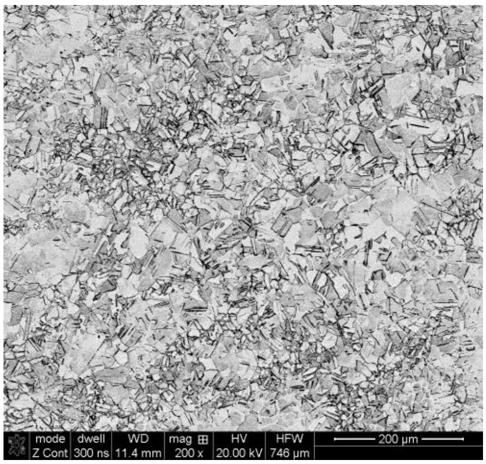

Fig. 4. Microstructure of CuZn10 fine-grained alloy with ~20µm average grain size

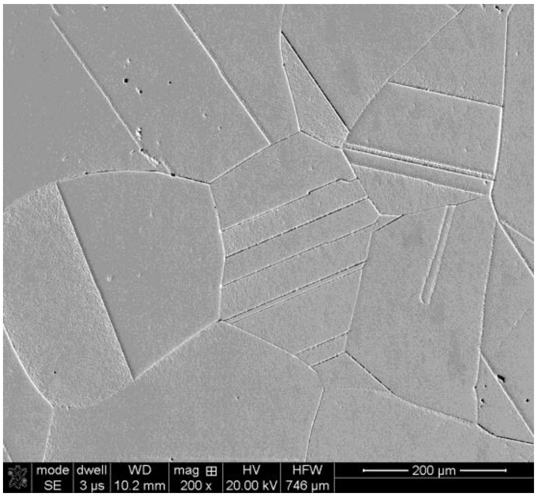

Fig. 5. Microstructure of CuZn10 coarse-grained alloy with ~200μm average grain size

Before cavitiational experiment surfaces of both samples were grinding with SiC papers, polished with diamond paste and finally polished with silica suspension to obtain shiny, metallographic surfaces.

3. Methods of investigation

Polished alloy samples CuZn10 were subjected to cavitational wear test on a jet-impact device (Fig. 6).

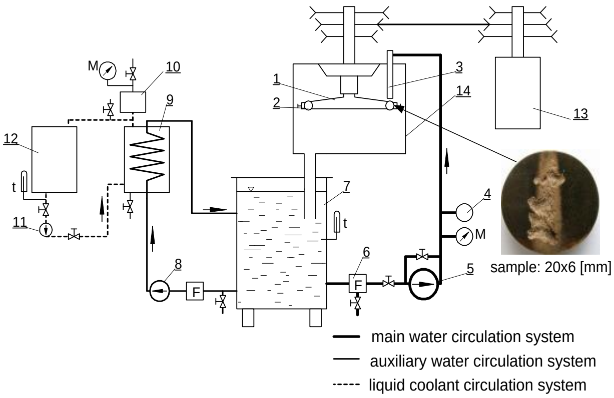

Fig. 6. A scheme of jet-impact measuring device: 1-rotor, 2-sample, 3-nozzle, 4-flow-meter, 5-pomp, 6-self-rinsing filter,7-circulating tank, 8-pomp of the cooling system, 9-cooler, 10-equalizing tank, 11-coolant pomp, 12-refrigerator, 13-elektric motor, 14-rotor casing

Examinated samples had cylindrical shape with 20 mm diameter and 6 ± 0,5 mm height. Surface roughness of samples before tests, measured by PGM-1C profilometer, was in range of 0,010÷0,015 μm. The samples were vertically mounted in rotor arms, parallel to the axis of water stream pumped continuously at 0,06 MPa through a 10 mm diameter nozzle located 1,6 mm away from the sample edge. The rotating samples were hitting by the water stream. Water flow of 1,55 m^3/h was constant during entire experiment. After 30, 60, 120, 180 and 300 seconds of exposition samples were removed to structural analysis.

Electron backscattered diffraction (EBSD) system coupled with field emission gun scanning electron microscopy (FEG SEM) was applied to estimate lattice strain introduced by action of cavitational beam. For each sample EBSD data were taken from 250 x 250 μm (for fine-grained alloy), or from 1200 x 1200 μm (for coarse-grained alloy) area. Assessment of lattice strain was conducted by local misorienation approach [5]. This method assumes that strain induced dislocation structure development is associated with rotation of "microvolumes" (cells, subgrains) leading to increase of local misorientation between adjacent points [6]. There are few metrics that may be use for quantitative analysis in this approach [4]. In present paper, following parameters were chosen:

- GOS (Grain Orientation Spread) - the average orientation of the grain is calculated, and then the misorientation between this average orientation and the orientation of each individual measurement point within the grain is calculated.
- KAM (Kernel Average Misorientation) – for a given data point the average misorientation between the data point and all of its neighbors is calculated (exclude misorientations greater than some prescribed value - 5° in this case).

4. Results and discussion

Results of EBSD strain analysis are presented in Fig. 7-15. Fig. 7, Fig. 8 and Fig. 9 show GOS maps obtained after different exposition time for CuZn10 alloy in as-cast state and coarse- and fine-grained material, respectively. Comparison of results presented in Fig. 10a , Fig. 10b and Fig. 10c indicates that surface of coarse-grained sample gets strengthened in shorter time than its fine-grained counterpart. It may be found that, after 30 seconds of cavitational wear, fraction of points with very low (0÷1°) GOS values drop to zero for coarse-grained specimen, but for fine-grained one was gradually lowered, but still present even after 300 seconds of exposition.

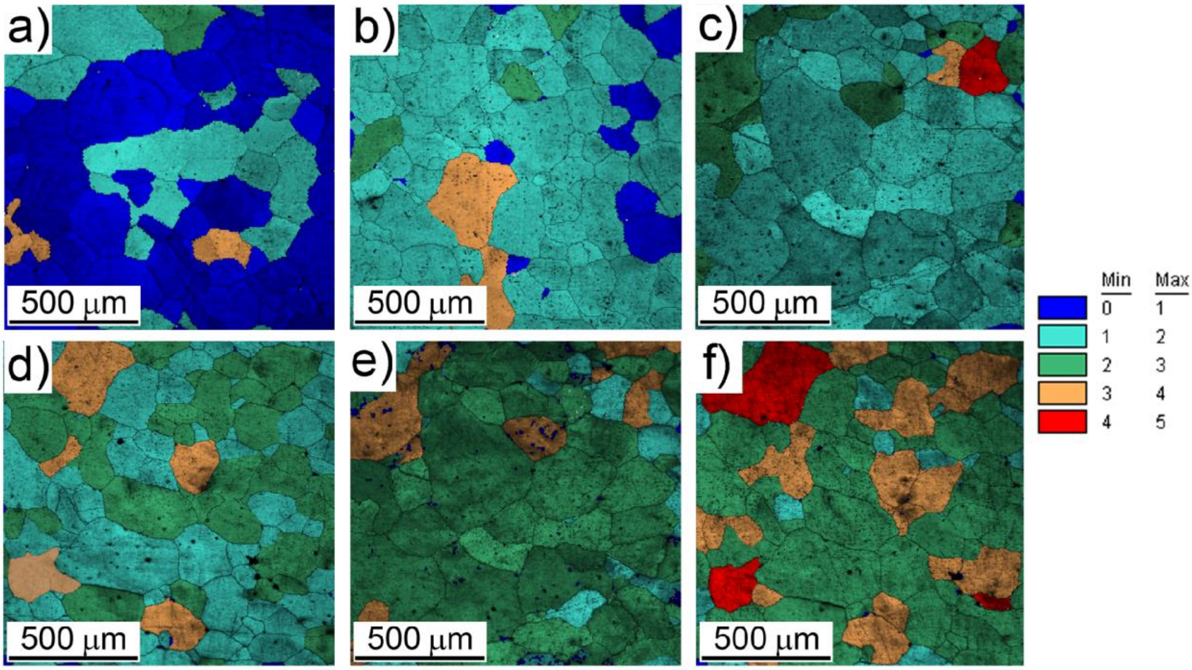

Fig. 7. Grain Orientation Spread (GOS) maps obtained for investigated alloy in as cast state after different time of cavitational exposure. a) initial state and after b) 30 seconds, c) 60 seconds, d) 120 seconds, e) 180 seconds, f) 300 seconds [7]

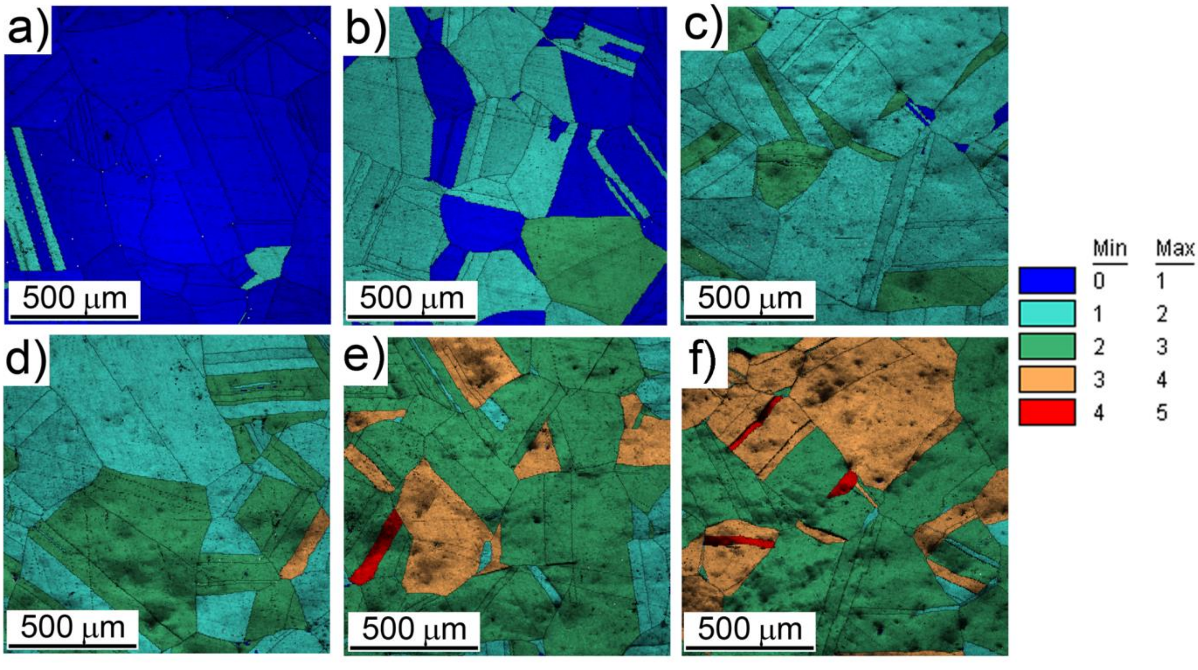

Fig. 8. Grain orientation spread (GOS) maps obtained for coarse-grained brass in as cast state after different time of cavitatational exposure. a) initial state and after b) 30 seconds, c) 60 seconds, d) 120 seconds, e) 180 seconds, f) 300 seconds

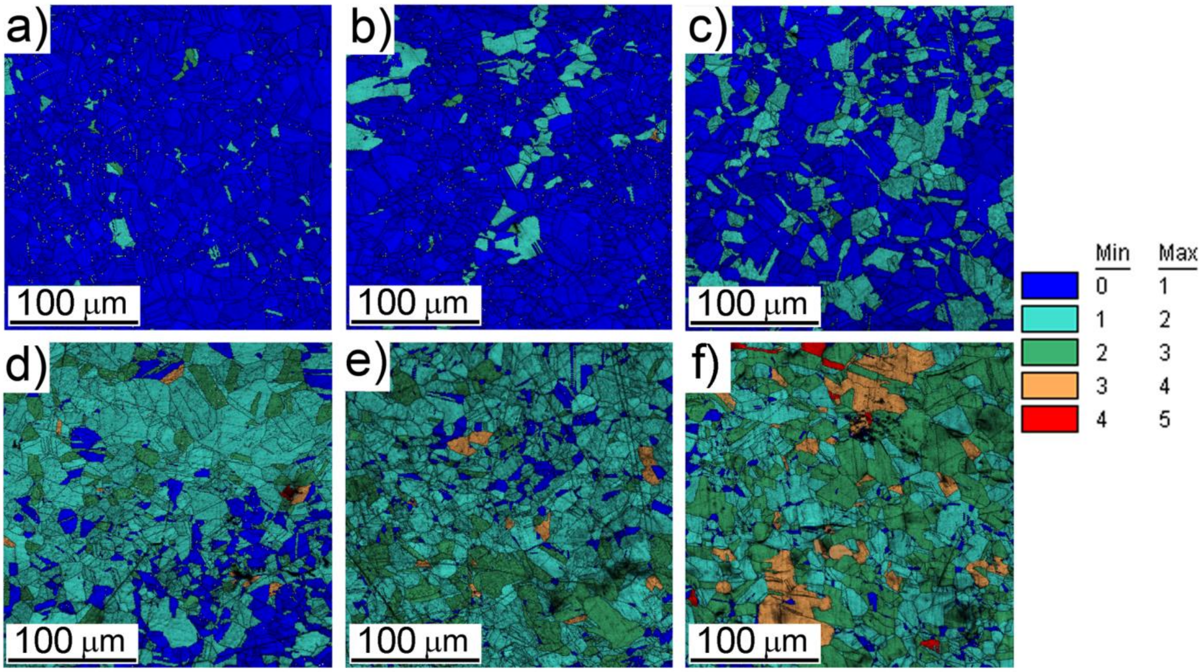

Fig. 9. Grain Orientation Spread (GOS) maps obtained for fine-grained brass in as cast state after different time of cavitational exposure. a) initial state and after b) 30 seconds, c) 60 seconds, d) 120 seconds, e) 180 seconds, f) 300 seconds

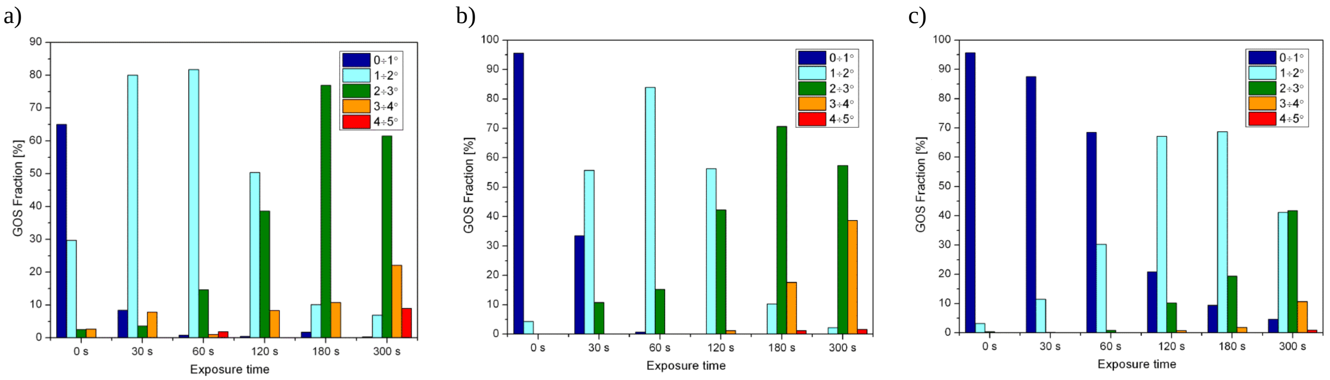

Fig. 10. Grain Orientation Spread (GOS) fractions in particular misorientation angle ranges as a function of cavitational exposure time for a) in as cast state and b) coarse-grained and c) fine-grained brass

Fig. 11 and Fig. 12 and Fig. 13 show KAM mapping for in as cast state and coarse- and fine-grained material, respectively. This parameter has different character than GOS – it is much more sensitive on very local misorientation change. Thus, mapping of KAM parameter may show strain localization in near grain boundary area, in neighborhood of small particles or in locally damaged micro-regions of material. By comparing results presented in Fig. 11 and Fig. 12 with that in Fig. 13 (confimed also by plots presented in Fig. 14), it may be found that for fine-grained material relatively large fraction of maximum KAM value was observed even for material before wear test, since this effect was not found for coarse grained sample. Above mentioned fact should be related with technological history of alloy after plastic working – due to lower value of temperature during recrystallization annealing fine-grained alloy exhibited some residual strains.

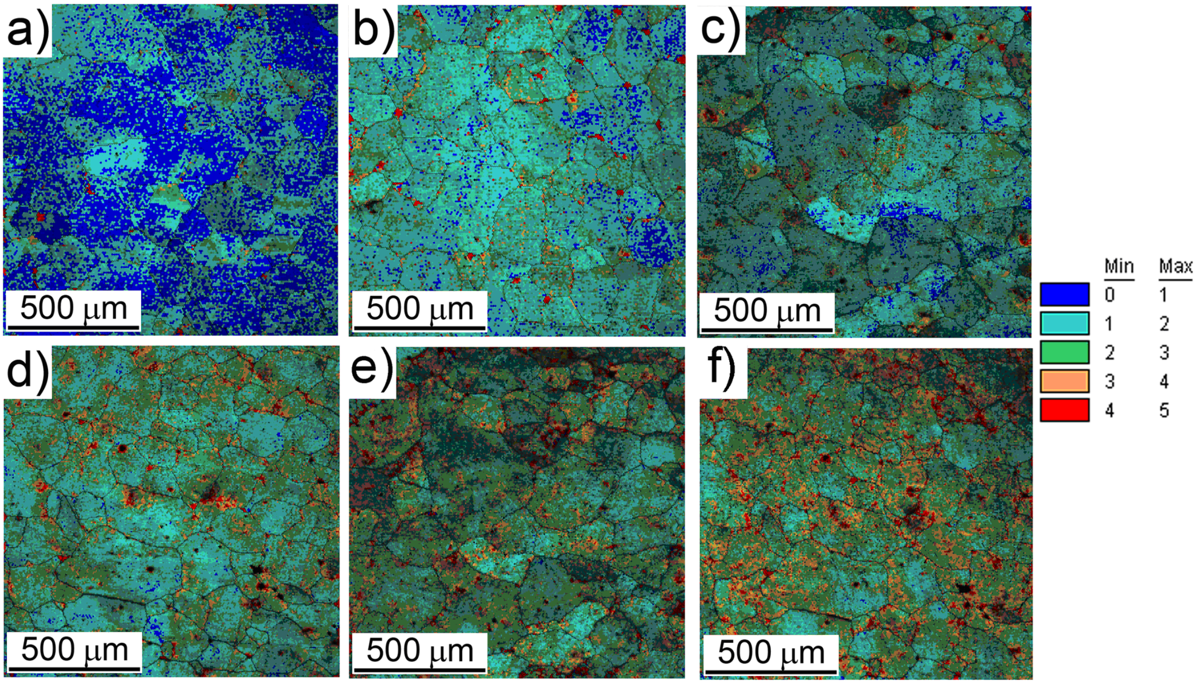

Fig. 11. Kernel Average Misoreination (KAM) maps obtained for investigated brass in as cast state after different time of cavitational exposure. a) initial state and after b) 30 seconds, c) 60 seconds, d) 120 seconds, e) 180 seconds, f) 300 seconds [7]

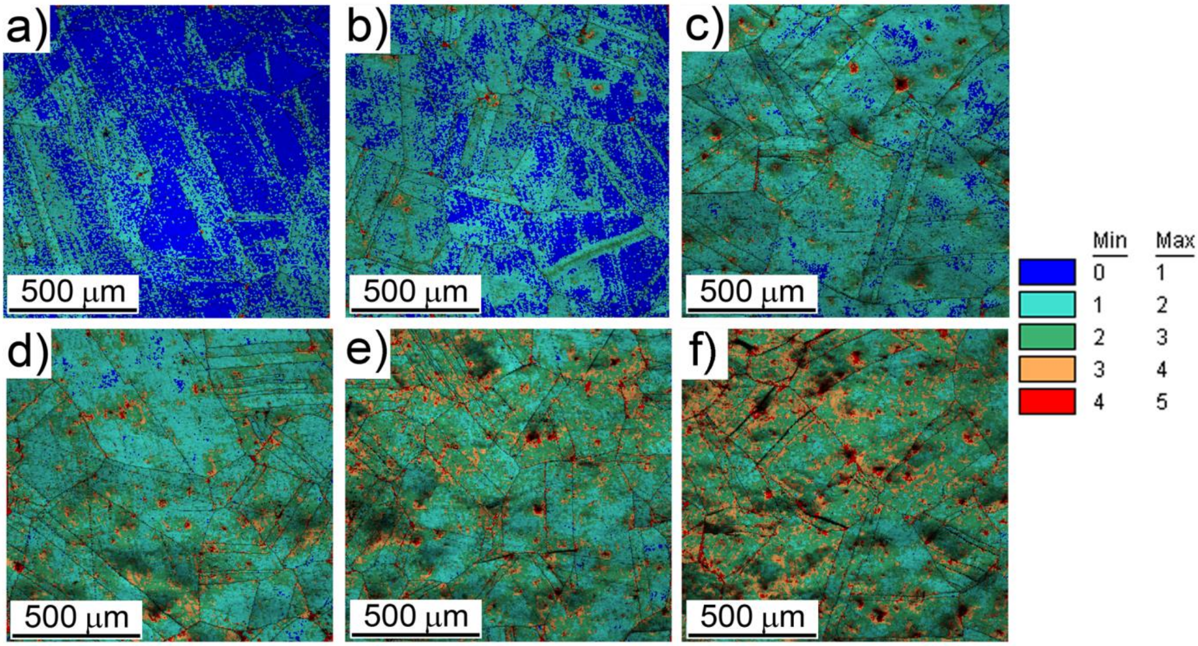

Fig. 12. Kernel Average Misorientation (KAM) maps obtained for coarse-grained brass in as cast state after different time of cavitational exposure. a) initial state and after b) 30 seconds, c) 60 seconds, d) 120 seconds, e) 180 seconds, f) 300 seconds

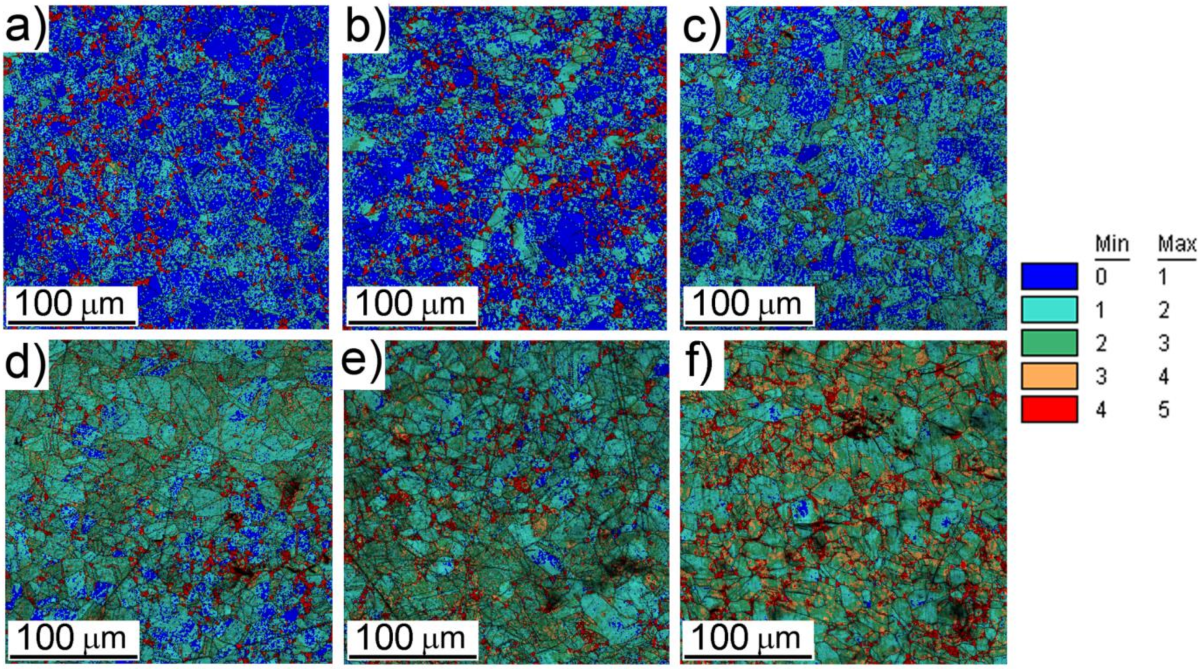

Fig. 13. Kernel Average Misorientation (KAM) maps obtained for finegrained brass in as cast state after different time of cavitational exposure. a) initial state and after b) 30 seconds, c) 60 seconds, d) 120 seconds, e) 180 seconds, f) 300 seconds

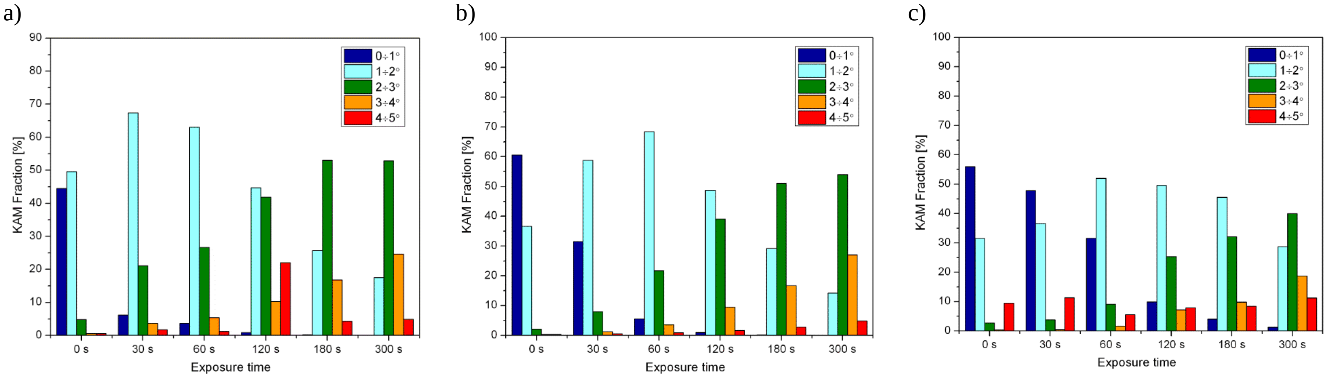

Fig. 14. KAM fractions in particular misorientation angle ranges as a function of cavitational exposure time for a) in as cast state and b) coarse-grained and c) fine-grained brass

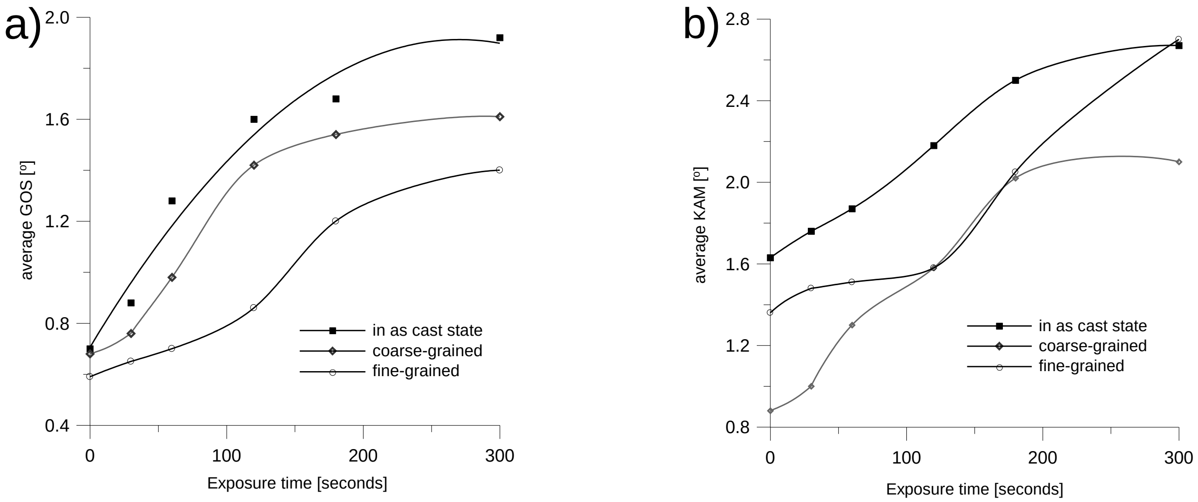

Fig. 15. Average a) GOS and b) KAM values as a function of cavitational exposure time for: in as cast state and coarse-grained and fine-grained brass

5. Conclusion

The process of the cavitation wear of structural material begins with an incubation period. The incubation period of the destruction is characterized by no mass decrement. On the surface of the material visible plastic strain effects and micro-cracks are visible. However, in the case of no mass loss of material it is difficult to determine the cavitational erosion resistance. Comparison of plastic strain in CuZn10 alloy analyzed by EBSD method performed through local misorientation approach (GOS and KAM parameters) as a function of cavitation time, show that the first local change appear on coarse-grained brass after shorter exposition time than for fine-grained one. In both cases, effect of strain localization in near grain boundary area and cavitation pitting was observed. However, confirmation of this thesis will require to carry out further research on a jet-impact device.

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