Pore Morphology Assessment of Oxide Coatings on AZ31B Magnesium Alloy

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Abstract

The shape and size of the pores in oxide coating plays significant role in the exploitation properties of the coatings. It determinates the size of the micro and nano prartcicles in lubricant that can be introduce inside of them due to enhance exploitation parameters [1-3]. The research material was an oxide coatings produced on an AZ31B magnesium alloy. Samples which size was $62.5 \times 16 \times 5$ mm were cut from 5 mm thick rolled sheet metal. Surface preparation involved grinding with SiC paper (grit 240) at 150 rpm, followed by ultrasonic degreasing in isopropyl alcohol (5 min), rinsing with distilled water, and drying. The oxidation process was carried out by plasma electrochemical oxidation (PEO) using a KIKUSUI PCR2000WEA power supply and a trapezoidal voltage waveform (max. 400 V in the positive cycle). The anode was a Mg alloy sample, and the cathode was stainless steel. The electrolyte was an aqueous solution containing 5 g/L of Na_2SiO_3 and 5 g/L of NaOH, maintained at a temperature of 303 ± 5 K using a cryostat. The electrolyte was continuously stirred during oxidation using a pump. The process parameters – current density, time, pulse frequency – were selected according to an experimental design based on the Hartley hypercube, considering:
– Current density: 10, 15, 20 A/dm^2
– Frequency: 50, 75, 100 Hz
– Time: 30, 45, 60 min.
To assess the surface porosity geometry 11 types of samples were exanimated in two fields of view each, using Hitachi TM3000 scanning electron microscope. Observation were conducted in Hitachi TM3000 at accelerating voltage of 15 kV, and magnification 1000x. Acquired SEM images had a resolution of 1280×1040 pixels, a pixel size of $0.128\,\mu$m, and were saved in grayscale TIFF format. Image processing and analysis was conducted using Aphelion software. Processing steps included noise reduction, thresholding based on maximal threshold detection, and and morphological correction (opening and closing operation) [4-6]. The accuracy of pore detection was validated by comparing detected pore edges with the original SEM images. For quantitative analysis, the following parameters were evaluated: (1) A_A – area fraction; (2) Pore surface area – statistical data on pores size; (3) N_A – the number of objects per unit area, determined using the Jeffries method; (4) Shape descriptors: (a) circularity – calculated as $4\pi \times$Area/Perimeter^2 with perimeter measured using the Crofton method; (b) Elongation – defined as the absolute difference between the major and minor axes divided by their sum; (c) BRFillRatio the ratio of the pore area to the area of its bounding rectangle.

Table. 1. Results of analysis the area of pores for all types of samples.

Sample s	A mean [μm²]	A max [μm²]	A median [μm²]	standard deviation	Circularity	Elongation	BR FillRatio
A	1.751	40,863	0,826	3,007	0,849	0,341	0,65
B	1.112	15,988	0,759	1,236	0,943	0,255	0,72
C	1.397	14,599	0,955	1,445	0,923	0,276	0,755
D	1.515	21,156	1,129	1,527	0,878	0,317	0,674
E	1.205	19,38	0,472	1,314	0,942	0,251	0,721
F	1.157	13,688	0,775	1,277	0,901	0,297	0,679
G	1.381	15,121	0,977	1,483	0,934	0,268	0,722
H	1.051	12,103	0,708	1,142	0,939	0,27	0,717
I	0.961	13,856	0,606	1,11	0,954	0,244	0,726
J	1.98	18,593	1,61	1,733	0,878	0,302	0,688
K	0.9301	8,9	0,64	0,961	0,954	0,244	0,091

As showed Table 1 obtained surface exhibit large variety of pores sizes. Shape descriptors are more consistent, and although few elongated pores, the majority were close to circular in shape. Since pores with an area larger than $1\,\mu\text{m}^2$ are the most relevant for lubricants containing nanoparticles, further research should focus on the geometry of pores $\geq 1\,\mu\text{m}^2$, analyzed at higher SEM magnification.

References

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