Structures and Properties in Varying Phase Composition of PEO Coatings

PURDUE MATERIALS ENGINEERING

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IBC Materials and Technologies, Inc. has developed a process for creating Plasma Electrolytic Oxidation (PEO) coatings on various aluminum alloy substrates. The purpose of the project was to understand the effects of IBC's proprietary process on the phase composition and mechanical properties of PEO coatings. The characterization was focused on the relationship between processing parameters and its effect on the alpha and gamma alumina volume fractions. Hardness, X-Ray Diffraction, and microscopy were performed to relate processing to properties.

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IBC Materials & Technologies INC

Project Background

Alpha phase aluminum oxide is preferred over gamma phase for many reasons. Alpha Al_2O_3 is much harder, thermodynamically stable, and more corrosion resistant. Conversely, gamma phase is thermodynamically unstable and has low surface energy [1].

To form alpha alumina requires a phase transformation from gamma phase at temperatures above 1000ºC. PEO applies a high voltage of over 100 V to the metal sample which creates a plasma at the surface. An electrostatic attraction is created between the metal substrate and the oxygen in the electrolyte bath. This lowers the phase transformation temperature and assists the nucleation growth of alpha alumina [2].

Figure 1: Surface plasma interacting with metal sample in electrolyte baths [2].

Optical Microscopy

Cells were analyzed according to ASTM E-562 standards. Sets of 25 points were randomly chosen and counted for intersections and number of cells. This data was then used to calculate the volume fractions and cell areas.

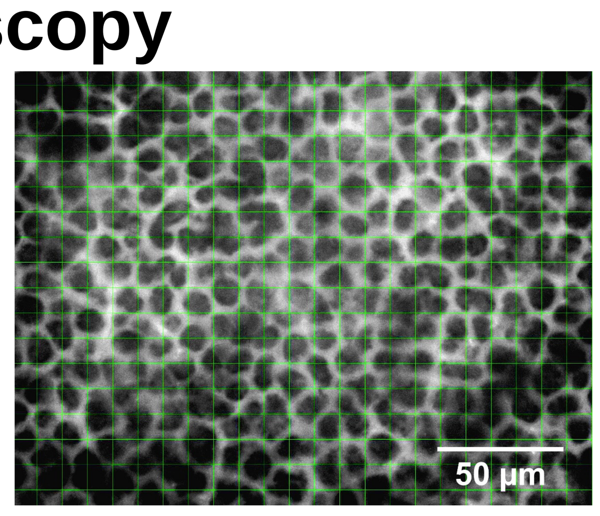
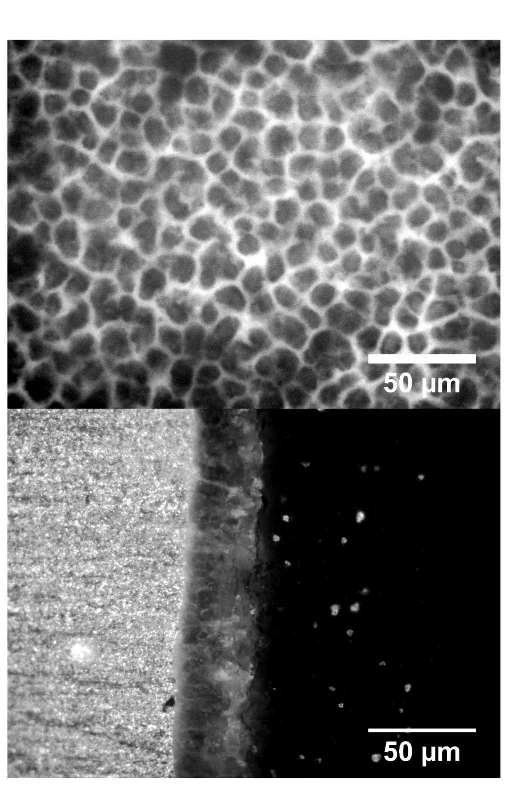


Figure 2: Optical micrographs were overlaid with a 100 μm spaced grid at a random offset.



The transparent nature of the PEO coating allows for specialized microscopy. Optical micrographs taken under polarized light show a cellular structure (top) within the PEO coating. Evidence suggest that the cells grow in a columnar pattern (bottom). SEM testing from previous research shows the cells contain higher copper and iron content than the surrounding walls.

Average cell sizes were compared with substrate alloy grade and process parameter changes. A change in process parameters and an increase in substrate alloy copper content directly relate to larger cells. Increased iron content in the substrate alloy has a negative correlation with cell size.

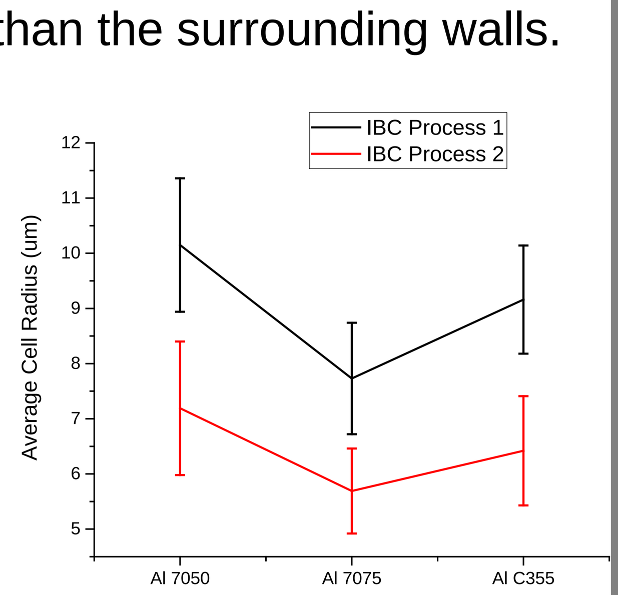


Figure 3: Average cell radii are compared by similar processing and substrate alloy grade.

Hardness

Hardness

A fixed load of 100g and 50g were applied onto the coating's surface and cross section respectively. Random points on the surface and points along the center of the cross section were evaluated.

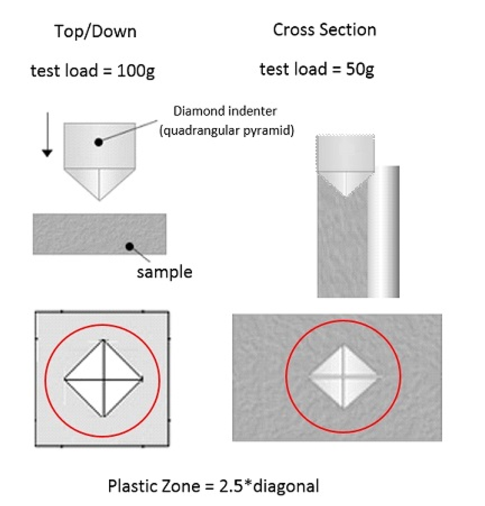


Figure 4: Micro-hardness indentation procedure using the Vickers method. Shown in red is the plastically deformed region which offsets data in close by indents.

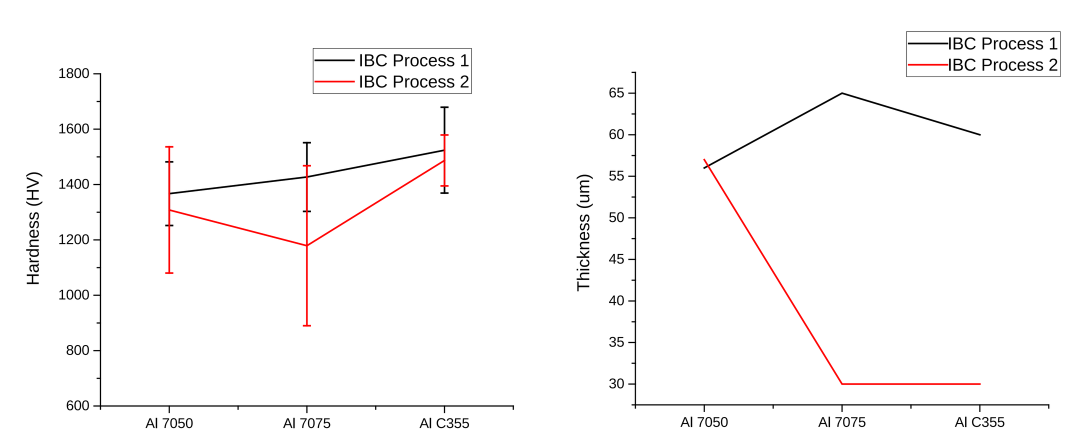


Figure 5: Hardness of the cross section was found to be more indicative of the coating. No significant difference was found between IBC processes 1 and 2 with the exception being that process 1 samples had thicker coatings.

Cell Hardness Comparison		
Location	Load (g)	Hardness (HV)
Cells	50	1434 ± 65
Cell wall	50	1266 ± 201

Using the microscope attached to the micro-indenter, the cell structure was identified on the surface of the coating. A proper load and points were selected such that indents would be able to sample the cell structure. It was found that the cells are significantly harder than the outside wall structure. The results were confirmed using a 95% significance t-test.

X-Ray Diffraction

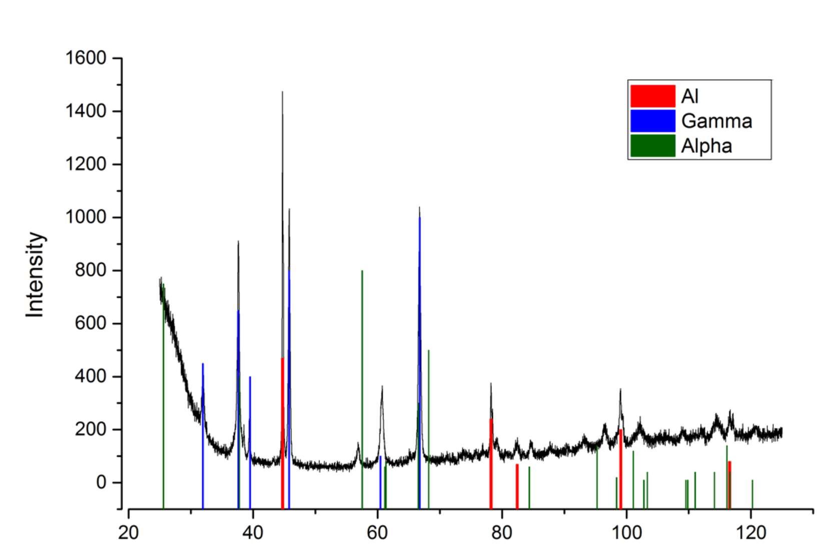


Figure 6: XRD spectra for Al 7050 A identifying the multiple phases present with the coating. The presence of Al peaks representing the substrate indicates the entire thickness of the coating was analyzed.

Coating Properties			
Alloy Grade	alpha%	Hardness (HV)	Cell Radius (μm)
Al 7050 A	6.5	1313	10.15
Al 7050 B	0.0	1300	7.19
Al 7075 C	2.7	1432	7.72
Al 7075 D	41.4	1248	5.69
Al C355 C83 E	89.6	1524	9.16
Al C355 C97 F	0.2	1487	6.41

Conclusions

The mechanical properties of PEO coatings are primarily dependent on the phase fraction of alumina present. In understanding phase growth, the substrate composition, processing parameters, and nucleation must be considered.

Hardness testing has shown that coating strength was unaffected by the different PEO processes provided. However, the cells display greater hardness than the surrounding wall. This suggests that the cells are comprised of greater amounts of alpha phase.

Cell size shows varying relationships with substrate alloying elements. Cell size increases between processes 1 and 2, also it increases with the substrate's copper content. Higher iron content within the substrate will decrease cell size.

Substrate dependence of the cell size may be due to iron and copper oxide nucleation. These inclusions provide a site for possible epitaxial growth through the coating [1][3].

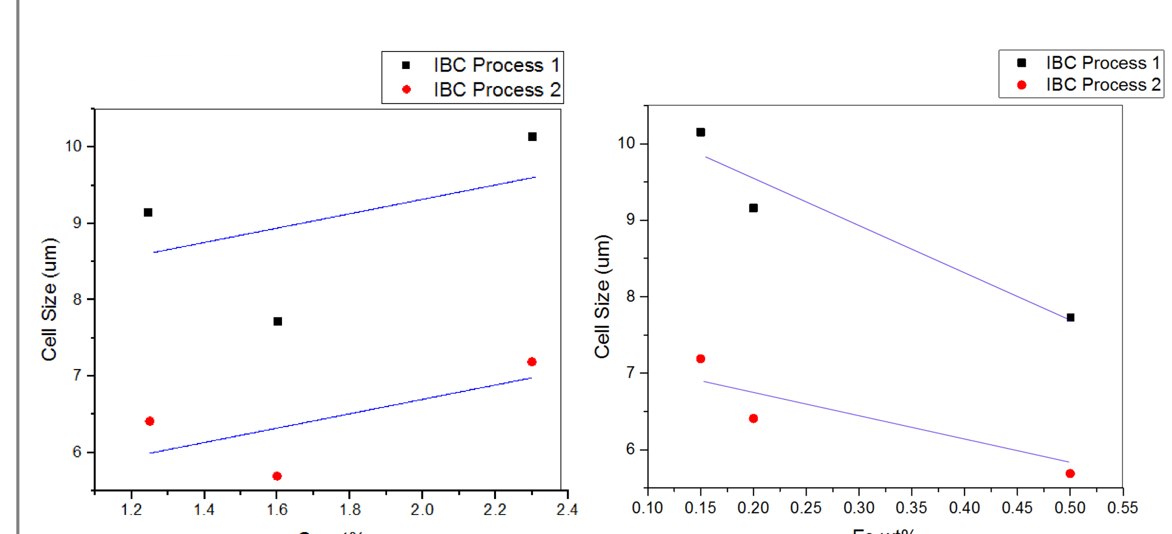


Figure 7: Cell radius is compared to the copper content within the substrate (left). Both IBC processes show and increase in the cell size as copper content increases. The iron content within the substrate alloy leads to a decrease in cell radius (right).

Recommendations

Further research should be focused on comparisons between phase composition and tribological properties. Scratch testing using a pin on disc method would be desired.

References

[1] - Andersson, J. M. (2005). *Controlling the Formation and Stability of Alumina Phases.* Linköping, Sweden: Linköping: Linköpings Universitet.

[2] - IBC Coatings. (n.d.). *Plasma Electrolytic Oxidation (PEO) | Micro Arc Oxidation | CeraTough™*. Retrieved from IBC Coatings: http://www.ibccoatings.com/plasma-electrolytic-oxidation-peo-ceratough

[3] - Wenbin X., Zhiwei D., Ruyi C., Tonghe Z. (2006). *Growth regularity of ceramic coatings formed by microarc oxidation on Al–Cu–Mg alloy*. Thin Solid Films.