Microstructural Characterization and Plasma Etching Resistance of Thermally Sprayed Al_2O_3 and Y_2O_3 Coatings

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Abstract

In this study, the plasma sprayed Al_2O_3 and Y_2O_3 coatings have been investigated for applications of microelectronic components. The plasma sprayed coatings had a well-defined splatted lamellae microstructure, intersplat pores and a higher amount of microcracks within the splats. The plasma sprayed Y_2O_3 coating had a relatively lower hardness of 300-400Hv, compared to 650-800Hv for Al_2O_3 coating, and would be readily damaged by mechanical attacks such as erosion, wear and friction. For a reactive ion etching against F-containing plasmas, however, the Y_2O_3 coating had a much higher resistance than the Al_2O_3 coating because of the reduced erosion rate of by-products.

Keywords : Al_2O_3, Y_2O_3, thermal spraying, plasma resistance

1. Introduction

Recently, plasma sprayed ceramic coatings including Al_2O_3 and Y_2O_3 have been employed as a reliable cost-effective solution in a reactive ion etching chamber where many components are exposed into physical and/or chemical attacks by corrosive gases and plasmas [1]. Plasma spraying is a complex process, which combines the injection of solid particles into a high temperature plasma flame, the melting of and acceleration of these particles, and consolidation of the sprayed molten droplets on a substrate to form a coherent coating.

In this study, ceramic oxide coatings including Al_2O_3 and Y_2O_3 have been produced onto aluminum substrates by plasma spraying. The plasma sprayed coatings have been investigated in terms of microstructural features and hardness. Furthermore, the plasma etching resistance of the Al_2O_3 and the Y_2O_3 coatings against CF_4/O_2 plasmas has also been evaluated for the application of microelectronic components.

2. Experimental and Results

Feedstock powders used were prepared in-house using conventional spray-drying from high purity (>99.9%) Al_2O_3 and Y_2O_3 raw materials. Plasma spraying was performed using a Praxair SG-100 gun with a mixture of Ar and He gases. Optimum plasma spray parameters for each ceramic oxide coating were used in order to minimize coating porosity. The plasma power used was 38.7kW for the Al_2O_3 coating and 36.0kW for the Y_2O_3 coating.

Microstructural investigation of the plasma sprayed coatings was carried out on polished cross-sections using a scanning electron microscopy. Microhardness was measured using a Vicker's indenter at an applied load of 200g. The plasma etching experiment was carried out using an inductively coupled plasma (ICP) system with CF_4/O_2 gases at a standard etching rate of 100nm/min for SiO_2.

Fig. 1 shows SEM micrographs of the coating cross-section for (a) Al_2O_3 and (b) Y_2O_3, respectively. Both plasma sprayed Al_2O_3 and Y_2O_3 coatings showed the characteristic lamellae microstructure built up many individual splats, and each splat was separated by thin layer of voids resulting from poor intersplat contact. Small amounts of unmelted particles were also incorporated in the coatings, and few micron-sized voids were formed around them. The individual splats contained several microcracks which were perpendicular to the splat boundary. The microcraks inside splats were attributed to a large thermal contraction of each splat during cooling. It is generally accepted that the sufficient powder melting in the high temperature plasma flame, and then the higher liquid fraction in the sprayed droplet leads to an increase in flattening of splat on impact to form a thin lamella [2]. There was no significant difference in microstructural features between the Al_2O_3 coating and the Y_2O_3 coating.

The average microhardness values of the Al_2O_3 and Y_2O_3 coatings are shown in Table 1. The Al_2O_3 coating had much higher microhardness than the Y_2O_3 coating.

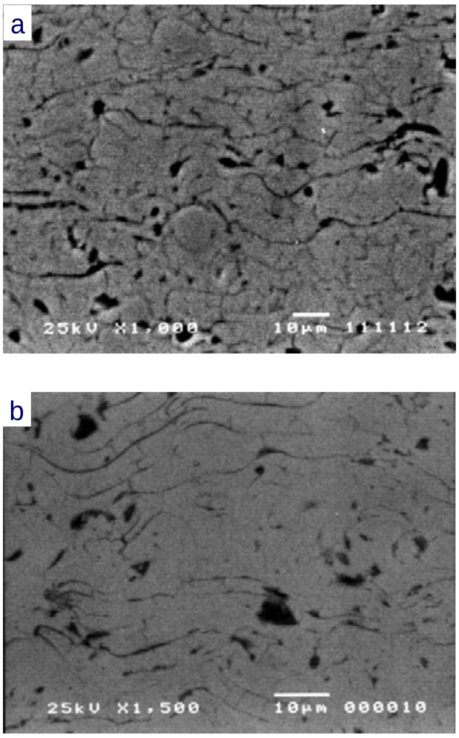

Fig. 1. Backscattered images of plasma sprayed (a) Al_2O_3 and (b) Y_2O_3 coatings.

The hardness data had non-lognormal distribution and exhibited a large amount of scatter, ranging 628-854Hv for the Al_2O_3 coating 268-385Hv for the Y_2O_3 coating. Therefore, the hardness data were presented using a two-parameter Weibull distribution, as shown in Fig. 2. Both plasma sprayed ceramic coatings had a bimodal distribution which was indicative of an inhomogeneous microstructural features in the coatings.

Table 1. Microhardness data of plasma sprayed oxide coatings

Al_2O_3 coating	724±54
Y_2O_3 coating	335±36

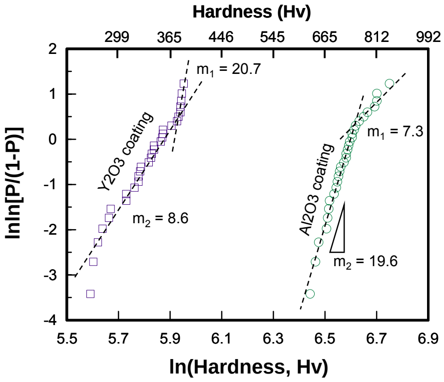

Fig. 2. Weibull plot of microhardness of plasma sprayed Al_2O_3 and Y_2O_3 coatings.

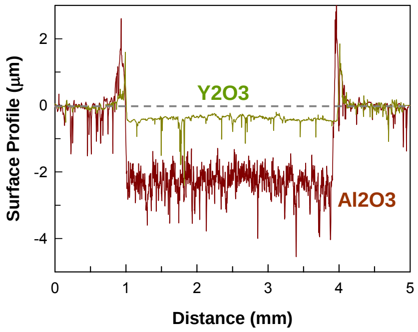

Fig. 3. Surface profiles of plasma sprayed Al_2O_3 and Y_2O_3 coatings after plasma erosion etching in ICP system.

Fig. 3 shows the surface profiles of the plasma sprayed Al_2O_3 and Y_2O_3 coatings after a plasma reactive etching with CF_4/O_2 gases in ICP system. The Y_2O_3 coating had a much reduced etching removal which indicated an improved resistance against corrosive plasmas, compared to the Al_2O_3 coating. When exposed to F-containing plasma, the Al_2O_3 reacted with F to form a mixture of AlF_3 and $Al_xO_yF_z$. Similarly, the Y_2O_3 also reacted and formed a mixture of YF_3 and $Y_xO_yF_z$. These reaction products were not volatile, and remained on the coating surface. Subsequent ion bombardment by rf biasing power would remove the reaction products from the coating surface. The enhancement of plasma resistance of the Y_2O_3 coating was attributed to the reduced erosion resistance of YF_3 compared to that AlF_3 in the Al_2O_3 coating.

3. Summary

The plasma sprayed Al_2O_3 and Y_2O_3 coatings had a well-defined splatted lamellae microstructure, intersplat pores and a higher amount of microcracks within the splats, resulting from the rapid solidification of small globules, flattened from striking a cold surface at high velocities. For evaluation of plasma etching resistance, the coatings were exposed to F-containing plasmas in an inductively coupled plasma chamber. The oxide coatings reacted with F to form AlF_3 or YF_3 as byproducts which were not volatile. The Y_2O_3 coating had a much lower plasma etching rate than the Al_2O_3 coating. (Acknowledgements: This research was supported by a grant (06K1501-00822) from CNMT under '21st Century Frontier R&D Programs', Korea.)

4. References

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