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Fabrication and Properties of Plasma Resistant YAS Frit-coated Al_2O_3

Eui-Keun Park, Hyun-Kwuon Lee
Department of Advanced Materials Science and Engineering/Kumoh National Institute of Technology
61 Daehak-ro (yangho-dong), Gumi, Gyeongbuk 730-701, Korea, Republic of
Czarkhan86@naver.com; hklee@kumoh.ac.kr

Abstract - Recently, as a realization of ultrafine circuit wafer process in semiconductor industry, highly plasma resistant ceramic materials have been continuously required for its severe etching process. Bulk Y_2O_3 ceramics and Y_2O_3-coated ceramic components by plasma thermal spray process have been paid great attentions as candidates. However, application of the bulk Y_2O_3 materials is highly expensive; furthermore, surface failure of Y_2O_3-coated ceramic components by plasma thermal spray process causes serious problems such as contamination and low production yield. In order to solve this problem should be studied on the surface reinforcement of ceramics consistently.

In this study, YAS (Y_2O_3-Al_2O_3-SiO_2) frit was fabricated by melting method. Then, fabricated YAS frit was coated on the surface of Al_2O_3 ceramics by simple coating process for improved plasma resistance. Effect of processing parameters on YAS frit-coated Al_2O_3 fabrication and effect of YAS frit composition on the plasma resistance were studied, and tried to be optimized. Plasma resistance of YAS frit-coated Al_2O_3 ceramics was improved with increasing Y_2O_3 content in YAS system; its plasma resistance was 6 times higher than quartz, 2 times than Al_2O_3 and a half of Y_2O_3.

Keywords**:** YAS, Y_2O_3-Al_2O_3-SiO_2, coating, plasma resistance

1. Introduction

Etching and deposition equipment are necessarily in use for surface micromachining or removal of impurities in semiconductor or display industry. Recently, as a realization of ultrafine circuit wafer process in semiconductor industry, highly plasma resistant ceramic materials have been continuously required for its severe etching process [1].

The typical plasma resistant ceramic is oxide material such as Al_2O_3, Y_2O_3, etc. Especially, Y_2O_3 has been reported to have highly plasma resistance [2]. Bulk Y_2O_3 ceramics and Y_2O_3-coated ceramic components by plasma thermal spray process have been paid great attentions as candidates [3]. However, application of the bulk Y_2O_3 materials is highly expensive owing to high price of the Y_2O_3 materials itself; furthermore, surface failure of Y_2O_3-coated ceramic components by plasma thermal spray process causes serious problems such as contamination and low production yield[2,4].

This study going to develop the plasma resistant ceramic materials by simple coating process on the surface of alumina ceramics using YAS(Y_2O_3-Al_2O_3-SiO_2) frit that include Y_2O_3. YAS glass is widely used in structural and functional ceramic field because of their promising high corrosion resistance, mechanical and optical properties [7].

In this study, YAS frit was coated on the surface of Al_2O_3 ceramics to solve contamination and high-cost of the semiconductor and display manufacturing process as fabricated low-cost plasma resistant materials. For its fabrication, YAS frit was designed by decreased or increased content of each oxide such as Y_2O_3, Al_2O_3 and SiO_2. And then, Effect of processing parameters on YAS frit-coated Al_2O_3 fabrication and effect of YAS frit composition on the plasma resistance were studied, and tried to be optimized.

2. Experimental

2.1. Raw materials

Commercial powders of Y_2O_3 (>99.99%, Kojundo Korea Co.,LTD, Japan), Al_2O_3 (>99.99%, Kojundo Korea Co.,LTD, Japan), SiO_2 (>99.99%, SukgyungAT, Korea) were used as raw materials.

2.2. Sample preparation

The raw materials were mixed according to each compositions (Fig. 1), and homogenized by ball milling with Al_2O_3 balls as medium and ethanol as solvent. Then, the slurry was dried to get the batch powder for YAS frit. Each batch powder was loaded into an alumina crucible and heated in an electric furnace. After then, the sample was naturally cooled down in the furnace. Fabricated YAS frit was coated on the surface of Al_2O_3 ceramics for improved plasma resistance.

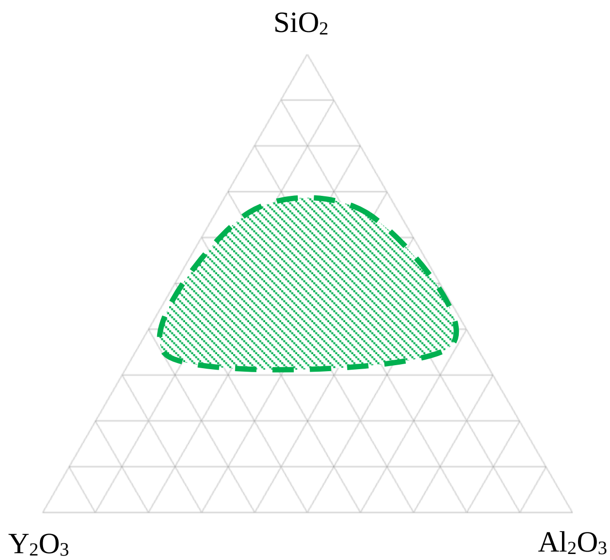

Fig. 1: Ternary phase diagram of the YAS (Y_2O_3-Al_2O_3-SiO_2) system. The batch compositions investigated in this work are marked by area (mol%).

2.3. Analysis

Prepared sample was tested according to the listed in Table 1. The surface of microstructure before and after plasma etching was analysed using a Field Emission Scanning Electron Microscope (FE-SEM, JEOL, JSM-6500F) with and energy-dispersive spectrometer (EDS). Etching rate of composition and materials that are Quartz, Y_2O_3, Al_2O_3 and YAS-frit coated Al_2O_3 was evaluated by weight loss before and after plasma etching.

Table 1: Parameters for anti-plasma test.

Parameter	Condition
Top RF Power, W	900
Bottom RF Power, W	200
CF4, Sccm	30
Ar, Sccm	10
O2, Sccm	5
Temperature, ℃	30
Pressure , mTorr	10
Operating time, hr	10

3. Results and Discussion

Degree of YAS frit crystallization and Y_2O_3 content were proportional to each other owing to decreasing bridge-oxygen that can be network forming as increased Y_2O_3 content. And effect of network forming oxide (NWF) was decreased to degree of crystallization as Al_2O_3 content [7, 8]. It is evident that the degree of YAS frit crystallization strongly depends on its composition.

Fig. 3.1 presents the cross-section of fabricated YAS frit was coated on the surface of Al_2O_3 ceramics. Dense and thick YAS-frit coated layer was observed is shown Fig. 3.1, its layer thickness was around 80 ㎛. In the every samples, Y-Al-Si-O glasses were obtained with crystalline of Y-Al-O and Y-Si-O. Is shown Fig. 3.2, YAS frit coated layer was joined strongly with Al_2O_3 ceramics.

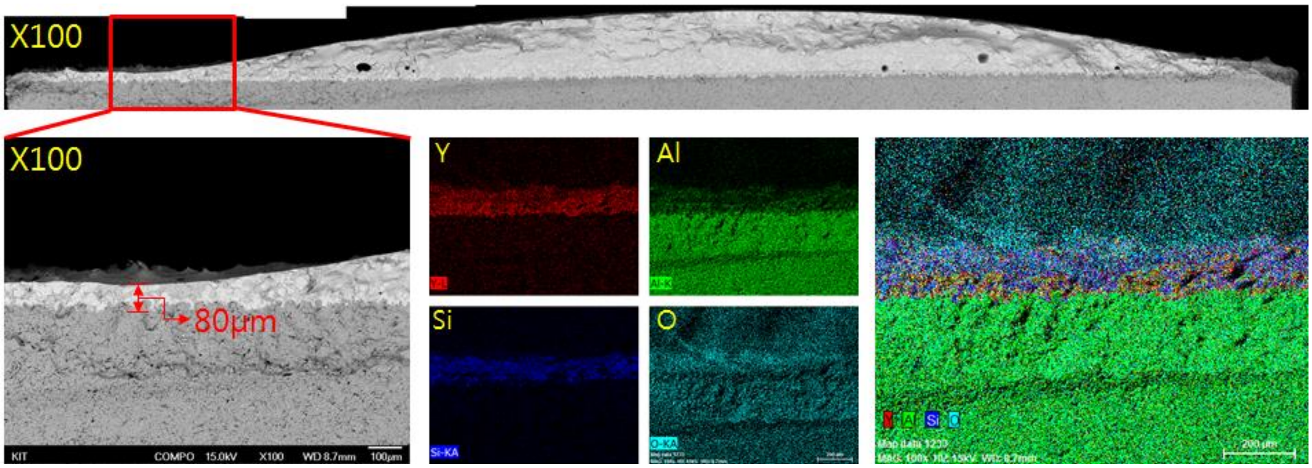

Fig. 3.1: FE-SEM and EDS mapping for cross-section of YAS frit-coated Al_2O_3 ceramics.

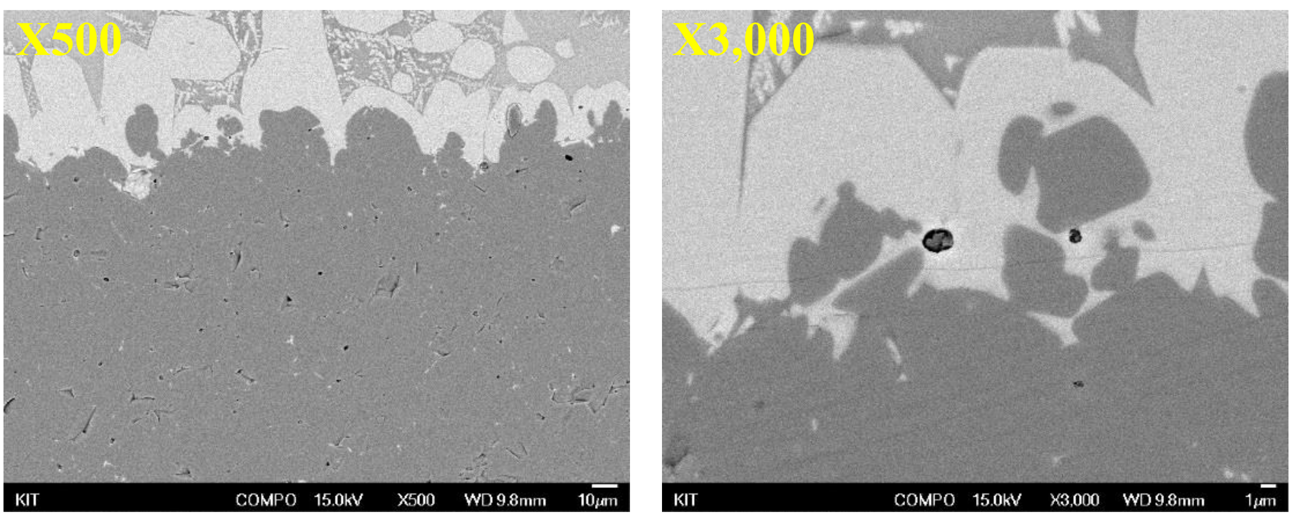

Fig. 3.2: FE-SEM image for joint interface of YAS frit-coated Al_2O_3 ceramics.

The plasma resistance and Y_2O_3 content were proportional to each other, composition of the highest Y_2O_3 content has highest plasma resistance. Fig. 4.1 presents comparison of the etching rates between commercial Y_2O_3, Al_2O_3, Quartz that the most commonly used plasma resistant ceramic materials with YAS-frit coated Al_2O_3 ceramics. Plasma etching rates of the YAS-frit coated Al_2O_3 ceramics (0.122%) was 6 times higher than quartz (0.739%), 2 times than Al_2O_3 (0.253%) and a half of Y_2O_3 (0.066%).

Is shown Fig. 4.2, comparison result between each materials using FE-SEM after anti-plasma test was shown that the quartz was chemically etched because of the chemical reaction between Si-element and fluorine gas and Al_2O_3 was physically etched. In Y_2O_3 material case, it nearly was not etched. In reality, It has been reported that the etch products are usually of the formula SiF_x owing to be produced by the interaction between radicals or ions of fluorine gas and silicon atoms of the materials being etched[9,10]. Because of that the Quartz was rapidly etched by plasma than the Y_2O_3, Al_2O_3, and YAS-frit coated Al_2O_3 ceramics.

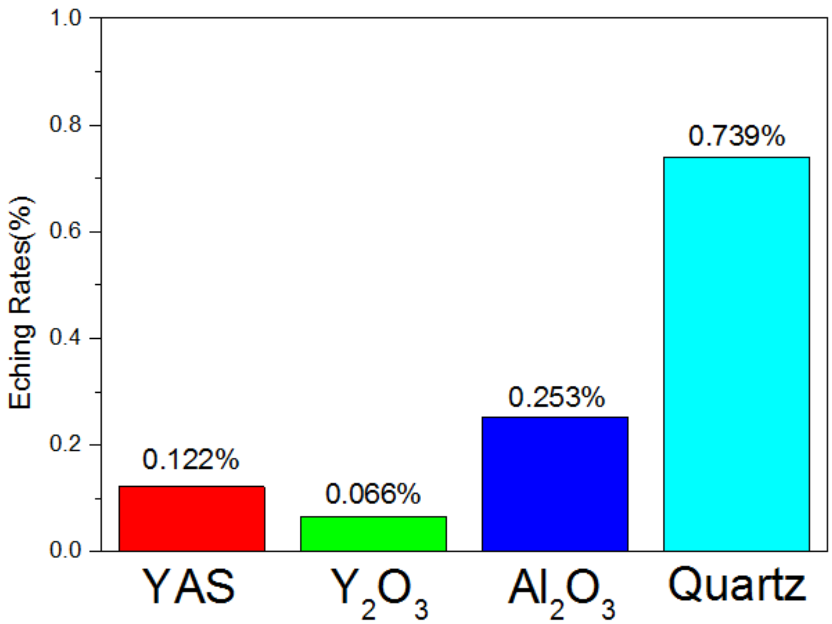

Fig. 4.1: Etching rates after fluorine plasma exposure for 10hr for YAS frit-coated Al_2O_3, Y_2O_3, Al_2O_3, Quartz, respectively.

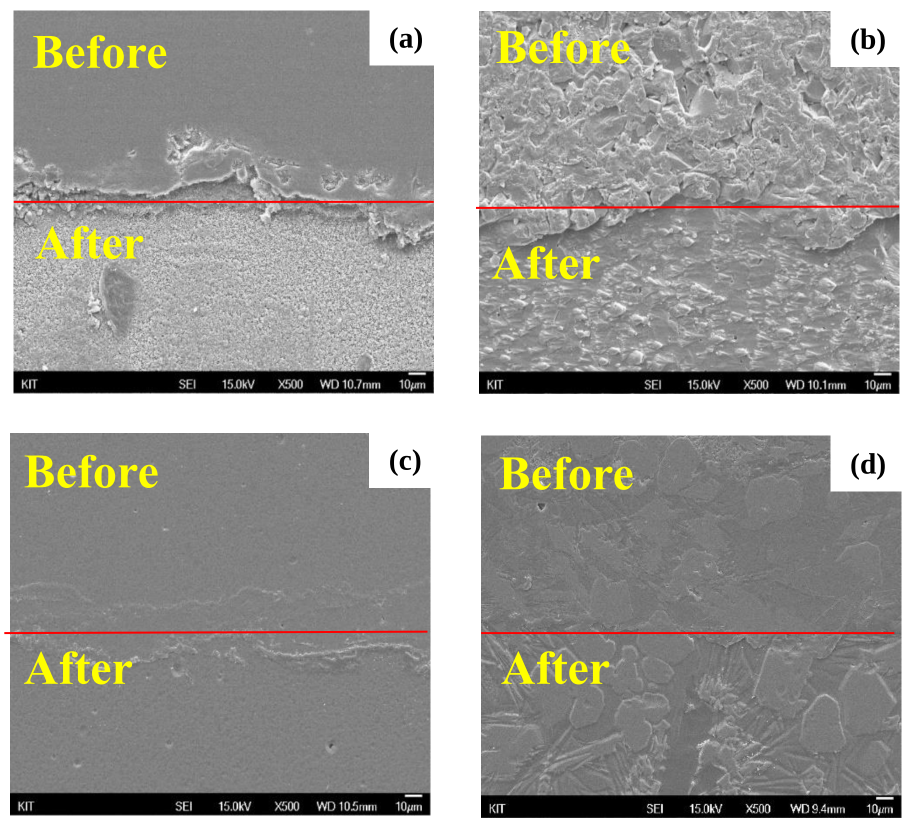

Fig. 4.2: FE-SEM images for (a) quartz, (b) Al_2O_3, (c) Y_2O_3, (d) YAS-frit coated Al_2O_3 after anti-plasma test.

4. Conclusion

The purpose of this study was to investigate that plasma resistance was improved by YAS frit coating on the surface of alumina ceramics. For this, fabricated YAS frit was coated on the alumina ceramics and then, plasma resistance was evaluated as each composition. The conclusions of the study were as follows. After YAS frit coated on the alumina ceramics, YAS frit became dense and thick coating layer (~80 ㎛) that was crystalline and amorphous of Y-Al-Si-O. The plasma resistance and Y_2O_3 content were proportional to each other, composition of the highest Y_2O_3 content has highest plasma resistance; its plasma resistance was 6 times higher than quartz, 2 times than Al_2O_3 and a half of Y_2O_3.

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