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Dense Yttria Film Deposited on a Plasma-Sprayed Al_2O_3 Coating by Aerosol Deposition

B.-K. Lee[1], J.-H. Jung[1,2], B.-D. Hahn[1], W.-H. Yoon[1], D.-S. Park[*1], J.-J. Choi[1], J. Ryu[1], J.-W. Kim[1], C. Ahn[1], K.-M. Song[2]

[1]Functional Ceramics Research Group, Korea Institute of Materials Science, Changwon, Gyeongnam, 642 – 842, South Korea
[2]Department of Applied Physics, Konkuk University, Chungju, Chungbuk, 380 – 701, South Korea
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Abstract

Dense yttria (Y_2O_3) film was deposited on a plasma-sprayed alumina (Al_2O_3) surface by means of aerosol deposition (AD). The average surface roughness value (Ra) of the plasma-sprayed alumina was varied from 0.3 μm to 4.3 μm by grinding and grit-blasting with fine and coarse alumina abrasives. The ground alumina surface with an Ra value of 0.3 μm was too smooth for deposition of the yttria film by AD. A 30-μm-thick yttria film with 95.3 % TD was successfully deposited on the alumina surfaces with Ra values between 0.5 μm and 1.8 μm. On the other hand, the alumina surfaces with Ra values larger than 2.4 μm exhibited local areas where part of the alumina layer was removed by the grit blasting and microscopically smooth areas were exposed. Deposition of yttria by AD was not accomplished on those local areas. The results suggested that the optimum surface of the plasma-sprayed alumina should be microscopically rough to ensure strong adhesion of the yttria film onto the alumina surface during AD. The yttria film exhibited superior resistance to CF_4 + Ar gas plasma erosion compared with the plasma-sprayed alumina coating.

Keywords: Yttria film, alumina, surface roughness, grit blasting, aerosol deposition

I. Introduction

Chamber parts of semiconductor processing and flat-panel-display processing (FPD) equipment often need electrical insulation and chemical protection. Sintered ceramics including alumina, aluminum nitride and others have been used for such parts owing to their excellent electrical property and chemical stability. However, recent progress requires scale-up of the production equipment and the chamber parts. This becomes more difficult, expensive and time-consuming as the size of part to be sintered is increased [1,2]. For making some of the large parts, ceramic coating might be an alternative. The electrostatic chuck (ESC) is an exemplary chamber part. It is a device that holds the wafer in semiconductor processing by means of high-voltage-driven electrostatic force. The voltage applied to the ESC ranges up to several kilovolts [3]. Thermal spraying has been considered for fast deposition of thick ceramic films on large-size parts of semiconductor and flat-panel-display equipment [1,2]. However, thick films deposited by thermal spraying often contain significant porosity that could deteriorate their properties as observed in the previous literature [1,2]. Furthermore, pores on the surface were subjected to an intensive attack by fluorine plasma as reported by Kim *et al.* [4].

AD has been reported to deposit dense nano-structured ceramic films on metals and glass at room temperature [5–7]. Akedo described how dense ceramic films were formed by AD [5]. Iwasawa *et al.* successfully deposited yttria film on quartz substrate that was over 95 % dense and consisted of crystallites smaller than 20 nm [6]. According to their report, the yttria film was up to 20 μm thick and there was an about 50-nm-thick anchor layer at the interface between the quartz and yttria film [6]. Interlocking between the substrate and the film in the anchor layer provided a strong adhesion of the film to the substrate as indicated by Akedo [5].

In this study, we employed AD for depositing dense yttria film on plasma-sprayed Al_2O_3. Surface roughness of the Al_2O_3 was varied in order to investigate its effect on deposition of yttria by means of AD.

II. Experimental Procedure

Aluminum alloy plates (20 mm × 20 mm × 5 mm (thickness)) with plasma-sprayed alumina were purchased from a local company (Acecoat Corp., Haman, Korea). The alumina was ground with a 600-grit diamond wheel until its thickness became 200 μm. The ground alumina surface was masked with scotch tape to expose only a quarter of the surface. The exposed surface was then subjected to grit blasting with either a 200-grit or 40-grit alumina abrasive (99.6 %, A&C Chemicals Pty. Ltd., Naranda, Aus-

* Corresponding author: pds1590@kims.re.kr

tralia). Air pressure for the grit blasting was varied from 0.1 MPa to 0.5 MPa. Surface roughness of the ground and grit-blasted alumina coating was measured with a surface profilometer (Perthometer PGK 120, Mahr GmbH, Göttingen, Germany). Samples were named according to the grit blasting conditions as shown in Table 1.

Table 1: Grit blasting conditions and corresponding sample numbers

Grit blasting condition	Grit number	Air pressure (MPa)	Sample name
	As-ground	As-ground	Sample 0
Condition 1	200	0.2	Sample 1
Condition 2	200	0.4	Sample 2
Condition 3	200	0.5	Sample 3
Condition 4	40	0.1	Sample 4
Condition 5	40	0.3	Sample 5
Condition 6	40	0.5	Sample 6

A commercially available yttria powder (AT grade, H. C. Starck, Goslar, Germany) was used for AD. The yttria powder was heat-treated at 1473 K for 2 h in air. Then, it was sieved using a 100-mesh screen for breaking large soft agglomerates. The average particle size (d_{50}) of the yttria powder after sieving was measured at 3.5 μm with a dry particle size analyzer (Helos & Rodos Windox 5, Sympatec GmbH, Clausthal-Zellerfeld, Germany). After sieving, the powder was poured in an "aerosol chamber" that was mechanically shaken to generate fine particle mist inside the chamber. Details of the AD procedure were described in the previous report [7]. Two scotch tapes with about 3 mm width were cross-applied for masking the middle of the alumina surface of the substrate to separate four areas with different surface roughness for AD of yttria (Fig. 1). Then, the substrate was fixed onto a motored stage using double-sided tape in the deposition chamber, which was evacuated by a rotary pump with mechanical booster pump. The substrate surface was positioned 10 mm away from the nozzle and reciprocally moved 15 times at 10 mm/s. Yttria particles were sprayed onto 20 mm x 20 mm surface of the alumina through a nozzle with slit-type opening of 0.8 mm x 35 mm in the deposition chamber. The schematic in Fig. 1 shows the arrangement of the nozzle slit and the substrate. Medical-grade compressed air was used for carrying the fine yttria particles and its flow rate was 10 L/min. During AD, pressure in the deposition chamber was about 920 Pa.

After AD of the yttria, the surface was analyzed with an X-ray diffractometer (D-Max 2200, Rigaku Co., Tokyo, Japan), scanning electron microscope (JSM-5800, Jeol Co., Tokyo, Japan) with EDS. Samples were cleaned in an ultrasonic bath and sputter-coated with gold for SEM observations. To examine the interface between the alumina and AD yttria film, a cross-section TEM sample was carefully prepared with FIB. HR-TEM was employed for examining the interface. Some samples were mounted using epoxy and polished down to 1 μm diamond paste for examining the cross-section. Density of the AD yttria film was measured with the liquid immersion method using xylene.

A dry etcher (BEP-5000, ICP-etching System, SN Tek, Gimpo, Korea) at the National Center for Nanomaterials Technology, Pohang, Korea was employed for etching by CF_4 + Ar gas plasma. Gas flow rates were 45 sccm and 15 sccm for CF_4 and Ar, respectively. Source power and bias power were 800 W and 500 W, respectively. The surface of the sample was partially covered with a small piece of silicon wafer during etching. The height difference between the etched and un-etched surface of each sample was obtained with a surface profilometer. The etching rate was obtained by dividing the etch depth by the exposure time (0.5 h).

III. Results

Ra values were 0.3, 0.5, 1.6, and 1.8 μm for the alumina surfaces before and after grit blasting under conditions 1, 2 and 3, respectively. When the abrasive particle size was increased from 200 grit (65 μm) to 40 grit (425 μm), the average surface roughness of the grit-blasted alumina was increased to 2.4, 2.9, and 4.3 μm under conditions 4, 5, and 6, respectively. Figs. 2(a) and (b) show SEM micrographs of typical alumina surfaces after grit blasting with 200-grit abrasive and 40-grit abrasive, respectively.

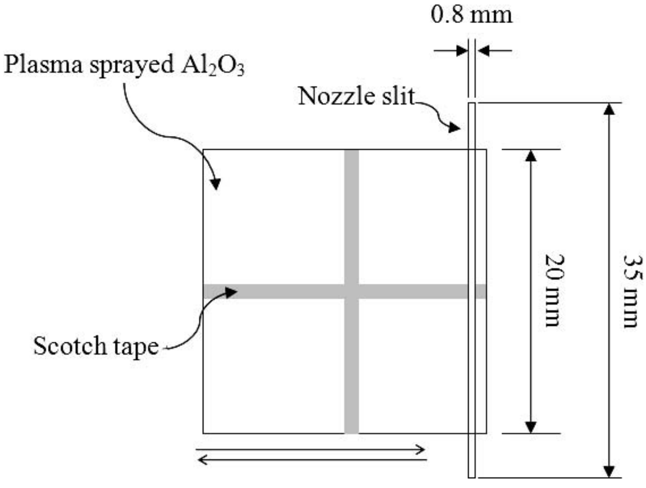

Fig. 1: Schematic diagram showing the arrangement of the substrate and the nozzle.

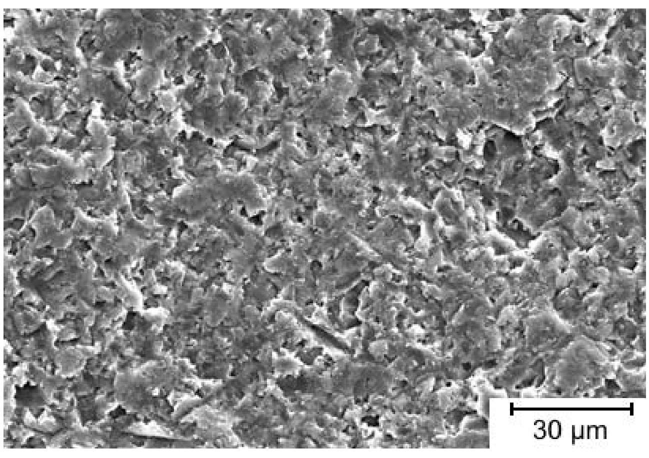

Fig. 2a: SEM micrographs of the plasma-sprayed alumina coating surface after grit blasting under condition 3.

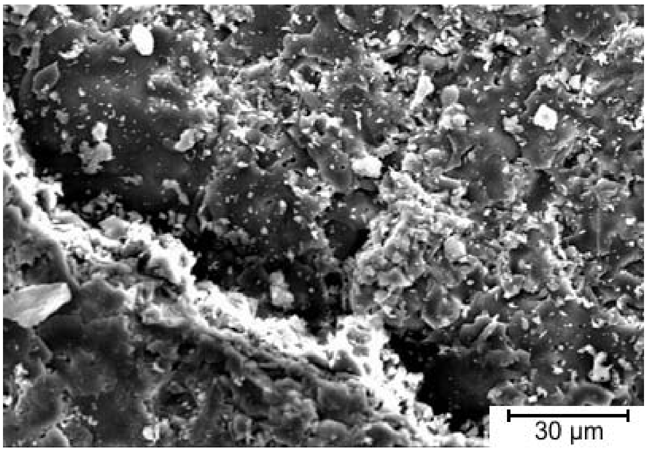

Fig. 2b: SEM micrographs of the plasma-sprayed alumina coating surface after grit blasting under condition 5.

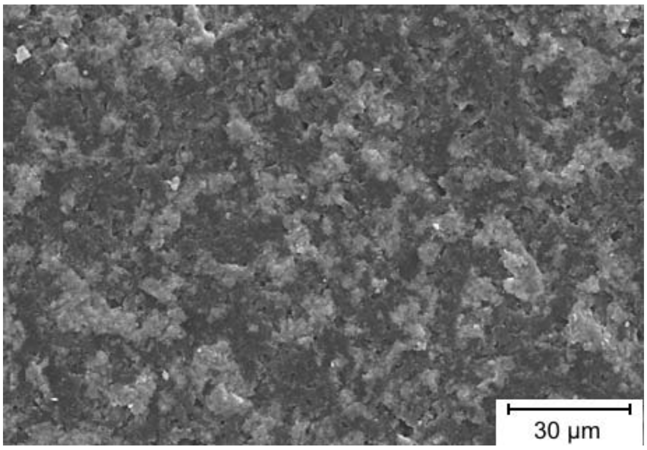

Fig. 3a: SEM micrographs of the samples after AD of yttria; sample 0.

While blasting with 200-grit abrasive generated small shallow craters on the plasma-sprayed alumina surface, blasting with 40-grit abrasive locally removed a portion of alumina, resulting in a deep crater on the surface. The yttria film of sample 0 was peeled off after deposition. As time went by, the area of yttria film that peeled off the alumina surface was increased, suggesting insufficient adhesion between the film and the alumina surface. Yttria films on the alumina surface blasted with 200-grit abrasive were sound. The yttria film was about 30 μm thick. It was also observed that the yttria films on the rough alumina surfaces blasted with 40-grit abrasive, i.e. yttria films on samples 4, 5, and 6, exhibited many localized defects. Fig. 3(a) shows the alumina surface after yttria film was peeled off. Many surface pores shown in Fig. 3(a) were filled with yttria but a major portion of the ground alumina surface was too smooth to form the anchor layer with the yttria film. Fig. 3(b) shows the surface of sample 3. A typical net-looking surface morphology of ceramic films deposited by AD was observed [7]. Fig. 3(c) shows the area near which the yttria film was absent. While area A of Fig. 3(c) is yttria film, area B shows a typical surface morphology of plasma-sprayed coating before grinding. Figs. 4(a) and (b) show cross-sections of sample 3 at low and high magnifications, respectively. Fig. 4(a) clearly shows aluminum, bond coating for plasma spraying of alumina, plasma-sprayed alumina coating and AD yttria film. It is readily recognized that plasma-sprayed alumina had quite high porosity while the microstructure of AD yttria film was uniform and dense. Also the interface between the alumina and yttria film was microscopically rough, increasing the contact area. The density of the yttria film was measured as 95.3 % TD. The CF_4 + Ar plasma etching test revealed that the etching rates of the plasma-sprayed alumina coating and sample 3 were 11.3 μm/h and 1.3 μm/h, respectively.

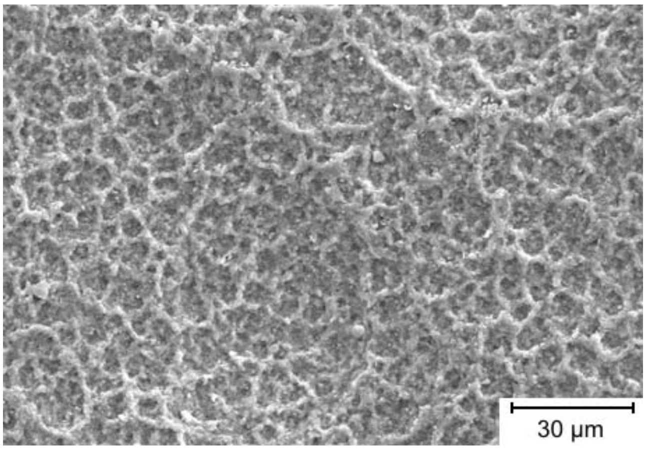

Fig. 3b: SEM micrographs of the samples after AD of yttria; sample 3.

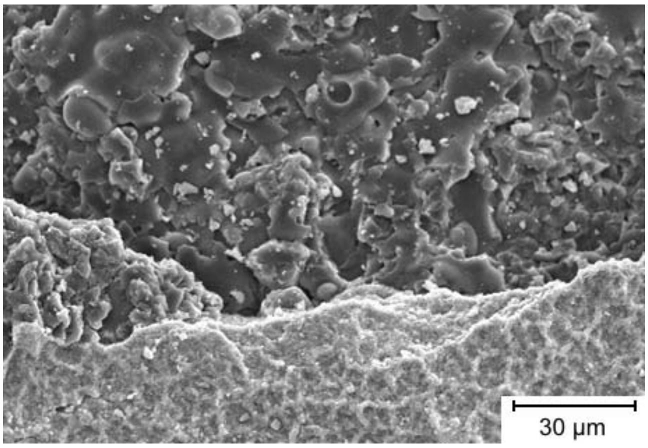

Fig. 3c: SEM micrographs of the samples after AD of yttria; sample 5.

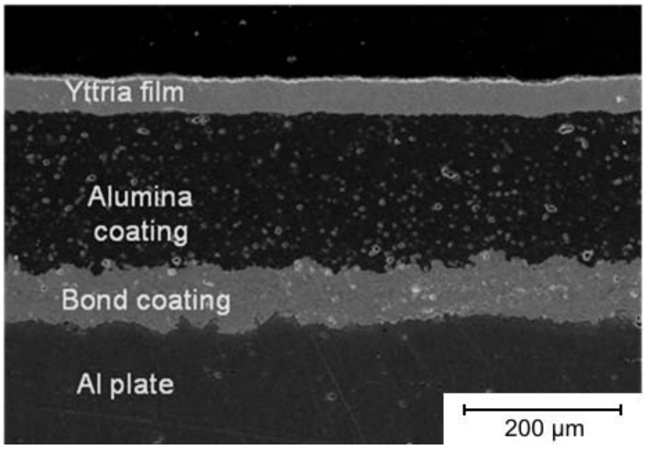

Fig. 4a: SEM micrograph of sample 3; low magnification showing aluminum plate, bond coating, plasma-sprayed alumina coating and AD yttria film.

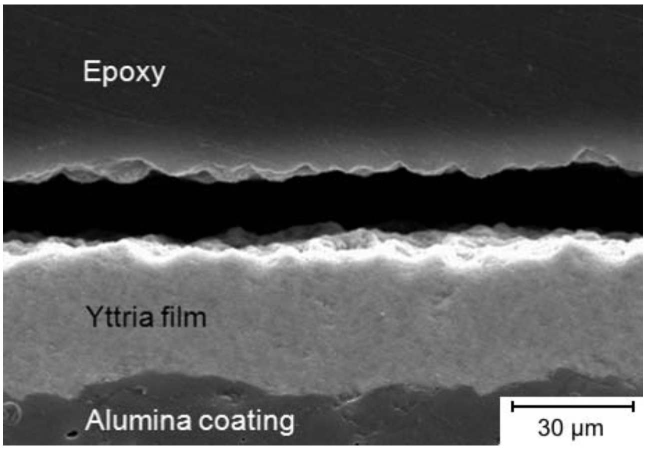

Fig. 4b: SEM micrograph of sample 3; high magnification showing the alumina coating and yttria film.

IV. Discussion

Plasma spraying has been considered as a method for manufacturing chamber parts for semiconductor processing equipment [1,2]. However, it often produced thick ceramic films with many pores. Since the pores provide weak points for electrical breakdown or plasma erosion, they need to be sealed. AD produced dense ceramic films but the thickness of the film was limited especially when the substrate was hard like Al_2O_3. An anchoring layer at the interface between the substrate and the AD film provided adhesion force that kept the film in place as indicated by Iwasawa *et al.* [6]. Unlike metal or quartz substrates, hard ceramic substrates like Al_2O_3 were difficult to penetrate in order to form a strong anchoring layer. Although the bonding mechanism between the substrate and the film and among the grains within the film is not clear, particles of the starting ceramic powder were broken into nanometer scale fragments to make the nano-structure of the film during AD as described by Akedo [5]. So, it is suspected that the nanometer-scale fragments adhered strongly to the hard ceramic substrate like alumina. Ceramic film deposited by AD was under residual stress as previously reported [8]. The residual stress increased as the film thickness was increased. So, the film was eventually peeled off when the thickness was larger than a critical value that might be determined by the residual stress and the adhesion strength at the interface. One strategy for increasing the adhesion strength at the interface was increasing the contact area between the substrate and the film.

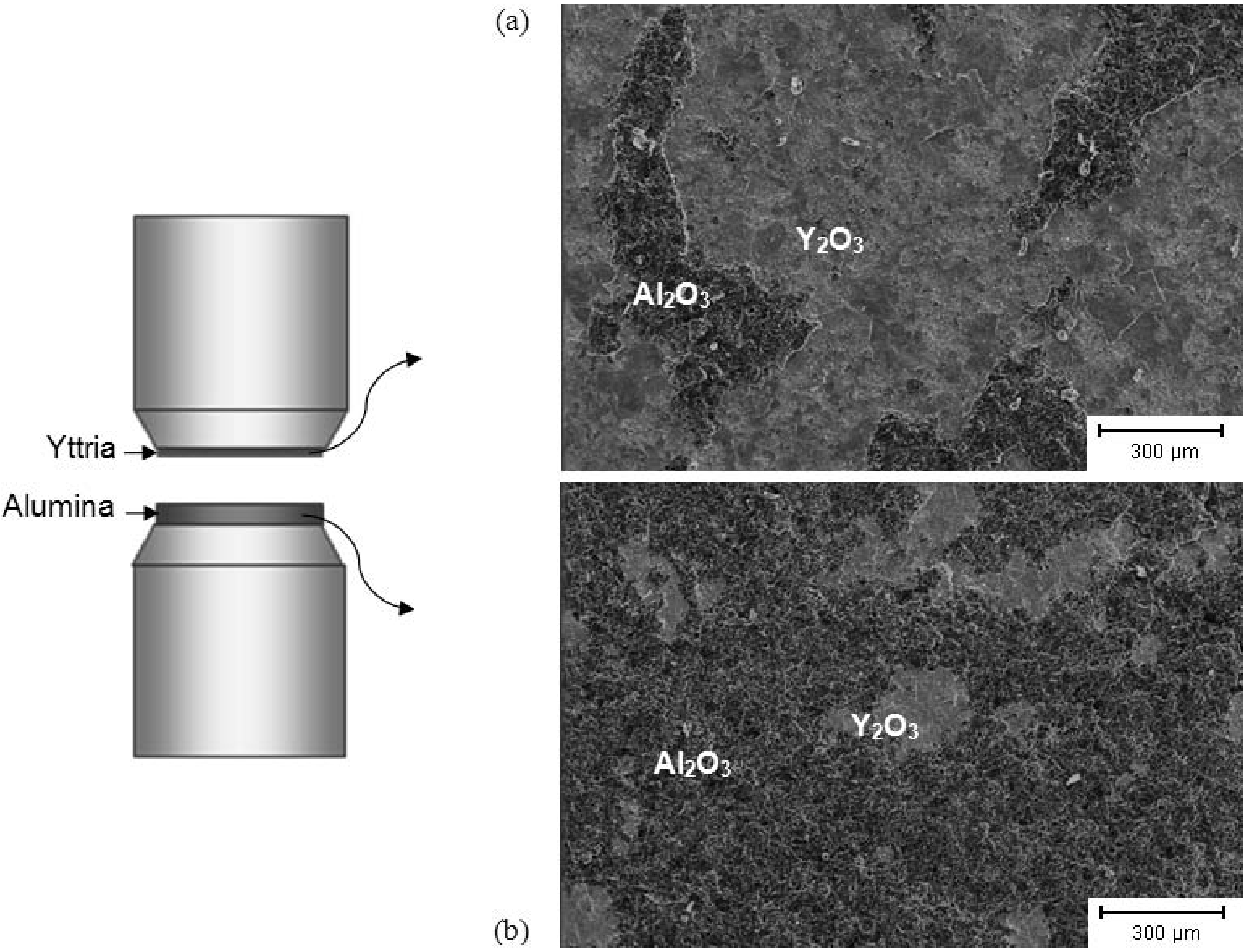

Fig. 5: SEM micrographs of sample 3 surfaces separated at the alumina-yttria interface after the tensile adhesion test; dark area and bright area representing alumina and yttria, respectively; (a) yttria side and (b) alumina side.

Rough surface of the alumina seemed advantageous over the smooth surface for deposition of dense yttria film by AD as revealed in Figs. 3(a) and (b). It is also confirmed from Fig. 4(b) that yttria film filled up the pores on the alumina surface, thus increasing the contact area between the alumina and yttria. Fig. 5(a) shows that a significant portion of yttria surface separated from the alumina by the tensile adhesion test was covered with alumina. On the other hand, only small localized areas of alumina was covered with yttria as shown in Fig. 5(b). It suggests that the adhesion strength at the alumina-yttria interface was comparable to the cohesion strength within the coating. High-resolution TEM observation of interface between the yttria and alumina revealed that the two ceramics were in direct contact without continuous amorphous phase as shown in Fig. 6. It was also observed that the plasma-sprayed alumina had many small regions of poor crystallinity as indicated by the circles. Grit blasting generated severe damage even in the sub-surface region of the alumina, which might weaken the alumina. The weak surface layer of the alumina coating was adhered to the yttria film during the tensile adhesion test.

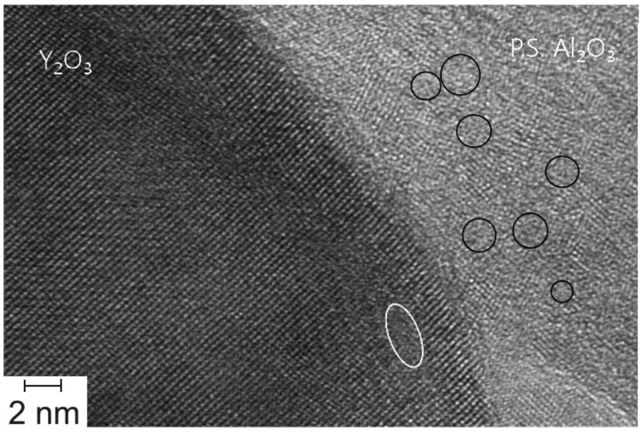

Fig. 6: High-resolution TEM micrograph of the interface between the AD yttria film and the plasma-sprayed alumina coating; circles indicate the severely damaged regions of poor crystallinity.

While blasting with the 200-grit abrasive generated only small surface craters as shown in Fig. 2(a), blasting with 40-grit abrasive removed a portion of the surface layer of the alumina coating, resulting in large and deep craters as shown in Fig. 2(b). Those craters were as large as hundreds of micrometers in both length and width as shown in Fig. 2(b) and several tens of micrometers in depth. The impingement angle of the particle with respect to the substrate surface had a strong influence on film deposition by AD as previously reported [7]. When the particle impinged perpendicular to the substrate surface, the deposition rate was the highest. On the other hand, hardly any film was deposited when the particle was sprayed parallel to the substrate surface. Therefore, there was a discontinuity of the yttria film at such an abrupt change in surface profile of alumina as shown in Fig. 3(c). Some of those large craters were not covered with the yttria film deposited by means of AD as shown for samples 4, 5 and 6. Closer examination of the large crater in Fig. 3(c) revealed that its bottom had a microscopically smooth surface where the yttria had not been deposited well. In other words, blasting with 40-grit abrasive not only increased the surface roughness of the Al_2O_3 but also exposed microscopically smooth surface area where yttria was not successfully deposited by AD. Therefore, for a successful deposition of dense yttria film by AD, it was desirable to have a controlled surface roughness and microscopically rough surface of the alumina coating as for samples 1, 2, and 3. In this study, the optimum average surface roughness (Ra) value was between 0.5 μm and 1.8 μm. Aerosol deposition of dense yttria film improved the CF_4 + Ar plasma erosion resistance of the porous plasma-sprayed alumina coating by about eight times.

V. Summary

Yttria film as dense as 95.3 % TD was deposited on a plasma-sprayed alumina coating by means of aerosol deposition. Blasting with 200-grit alumina abrasive produced the surface with Ra values between 0.5 μm and 1.8 μm on which the yttria film with 30-μm thickness was deposited. On the other hand, the surface of the alumina coating was heavily damaged by blasting with 40-grit alumina abrasive not only to obtain an Ra value between 2.4 μm and 4.3 μm but also to expose a microscopically smooth area after removal of a portion of surface layer. It was important for the plasma-sprayed alumina coating to have a microscopically rough surface for deposition of the yttria film by means of AD. The microscopically rough surface provided a large contact area at the interface and strong adhesion between the alumina and yttria film that allowed deposition of the thick and dense yttria film. The dense yttria film deposited by means of aerosol deposition remarkably improved the erosion resistance to CF_4 + Ar plasma of the porous plasma-sprayed Al_2O_3 coating.

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