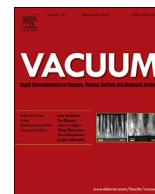




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Alumina and titania films deposition by APS/ASPPS dual mode thermal spray equipment using Ar added N₂ working gas

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ABSTRACT

In order to develop a low cost oxide film deposition process with short duration time, a 1 kW class Atmospheric thermal plasma spray (APS)/Atmospheric solution precursor plasma spray (ASPPS) dual mode thermal spray equipment was manufactured and film depositions of alumina (Al₂O₃) and titania (TiO₂) by APS and titania film deposition by ASPPS were carried out. Consequently, though intensive fluctuation with intensive abrasion of electrodes occurred during plasma jet generation in case of N₂ working gas, the plasma jet was stabilized and the abrasion was dramatically diminished by slight addition of Ar to N₂ working gas. Since the suction type feedstock feeder could be confirmed to be available not only for powder feedstock but also solution precursor feed stock. In the case of APS, lamellar structure alumina and titania films could be deposited. However, in the case of titania films deposited by APS, a phase transformation from anatase to rutile occurred partially during film deposition. Also in the case of ASPPS, titania films including rutile and anatase were deposited. From these results, the developed equipment was proved to be available as an APS/ASPPS dual mode thermal spray equipment and this technique was found to have high potential for a low-cost oxide film deposition process.

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1. Introduction

Oxide films have been used for various applications including corrosion-resistive, abrasion-resistive and thermal barrier coatings of bridges [1], semiconductor manufacturing equipment, and engines [2–4]. In addition they start to be utilized as functional film such as a photo-voltaic device, solid electrolyte, and gas sensor. Especially, because of its excellent chemical stability, alumina (Al₂O₃) film has been used in practice. Recently, because of its excellent photo-catalytic properties [5], titania (TiO₂) film is successfully applied as an antimicrobial coating, or, photo-voltaic device for dye-sensitized solar cells (DSSC) [6].

As the oxide film deposition process, chemical vapor deposition

(CVD) [7], physical vapor deposition (PVD) [8] and sol-gel method [9] have been widely and dominantly used. However, since CVD and PVD have some disadvantages such as low deposition time, high initial cost due to requirement of vacuum equipment, limitation of the sample size due to dimension of the vacuum chamber, a low cost film deposition process with short duration time is demanded. As for the sol-gel method, although film deposition can be conducted by use of simple equipment, this process also has some disadvantages such as low deposition rate, limitation of the sample size due to dimension of the water bath for hydrolysis, difficulty of thick film deposition due to factors such as the internal stress generated by volume variation of the film during crystallization. On the other hand, in atmospheric thermal plasma spray (APS), since the high rate (over several hundred microns/min.) film deposition can be conducted by simple equipment in open air without any chambers, low cost film deposition with short duration time can be carried out. Also, solution precursor plasma spray (SPPS) [10–14], which is one of the plasma spray processes, can create a dense film

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with control of film component and crystal structure as in conventional CVD because the film is created by chemical reaction among feedstocks, working gas and ambient gas. Although use of SPPS has so far been mainly conducted under low pressure environments in order to avoid oxidation of the film during operation, atmospheric SPPS (ASPPS) has started to be conducted for oxide film deposition by various laboratories only recently. Previously, authors used to deposit titania films using the developed ASPPS equipment with titanium tetra iso-butoxide (TTIB) as feedstock [15–18]. Subsequently, it was proved that the anatase film which had enough photo-catalytic properties to decolor methylene-blue droplets with 8 h UV irradiation and could generate electric power as the photo-voltaic device of the dye-sensitized solar cell, could be deposited.

Maranga, Ando et al. have been developing electric power generating systems using renewable energy devices such as small hydro, Dye-Sensitized Solar Cell (DSSC) and so on for non-electrified rural areas in the Project for Capacity Development for Promoting Rural Electrification as part of the Renewable Energy (BRIGHT project) [19]. For lifetime elongation of the small hydro and low cost (DSSC) Solar Cell manufacturing in this area, development of alumina film and titania film deposition processes using APS is thought to be effective. However, since solid materials such as powder and wires are used as feedstock in the case of thermal spray, the flame should have enough thermal energy to melt the feedstock during flight and high power (over 30 kW class) thermal spray generating equipment is considered necessary. Therefore, it is not suitable for the non-electrified rural area because of its high equipment cost and energy consumption. Therefore, we developed a 1 kW class APS equipment [20], which will be able to be driven by electric power from batteries charged by renewable energy devices. Consequently, it could be confirmed that stainless films could be deposited using Ar working gas and alumina films also could be deposited by addition of N₂ to the Ar working gas using 20 l/min. and 2 l/min. in Ar and N₂ working gas flow rates, respectively. Nevertheless, since Ar is a very expensive gas, a low running cost deposition condition such as using low price N₂ dominant working gas is required.

In this study, in order to develop a low cost and low power oxide film deposition process with short duration time for non-electrified rural area, a 1 kW class APS/ASPPS dual mode thermal spray equipment was manufactured. Using this dual mode alumina and titania film depositions by APS and titania film deposition by ASPPS were carried out.

2. Experimental procedure

Fig. 1 shows the schematic diagram of the thermal spray equipment used in this study. This equipment consists of plasma torch, DC power source, feed-stock supplying system and working gas supply system. Except the feedstock supplying system, the constitution of this equipment was the same as for the APS and the ASPPS equipment used in our previous studies and for conventional high power thermal plasma spray equipment. In the case of the conventional thermal spray equipment, the mechanically and electrically driven type powder feeder is generally used. Because the powder feeder is very expensive, a suction type powder feeder, which can feed the powder into the plasma jet by negative pressure generated by the thermal plasma jet, was developed in this study. Tables 1–3 show film deposition conditions for alumina film deposition by APS, titania film deposition by APS and titania film deposition by ASPPS, respectively. As the feedstock, alumina powder (PRAXAIR Al-1010-HP) and titania (anatase) powder were used in case of APS and TTIB (Ti(OC₄H₉)₄) was used for ASPPS. A 15 mm × 15 mm × 1 mm SUS304 stainless steel plate with grit

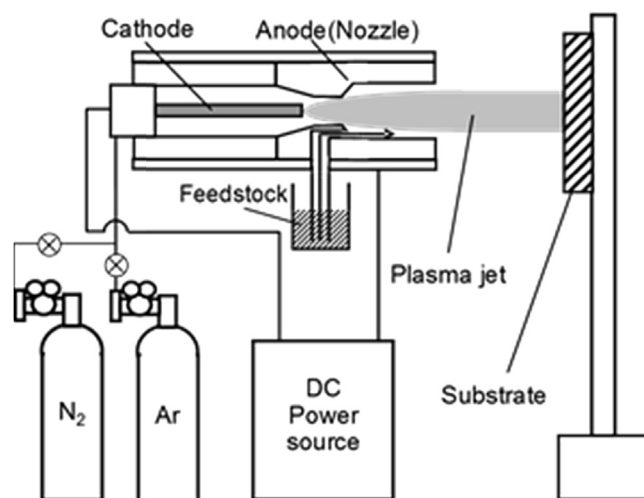


Fig. 1. Schematic diagram of the APS/ASPPS dual mode thermal plasma spray equipment.

Table 1
APS alumina film deposition conditions.

Substrate	SUS304 stainless steel
Working gas (Flow rate)	Ar (0.5 l/min.)/N ₂ (0.5–2.5 l/min.)
Spray distance	70 mm
	50 mm
Discharge Current	50 A, 20 V
Deposition time	30 s.
Feedstock material	Al ₂ O ₃ powder

*Conditions for the sample shown in Figs. 4 and 5.

Table 2
APS titania film deposition conditions.

Substrate	SUS304 stainless steel
Working gas (Flow rate)	Ar (0.5 l/min.)/N ₂ (0.5–2.5 l/min.)
Spray distance	100 mm
	50 mm
Discharge Current	50 A, 20 V
Deposition time	30 s.
Feedstock material	Anatase powder

*Conditions for the sample shown in Figs. 6 and 7.

Table 3
ASPPS alumina film deposition conditions*.

Substrate	SUS 304 stainless steel
Working gas (Flow rate)	Ar (0.5–1 l/min.)/N ₂ (0.5–2.5 l/min.)
Spray distance	50 mm
Discharge Current	50 A, 20 V
Deposition time	1 min.
Feedstock material	Ethanol diluted TTIB**

*Conditions for the sample shown in Figs. 9 and 10.

** Titanium tetra iso butoxide (Ti(C₄H₉)₄)(Volume ratio of TTIB/Ethanol = 1/1).

blasted surface was used as the substrate. Fig. 2 shows the X-ray diffraction patterns of the Al₂O₃ powder, TiO₂ powder and SUS304 stainless steel substrate used in this study. The substrate was horizontally set on the substrate holder and the central area of the sample was placed perpendicular to the axial center of the plasma jet. The input power for the discharge was fixed at 20 V, 50 A. After oxide film deposition, the microstructures of the films were investigated using an optical microscope and X-ray diffraction with CuK α at 40 kV and 100 mA. For confirming the hardness and adhesion strength of the deposited films, pencil scratch testing (ISO

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