

低電力大気圧マイクロ波プラズマ溶射による硬質皮膜の作製および 特性評価

Deposition of high hardness coating by low power atmospheric pressure microwave plasma spray and the characteristics evaluations

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# Deposition of high hardness coating by low power atmospheric pressure microwave plasma spray method and the characteristics evaluations

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#### Abstract

Atmospheric microwave discharge easily generates plasma at a microwave power of not more than 1kW. Low power atmospheric pressure plasma spraying expects promising way to deposit coatings with reduced microstructural change. In the research findings, we investigated that by 1.0kW of input power, it is possible to generate about 4000 K plasma for the deposition of metal and ceramics coatings by microwave plasma spray. Moreover, low power atmospheric pressure microwave plasma spray is also expected as a promising way to deposit coatings onto heat susceptible substrates such as high carbon steel and CFRP (carbon fiber reinforced plastic).

In this research, low power microwave plasma spraying system under the atmospheric pressure was studied. The substrate temperature during spraying, particle velocity and the thermal efficiency of plasma was measured as the operational characteristics of the system. Furthermore, high hardness coatings were deposited with metallic (Cr) and ceramics ( $Al_2O_3$ ) materials onto heat susceptible substrates. The characteristics of the coatings obtained by this research were evaluated.

The results obtained in this study are summarized as follows:

[ Evaluation of the operating characteristics of low power atmospheric pressure microwave plasma ]

- 1. Plasma length were shortened by the increase of working gas flow rate and the reduction of antenna outlet diameter, and temperatures were uniform at about 4000 K at 13 mm from the tip of the antenna.
- 2. Decrease in antenna outlet diameter and increase in working gas flow rate will increase the particle velocity, where the maximum Cr particles velocity is 135 m/s.

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- 3. Atmospheric pressure microwave plasma spraying device is able Cr coating on high carbon steel and CFRP substrate without the heat damage. The coating hardness was more than 1000Hv<sub>0.05</sub>.
- 4. Al<sub>2</sub>O<sub>3</sub> coating is able to be deposited by microwave plasma spray. However, the hardness of the coating was low at 58.1 Hv due to the insufficient melting of the spray particles.

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## **First Chapter**

## Introduction

#### 1-1 Fundamentals of plasma spray method

Plasma spray is one of the methods to fabricate thick coatings by utilizing the electrically formed thermal energy which produced from the combustion of gas plume, thermal arc etc. The coatings is fabricated by involving the following mechanisms, which is started by heating and accelerating the spray particle inside the plasma plume. then the melted particles are impinged onto the surface of the substrates, and finally flattened particles are then rapidly solidified to form the coatings. Plasma spray process which is generally been represented by direct current (DC) plasma where the plasma is generated by direct-current arc electric discharge while radio frequency (RF) plasma, where the plasma is generated by the discharge of radio-frequency are able to produce approximately 5000~20000K of extremely high temperature plasma. This feature of the thermal plasma made it possible to melt even the high melting point materials such as ceramics and cermet, in order to fabricate the coatings <sup>1)</sup>. However, it has been mentioned as a problem of the plasma spray where the superfluous heat input from plasma cause a remarkable change of material structure to the spray materials, which are spray particles and substrates, deliver to the target coatings is not obtainable. For an example, hydroxyapatite, which is a biomedical material could be decomposed to a harmful phase by the thermal spray deposition using high input power of 40kW<sup>2), 3)</sup>. Thus, the thermal spray method which is able to control the change of the material micro-structure of these thermal spray materials is called for. Therefore, the research of the low input power plasma spraying process which expects the control of the heat input to the spray material is advancing in recent years  $^{4), 5)}$ .

### 1-2 Low power atmospheric pressure microwave plasma spray method

Low input power plasma spray method <sup>6)</sup> is defined as a thermal spray method by using the thermal plasma generated with low input power in the heat source. The effects of lowering the input power of the thermal spraying equipment by the plasma production at low electric power as well as the effects of controlling the heat input to the spray material (control of the significant change of material structure) by low input power plasma are expected and the research is advancing in recent years. However, the input power of Cu coating deposited by conventional DC plasma spray method under atmospheric pressure condition which was reported is approximately 5kW <sup>7), 8)</sup>, while deposition of coating by using RF plasma spray under atmospheric pressure condition is reported to be difficult due to the difficulty in stabilizing the plasma. However, on the other hand, with the input power of several kilowatts or less, thermal plasma generation under atmospheric pressure is possible for microwave plasma spray method. For this reason, it is thought that the coating deposition in which the heat input to the thermal spray material can be controlled is possible by applying microwave plasma as a low input power plasma spray process. Moreover, this microwave plasma does not require electrode for electric discharge, which made it possible to generate plasma from chemically reactive type of gases if being compared to DC plasma in which the electrodes are needed for electric discharge. Furthermore, compared to DC plasma arc, microwave plasma has a lot of features such as plasma can be produced with relatively low input power, discharge power, plasma intensity, discharge frequency are wide, etc. <sup>9)</sup>. Since it has such a feature, in recent years, microwave plasma was applied in a wide range of fields, such as decomposition processing of harmful gas, heat treatment of waste, sterilization of medical material, and deposition of thin film <sup>10</sup>. However, since the efficiency of microwave plasma discharge is bad and the difficulty of producing high temperature plasma, there are few examples of application to plasma spray. However, in our laboratory, the problem of the microwave plasma in the abovementioned was solved, and the low input power atmospheric pressure microwave plasma spraying device which used microwave plasma for the heat source was invented <sup>11)</sup>. Detailed explanation of this thermal spraying equipment is specified in 2nd chapter. In our laboratory, research is advancing in order to make the low input power atmospheric pressure microwave plasma to be applied the plasma spray process.

# 1-3 Characteristics evaluation and the expected application field of low power atmospheric pressure microwave plasma spray

Under atmospheric pressure, the plasma production of approximately 1 kW of low input power is made possible by the atmospheric pressure microwave plasma spraying device at our laboratory. Compared with plasma called DC plasma and RF plasma which are used conventionally, the generated plasma is about 3 mm in diameter, and small in shape. As the characteristics evaluation of atmospheric pressure microwave plasma spraying device, the investigation of plasma production conditions, the measurement of the temperature of plasma plume and the coating deposition of metallic material which are copper and aluminium has been made. From the plasma temperature measurement result by optical emission spectroscopy measurement, the microwave plasma of approximately 1 kW of input power had the hollow structure where the luminescence of excited Ar atom did not exist in the central axis of the upper stream part, and the plasma changed to solid structure in the downstream part with maximum temperature of 5000K. From these findings, it was clear that the structure is different with the plasma currently used for the conventional plasma spray. Moreover, in order to investigate whether the heat input reduction to the spray substrates is possible, the metal (Cu) coating onto low melting point material called carbon fibre reinforced plastics (CFRP) and fibre-reinforced plastic (FRP) which are susceptible to heat, and it already became clear that the deposition of copper coating onto low melting point substrate is possible. In case of coating deposition of hydroxyapatite as a biomedical material , emergence of decomposition phase harmful to human body caused by the heat input from the plasma will occur for the conventional plasma spray process  $^{2), 3), 12}$ . However, it turned out that it is possible to control the emergence of the decomposition phase by coating deposition using this plasma spraying method  $^{13}$ .

Because of the deposition of coating with the controllable heat input into the spray material is possible, coating deposition of spray material that were difficult to be obtained by conventional plasma spray result in the widening of expected field of application for microwave plasma spray.

#### 1-3-1 Deposition of high hardness metal coating

Functional hard chrome plating is a critical process associated with manufacturing and maintenance operations on aircraft, vehicles and ships, both in civilian and military sectors. Hard chrome electroplating is commercially used to produce wear-resistant coatings, but the plating bath contains hexavalent chromium, which has adverse health and environmental effects. For this reason, the use of hexavalent chromium will be limited. As a results, the European Parliament and the Council on end-of-life vehicles expressly prohibits the use of lead, cadmium, hexavalent chromium and mercury in all vehicles put on the market after 1<sup>st</sup> July 2003 (passenger cars and commercial vehicles up to 3.5 tonnes)<sup>14</sup>. The total permissible amount of hexavalent chromium is limited to 2 g per vehicle. This directive must be implemented as national law in the Member States of the European Union<sup>15</sup>.

The types of coatings that are most widely viewed as being capable of replacing hard chrome plating are the thermal spray technologies <sup>16</sup>. For this reason, the research of depositing hard chrome coating by low input microwave plasma spray method has been brought upon.

#### 1-3-2 Deposition of high hardness ceramics coating

Plasma spray coatings of component surfaces are used to prevent the corrosion of certain substrate. Thermal spray coatings have been highly successful in industry due to their versatility. As far as anticorrosion and anti-wear applications are concerned, the most frequently used coating materials are oxide ceramic coatings <sup>17</sup>. Aluminium oxide, Al<sub>2</sub>O<sub>3</sub>, more often referred to as alumina, is an exceptionally important ceramic material which has many technological applications. It has several special properties like high hardness, chemical inertness, wear resistance and a high melting point. Alumina

ceramic can retain up to 90% of their strength even at 1100 degree Celcius. Because of excellent properties of alumina ceramics, they are widely used in many refectory materials, grinding media, cutting tools, high temperature bearings, a wide variety of mechanical parts, and critical components in chemical process environments, where materials are subject to aggressive chemical attack, increasingly higher temperature and pressures <sup>18</sup>. It is reported that the corrosion resistance of alumina coatings are higher than that of cermet and metallic coatings <sup>19</sup>. Therefore, in order to achieve the coating which possess better properties compare to metal, alumina coating deposition is also brought upon this research.

#### 1-4 Research objectives

The research objective is the deposition of high hardness coatings of two different types of materials which are metal and ceramics onto heat susceptible substrates.

As the candidate of metal material, Cr was selected as the spray powder in order to make comparison between Cr coating deposited by microwave plasma spray and hard chrome plating method. Furthermore, to study the possibility of the deposition onto heat susceptible substrates, high carbon steel and CFRP substrates was used as the candidates in which high carbon steel possess the higher phase transformation temperature.

Then, the investigation of hard ceramics coating by using microwave plasma spray was conducted. In this research, alumina material was used as the spray material.

#### 1-5 Structure of thesis

In the first chapter (Introduction), the fundamentals of plasma spray method, the progress of research by using low power microwave plasma spray device, the expected application field of the device, and the research objective were included.

In the second chapter (Characteristics evaluations of low power atmospheric pressure microwave plasma spray device), the study of plasma production conditions by using input power below 1kW, temperature distribution of the plasma torch, the measurement of substrates temperature, and the characteristics study of the microwave plasma spray below 1kW of input power were conducted.

In the third chapter (Deposition of hard chrome coating by low power atmospheric pressure microwave plasma spray and the characteristics evaluation), high hardness material, Cr coating was deposited onto SUS304, high carbon steel and CFRP substrates, and the evaluation of the coating obtained by the experimentation was conducted.

In the fourth chapter (Deposition of alumina coating by low power atmospheric pressure microwave plasma spray), the deposition of alumina coating was tried and the evaluation of the coating obtained by the experimentation was conducted.

In the fifth chapter (Research task and future plan), the problems realized after the research being done were summarized and problem-shooting method was discussed align with further studies needed for microwave plasma spray method.

In the sixth chapter (Conclusions), all of the results obtained from this research are summarized.

### Second Chapter

# Characteristics evaluations of low power atmospheric pressure microwave plasma spray device

In this research, plasma spray device that use low input power microwave plasma in which is able to be operated in atmospheric pressure was invented. The main objective of this chapter is to investigate the characteristics of the atmospheric pressure microwave plasma torch. The plasma ignition conditions and the thermal efficiency of this microwave plasma spray device were investigated. Furthermore, measurement of substrate temperature, plasma temperature by optical spectroscopic measurement and particle velocity measurement by using high speed camera were conducted.

#### 2-1 Low power atmospheric pressure microwave plasma spray device system

The schematic diagram of the low input power atmospheric pressure microwave plasma spraying system used for this research is shown in Fig. 2-1-1. Microwave is oscillated by microwave generation equipment (MICRO-DENSHI, MMG-213V-2P) on the frequency of 2.45 GHz, and then transmitted in the TE<sub>10</sub> mode through rectangular waveguide (with cross-sectional size: 109.2 mm x 54.6 mm). The maximum continuous output of microwave generator equipment is 1.2 kW. The matching of the impedance between microwave generation equipment and plasma torch and also the turning down of the reflect power  $(P_x)$  that came from the plasma torch is controlled by the use of an E-H tuner. In the rectangular waveguide, through a directional coupler, the incident wave and reflected wave of microwave are separated, and forward power  $(P_t)$  and reflect power are measured with a wattmeter. By ignoring the power attenuation in the transmission line, electric power consumed by the plasma source is calculated as  $P_f - P_r$ . When this reflective power becomes the minimum, the dielectric breakdown of the gas can be carried out at the optimum electric power efficiency. Thus, the efficiency of the microwave electric power used by plasma torch which defined as coupling efficiency  $(\eta_p)$  is described as the following formula.

$$\eta_p = \frac{P_f - P_r}{P_f} \quad (2-1)$$

Generation of plasma is attained by carrying out the dielectric breakdown of the plasma working gas using an E-H tuner, after supplying Ar which is the plasma working gas to the plasma torch. The plasma working gas was directly supplied from the gas cylinder, and the gas mass flow was adjusted using the mass flow controller (KOFLOC, 3755).



Fig. 2-1-1 Schematic diagram of low power atmospheric pressure microwave plasma spray system.

# 2-2 Structure of plasma torch of atmospheric pressure microwave plasma spray

The schematic view of an atmospheric pressure microwave plasma torch is shown in Fig. 2-2-1. Microwave oscillated from microwave generation equipment is transmitted in a cylindrical resonant cavity (  $\phi$  120 x L 113), and an antenna which is a metal pipe is arranged on the central axis of the resonant cavity in which the microwave electric field concentrates on the apical portion. Moreover, the plasma working gas blows off from the antenna apical portion, the dielectric breakdown of the plasma working gas is carried out by the microwave electric field concentrated at the antenna apical portion, and the plasma is generated. The photograph of the antenna used for this equipment is shown in Fig. 2-2-2. The side linked to the rectangular waveguide of the antenna was made of Cu-Zn, while the side of antenna apical portion was made of Cu-W which is excellent in heat resistance. For the outside diameter and inside diameter of Cu-Zn is fixed to 6 mm and 4 mm, while for the Cu-W antenna apical portion, outside diameter is fixed to 4 mm, but the inside diameter of 2.5 mm, and 1.5 mm is prepared for the experimentation. As for the reason of changing the inside diameter of the antenna is because it can be thought that by the change of antenna diameter, at the same working gas flow rate, the rise of the gas flow can be expected, which resulting in the effect of the increase of particle velocity. The schematic diagram of the structure of the antenna apical portion is shown in Fig. 2-2-3.



Fig. 2-2-1 Schematic view of atmospheric pressure microwave plasma torch.



Fig.2-2-2 Photograph of the antenna.



Fig. 2-2-3 Schematic diagram of Cu-W pipe parts of the antenna.

#### 2-3 Investigation of plasma ignition conditions of plasma torch

#### 2-3-1 Experimental conditions

This experiment investigated the plasma ignition conditions of the low power atmospheric pressure microwave plasma torch. The optimal condition of the plasma ignition in which this experiment defines is the generation (ignition) of plasma is easy (plasma can be lit and stabilized in a short time), and it is considered as the conditions which are stable. The first objective of the definition given beforehand is because when the plasma ignition is not occurred for a long time with the  $P_f - P_r$  states remain high, plasma torch might be damaged by the heat cause by the microwave. Moreover, and for the latter objective is because of the aim of finally using this equipment as a thermal spraying equipment, it becomes more unstable by supplying a spray particle into plasma when the generated plasma is unstable, and it is expected that the maintenance of the plasma becomes difficult.

As the plasma production method, after generating the plasma at 1 kW, the possibility of maintaining the plasma was investigated by performing operation which lowers input power to meet the experimental conditions. The ignition conditions of plasma are shown in Table 2-3-1. Two conditions of plasma working gas flow rate, two size of inside diameter of an antenna and three conditions of input power were prepared to investigate the production and the maintenance of plasma. The trial of plasma production of each condition was done 5 times and the propriety of the maintenance of plasma production was judged. The reason of the experiment time was made into a maximum of 3 minutes is because the damage to the antenna by microwave when plasma cannot be generated or maintained in the state where the reflect power was made small as much as possible, was taken into consideration. Argon gas was used as the plasma working gas due to the reason that it is a monoatomic gas, ionization potential is low and the generation of plasma is comparatively easy. The stability of plasma was evaluated from the existence of plasma and the observation of the appearance of the generated plasma. Furthermore, the investigation of the coupling efficiency on each condition was also conducted simultaneously.

Forward power (kW) .	0.1, 0.3, 0.5, 1.0		
Working gas flow rate (l/min)	11, 15, 19		
Antenna outlet diameter (mm)	1.5, 2.5		
Operating time (s)	300		

Table 2-3-1 Experimental conditions for plasma ignition

## 2-3-2 Experimental results and discussions

Result of the investigation of plasma production conditions is shown in Fig. 2-3-1. Plasma ignition that was able to be produced 5 out of 5 times and maintained for 5 minutes are shown as  $\bullet$  mark.  $\bigcirc$  mark shows that  $3\sim 4$  times out of 5 times of the plasma production, plasma was able to be maintained for 5 minutes,  $\triangle$  mark shows that  $1 \sim 2$  times out of 5 times of the plasma production, plasma was able to be maintained for 5 minutes, × mark shows that 5 times out of 5 times of the plasma production, plasma was not able to be maintained. From Fig. 2-3-1, although it was confirmed that the plasma is able to be stabilized and maintained on the conditions of input power 0.5kW, and antenna outlet diameter 2.5 mm, the maintenance of plasma is difficult at antenna inside diameter 1.5 mm. From the results, it can be clarified that the maintenance and stabilization of plasma is difficult with reduction of the antenna outlet diameter. On the input power 0.3 and 0.5 kW of the plasma production conditions, it became clear that for the generation and maintenance of plasma to be possible. However, on 0.1 kW of input power, plasma production and maintenance cannot be performed and the maintenance of plasma becomes difficult on the input power of 0.3 kW, antenna outlet diameter 1.5 mm, and the ignition conditions of working gas flow rate 19 l/min. Furthermore, coupling efficiency is above 99% at the time of the plasma has been stabilized and it was made clear that the microwave generated plasma possesses high coupling efficiency.



Fig. 2-3-1 Result of plasma ignition experiment.

#### 2-4 Measurement of thermal efficiency of microwave plasma spray torch

## 2-4-1 Experimental method

In this experiment, the objective is to evaluate the characteristics of the thermal efficiency of low power atmospheric pressure microwave plasma spray torch. Thermal efficiency of the plasma torch is defined as an index which shows the rate of the energy (the amount of heat transfer) used for heating of material to the energy used for the plasma ignition, and it shall be effective to the evaluation of the thermo-physical properties of plasma <sup>20</sup>. In this experiment, thermal efficiency ( $\eta_T$ ) is defined as the formula shown below, and the schematic diagram of thermal efficiency measurement system is shown in Fig. 2-4-1.

$$\eta_T = \frac{Q_2}{Q_1} = \frac{(T_2 - T_1)C_w S}{P_f - P_r} \times 100$$
(2-2)

$Q_1$	:	Energy used for the plasma ignition [W]
$Q_2$	:	Recovered energy by heat exchanger [W]
$T_1$	:	Water temperature of the entrance of heat exchanger [K]
$T_2$	:	Water temperature of the exit of heat exchanger[K]
$C_w$	:	Specific heat of water [J/kgK]
S	:	Flow rate of water [m <sup>3</sup> /s]
$P_f$	:	Forward power [W]
$P_r$	: 1	Reflect power [W]
C	1	

Cu plate was heated by the plasma, and the temperature change of the water flowed inside the Cu pipe that was set at the lower side of the Cu plate was used to calculate the heat efficiency of the plasma. The temperature of the water flowed at the entrance and the exit of the Cu pipe was measured by K-type thermocouple. The ignition conditions, as was shown in the Fig. 2-4-1, and the working gas flow rate was made as the changing parameter and the heat efficiency was measured. Furthermore, measurement position was set to 30 mm from the tip of the antenna in which the plasma was produced, and the Cu plate is fully heated by the contact to the plasma.



Fig. 2-4-1 Schematic diagram of the thermal efficiency measurement system.

Forward power [kW]	0.5	
Working gas	Ar	
Measurement distance (mm)	30	
Working gas flow rate [1/min]	11, 15, 19	
Operating time [s]	300	

Table 2-4-1 Experimental conditions for thermal efficiency measurement

#### 2-4-2 Experimental results and discussions

The photograph of the thermal efficiency measurement experiment of atmospheric pressure microwave plasma torch is shown in Fig. 2-4-2, and the thermal efficiency measurement result is shown in Figure 2-4-3. From the result in Fig. 2-4-3, the highest thermal efficiency is at antenna inside diameter 2.5mm and working gas flow rate 11 l/min, where the average of the heat efficiency is 28.1 %. From this result, it is confirmed that the heat efficiency of this microwave plasma spray is at comparatively at the same level of the heat efficiency of atmospheric pressure DC plasma torch (plasma gun) which is widely and generally used where the heat efficiency are about  $10 \sim 40\%^{20/21}$ . Furthermore, from the results, the heat efficiency of the plasma torch is decreasing with the increase of working gas flow rate. This can be considered that there is the influence of plasma temperature, in which plasma temperature rises with the increase of the energy given per working gas unit volume under atmospheric pressure. From this, it is thought that plasma temperature fell by the increase in a working gas flow rate, and thermal efficiency fell because of the decrease of the amount of heat input into the Cu plate.

It became clear from the above result that this equipment has thermal efficiency comparable as generally used DC plasma. For this reason, the application as a heat source for plasma spray coatings is expected from this device.



Fig.2-4-2 Photograph of thermal efficiency measurement system.



Fig. 2-4-3 Results of thermal efficiency measurement of microwave plasma spraying device.

\*21), \*22) The thermal efficiency value was obtained by the experimental conditions shown below.

\*21)

Device	:	Atmospheric pressure DC plasma torch
Thermal conductivity	:	$10 \sim 25 \%$
Plasma working gas		
Primary	:	Ar (66.3 $dm^3/min$ )
Secondary	:	He $(35.7 \text{ dm}^3/\text{min})$
Arc current	:	Current 275A
Arc voltage	:	Voltage 175V
*22)		
Device	:	Atmospheric pressure DC plasma gun
Thermal conductivity	:	40 %

Plasma working gas : Ar Arc current : Current 1000A

Arc voltage : Voltage 40V

#### 2-5 Measurement of substrates temperature

#### 2-5-1 Experimental method

In this experiment, the effect of the heat input from the plasma plume to the substrates was investigated by measuring the temperature of substrates during plasma spraying was performed. For the measurement of substrates temperature, the temperature is measured with the sheath type thermocouple (K-type) by collecting the signals from the thermocouple by the data logger GR-3000 (KEYENCE CORP). The substrate material was set to SUS304 with the dimension size of 20x20x3 mm. The thermocouple with \$1.5mm in diameter was inserted inside the slit opened in the substrates with the distance of 1mm from the surface of the substrates and 2mm width. By this positioning of the thermocouple, inside temperature of the substrates are able to be measured. Experimental conditions of substrates temperature measurement are shown in Table 2-5-1, while Fig. 2-5-1 shows the measurement method. In this experiment, substrate temperature by the change of spraying distance, working gas flow rate, and input power were measured. Spraying distance is the distance from the antenna tip to the substrate surface. Generally, for the conventional plasma spraying method which consumed high input power, to protect the substrates from excessive heat, cooling device is normally installed at the spray stage. However, by using low input power plasma spraying method, spray substrates are able to be protected against the excessive heat without the cooling device and this will also widened the type of spray substrates materials to be applied with. Measurement time is set to the time the measurement started until the temperature is stable which is approximately 120 second. The reason of spraying distance is set to be at least as near at 30 mm is because at the spraying distance below 30 mm, it was checked that the spray particles which collided with the substrates and were not impinged onto the substrates will soars in the direction of the antenna with the reflected working gas. This results in the difficulty of making the plasma to be stabilized. Therefore, in this research, the distance of 30 mm or more which has little influence by the un-impinged spray particles on the plasma was used as the experimental condition. Moreover, since substrates temperature aimed at the coating deposition in which inhibited the heat influence on a low melting point substrates which discussed in the third chapter, the maximum temperature was measured, and the average value was calculated from 3 times of the measurements.

Table 2-5-1 Experimental conditions for substrate temperature measurement

Forward power (kW)	0.5		
Working gas flow rate (l/min)	11, 15, 19		
Spray distance (mm)	30, 35, 40		
Traverse speed (mm/s)	5		
Antenna outlet diameter (mm)	1.5, 2.5		



Fig. 2-5-1 Schematic drawing of substrate temperature measurement.

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#### 2-5-2 Experimental results and discussions

The results of substrate temperature at experimental conditions of Fig. 2-5-1 at antenna outlet diameter 2.5 mm are shown in Fig. 2-5-2. Fig. 2-5-3 shows the photograph of the plasma at the time of measurement. Furthermore, the plasma length results which were derived from the photograph of the plasma are shown in Table 2-5-2. From the results, the substrates temperature is decreased with the increase of working gas flow rate and the increase of spray distance. As a result, at working gas flow rate 11 l/min and spray distance 30 mm, the substrates temperature was at the highest at the temperature of 678 K while the lowest substrate temperature was at spray conditions of working gas flow rate 19 l/min and spray distance 40 mm at 389 K. In the reduction of the plasma length by the increase in working gas flow rate, it is thought that the energy given per unit working gas volume is decreased with working gas flow rate while the energy from microwave was constant, and plasma length decreased by the pinch effect <sup>23)</sup>. With the plasma generated using the antenna with an antenna outlet diameter of 2.5 mm, the experimental result shows that at any conditions of working gas flow rate, the highest substrates temperature is shown at 30 mm of spray distance.



Fig. 2-5-2 Effect of working gas flow rate and spray distance on substrate temperature at antenna outlet diameter 2.5 mm.

	1.5 mm	2.5 mm
11 l/min	Plasma length	
15 l/min		
19 l/min		

Fig. 2-5-3 Photographs of the plasma generated by microwave plasma torch for each experimental conditions

		Working gas flow rate (l/min)		
		11	15	19
Antenna	1.5	33.1	30.9	27.4
(mm)	2.5	34.3	32.6	28.3

Table 2-5-3 Plasma length (mm) of each experimental condition

The result of the measurement of substrates temperature obtained by the plasma generated at antenna inside diameter 1.5 mm is shown in Fig. 2-5-4. From the result, by reducing the antenna inside diameter, it is confirmed that the substrates temperature are able to be decreased comparing to antenna inside diameter 2.5 mm. Moreover, even at antenna outlet diameter 1.5 mm, the decrease of plasma length with working gas flow rate is observed. It can be thought that the reason of this phenomenon is because of the decreasing of the plasma length as the result of the reduction of antenna size from the observation of the photograph of plasma in Fig. 2-5-3. In this research, in third chapter, the objective of the study is to deposit coating by controlling the heat input onto the substrates by using low input atmospheric pressure microwave plasma spraying method. From the results shown in Fig. 2-5-4, the glass transition temperature of CFRP is 523 K, means that by choosing the spray conditions, the deposition of coating without heat effect onto CFRP substrates is also able to be conducted.



Fig. 2-5-4 Effect of working gas flow rate and spray distance on substrate temperature at antenna outlet diameter 1.5 mm