

Boron Carbide Coating by Electromagnetically Accelerated Plasma Spraying

J. Kitamura, S. Usuba, Y. Kakudate, H. Yokoi, K. Yamamoto, A. Tanaka, and S. Fujiwara

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A new system of electromagnetically accelerated plasma spraying (EMAPS) consisting of a pulsed high-current arc-plasma gun and a large flow rate pulsed powder injector has been developed to synthesize a hard and dense coating of boron carbide (B_4C) with a high adhesion. The plasma gun with a co-axial cylindrical electrode configuration generates electromagnetically accelerated arc plasma with a typical velocity and maximum pressure of 1.5-3.0 km/s and 1 MPa, respectively, by discharging a pulsed high current of about 100 kA in peak and about 300 μ sec of duration. The heating and accelerating of source powder are accomplished by injecting it into the inter-electrode space of the gun prior to the plasma generation using a newly developed pulsed powder injector that enables a gram of powder to be injected within 1 ms with precisely controlled time delay. With this system, hard B_4C coatings with a high adhesion and crystallinity were successfully formed on mirror-polished stainless (SUS304) substrates without a binder.

Keywords boron carbide, electromagnetically accelerated plasma, plasma spraying, pulsed powder injection

1. Introduction

Boron carbide (B_4C) is an attractive material as a super-hard coating under high temperature and high heat flux conditions.^[1,2] Sintered bulk B_4C is one of the hardest materials, only surpassed by diamond and cubic boron nitride.^[3] It also has a low density and a good resistance to chemical agents. For example, it can be applied as a coating material on cutting tools and as protection for a tokamak wall in a nuclear fusion reactor.^[1] Several methods have been investigated to synthesize the boron carbide coating; they include radio-frequency plasma chemical vapor deposition (CVD),^[4] microwave-plasma CVD,^[5] laser-assisted CVD,^[6,7] plasma-jet CVD,^[8] sputtering,^[9,10] gas conversion techniques,^[11] pulsed laser deposition,^[12] plasma spraying,^[13-16] and ion beam evaporation techniques.^[17]

In the case of using the B_4C coating under high temperature and/or heat flux conditions, it seems reasonable to use thick coatings (~100 μ m or thicker) to protect the base substrate materials from the ambient gas.^[1] To make such a thick coating, plasma spraying is considered to be suitable due to its high growth rate and high particle-heating rate. However, the density and the adhesion of coatings formed using plasma spraying are generally low due to insufficient velocity of the powder jet. A substrate coated so sparsely would be damaged by the ambient gases passing through the pores, and the coating would be easily

detached. High velocity oxygen-fuel (HVOF) flame spraying or a detonation gun, on the other hand, are also considered to be difficult to adapt because the particle heating rate is insufficient for coating formation of high melting materials with high quality in terms of uniformity, adhesion, and high density, although either method is capable of generating a high-speed powder jet.^[18] Therefore, the development of a spraying method that can generate a high-speed powder jet while retaining a high temperature is important for the formation of a boron carbide coating that is dense and thick.

Dense and highly adhesive coatings of tungsten carbide, tantalum carbide, and zirconium boride are produced by electrothermally exploded powder spraying (ELTEPS) developed by Tamura et al.^[19] The velocity of the fastest particle jet accelerated by the electrothermal explosion is estimated to be 900 m/s. This method is only applicable to conductive materials because the source materials are heated and accelerated by pulsed high current. The pulsed plasma spraying method using a railgun electrode^[20-23] or a coaxial cylindrical electrode^[23] is similar to the ELTEPS method in terms of using pulsed high current to heat source materials. In this method, source materials are accelerated by electromagnetic force and heated by both joule heating in itself and heat flux from the surrounding plasma. The source materials are accelerated about 2 km/s. With this method, dense and highly adhesive coatings of tungsten carbide, tungsten, and TiAl are produced.^[20] Tahara et al. reported coatings of insulators such as Al_2O_3 and ZrO_2 using a magneto-plasma-dynamic (MPD) arc jet generator.^[24] With this method, insulating source materials placed near a cathode are ablated by a concentrated plasma jet and sprayed onto the substrates.

An electromagnetically accelerated plasma spraying (EMAPS) system has been developed. The purpose of the project is to intensely accelerate and heat powders with high current arc plasma without limitation to the electrical conductivity of the source powders. This system consists of a pulsed high-current arc-plasma gun, which is a coaxially cylindrical electrode, and a

J. Kitamura, Joint Research Consortium of FCT, Japan Fine Ceramics Center, c/o AIST 1-1-1 Higashi, Tsukuba, Ibaraki 305-8565, Japan; S. Usuba, Y. Kakudate, H. Yokoi, K. Yamamoto, A. Tanaka, and S. Fujiwara, National Institute of Advanced Industrial Science and Technology (AIST) 1-1-1 Higashi, Tsukuba, Ibaraki 305-8565, Japan. Contact e-mail: j.kitamura@aist.go.jp.

large flow rate pulsed powder injector. This article presents the basic characteristics of this system and preliminary results with insulating boron carbide (B_4C) coatings on stainless steel (SUS304) substrates without a binder.

2. Experimental

The concept of the typical spraying process of the present system is illustrated in Fig. 1: (a) source powders are charged

in the powder injector, and the condenser of the discharge circuit is charged; (b) the powder is injected with pressurized gas into the inter-electrode space of the gun, which is initially evacuated; (c) while the injected powder fills the inter-electrode space, the high current discharge is initiated at the position where inner and outer electrodes are initially shorted with a fine metallic wire (the plasma initiation point), and the magnetic field is established; (d) the powders are accelerated and heated by the electromagnetically accelerated plasma; and (e)

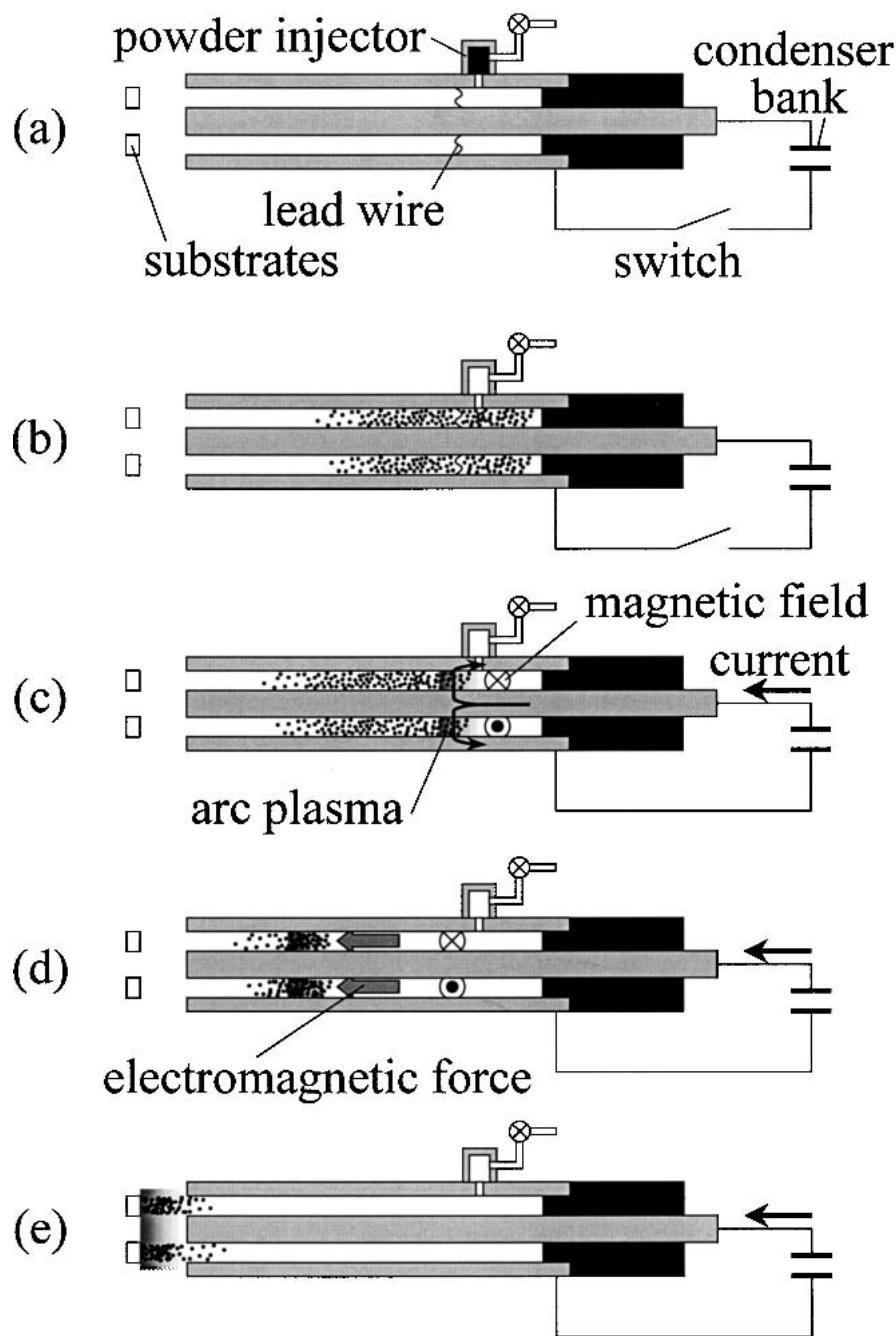


Fig. 1 The concept for the spraying process in the EMAPS system. (a) Source powders are charged in the powder injector, and the condenser of the charge circuit is charged. (b) The powders with the pressurized gas are injected into the inter-electrode space of the gun. (c) The high-current discharge is initiated at a fine lead wire to establish arc plasma and magnetic force simultaneously. (d) The powders are accelerated and heated by the electromagnetically accelerated plasma. (e) The deposition is formed on the substrate.

the deposition is formed on the substrate placed near the gun muzzle.

The advantage of this method is that the powders can be intensely accelerated and heated by high current arc plasma without any limitation to the electrical conductivity of the powders. With this method, the powder injector is considered to be one of the key elements because the response and the reproducibility of the injection must be high enough to control the delay time precisely from the powder injection to the initiation of the high-current discharge, which is considered to be one of the important factors that permits the characteristics of the coating to be controlled. It is, however, impossible to use a conventional electromagnetic valve that is widely used as a fuel injector due to its small flow rate and inadequacy for reproducing the desired response. For this reason, a simple method for powder injection was developed as shown in Fig. 2, in which (a) the powder is initially charged in a pressurized gas reservoir, which has a nozzle that is closed by a diaphragm made of thin resinous film; (b) at a given time, the fine metallic lead wire located near the diaphragm is exploded by pulsed current; and (c) the wire explosion causes instantaneous rupture of the diaphragm and subsequent injection of the powder with pressurized gas into the inter-electrode space. This method is expected to achieve pulsed powder injection with a large flow rate and high reproducibility.

Copper was used to construct the gun electrodes and the fine lead wires for the initiation of high current arc plasma. The diameter of the center anode and the inside diameters of the outer cylindrical cathode are 15 mm and 40 mm, respectively. The length from the plasma initiation point to the gun muzzle corresponding to the plasma travel length is 36 cm. The powder injection point is located 4 cm behind the plasma initiation point. These dimensions are not fixed and should be optimized if necessary. Three optical sensors are set to measure the velocity of the accelerated plasma by detecting the plasma emission. The distances between the plasma initiation point and the optical sensors are 10 cm, 20 cm, and 36 cm (gun muzzle). The condenser bank of 3 mF, whose maximum charged voltage and stored electric energy are 10 kV and 150 kJ, respectively, is used for the high-current source. The minimum discharge interval corresponding to the time between shots of 10 min comes from the specification of the ignitron switch used for the high-current start switch and clamp (crowbar) switch (Richardson Electronics, Ltd., IL, USA). The handmade gas reservoir of the powder injector, made of brass and polycarbonate, has a capacity of 17 cm³ and a nozzle of 8 mm in diameter. Polyvinyl chloride film is used for the diaphragm. The copper wire explosion and argon gas are used for the rupture of the diaphragm and the powder injection, respectively. The actual shot cycle of the present system is about 15-20 shots/day, which is limited by the manual installations of the powder and diaphragm for the powder injection and the lead wire for the plasma initiation. While the cycle is low, the methods of the installations are suitable for basic experiments on a laboratory scale due to low cost and simplicity.

Typical experimental conditions of B₄C coating formation are summarized in Table 1. The condenser bank was charged up to 5.9 kV to store electric energy of 52.2 kJ. The diameter of the copper wire for the initiation of the arc plasma was 100 μm. B₄C powder (particle size: 60 μm, purity: 98%, Nilaco Corp., Japan) was used for source material. As substrates, stainless steel

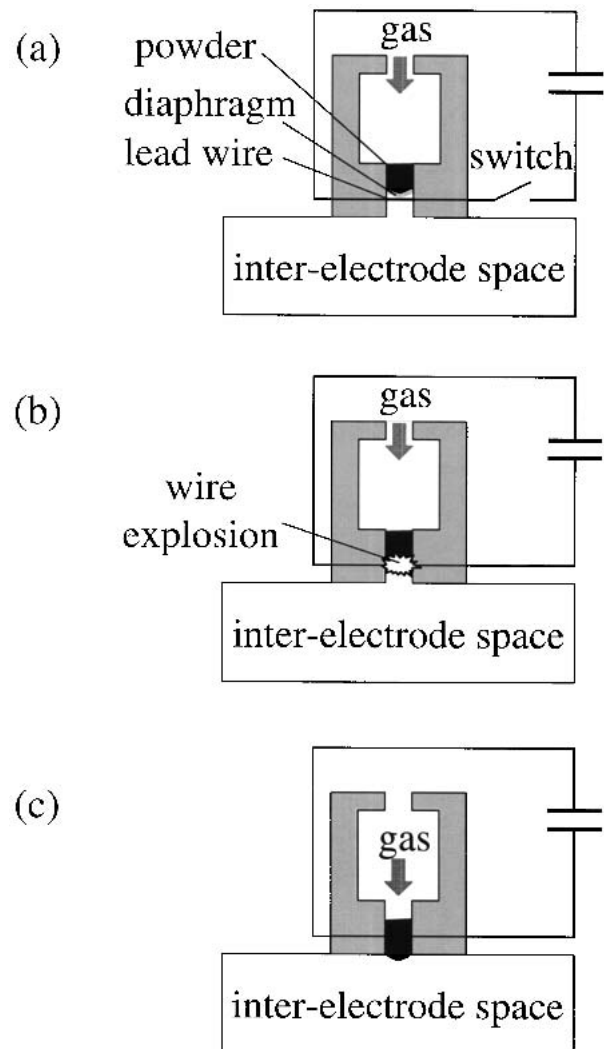


Fig. 2 Illustrations of the large flow rate pulsed powder injection process. (a) The powder is charged in a pressurized gas reservoir, which has a nozzle that is closed with a diaphragm made of thin resinous film. (b) At a given time, the fine metallic lead wire located near the diaphragm is exploded by pulsed current. (c) The wire explosion causes an instantaneous rupture of the diaphragm and a subsequent injection of the powder with pressurized gas into the inter-electrode space.

Table 1 Typical Experimental Conditions

Parameters	Optimal Conditions
Capacitance of the condenser bank	3 mF
Applied voltage of the bank	5.9 kV
Stored energy	52.2 kJ
Minimum interval between discharges	10 min
Diameter of the lead copper wire	100 μm
Source powder	Polycrystalline B ₄ C (60 μm, 98%)
Substrate	SUS304 (10 × 10 mm ²)
Substrate temperature	Room temperature
Ambient pressure	1 Pa

(SUS304) plates with a base of 10 mm × 10 mm and a thickness of 3 mm that were mirror-polished with a diamond slurry of 1 μm were used. To increase the growth rate by hitting the most of the

Table 2 The Optimal Conditions of the Powder Injector

Parameters	Optimal Conditions
Diameter of the lead copper wire	100 μm
Diaphragm	Polyvinyl chloride (Asahi Kasei Corp., 40 μm)
Argon gas pressure	0.8 MPa

powders onto the substrates, six plates are placed equally 10 mm away from the gun muzzle on the projected plane of the cross section of the inter-electrode space of the gun because the spray pattern is considered to be annular.

The spraying process was performed without any pretreatment of substrates, such as heating or sand blasting. The ambient air pressure, which was typically less than 1 Pa in the chamber, including the inter-electrode space, was also a factor to be optimized for proper operation of the pulsed powder injector and subsequent B_4C coating formation. For the characterizations of the coatings, scanning electron microscopy (SEM), optical microscopy, Vickers hardness testing (Hv), X-ray diffraction (XRD), and electron probe microanalysis (EPMA) are used.

3. Results and Discussion

The parameters of the optimal condition of the pulsed powder injector are summarized in Table 2. The typical waveforms of the pressure change in the injector and the discharge current in the condition are shown in Fig. 3, where the amount of the injected powder is 1.5 g. The beginning of the pressure change is clearly seen in Fig. 3(a). The time between the trigger of the wire explosion and the beginning time of the pressure change was defined as the acting time. The average of acting times measured in seven tests under the same conditions is $320 \pm 10 \mu\text{sec}$. To obtain such a highly reproducible response, the peak current for the wire explosion should be at least 1 kA, as shown in Fig. 3(b). The increase of the pressure change, in other words, the increase of the argon gas jet velocity, is observed at about 1800 μsec (white arrow in Fig. 3a). No increase of such a pressure change is observed when no powders are charged to the injector. This suggests that most powders that obstruct the gas jet have been injected into the inter-electrode space from the injector at this time. Therefore, the flow rate of the powder is estimated to be 1 g/ms. It is assumed that this large flow-rate powder injector instantaneously supplies enough source material for the coating formation with high reproducibility. The diaphragm is primarily fragmenting at the rupture. The debris mainly remains in the inter-electrode space near the injector.

A typical waveform of high-current discharge to generate the electromagnetically accelerated plasma and its light emissions detected by the optical sensors during the spraying process are shown in Fig. 4, where the delay of plasma initiation from the powder injection is 3.75 ms. The maximum current is approximately 100 kA, and the discharge duration is approximately 300 μsec . The average velocity of the plasma from point A (10 cm) to point C (the gun muzzle) is estimated to be 2.2 km/s from the signals of the optical sensors. The estimated maximum electromagnetic pressure is 1 MPa. The measured plasma voltage ranges from 100-150 V, and the estimated applied energy ranges from 1.0-1.5 kJ. When the delay time is changed from 3 to 20 ms,

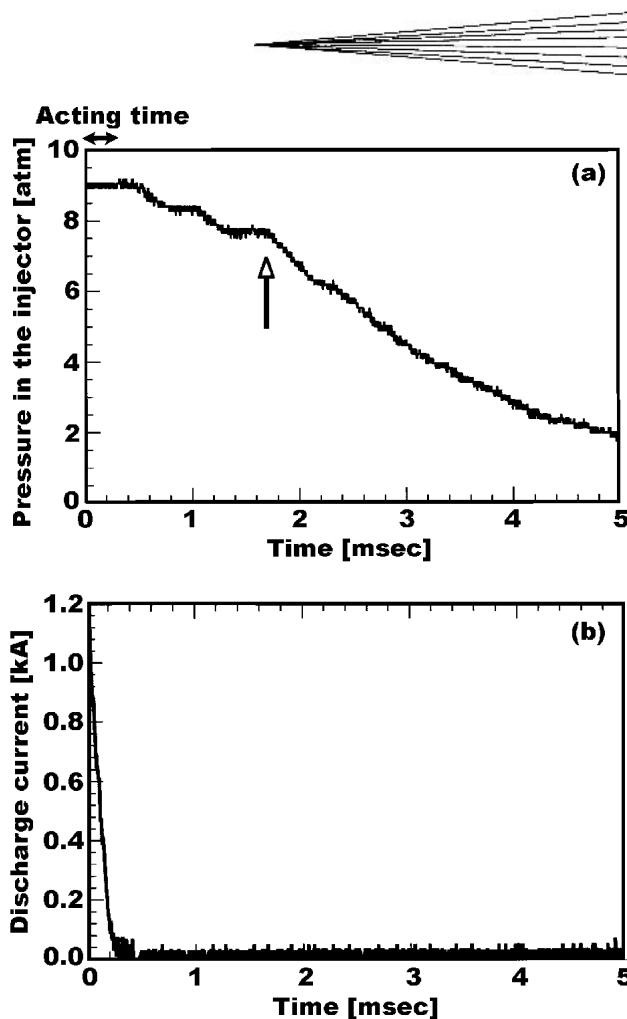


Fig. 3 (a) Pressure change in the injector during B_4C powder injection process; (b) the discharge current for the copper wire explosion

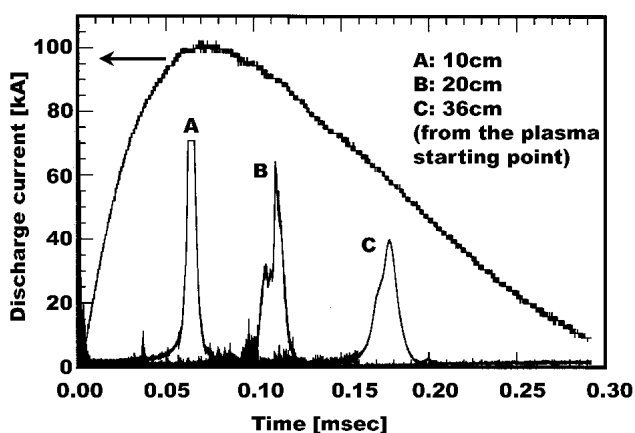


Fig. 4 Typical waveform of high current discharge to generate the electromagnetically accelerated plasma and its light emissions detected by the optical sensors during the spraying process

the velocity of the accelerated plasma also changes, ranging from 1.5-3.0 km/s. The high reproducibility of the coating formation is expected because the reproducibility of the acting time of the powder injector is less than $\sim 1/10$ of the discharge time.

Boron carbide coatings are formed on mirror-polished SUS304 substrates. Although one or two substrates in the six substrates are partially uncovered, other substrates are fully covered when the amount of the powder supply is 0.1 g. At this amount, the growth rate of the fully covered area ranges from 0.5–1.0 $\mu\text{m}/\text{shot}$. The growth rate increases to ranging from 1.5–2.0 $\mu\text{m}/\text{shot}$ as the powder supply is increased to 0.5 g and saturated when the amounts are larger. The uncovered area also becomes smaller as the powder supply is increased, and almost disappears at the supply of 1.5 g. These conditions of the coating formation are found to be highly reproducible from the shots of several hundred times. This indicates the action of the powder injection is highly reproducible.

SEM images of source B_4C powders and a surface of an as-sprayed boron carbide coating are shown in Fig. 5. Deformation of the powder is clearly seen in Fig. 5(b). This suggests that the boron carbide powders melt during the spraying process. No segregated area of boron or carbon was observed from map analysis using EPMA. This suggests there was no decomposition of boron and carbon. The white droplets observed in Fig. 5(b) are determined to be copper by EPMA, which is considered to come from either the gun electrodes or the lead wire used for the plasma initiation. The thickness of the coating increases almost linearly with further spraying onto the coating. Figure 6(a) and (b) shows the optical microscopic images of cross sections of the unsprayed mirror-polished stainless substrate and the boron carbide coating synthesized by 50 multiple shots with 1.0 g/shot of the source powder supply. The thickness of the coating is approximately 80 μm from Fig. 6(b). The growth rate and the deposition efficiency are estimated to be 1.6 $\mu\text{m}/\text{shot}$ and 0.5%,

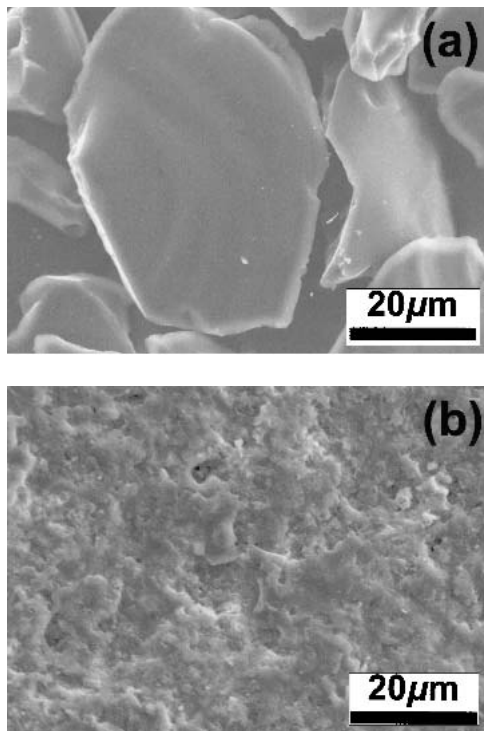


Fig. 5 Secondary electron microscopic images of (a) source B_4C powders and (b) as-sprayed coating surface

respectively. Although there are several cracks, the inter-crack area, that is, the coating structures between the cracks, is relatively uniform with a low porosity compared with the other spray coatings of boron carbide.^[13–16] The width and the number of the cracks in the coating are larger and fewer compared with the growth rate of 1.6 $\mu\text{m}/\text{shot}$ and the number of shots (50), respectively. This suggests the cracks are derived from no original morphological features of the source powder. The cracks are considered to be caused by the internal stress, which is generated by the repetition of the expansion and shrinkage of the coating by repetitive heating during ablating plasma and cooling to room temperature after sprayed, when the coating thickness exceeds critical thickness. From these observations, the density of the inter-crack area is conjectured to be higher than the other boron carbide coatings obtained by conventional spraying.^[13–16] In Fig. 6(b), no vacancy at the interface between the coating and the substrate is observed. The roughness of the interface suggests that the coated materials are anchored strongly on the originally smooth surface of the SUS304 substrate by the high-velocity impact of the powder. Therefore, the coating is expected to have good adhesion to the substrate.

The crystal structure of the coating was investigated using XRD. Figure 7(a) and (b) shows the XRD patterns of the source B_4C powder and the coating of 50 shots, respectively. Glancing incidence XRD ($\theta = 0.3^\circ$) measurement is used for the coating. The diffraction pattern in Fig. 7(a) is matched with the Joint

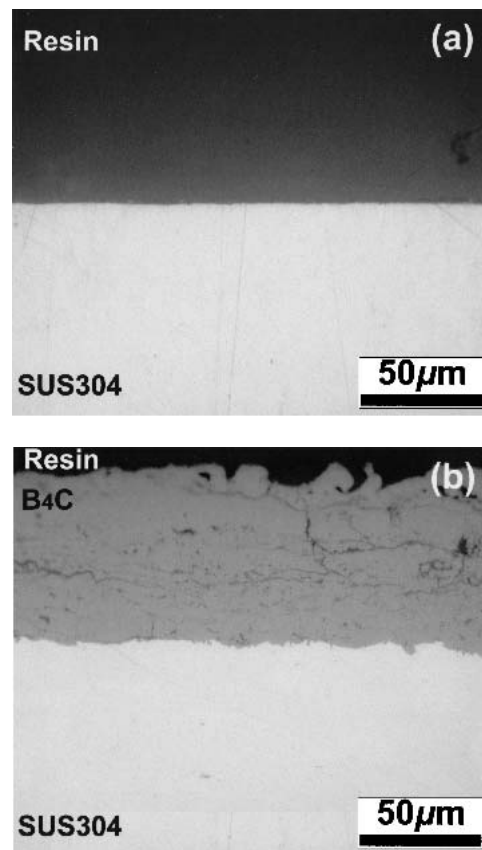


Fig. 6 Cross-sectional optical microscopic images of (a) the unsprayed mirror-polished SUS304 substrate and (b) the boron carbide coating on the SUS304 substrate (50 shots)

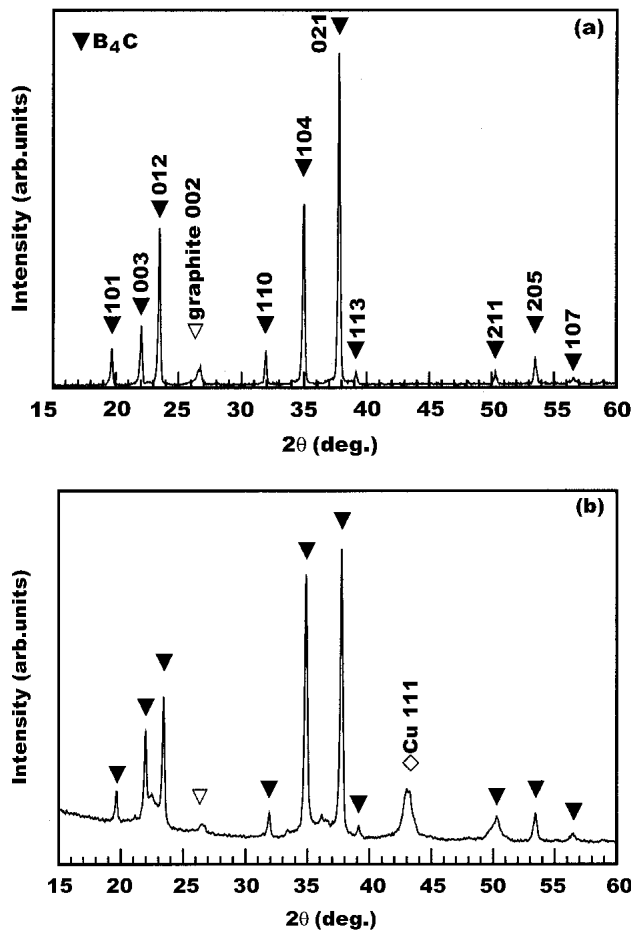


Fig. 7 XRD patterns of (a) the source B_4C powder and (b) the B_4C coating on the SUS304 substrate (50 shots)

Committee of Powder Diffraction Standards (JCPDS) data of rhombohedral boron carbide (card no. 35-0798) except for a peak at $2\theta = 26.35^\circ$, which is identified as belonging to graphite. Compared with Fig. 7(a), the crystallized B_4C pattern that is clearly observed in Fig. 7(b) indicates that the original crystalline structure of the powder is duplicated in the coating. The peak from the graphite is still observed in Fig. 7(b). The peak at $2\theta = 43.3^\circ$ in Fig. 7(b) is identified as belonging to copper. Compared with the other boron carbide coatings,^[7,8,10,11] except for the coating synthesized using the ion-beam evaporation technique,^[17] the coating in this work has a highly crystalline structure. From quantitative EPMA, the chemical composition ratio of boron and carbon of the powder ($B/C \sim 4$) is preserved in the coating. The copper and oxygen atom concentrations in the coating measured by EPMA are approximately 5 at.% and less than 1 at.%, respectively. No incorporation of the diaphragm (polyvinyl chloride, $CH_2 = CHCl$) into the coating is considered due to the preservation of the B/C composition ratio and no observation of chlorine from EPMA.

The Vickers hardness (Hv) of the 50-shot coating and sintered bulk B_4C as a reference is shown in Fig. 8, where the load and loading time are 50 gf and 10 s, respectively. The values of Hv of the inter-crack area of the coating range from 21.6–31.4 GPa. The average value of 25.5 GPa (2600 kgf/mm²) is rela-

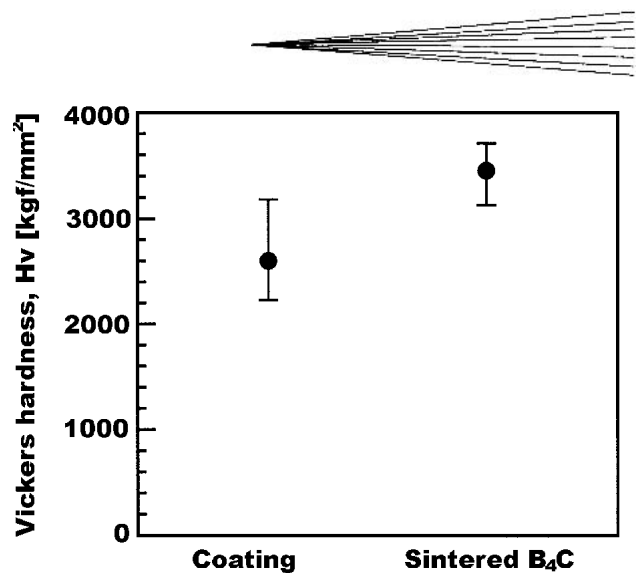


Fig. 8 Vickers hardness of the B_4C coating of 50 shots and sintered bulk B_4C as a reference

tively high among the other boron carbide coatings.^[17] It is assumed that the high Hv values result from the high degree of crystallization, as shown in Fig. 7(b), and the low density of the pores in the inter-crack area, as shown in Fig. 6(b). However, the Hv values are still smaller than the sintered B_4C whose Hv values range from 30.4–36.3 GPa [33.8 GPa (3450 kgf/mm²) average]. The smaller values of the coating are considered to result from insufficient coating density and/or the mixing of copper (Hv ~ 37 kgf/mm²).

4. Conclusion

A system for EMAPS consisting of a pulsed high-current arc-plasma gun and a large flow rate pulsed powder injector has been developed for the coating of hard materials with a high melting point such as B_4C . The advantage of this system is that powders can be intensely accelerated and heated by the high current arc plasma without limitation to the electrical conductivity of the powders. The velocity of the accelerated plasma ranges from 1.5–3.0 km/s, and the estimated maximum electromagnetic pressure is ~ 1 MPa during the high current pulse of 100 kA peak and ~ 300 μ sec of duration. The high reproducibility of the coating formation is achieved due to the highly reproducible action and the large flow rate of the powder injector. Hard and thick boron carbide (B_4C) coatings are formed on mirror-polished SUS304 substrates without binder materials or pretreatment of the substrate. The crystal structure and B/C composition ratio of the source powder are reproduced in the coating despite the loss of morphological features. Further investigations, such as measurements of plasma density, plasma temperature, and powder velocity are necessary to understand the mechanism of this coating process. Also, to increase the growth rate by increasing the shot cycle, improvement of the switch and automating of the powder supply and the plasma initiation are necessary. The formation of rough interface between the coating and the substrate suggests a high adhesion. Also, the loss of the morphological features of the source powder in the coating indicates a large deformation of the powder. It is considered that these are caused by high velocity impact of the powder.

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