

## MANUFACTURING AND WEAR PROPERTIES OF SiC COATING LAYER ON ZR ALLOY FABRICATED BY VACUUM KINETIC SPRAY PROCESS

This study manufactured a SiC coating layer using the vacuum kinetic spray process and investigated its microstructure and wear properties. SiC powder feedstock with an angular shape and average particle size of 37.4  $\mu\text{m}$  was used to manufacture an SiC coating layer at room temperature in two different process conditions (with different degrees of vacuum). The thickness of the manufactured coating layers were approximately 82.4  $\mu\text{m}$  and 129.4  $\mu\text{m}$ , forming a very thick coating layers. The SiC coating layers consisted of  $\alpha$ -SiC and  $\beta$ -SiC phases, which are identical to the feedstock. Cross-sectional observation confirmed that the SiC coating layer formed a dense structure. In order to investigate the wear properties, ball crater tests were performed. The wear test results confirmed that the SiC coating layer with the best wear resistance achieved approximately 4.16 times greater wear resistance compared to the Zr alloy. This study observed the wear surface of the vacuum kinetic sprayed SiC coating layer and identified its wear mechanism. In addition, the potential applications of the SiC coating layer manufactured using the new process were also discussed.

*Keywords:* Vacuum kinetic spray, Silicon carbide, Microstructure, Wear

### 1. Introduction

Silicon carbide (SiC) has outstanding hardness, wear resistance, heat resistance and oxidation resistance properties [1,2]. Based on these properties, silicon carbide has been used in various industrial applications such as turbine parts, silicon carbide liners and heat exchanger tubes. SiC structural ceramic materials are mostly manufactured using powder metallurgy and sintering process. However, the high melting point of silicon carbide makes sintering difficult, and high pressure and temperature (2100°C, 30~100 MPa) and the addition of additives are mandatory to perform sintering densification [3].

Silicon carbide can be used as a functional material by forming a coating layer on an existing metal surface for the purpose of improving physical properties. In the past, attempts were made to use chemical vapor deposition (CVD), physical vapor deposition (PVD) and plasma spray, which are processes that require a high energy source to manufacture a SiC coating layer [4-6]. It is possible to obtain a dense, pure silicon carbide coating layer with CVD and PVD, but due to the high energy consumption and increased processing time according to the low deposition rate (about 1  $\mu\text{m/hr}$ ), the overall process cost increases. In the case of plasma spraying, the high thermal energy of the plasma causes the silicon carbide phase to decompose, limiting its capability to manufacture a SiC coating layer. Due to this, there is a demand to develop a new process with a low process cost and no silicon carbide phase decomposition.

One of the alternatives is vacuum kinetic spray, which can laminate ceramic or compound materials at room temperature. Vacuum kinetic spray is a new, powder-based coating process that uses the pressure differences between the aerosol chamber and deposition chamber to accelerate solid powder (ceramic or compound powder with hundreds nm ~ tens  $\mu\text{m}$  size) up to approximately 100~500 m/s, and the accelerated powder collides with the substrate.

This study manufactured a silicon carbide coating layer using vacuum kinetic spray and investigated the critical wear properties of the material. In addition, the microstructure and properties of a vacuum kinetic sprayed SiC coating layer were compared according to different process conditions, and possible application as a high wear resistance coating layer was discussed.

### 2. Experimental

Figure 1 shows the morphology and particle size of the SiC powder used in this study. The SiC powder (Stanchem, Poland) had an angular shape with a particle size in the range of 19.9  $\mu\text{m}$ -79.2  $\mu\text{m}$ , and the average particle size was calculated as 37.4  $\mu\text{m}$ . The vacuum level of the deposition chamber was set as 2.8 Torr in the #1 condition and 0.7 Torr in the #2 condition, the flow rate was set as 30 L/min in #1 and 10 L/min in #2, and the nozzle movement speed was set as 3 mm/sec in #1 and 1 mm/sec in #2. The distance between the substrate and nozzle was

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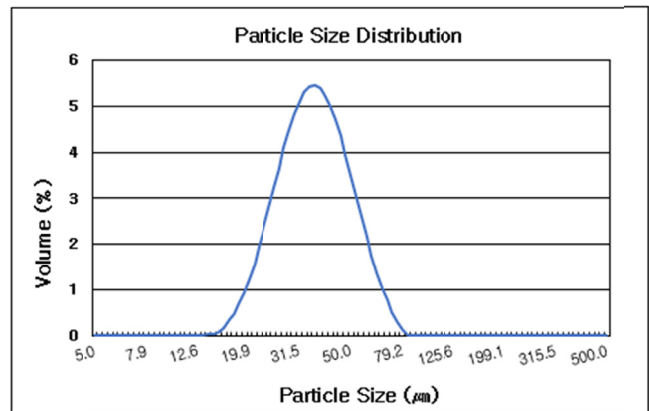
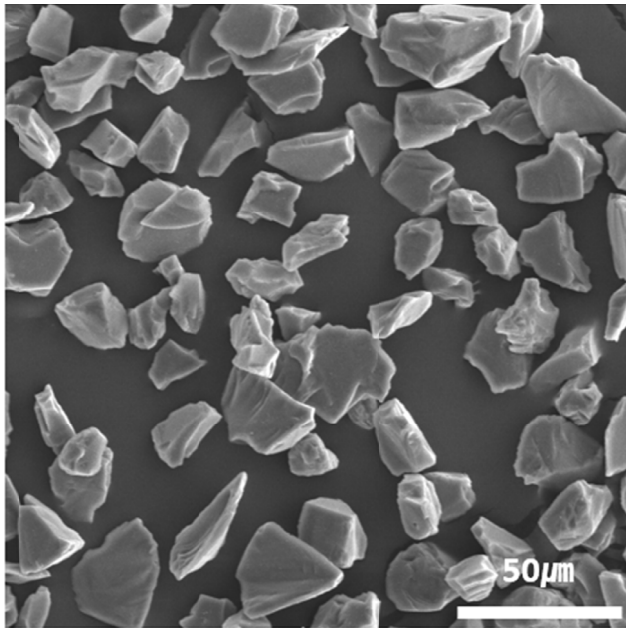


Fig. 1. Morphology (a) and grain size analysis (b) results of initial powder

set as 5 mm in both conditions, and 10 passes were performed at room temperature. A Zr-Nb based alloy substrate was used. From hereinafter, the #1 condition specimen will be referred to as #1 material and the #2 condition specimen will be referred to as #2 material.

The coating specimens were cut and mounted to analyze the microstructure and perform phase analysis. The coating layer thickness and microstructure of the polished specimens were examined using SEM/EDS (scanning electron microscope, Tescan, VEGA II). XRD (X-ray diffractometer, Ultima VI) was used to analyze the phases of the initial powder and coating layer, and SPM (scanning probe microscope, SPA-400) analysis was performed to measure the surface roughness of the coating layer.

A wear test was performed with a three body wear test (ball crater test). A 12.5 mm radius stainless ball was used as the rotation ball. 2  $\mu\text{m}$  SiC powder and water mixture of 3:4 ratio was used as the slurry, and the slurry was supplied at a rate of 1.15 g/min. The test was performed with a sliding distance of 3 m, sliding speed of 0.1 m/s and load of 0.2 N. After the wear test, the worn surface and wear behavior were observed and analyzed using SEM.

### 3. Results and discussion

Fig. 2 shows the XRD phase analysis results of the initial SiC powder feedstock and two manufactured SiC coating layers. It was confirmed that the SiC powder feedstock was composed of  $\alpha$ -SiC and  $\beta$ -SiC phases. The vacuum kinetic sprayed SiC coating materials were also composed of  $\alpha$ -SiC and  $\beta$ -SiC peaks. There were no additional phase transformations or decomposition after coating, but the SiC peak became broader after coating. Vacuum kinetic spray is a deposition process performed by applying high kinetic energy to solid powder at room temperature and caus-

ing a collision between the powder and substrate. Therefore, the phases of the initial powder are maintained, and as the SiC powder collides with the substrate, it is suspected that the fine collisions resulted in the broadening of the peak.

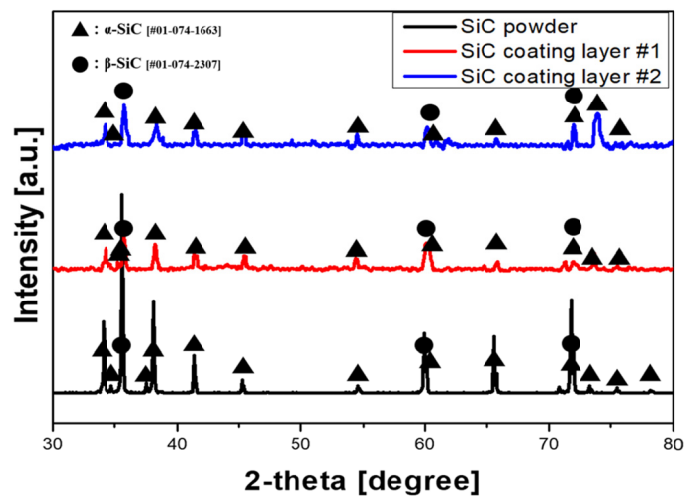


Fig. 2. XRD analysis results of initial SiC powder and vacuum kinetic sprayed SiC coating layers

To measure the thickness and to perform microstructural observation of the vacuum kinetic sprayed SiC coating layers, the cross-section microstructure was observed, and the results are shown in Fig. 3(a), (b). Both the two specimens did not feature initial SiC powder morphologies. The coating layer thickness of #1 material measured 82.4  $\mu\text{m}$ , and porosity was 0.29%. Meanwhile, #2 material, which had a greater vacuum level, had thickness of 129.4  $\mu\text{m}$  and porosity of 0.19%. In closer observation of the coating layer, #1 material had coarse SiC particles of 5–8  $\mu\text{m}$  size (yellow arrows) throughout the specimen, and a lateral crack (green arrow) was present in the coating layer.

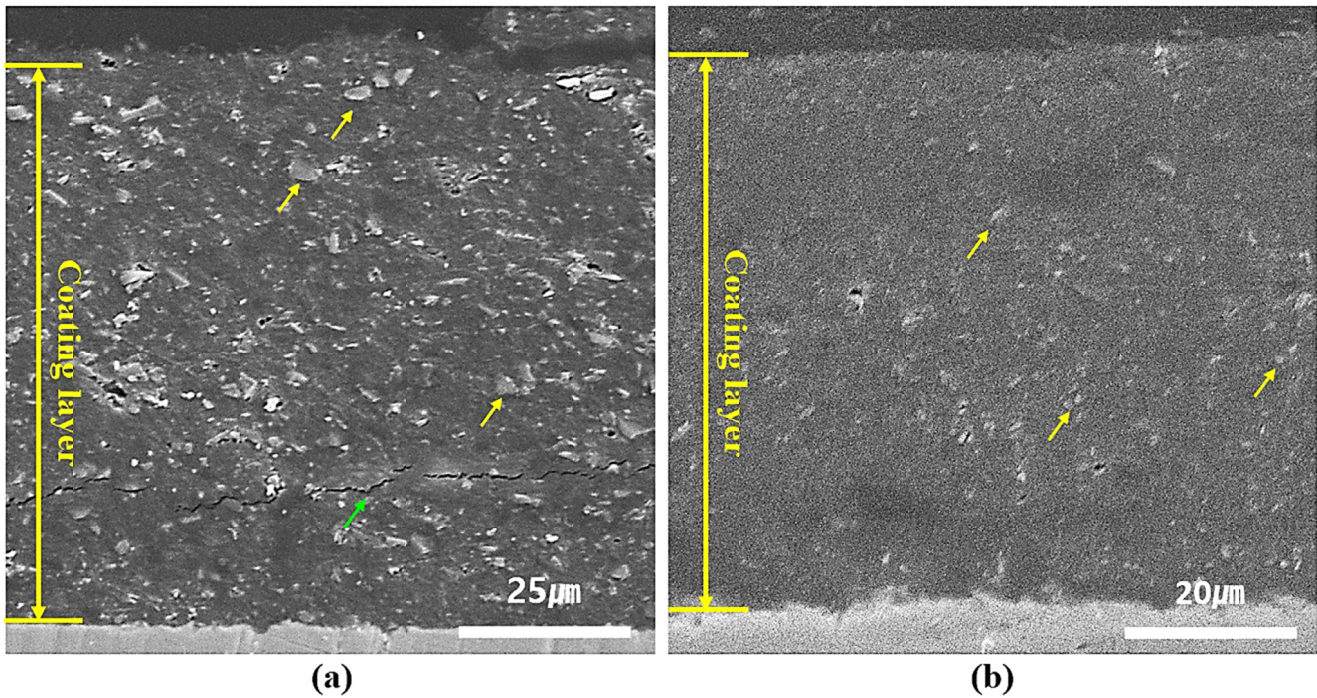


Fig. 3. Cross-sectional microstructure observation results of vacuum kinetic sprayed SiC: (a) #1 SiC coating material, (b) #2 SiC coating material

#2 material had fine fracturing of initial SiC powder, resulting in difficulty of identifying the morphology and size of particles (dark area). However, there were SiC particles of approximately 3~4  $\mu\text{m}$  size (relatively less fractured) in some areas (yellow arrow). In other words, if the vacuum level, which is known to influence particle velocity, is higher (lower Torr), the initial SiC powder (37.4  $\mu\text{m}$ ) receives greater kinetic energy, making it fracture more finely, and this is suspected to result in a coating layer with higher density (lower porosity).

Fig. 4 is the SEM observation results ((a): #1, (b): #2) and SPM analysis results ((c): #1, (d): #2) of the SiC coating layers. Similar to Fig. 3, the shape of initial SiC powder was not present in both SiC coating layer surfaces. On the surface of #1 material, coarse particles of 5~8  $\mu\text{m}$  size were observed, and it was confirmed that the surface was rough. In the case of #2 material, the particles were fractured into a finer size. It was confirmed that less fractured particles of 3~4  $\mu\text{m}$  were present in some areas like that in the microstructural observation. In the surface roughness of the SiC coating layer measured using SPM, RMS values measured #1: ~273 nm and #2: ~112 nm. Considering the initial powder size, the SiC coating layer surface was relatively smooth, but #2 material, which was manufactured at a higher vacuum level with lower Torr (higher kinetic energy), had 2.34 times lower surface roughness. Looking at the SPM image, #1 material had many areas with high height, while in #2 materials, there were only a few areas with high height. Such a high height is suspected to have formed from the less fractured SiC particles seen in Figs. 3 and 4.

The wear properties of SiC coating layers manufactured with vacuum kinetic spray (#1, #2) are shown in Fig. 5. A ball crater wear test measured the wear volume of the substrate Zr alloy as  $12.9 \times 10^{-5} \text{ mm}^3$ , #1 SiC coating layer as  $22.4 \times 10^{-5} \text{ mm}^3$

and #2 SiC coating layer as  $3.1 \times 10^{-5} \text{ mm}^3$ . In other words, #2 material had wear resistance property 4.16 times higher than the substrate Zr alloy, and 7.22 times higher than #1 material. Meanwhile, it is worth noting that the wear volume in #1 material was 1.73 times greater than Zr alloy. In general, Zr alloy is reported to have lower wear resistance than Ni and Fe alloy [7]. Despite this, #1 material had lower wear resistance than Zr alloy, and this is suspected to be due to the microstructure quality (lateral cracks, relatively large fractured SiC particles) caused by the condition. Meanwhile, the wear test results of #2 material show that the vacuum kinetic sprayed SiC coating layer was able to improve the physical properties of Zr alloy, which has low wear resistance, and this is proof that the control of process condition is critical.

Observation results of the wear surface of Zr alloy and SiC coating layers (#1, #2) are shown in Fig. 6(a), (b). In Zr alloy, abrasive wear behavior was shown in all areas of the wear specimen, and a very smooth surface was formed. In the wear surface of SiC coating layers, #1 material had peeling between the substrate and SiC coating layer (the interface between the matrix and SiC coating layer or crack propagated through lateral cracks), 5~8  $\mu\text{m}$  particles (yellow arrow) were eliminated, and a deep wear trace in the coating layer was observed. On the other hand, #2 material, which had a higher vacuum level, did not show signs of peeling between the SiC coating layer and substrate, and a smooth wear surface was formed in most areas. Relatively rough wear surfaces were found in some areas. 3~4  $\mu\text{m}$  particles were observed in these areas, and elimination of particles (yellow circles) was also observed. Lopez et al. [8] reported that smaller grain size in silicon carbides can reduce wear loss (increased wear properties). In other words, the outstanding wear resistance of #2 material is due to i) smaller particle size caused by fine

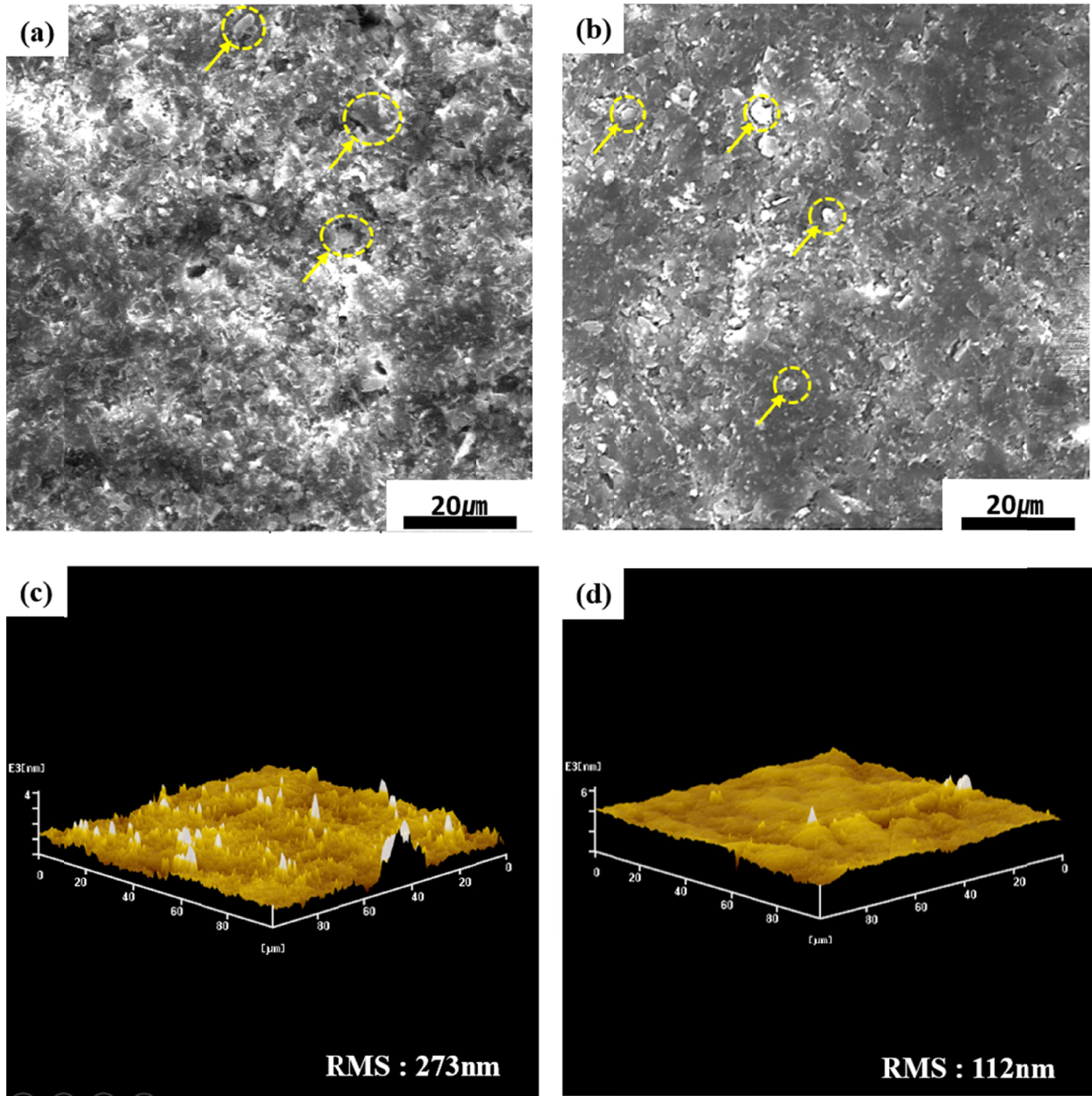


Fig. 4. SEM surface analysis and SPM analysis results of vacuum kinetic sprayed SiC coating layer: (a) #1 SEM, (b) #2 SEM, (c) #1 SPM, (d) #2 SPM

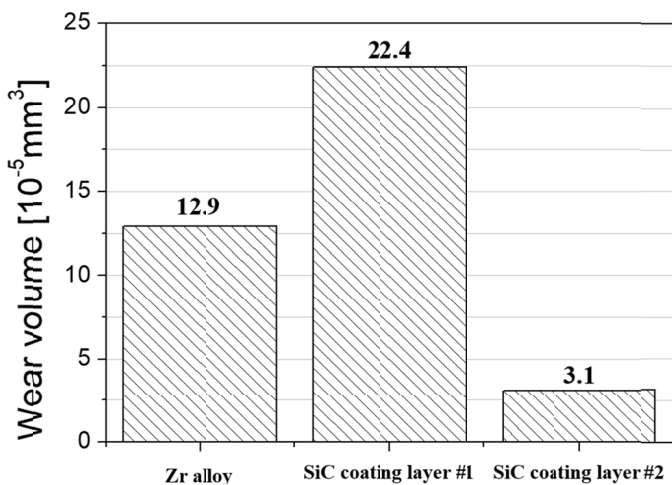


Fig. 5. Ball crater wear test of Zr alloy and SiC coating layers

fracturing of powder deposited by kinetic energy and ii) a low porosity and dense microstructure. In the case of #1 material, the initial defects (cracks, porosity) along with the elimination of 5~8  $\mu\text{m}$  particles in the coating layer resulted in lower wear resistance properties than Zr alloy.

Based on the above findings, it was confirmed that process condition control is critical and vacuum kinetic spray can be used to deposit a sound SiC coating layer that can improve the low wear resistance of Zr.

#### 4. Conclusions

The thickness of the SiC coating layer manufactured using vacuum kinetic spray measured #1: 82.4  $\mu\text{m}$ , #2: 129.4  $\mu\text{m}$ . The

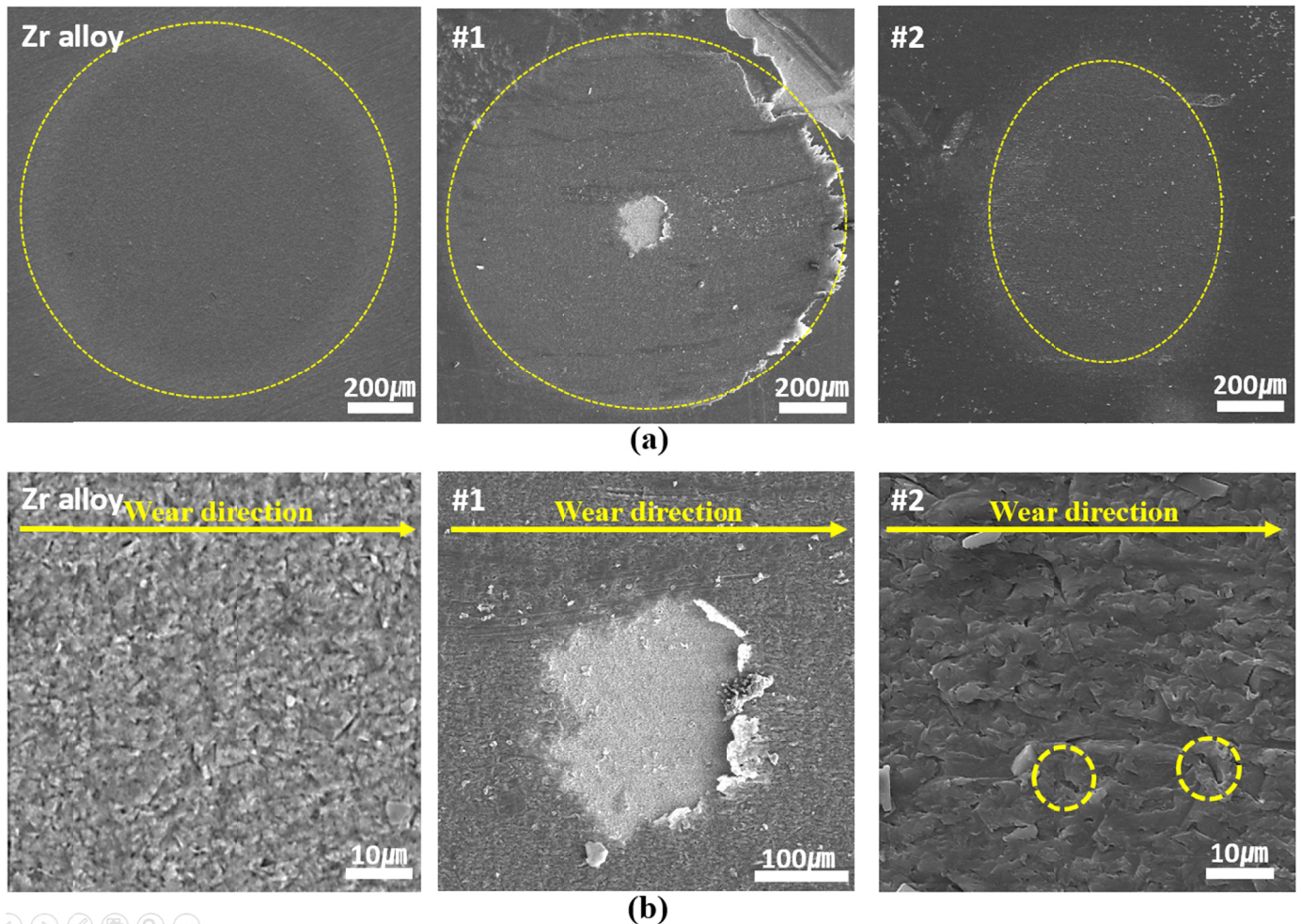


Fig. 6. Wear surface SEM observation results of Zr alloy and SiC coating layers: (a) low magnification images, (b) high magnification images

SiC coating layer consisted of  $\alpha$ -SiC and  $\beta$ -SiC phases that were identical to the initial powder. The shape of initial SiC powder (37.4  $\mu\text{m}$ ) was not present in the SiC coating layer, indicating that most of the SiC powders were fractured and deposited, but there were some areas with relatively less fractured particles of approximately 3–4  $\mu\text{m}$ .

The wear volume measured  $12.9 \times 10^{-5} \text{ mm}^3$  in the substrate Zr alloy and  $3.1 \times 10^{-5} \text{ mm}^3$  in the SiC coating layer, indicating that the SiC coating layer had 4.16 times higher wear resistance property compared to the substrate Zr alloy. In #1 material, peeling between the substrate and SiC coating layer was present in some areas, 5–8  $\mu\text{m}$  particles were dislocated, and a deep wear trace was formed on the coating layer. #2 material, which received higher kinetic energy (due to a higher vacuum level (major factor)), did not show signs of peeling between the SiC coating layer and substrate, and a smooth wear surface was formed in most areas. However, there were some areas with relatively rough surfaces. 3–4  $\mu\text{m}$  particles were observed in these areas, and elimination of particles was also observed.

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