Study of improvement of TiC layer by electrical discharge coating

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Abstract
A titanium carbide (TiC) layer formed by electrical discharge coating has high hardness and excellent wear resistance. Therefore, this layer has been applied to molds, jigs and tools to extend the lives of these devices. On the other hand, depending on prospective application, it may be difficult to apply the layer because of defects and roughness on its surface. In this study, to reduce the number of defects and roughness, an improved TiC layer was formed using a TiC electrode containing silicon (Si), and the characteristics of the layer were investigated. It was found that the defects of the improved TiC layer were fewer than those of the TiC layer. The more Si the electrode contained, the fewer the defects in the improved TiC layer. The roughness of the improved TiC layer was lower than that of the TiC layer, and the hardness of the improved TiC layer was higher than that of the Si layer by electrical discharge coating. The more Si the electrode contained, the lower the roughness and hardness of the improved TiC layer. Furthermore, the improved TiC layer had higher corrosion, erosion and oxidation resistance than the TiC layer, the same as the Si layer.

Key words: electrical discharge coating, improved TiC layer, Si, corrosion resistance, erosion resistance, oxidation resistance

1. INTRODUCTION
The coating technology using pulsed electrical discharge is the new technology based on the technology of electrical discharge machining. It is performed in a dielectric fluid using the electrode made of semi-sintered powder. The micro pulsed discharge is generated between the electrode and the workpiece, and melts the surface of the material of the electrode and the workpiece. The coating layer is formed by moving the melting electrode material to the melted workpiece surface. Since micro pulsed discharge repeated several thousand times a second is used, the heat is hardly concentrated. In addition, since the compositions of the coating layer and the workpiece are mixed at the interface, high adhesion between the layer and the workpiece is possible. Partial coating without pre-processing such as masking, and coating of inner surface of pipes are possible. It is possible to form the hard TiC layer of about 10μm thickness which has high wear resistance when the electrode is made from the semi-sintered powder of titanium carbide. Therefore, this layer has been applied to molds in order to extend the lives.

On the other hand, depending on the candidate for application, the TiC layer may be inapplicable because the roughness of the layer is too high. Although processes after the coating such as a lapping process has been examined in order to improve the roughness of the layer, they have some problems about layer thickness or cost. Although when the roughness of the layer is improved only by improving the TiC electrode, it is conceivable that the above-mentioned problems can be solved and the candidate for application can be extended, such researches have not been performed enough.

In order to make low the roughness of the layer by electrical discharge coating, it is required to make low the roughness of the crater by single pulse discharge. It was reported that the molten material spreads flatly when the coefficient of viscosity of that was low. Then, in this study, an improved TiC layer was formed by electrical discharge coating using a TiC electrode containing silicon (Si) whose coefficient of viscosity is relatively low. Furthermore, the characteristics of the layer were investigated.

2. EXPERIMENTAL METHODS AND CONDITIONS
2.1 Electrical Discharge Coating Method
The schematic diagram of electrical discharge coating is shown in Figure 1 and the coating conditions are shown in Table 1. The electrode used in this study is an improved TiC electrode containing 20, 30 or 50wt% of Si. The improved TiC layer is formed on the workpiece by pulsed electrical discharge whose current is 8A and duration is 4μs between the electrode and the workpiece. The electrode is minus polarity and the workpiece is plus polarity. Open circuit voltage is 220V and Pulse interval is 64μs. When the electrode wears 0.1mm in
2.2 Metallographic Observation Method

The surface of the layer is observed by Scanning Electron Microscope (SEM) to investigate the surface texture of the layer. The workpiece is austenitic stainless steel SUS304.

2.3 Elemental Analysis Method

Point analysis of the surface of the layer and elemental mapping of the cross-section of the layer are performed by Electron Probe Micro Analyzer (EPMA) to investigate the elemental distribution of the layer. Before the mapping analysis, the cross-section of the layer is ground by wet abrasive paper #1000, and finished up by buffing. The workpiece is cold work tool steel SKD11.

2.4 Phase Identification Method

X-ray diffraction pattern of the surface of the layer is measured by X-ray diffractometer (XRD) in order to identify phases of the layer. Before the mapping analysis, the cross-section of the layer is ground by wet abrasive paper #1000, and finished up by buffing. The workpiece is cold work tool steel SKD11.

2.5 Hardness and Roughness Measurement Method

The hardness of the surface of the layer is measured by Micro Vickers Hardness Tester using the load of 0.098N. The roughness of the surface of the layer is measured by a surface texture measuring instrument. The workpiece is cold work tool steel SKD11.

2.6 Corrosion Resistance Evaluation Test Method

Corrosion resistance of the layer is evaluated by aqua regia dip test. Dip time is 1 hour. After dipping, the corrosion wastage is measured and the surface is observed by SEM. The workpiece is precipitation hardening stainless steel SUS630.

2.7 Erosion Resistance Evaluation Test Method

Erosion resistance of the layer is evaluated by water jet test. Fig. 2 shows the schematic diagram of water jet test, and Table 2 shows the conditions of water jet test. After the test, the depth of the maximum damage is measured. The workpiece is SUS630.

2.8 Oxidation Resistance Evaluation Test Method

Oxidation resistance of the layer is evaluated by the heating test. In order to clarify whether the layer oxidizes or not at 800 degrees in the atmosphere, the X-ray diffraction patterns, after the layer is maintained at 800 degrees in the atmosphere for 10 minutes and cooled in the furnace, are measured. The workpiece is hot work tool steel SKD61.

3. RESULTS AND DISCUSSION

3.1 Metallographic Analysis Result

SEM micrographs of the improved layer surfaces (Si content ratio in an electrode: 20, 30 or 50 wt%) are shown in Figure 3. As comparison, those of the TiC layer and the Si layer are also shown. Although it is inferior to the Si layer, the defects of the improved TiC layers are fewer than those of the TiC layer. It is conceivable that this is because the improved TiC layers contain Si whose coefficient of
viscosity is relatively low (0.94 mN \cdot m/s²), so that the coefficient of the molten material of the electrode and workpiece under the coating process is lower and the molten material spreads and solidifies more flatly than the TiC layer. The more Si the electrode contains, the fewer the defects in the improved TiC layer. It is conceivable that this is because the more Si the molten material contains, the lower the coefficient of viscosity of the improved TiC layer.

Next, Figure 4 shows the chemical compositions of the improved TiC layers (Si content ratio in an electrode: 20, 30 or 50 wt%). C (Carbon), Si and Ti of contents of the electrode and Cr (Chromium) and Fe (Iron) of contents of the workpiece exist in all the layers. The more Si the electrode contains, the more Si and the less C and Ti the improved TiC layer contains.

Figure 5 shows the elemental mapping in cross-section of the improved TiC layer (Si content ratio in an electrode: 30 wt%). Ti, C and Si of the contents of the electrode are uniformly concentrated in the layer whose thickness is about 7μm. On the other hand, Fe and Cr of the contents of the workpiece are uniformly distributed in the layer.

X-ray diffraction patterns of the improved TiC layers (Si content ratio in an electrode: 20, 30 or 50 wt%) are shown in Figure 6. As comparison, the pattern of the workpiece is also shown. In the diffraction patterns of all the improved TiC layers, the diffraction peaks of Si and alpha iron(α-Fe) are not observed, but only the diffraction peaks of TiC are observed. It is conceivable that this is because the improved TiC layers are amorphous Fe-Si alloys, the same as the Si layer, and TiC disperses homogeneously in the layers. The more Si the electrode contains, the lower the diffraction intensity of each diffraction peak of TiC.

3.2 Evaluation Results of Characteristics
3.2.1 Evaluation Result of Roughness and Hardness

Figure 7 shows the roughness and hardness of the improved layer surfaces (Si content ratio in an electrode: 20, 30 or 50 wt%).
of the TiC layer and Si layer are also shown. Before
the coating, the roughness of the workpiece is about
1 \mu mRz and the hardness of the workpiece is about
800HV.

Although it is inferior to the Si layer, the
roughness of the improved TiC layers is lower than
that of the TiC layer which is about 7 \mu mRz. Meanwhile, although it is inferior to the TiC layer
which is about 1700HV, the hardness of the
improved TiC layers is higher than that of the Si
layer which is about 700HV. In addition, the more Si
the electrode contains, the lower the roughness of
the improved TiC layer. It is conceivable that it is
because the more Si the molten material contains,
the lower the coefficient of viscosity of the improved
TiC layer. In addition, the more Si the electrode
contains, the lower the hardness of the improved TiC
layer. It is conceivable that this is because the more
Si the electrode contains, the fewer the layer
contains TiC.

3.2.2 Evaluation Result of Corrosion Resistance

Figure 8 shows the reduction in thickness of the
improved layers (Si content ratio in an electrode: 20,
30 or 50 wt%) after the aqua regia dip test. As
comparison, that of the workpiece, the TiC layer and
Si layer are also shown. Although the reduction in
depth of the workpiece is 0.30mm and that of the
TiC layer is 0.19mm, that of all the improved TiC
layers is 0mm, the same as the Si layer.

Figure 9 shows the SEM micrographs of the
specimen surfaces after the aqua regia dip test. It is
observed that the grain boundary is exposed and the
surfaces are corroded in the area without a layer and
with the TiC layer. Meanwhile, it is not observed
that the area with the improved TiC layers is
corroded, the same as the Si layer.

This result shows that the improved TiC layers (Si
content ratio in an electrode: 20, 30 or 50 wt%) have
higher corrosion resistance than the TiC layer. It is
conceivable that this is because the improved TiC
layers are amorphous, so that the grain boundary
corrosion does not occur, and the layers have few
numbers of defects, so that the corrosion from
defects does not occur.

3.2.3 Evaluation Result of Erosion Resistance

Figure 10 shows the maximum damage depth of
the improved layers (Si content ratio in an electrode: 20,
30 or 50 wt%) after the water jet test8). As
comparison, that of the workpiece, the TiC layer and
Si layer are also shown. Although the maximum
damage depth of the workpiece is 1.5mm and that of the
TiC layer is 0.03mm, that of all the improved TiC
layers is 0mm, the same as the Si layer.

Figure 11 shows the optical micrographs of the
specimen surfaces after the water jet test. The
damages in the direction of depth whose diameter is
0.95mm in the area without a layer and is 0.15mm in
the area with the TiC layer are observed. The area
with the Si layer is not damaged in the direction of
depth at all although discoloration is observed. On
the other hand, it is not observed that the area with
the improved TiC layers is damaged, the same as the
Si layer.

This result shows that the improved TiC layers (Si
content ratio in an electrode: 20, 30 or 50 wt%) have
higher erosion resistance than the TiC layer. It is
conceivable that this is because the improved TiC layers are amorphous, so that the grain boundary erosion does not occur, and the layers have few numbers of defects, so that the erosion from defects does not occur.

3.2.4 Evaluation Result of Oxidation Resistance

Figure 12 shows the X-ray diffraction pattern of the improved TiC layer (Si content ratio in an electrode: 30 wt%) before and after heating to 800 degrees. As comparison, the patterns of the TiC layer and the Si layer are also shown. Before heating,
diffraction peaks of oxide are not observed in the pattern of all the layers. After heating to 800 degrees, although the diffraction peaks of titanium oxygen and $\alpha$-Fe are observed in the pattern of TiC layer, those of TiC are not observed. In the pattern of Si layer, the diffraction peaks of oxide are not observed, and those of $\alpha$-Fe are observed because the layer crystallizes$^{10}$. On the other hand, in the pattern of the improved TiC layer, the diffraction peaks of oxide are not observed and those of $\alpha$-Fe are observed, the same as the Si layer. In addition, the diffraction peaks of TiC are observed although the diffraction intensity after heating is lower than that before heating.

Figure 13 shows the SEM micrographs of the layer surfaces before and after heating to 800 degrees, and Figure 14 shows hardness of the layer surfaces before and after heating to 800 degrees. The surface of the TiC layer is damaged severely and the hardness of the TiC layer decreases by the heating. The surface of the Si layer is not damaged and the hardness of the Si layer does not decrease by the heating$^{10}$. On the other hand, the surface of the improved TiC layer is not damaged and the hardness
of the improved TiC layer does not decrease although white spots appear by the heating. This result shows that the improved TiC layer (Si content ratio in an electrode: 30 wt%) have higher oxidation resistance than the TiC layer.

4. CONCLUSIONS

In this paper, an improved TiC layer was formed by electrical discharge coating using a TiC electrode containing Si whose coefficient of viscosity is relatively low. Furthermore, the characteristics of the layer were investigated. The following conclusions were obtained.

(1) The defects of the improved TiC layers are fewer than those of the TiC layer. The more Si the electrode contains, the fewer the defects in the improved TiC layer.

(2) Although C, Si, Ti and Fe exist in the improved TiC layer, the diffraction peaks of Si and alpha iron (\(\alpha\)-Fe) are not observed, but only the diffraction peaks of TiC are observed in the diffraction pattern of the improved TiC layer.

(3) The more Si the electrode contains, the more Si and the less C and Ti the improved TiC layer contains. The more Si the electrode contains, the lower the diffraction intensity of each diffraction peak of TiC.

(4) The roughness of the improved TiC layers is lower than that of the TiC layer, and the hardness of the improved TiC layer is higher than that of the Si layer.

(5) The more Si the electrode contains, the lower the roughness of the improved TiC layer. The more Si the electrode contains, the lower the hardness of the improved TiC layer.

(6) The improved TiC layer has higher corrosion, erosion and oxidation resistance than the TiC layer, the same as the Si layer.

REFERENCES


