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Microstructure of Cr_3Si coatings on austenitic stainless steel by spark plasma sintering[†]

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KEY WORDS: (Austenitic stainless steel) (Coatings) (Hot hardness) (Intermetallic compound) (Reaction layer) (Silicide) (Spark plasma sintering) (Surface modification) (Wear resistance)

1. Introduction

The system requirements of high temperature materials become ever more severe, material that has both excellent oxidation resistance and corrosion resistance is demanded. Surface modification is one of the effective ways. In surface modification processes, there are many coating processes, such as thermal spraying, nitriding and cementation.^{[1][2]} To protect the substrate against severe environments, the surface layer is required to be thick and well joined with the substrate.^[3] In this background, recently, works on the application of spark plasma sintering (SPS) or pulsed electric current sintering (PECS) to the joining or surface modification process have been reported, though much attention has been paid to this process as a fabrication method for new functional graded materials and composite materials. This process enables one to sinter these materials at lower temperatures and in shorter times than conventional sintering techniques.^[4]

In this investigation, Cr-Si intermetallic compound layers were coated on an austenitic stainless steel SUS310S substrate by the SPS process to improve its wear and heat resistances and the resulting properties of the obtained coatings were investigated.

2. Experimental Procedure

Cr (99.5 %) and Si (98 %) with average particle sizes of 45 μm and 150 μm , respectively were used as starting powders and were mixed at a molar ratio of 76:24. An austenitic stainless steel, SUS310S ($\phi 19 \times 5 \text{ mm}$), was used as a substrate. Substrate surfaces were ground with 1.0 μm alumina powders and were degreased by washing in acetone just before coating. The coating was carried out with a spark plasma sintering (SPS) apparatus. SUS310S substrate and a powder mixture of Cr and Si were loaded into a graphite die. Sinter-coating was carried out at 1073-1323 K for 0.6-10.8 ks at a coating pressure of 60 MPa in a vacuum of $4 \times 10^{-3} \text{ Pa}$.

The microstructure of the Cr-Si coating layer and the interface between the Cr-Si coating layer and SUS310S substrate was observed with optical microscopy (OM) and scanning electron microscopy (SEM). The surface layer was also examined by X-ray diffraction (XRD).

Microhardness measurement was carried out using a Vickers microhardness tester under 0.98 N loads. 22 indentations were performed on each sample, and 20 points average value except both maximum and minimum values was used for hardness. Wear testing was carried out at room temperature with a pin-on-disk tribometer. The conditions for wear testing were a running distance up to 300 m, wear load of 5 N, rotating speed of 0.047 m/s, wear radius of 3 mm, and diameter of 6 mm alumina ball used as counter material. Hot hardness test was performed to evaluate the high temperature property of the obtained coating layer.

Table 1 Chemical composition of SUS310S (mass%).

C	Si	Mn	P	Ni	Cr	Fe
0.01	0.64	1.05	0.025	19.02	24.00	Bal.

3. Results and Discussion

Figure 1 shows X-ray diffraction (XRD) patterns of Cr-Si coating layer. XRD studies revealed that the coating layer treated for 10.8 ks at 1173-1323 K consisted of Cr_3Si , Cr_5Si_3 , and unreacted Cr, and the coating layer treated at 1073-1123 K consisted of Cr_3Si , Cr_5Si_3 , and unreacted Cr and Si.

Figure 2 shows the relationship between hardness and density of Cr-Si coating layer treated for 10.8 ks at 1073-

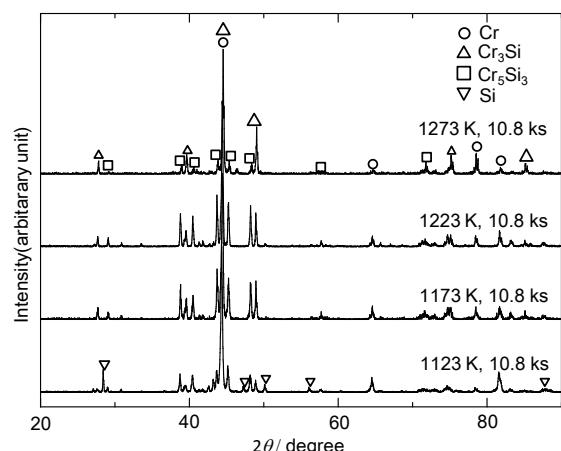


Fig. 1 X-ray diffraction pattern of Cr-Si coating layer.

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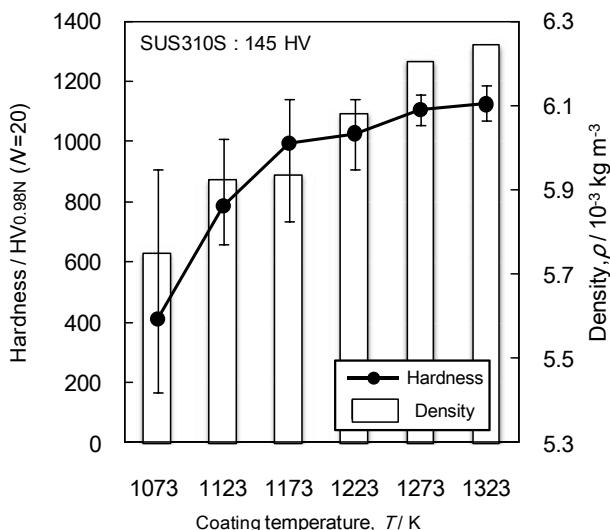


Fig. 2 Relationship between hardness and density of Cr-Si coating layer treated for 10.8 ks at 1073–1323 K by the SPS process.

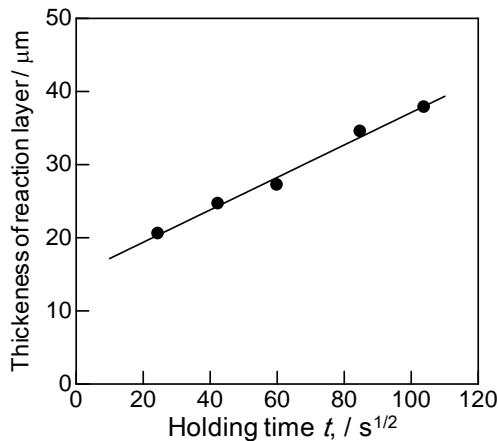


Fig. 3 Effect of holding time on the thickness of reaction layer in samples coated at 1273 K.

1323 K by the SPS process. As the coating temperature increased, the densification was developed and the hardness of these coating layers also increased up to 1150 HV.

Also, a reaction layer with the thickness of 40 μm and the hardness of 700 HV was formed between the coatings and the substrate in the sample treated for 10.8 ks at 1273 K.

Figure 3 shows the relationship between the thickness of the reaction layer and holding time. The growth of the reaction layer showed a parabolic relation against holding time.

Figure 4 shows the wear loss of Cr-Si coating layer and SUS310S substrate. The SUS310S substrate had poor wear resistance. In comparison, Cr-Si coating layer shows considerably less wear. This confirmed that the wear property of SUS310S was improved by the SPS process.

Figure 5 shows a hot hardness result tested at temperatures ranging from room temperature to 1073 K for Cr-Si coating layer and SUS310S substrate. Cr-Si coating layer showed higher hardness than SUS310S substrate, even at high temperature. A gradual decrease in hardness up to a transition temperature, followed by a rapid drop above

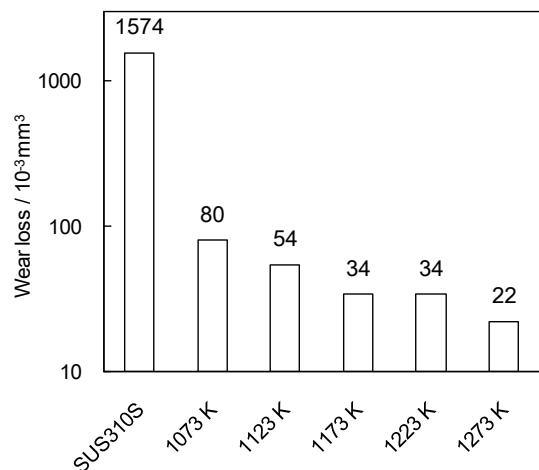


Fig. 4 Wear loss of Cr-Si coating layer and SUS310S substrate.

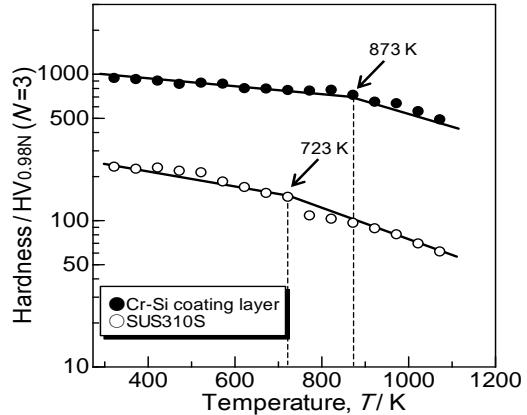


Fig. 5 Hot hardness test of Cr-Si coating layer and SUS310S.

higher hardness than SUS310S substrate, even at high temperature in both Cr-Si coating layer and SUS310S substrate was observed, as shown in Fig. 5. Transition temperatures of Cr-Si coating layer and SUS310S substrate were 873 K and 723 K, respectively. These results indicate that the hardness of 700 HV relatively up to 873 K. Therefore, it was concluded that the Cr-Si coating layer could be applied to some parts that require wear resistance at high temperature.

4. Conclusions

Cr-Si intermetallic compound layers were coated on SUS310S substrate by the SPS process to improve its wear and heat resistance. The conclusions of this investigation are summarized as follows.

- (1) XRD studies revealed that the coating layer treated for 10.8 ks at 1173–1323 K consisted of Cr_3Si , Cr_5Si_3 , and unreacted Cr and coatings treated at 1073–1123 K consisted of Cr_3Si , Cr_5Si_3 , and unreacted Cr and Si.
- (2) The hardness of the coating layer showed 1150 HV at the maximum.
- (3) The reaction layer with a thickness of 40 μm and a hardness of 700 HV was formed between the coatings

and the substrate in the sample treated for 10.8 ks at 1273 K.

(4) Results of pin-on-disk wear tests and hot hardness tests show that the wear and high-temperature properties of SUS310S wear improved by the SPS process.

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