Characteristics of Products of Thermo Reactive Deposition Surface Treatment on JIS-SUJ2 Steel using Fe-Cr Coating Powder with Process Temperature Variation

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Abstract. In this study the effect of temperature variation of the thermo-reactive deposition (TRD) process on carbide layer formation on SUJ2 steel substrates by pack cementation using Ferrochromium powder as carbide former was studied. This process was carried out on SUJ2 steel substrates at temperatures of 900, 980 and 1060 °C for 6 hours. The effect of temperature on layer thickness, homogenity and hardness was studied. Results show that the higher the temperature the thicker the layer formed on the substrate surfaces. Furthermore the XRD results show that the layer formed by this process is chromium carbide. The average microhardness of the layer for 3 process temperature variations is around 1750 HV while the wear rate is around 7 x 10⁻⁴ mm³ / m.

Keywords: Thermo reactive deposition, SUJ2 steel, chromium carbide

1. Introduction

Thin hard carbide coating deposition is one common surface treatment applied to steels to improve life time of tools, dies, and other mechanical parts. As of now, three methods of producing hard carbide are widely used: chemical vapor deposition (CVD), physical vapor deposition (PVD) and thermo-reactive diffusion (TRD) [1]. CVD and PVD, however, have a number of drawbacks, namely high plant investment costs and the requirement of a vacuum or very controlled atmosphere. To obtain the same surface hardness as the CVD and PVD process the TRD process could be an easier, environment-friendly and lower-cost alternative compared to the other processes [2].

TRD process can be done on parts by three methods: molten salt bath [3,4], fluidized bed [5,6] and pack cementation [7,8]. In this process, the carbon and nitrogen in steel substrate diffuse into a deposited layer with carbide forming and nitride forming element such as vanadium, niobium, tantalum, chromium, molybdenum or tungsten. Diffused carbon or nitrogen reacts with carbide and nitride forming elements in the deposited layer to form carbide or nitride layer which is solid and with metallic bonding [9].

TRD coating process can be carried out using chromium. Chromium has good oxidation and corrosion resistance; furthermore CrC has superior hardness, strong adhesion properties and wear resistance which can replace electroplated metal as protection for the mold [5,10]. Several coatings using chromium produce good characteristics for heavy-wear applications such as high hardness and...
toughness, high Young modulus, and high melting temperatures suitable for applications at relatively high temperatures [5,10]. The aim of this study is to produce hard carbide coating on substrate steel by pack cementation. Ferrochromium powder is used to form chromium carbide layers in SUJ2 steel.

2. Experimental method
In this study the coating process is carried out on JIS-SUJ2 steel substrates, with a sample diameter of 1.00 mm and a height of 6.07 mm. The chemical composition of SUJ2 steel is shown in Table 1, all samples were prepared.

Table 1. Chemical composition of SUJ2 Steel (wt %).

| Element | Fe  | C   | Si  | Mn  | Ni  | Cr  |
|---------|-----|-----|-----|-----|-----|-----|
| Composition | Bal | 1.03 | 0.24 | 0.28 | 0.06 | 1.68 |

The mass ratio of steel and powder used was 1:1 where the mass of SUJ2 steel is 300 grams. In addition to carbide-forming powders, Al₂O₃ powder as an anti-sintering agent and NH₄Cl as a catalyst was respectively added 37% and 3% of the amount of powder used. The TRD process was carried out by the cementation pack method in a powder mixture consisting of 180g ferrochromium, 111g Al₂O₃, and 9g NH₄Cl on Table 2. The process was carried out in a furnace for 6 hours at temperatures of 900, 980 and 1060 °C.

Table 2. Powder Coating Composition.

| Material | Percentage (%) | Amount (300 g x percentage) |
|----------|----------------|-------------------------------|
| Al₂O₃    | 37%            | 111 grams                     |
| NH₄Cl    | 3%             | 9 grams                       |
| FeCr     | 60%            | 180 grams                     |

The cross-section of the carbide coating produced on the sample surface was observed, and layer thickness measured, by optical microscopy, using Nital (2% HNO₃ and 98% alcohol) as etchant. Vickers microhardness testing was performed to measure coating layer microhardness with load of 100g and holding time of 10 s.

Wear resistance testing of the coated SUJ2 steel samples was carried out using Ogoshi high speed universal wear testing machine with sliding distance of 400m, load of 120 N and 2.38 m/s sliding speed. The revolving disk used for testing had a radius and thickness of 14.87 and 3.53 mm, respectively. Identification of phases formed on the coating was carried out by XRD analysis with 2θ varying from 10° to 100°, while identification of chemical composition was carried out by EDS linescan in conjunction with SEM.

3. Results and discussion

3.1. The influence of treating temperature on the formation of carbide layer
The effect of treatment temperature on layer thickness is shown in Figure 1. This shows that the average thickness of the carbide layer increases with an increase in treatment temperature from 900 to 1060°C. The correlation can be expressed in the Arrhenius equation:

$$\frac{d^2}{t} = K = K_0 \exp \left( -\frac{Q}{RT} \right)$$  \hspace{1cm} (1)

where d represents layer thickness (m), t is time (s), K₀ is the pre-exponential constant, K is the diffusion coefficient (m / s²), Q is the activation energy in joules and R is the ideal gas constant (8.314 J / mol K).
As can be seen from the equation, changes in temperature theoretically will affect the rate of diffusion of carbon and carbide elements [3,5]. The increase in temperature will accelerate the reaction of the carbide layer formation. As a result, the thickness of the formed coating layer increased with the increase of treatment temperature.

3.2. Influence of Temperature on Hardness
The effect of temperature on the average hardness of the formed layer is shown in Figure 2. From the figure it can be seen that temperature does not have a significant effect on the hardness of the resulting layer. In this study, the hardness for samples treated at temperatures 900, 980 and 1060 °C are 1746.8, 1751.6 and 1753.4 HV, respectively.

3.3. Characterization of Carbide Layer
In Figure 3, the results of optical microscopy on SUJ2 steel cross section coated with Fe-Cr powder at 980°C for 6 hours are shown. There is a clear difference between the substrate and the surface carbide coating without the formation of an interdiffusion layer. The same results were obtained by X.S Fan et al. where in the study no interdiffusion layers were formed between the substrate and the formed coating. However, the aforementioned study used a vanadium-based carbide former, V2O5, to form the carbide layer and the lack of the interdiffusion layer formation was caused by the low solubility of vanadium and carbon in austenite at the TRD process temperature [4].
Figure 3. Cross section optical micrograph.

Figure 4 compares thickness of carbide coatings under different process temperatures. The linescan results further show the diffusion capability of chromium based carbide former with respect to the process temperature. The higher the process temperature, the faster the speed of diffusion to form carbides from this element as shown in Figure 5. This can be seen from the CPS of the element which increases as process temperature increases.

Furthermore, based on calculation of elemental increase using EDS-linescan, in Figure 5 it is shown that Fe content from the layer to the substrate tends to increase. Conversely, the chromium content tends to decrease. Meanwhile chromium content has increased significantly in the substrate/coating interface area.

Figure 6 shows the result of the carbide layer compound chemical composition analysis with a temperature of 1060°C. It can be seen that the compounds found in the layer consist of chromium carbides. The chromium carbides detected are Cr$_{23}$C$_6$, Cr$_7$C$_3$, and Cr$_3$C$_2$.

Figure 4. Cross-section of sample at: (a) 900°C and (b) 980°C.
3.4. The effect of temperature on wear properties

Figure 7 shows the linear relationship between the wear rate and the TRD process temperature. The higher the process temperature, the better the wear resistance of the material. At process temperature of 900°C, the wear rate as measured is $7.25 \times 10^{-4}$ mm$^3$ / m. The wear rate decreases at process temperatures of 980 and 1060°C with a wear rate of $7 \times 10^{-4}$ mm$^3$ / m and $6.875 \times 10^{-4}$ mm$^3$ / m. This corresponds to the hardness values measured on each TRD process temperature.
The highest wear rate is seen in the layer formed at 900°C TRD process temperature with an average hardness of 1587.6 HV. Meanwhile the lowest wear rate is seen at 1060°C, which has the highest hardness value among the other samples. Thus, it can be concluded that the wear rate is inversely proportional to the average hardness value.

4. Conclusion
The coating of chromium carbide to SUJ2 steel substrate by means of pack cementation process performed in this study may be summarized as follows: wear rate is inversely proportional with the average hardness value from the experiment; process temperature has no significant effect on the average hardness where hardness values obtained tend to be similar at 1746.8 HV at 900°C, 1751.6 HV at 980°C and 1753.4 HV at 1060°C. This also impacts wear rate which shows a similar trend in all three process variations at approximately 7 x 10^{-4} mm³/m. The higher the process temperature, the thicker the carbide layer formed, where increasing temperature speeds up diffusion where the average thickness obtained at temperatures of 900, 980 and 1060 °C are 11.35 19.89 and 25.86 μm, respectively. With FeCr as carbide former, compounds found in the layer consisted of chromium carbides (Cr₂₃C₆, Cr₇C₃ and Cr₃C₂).

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