Improving surface properties and wear behaviors of duplex stainless steel via pressure carburizing

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A R T I C L E   I N F O

Article history:
Received 21 May 2012
Accepted in revised form 5 September 2012
Available online 12 September 2012

Keywords:
Carburizing
Surface roughness
Diffusion
Duplex stainless steel
Surface properties

A B S T R A C T

In this research, the surface properties and wear behavior of duplex stainless steel (DSS) carburized using pressure carburizing (PC) method are studied. Carburizing on DSS having different microstructures was carried out using conventional carburizing and pressure carburizing methods. The surface properties namely, the microstructure, hardness and wear of DSS prior to and following the carburizing process were investigated. The results showed that the initial microstructure condition affects the carburizing process wherein the DSS with a finer microstructure exhibits better surface properties than the coarser one. It was found that the surface hardness and wear properties of DSS are enhanced significantly for the sample carburized via PC method. The enhancement was attributed to the formation of a thicker and harder carburized layer. Elemental analyses revealed that the concentration of carbon atoms in the PC sample was highly packed and that the atoms diffused further into the substrate compared with the conventional carburizing sample.

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1. Introduction

Treating steel components to improve the surface properties such as hardness via carburizing process is a well-established technology. Carburizing process is practiced to increase the surface hardness and wear resistance of metals by the diffusion of carbon atoms into the metal substrate to form a carbide layer. Carburizing is usually carried out at elevated temperatures in a carbon medium such as solid, liquid or gas. The carbon medium functions to release carbon atoms into the surface of the workpiece such that the atoms are absorbed into the metals at the carburizing temperature [1–6].

Basically, carburizing under a solid medium involves direct interaction between the substrate’s surface and the surface of the carbon powders. In conventional carburizing methods, the diffusion of carbon atoms into the substrate relies primarily on the temperature and time parameters. Pressure carburizing (PC) is a new surface engineering technique whereby external initial pressure is applied to the substrate during the carburizing process. The application of an external force compacts the carbon powders which increase the interaction areas between the powders and the substrate. It is understood that during the early stage of the solid-state diffusion bonding process, the asperities on each of the faying surfaces deform plastically as the pressure is applied. This is followed by a stage whereby mainly creep and diffusion of atoms are present. Diffusion bonding using superplastic materials has been studied extensively as such materials yield a higher bonding process rate compared with non-superplastic materials [7–10]. This is due to the fact that the surface asperities of superplastic materials can be plastically deformed easily by the applied pressure, which in turn, expedites the early stage. The fine grain microstructures of superplastic materials also accelerate the following stage. Thus, it is expected that the concept based on the solid-state diffusion bonding of superplastic materials can also be applied in the carburizing process under a solid medium, which involves direct interactions between the powder’s surface and the substrate.

In this research, PC is conducted on fine grain DSS in order to enhance its surface properties and wear behavior. DSS possesses superior mechanical properties and excellent corrosion resistance, and is capable of showing superplasticity of up to 2000% elongation [11].

2. Experimental procedure

2.1. Material preparation

The chemical compositions of the duplex stainless steel (JIS SUS329J1) used in this research is shown in Table 1. The specimens were prepared from as-received and thermo-mechanically treated DSS consisting of approximately 50% ferrite and 50% austenite. Each specimen was classified from its grain microstructure size, i.e. coarse

Table 1
The chemical composition of duplex stainless steel (JIS SUS329J1) in wt%.

<table>
<thead>
<tr>
<th>Element</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight (%)</td>
<td>0.06</td>
<td>0.42</td>
<td>0.30</td>
<td>0.03</td>
<td>0.06</td>
<td>4.18</td>
<td>24.5</td>
<td>0.49</td>
<td>Balance</td>
</tr>
</tbody>
</table>
and fine. The as-received DSS has coarse grain microstructure with elongated and longitudinal shape. Meanwhile, fine equiaxed grain microstructure with an average grain size of 3 μm was obtained from the thermo-mechanically treated DSS. Thermo-mechanical treatment was conducted by solution-treating the as-received DSS at 1573 K for 1 h, followed by water-quenching. The specimen was then cold-rolled to a plate through a reduction area of 75%. All specimens were cut from the as-received and thermo-mechanically treated DSS to a dimension of (10 × 10 × 8) mm.

2.2. Conventional carburizing procedure

For conventional carburizing, specimens from the as-received and thermo-mechanically treated DSS were used. Prior to the process, the specimens were ground using emery papers with a grit size ranging from 240 to 1200 in order to remove the oxide layers and irregularities. Following this, the specimens were cleaned using alcohol to remove grease and other contaminants. The specimens were carburized in a stainless steel container, as shown in Fig. 1a.

2.3. Pressure carburizing procedure

For the PC process, only the thermo-mechanically treated specimens were used. Upon the same sample preparation methods as in conventional carburizing, the specimens were arranged inside a clamp, as shown in Fig. 1b. It can be seen that the carburizing powders were filled completely within the center hole of the clamp. An initial pressure of 74 MPa was applied on the specimens by tightening the screws and nuts of the clamp using a torque wrench. The pressure was applied in order to superplastically deform the surface asperities of the material. Both processes were performed in a tube furnace under a controlled atmospheric condition at a temperature of 1223 K for 4 h. Finally, the specimens were air-cooled to room temperature.

2.4. Hardness and microstructure evaluation

The surface hardness of the carburized layers was measured using a microhardness tester fitted with a Vickers indenter under a load of 2 N. The carburized layer thickness and microstructure were examined using an optical microscope and a scanning electron microscope (SEM). X-ray diffraction (XRD) analysis was performed using Bruker AXS-D8 Advance X-ray diffractometer with CuKα radiation at 1.54056 Å X-ray wavelength in order to confirm the presence of carbides on the surfaces. The specimens were scanned from 10 to 80° 2θ angle at a step size of 0.020 and a count time of 1 s at each step.

2.5. Elemental mapping analysis

Elemental mapping analysis was performed using Field Emission scanning electron microscope (FE-SEM) outfitted with secondary electron and backscatter electron detection capability in order to confirm the distribution of carbon on the carburized layers. Elemental imaging of FE-SEM was accomplished using energy dispersive spectrometer with elemental mapping capability.

2.6. Wear tests

Wear tests were carried out under a dry sliding condition using plate-on-flat configuration on a reciprocating wear testing machine (Biceri). Boronized steel with a hardness of 2000 HV was used as the counterface material (sliding plate). Wear tests were also carried out under unlubricated condition at room temperature for 5 min (300 s). The drive speed was set to 280 rpm and the sliding distance was 90 m for an applied load of 50 N. In order to quantify the weight loss during experiments, each specimen was weighed before and after the tests using an electronic balance having a sensitivity of 0.1 mg. The wear rate of each specimen was calculated from the worn surface. Following the wear tests, the wear groove and worn rolling of the specimens were characterized using FE-SEM and the roughness of the wear groove was measured by Taylor Habson Roughness Measurement.

3. Results and discussion

3.1. X-ray diffraction

The presence of carbide phases on the carburized specimens was confirmed by XRD analysis. The typical XRD of fine DSS before and after the carburizing process is shown in Fig. 2, for process duration of 4 h at 1223 K. From the relative peak intensities in the XRD

![Fig. 1. Schematic diagram of (a) conventional carburizing container and (b) pressure carburizing clamp.](image)

![Fig. 2. XRD patterns of 3 specimens after carburizing (a) sample E, (b) sample D, and (c) sample B.](image)
patterns, the presence of carbide phases $\text{Fe}_3\text{C}$, $\text{FeC}$ and $\text{Cr}_2\text{C}_6$ was detected on the fine DSS and as-received materials. This indicates that the carburizing process was successful. Although uncommon, it is interesting to note that the FeC phase was detected here. Study has shown that the FeC also was detected at the interface of diffusion bonded duplex stainless steel and medium carbon steel couple [12].

3.2. Microstructure and carburized layer profile

FE-SEM examinations of the microstructures and carburized layer profiles for both carburizing processes are shown in Fig. 3. The different size microstructure for both grain sizes can be clearly seen in Fig. 3a and b, whereby the average grain sizes are 3 $\mu$m and 30 $\mu$m for fine and coarse DSS respectively. It can be observed that the morphologies for all carburized layers are uniform and smooth. It was reported elsewhere [13] that the compact and smooth morphology is due to the high alloying elements in stainless steel. It can be noted that the formation of carbides is more uniform in the fine grain samples compared with the coarse ones. The carburized layer in the PC sample is obviously thicker. The grain microstructure condition, treatment condition, carburized layer thickness and surface hardness for the samples are summarized in Table 2. It shall be noted that the PC sample has the thickest carburized layer and highest surface hardness, with a value of 64 $\mu$m and 1512 HV respectively. The introduction of force to the surface of the carburized material is expected to deform the asperities plastically, which consequently accelerates diffusion of carbon atoms into the material. This eventually leads to a thicker carburized layer and higher surface hardness for the material. Although the fine grain microstructure also contributes to the increment in carburized layer and surface hardness (comparing samples C and D), it is notable that the effect of super-plasticity is more pronounced (comparing sample D and E).

Fig. 4 shows high and low magnification of the carburized layer for fine grain DSS. The cross-section microstructure consists of a mixed phase structures, particularly near surface region. High carbon super-saturation would be expected at the upper part of layer.
carburizing stage, the austenitic grains (γ) are transformed to carbon expanded austenite (γ′) while ferrite grains (α) are first transformed to austenite and then changed to carbon expanded austenite due to carbon diffusion. Carbide precipitates appear in the layer when the solubility of austenite is above the boundary solubility in austenite. As can be seen in the figure, the carburized layers produced below the critical temperature are free from precipitation and contain supersaturated carbon. On the other hand, the layers produced above the critical temperature revealed a mixed phase structures, particularly in the upper part of the layer. The cooperation with the existence of coagulated and uniformly distributed carbides gives a high hardness on the surface.

FE-SEM micrograph in Fig. 5 indicates that the surface is composed of microcrystalline grains of carbide formation on top of the surface. The output carbides grow in the over-saturated austenite and form iron carbides. These may be associated with a higher carbon diffusion rate on the surface. As a result of these, forming carbides is responsible for the intense surface hardening up to 1500 HV.

3.3. Elemental mapping analysis

Elemental mapping analysis was performed to verify the distribution of carbon atoms. The substrate’s surface area and the results are displayed in Fig. 6. The white dots symbolize the existence of carbon atoms. It is clearly observed that the concentration of carbon atoms is highly packed and uniform in the PC sample and that the atoms are diffused further into substrate compared to the conventional carburizing samples. The results confirm to the hardness gradient of the sample’s cross-sections as shown in Fig. 7. As expected, the PC sample exhibits the highest surface hardness. Meanwhile, the hardness decreases towards the core which indicates a reduction in the concentration of carbon.

<table>
<thead>
<tr>
<th>Sample code</th>
<th>Grain structure</th>
<th>Treatment</th>
<th>Carburized layer (μm)</th>
<th>Surface hardness (HV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Coarse</td>
<td>No carburizing</td>
<td>–</td>
<td>340</td>
</tr>
<tr>
<td>B</td>
<td>Fine</td>
<td>No carburizing</td>
<td>–</td>
<td>426</td>
</tr>
<tr>
<td>C</td>
<td>Coarse</td>
<td>Conventional carburizing (CC)</td>
<td>25</td>
<td>1116</td>
</tr>
<tr>
<td>D</td>
<td>Fine</td>
<td>Conventional carburizing (CC)</td>
<td>35</td>
<td>1243</td>
</tr>
<tr>
<td>E</td>
<td>Fine</td>
<td>Pressure carburizing (PC)</td>
<td>64</td>
<td>1512</td>
</tr>
</tbody>
</table>

Fig. 4. Microstructure of carburized layer on fine grain DSS (a) high magnification (b) low magnification.

Fig. 5. FE-SEM microstructure of carbide formation on the top surface of fine grain DSS.

Fig. 8 shows the EDX analyses of carbon concentration at some region of carburized layer on PC sample. It can be seen that the percentages of carbon decrease towards the substrate proving that the elemental diffusion occurred at the surface into the interior. In addition, a large number of grain boundary in PC sample which has fine grain DSS could enhance the movements of carbon into the substrate as it could provide more diffusion paths.

3.4. Wear behavior

The calculated wear volume loss is presented in Fig. 9. From this figure, the lowest values of wear volume are attained for sample E (PC sample), with an approximate value of $1.54 \times 10^{-10}$ m$^3$. The specific wear rate for PC sample is roughly 6 times lower than sample A and approximately 3 times lower than sample B. It was noted that samples A and B are non-carburizing samples. As can be seen, the PC sample (E) still produces the lowest values for specific wear rate and volume when compared with samples C and B, which were carburized using the conventional method. This is mainly due to the increased surface hardness and existence of hard carburized layer Fe$_3$C and FC on the surface. It is thus obvious that the wear resistance was enhanced appreciably via PC process on fine grain. The relationship
between the specific wear rate and surface hardness for the carburized samples is shown in Fig. 10. It is observed that the specific wear rate decreases with an increasing surface hardness. Therefore, the satisfactory wear performance through the test can be achieved by PC sample compared with others.

The worn surfaces of the specimens were examined using FE-SEM to elucidate the modes of the wear process. Fig. 11 shows the images of wear tracks at lower magnification and higher magnification (inset) for samples A, B, C, D, and E. From this figure, the worn tracks of the carburized samples indicate that the shallower groove wear pattern is dominant on PC sample. Again, this proven excellent wear resistance was produced on the PC sample.

4. Conclusions

In this research, it is found that the carburized layer thickness ranges from 25 μm to 64 μm and the surface hardness ranges from 340 HV to 1512 HV. The PC sample exhibits a significant enhancement in wear behavior, whereby the samples attain the lowest specific wear rate and wear volume. In conclusion, it is proven that the surface properties and wear behavior of DSS can be enhanced appreciably via PC process using fine grain microstructure. It is noteworthy that the application of an external force in the carburizing process could produce such a different result. This research can be extended to investigate and exploit the external force factor in...
surface hardening using a solid medium to further improve the
goal of the process.

Acknowledgments

The authors would like to extend their greatest appreciation to the
Institute of Research Management and Consultancy, University of
Malaya (IPPP) for funding this research under grant number: PS052/
2007B, University of Malaya under High Impact Research (HIR), grant
no. (J-16001-73804).

References

643.
Fig. 8. EDX analyses of carbon concentration on some region towards the substrate for sample E.
Fig. 9. Wear volume losses for all samples.

Fig. 10. Relationship between specific wear rate, $K_w$, and surface hardness value.
Fig. 11. FE-SEM micrograph showing a portion of the wear scar region at lower magnification (bigger picture) and higher magnification (inset) for (a) sample A, (b) sample B, (c) sample C, (d) sample D, and (e) sample E.