Enhancement of Erosion Resistance on AISI H13 Tool Steel by Oxynitriding Treatment

Shih-Hsien CHANG, Tzu-Piao TANG, Yi-Chin CHEN and Jhewn-Kuang CHEN

Department of Materials and Mineral Resources Engineering, National Taipei University of Technology, Taipei, 106, Taiwan, R. O. China. E-mail: changsh@ntut.edu.tw

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In order to effectively improve erosion resistance and evaluate the effects of an oxy layer on AISI H13 tool steel after the oxynitriding process, this study used three different nitriding surface treatments, namely oxynitriding process 1 (using air), oxynitriding process 2 (using steam) and normal gas nitride. To evaluate the effects of microstructure and the erosion resistance of AISI H13 tool steel after different nitride processes, evaluated micro hardness, erosion tests and SEM microstructure inspections were conducted. Experimental results showed that the oxide layer can protect and improve the aluminum erosion for AISI H13 tool steel. Erosion tests of 2 and 4 h for oxynitriding process 1 could produce a thicker and complex oxide layer, which has higher hardness (HV 1021.9) and optimal weight loss (0.16%). This procedure is proven to effectively reduce the ratio of Al-Fe-Si compounds during the A380 alloy erosion test.

KEY WORDS: erosion resistance; AISI H13 tool steel; oxynitriding; A380 alloy.

1. Introduction

During hot work steel processes, the die surface is rapidly heated and then quenched, thus resulting in a decrease of surface hardness and toughness. In addition, thermal gradients lead to dimensional variations, and generate stress and deformation. Thermal fatigue and erosion are the most important factors limiting tool life and resulting in tool failure of hot work tool steels. In the case of most steels, nitriding can be used for wear resistance and has some improvement on corrosion resistance. Using nitriding to enhance the wear, fatigue and corrosion resistance is a complex problem because of the results may be affected by related factors in the process.

In the die casting process, molten aluminium alloy at temperatures ranging from 943–983 K is injected into the die cavity at high velocity from 30 to 100 m s⁻¹. The injection pressure is 50–80 MPa. In aluminium die casting, tools are exposed to erosion, corrosion and soldering under frequent contact of the tool surface to the casting alloy, as well as heat checking and gross cracking due to thermal fatigue, and oxidation due to high pouring temperature. The gradual destruction of die surfaces during the service process decrease the casting piece quality and limit the die lifetime.

Oxynitriding processes use air or steam at the end of the nitriding stage and it is an integral part of the treatment, used as an oxidizing medium. The complex oxide layer with Fe₃O₅ and Fe₇O₈ structures are formed on the surface, improving the corrosion and erosion properties of the steel. In die casting operations, a hot work tool steel AISI H13 is commonly used as die material, and an aluminum silicon alloy A380 as a cast material. In this study, the chemical composition (mass %) of the AISI H13 tool steel was shown in Table 1. Similarly, the composition of A380 alloy was shown in Table 2, respectively.

2. Experimental

The objective of this study was to explore the possibility of surface engineering of the die steel surface to reduce the erosion of the die surfaces. One gas nitriding and two different oxynitriding treatments were experimented to determine the feasibility of improving and enhancing the erosion resistance of hot work tool steel. The results indicated that oxynitriding treated with an air procedure had showed better erosion resistance after long-cycle erosion tests.

In this study, the erosion resistance of nitride was evaluated by its weight loss percentage: weight loss of the specimens dipped in molten aluminum alloy for a predetermined length of time, as shown in Fig. 1(a). AISI H13 tool steel (quenched at 1303 K and tempered at 853 K, and repeated for 3 times to reach a hardness of HRC 47–48) was used as the substrate material. Moreover, the specimen sizes of the

| Table 1. Chemical composition of AISI H13 tool steel (mass%). |
|-------------|-----|-----|-----|-----|-----|-----|-----|
| C           | 0.39 | 0.81 | 0.02 | 0.38 | 0.14 | 5.0  | 1.15 | 0.82 | 91.29 |

| Table 2. Chemical composition of A380 alloy (mass%). |
|-------------|-----|-----|-----|-----|-----|-----|-----|-----|
| Cu          | 1.62 | 11.22 | 0.3  | 0.78 | 0.86 | 0.12 | 0.05 | 0.02 | 83.03 |
erosion test as shown in Fig. 1(b). The specimen was then dipped in aluminum alloy A380 for melting and maintained at 1023 K. The rotational speed of the specimen was kept at 50 rpm. The dip time was 2 and 4 h for specimens to evaluate the erosion resistance and weight loss. After removing the aluminum, the specimens were cleaned with NaOH to remove oxide or other residues. The weight loss percentage of the erosion test was calculated as follows:

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\text{Weight loss} \% = \left( \frac{W - AW}{IW} \right) \times 100
\]

IW is the initial weight and AW is the weight of after erosion test.

In the experiment, three different kinds of surface treatments were: 1) normal gas nitriding: at temperature of 843 K for 1 h; 2) oxy-nitriding process 1: gas nitriding was carried out at temperature of 843 K for 1 h, and air oxidation at 823 K for 3 h; 3) oxy-nitriding process 2: gas nitriding was carried out at temperature of 803 K for 1 h, and steam oxidation at 798 K for 1 h. To evaluate the effects of erosion resistance on AISI H13 tool steel by nitriding process, erosion test, surface hardness and microstructure inspections were performed. Microhardness tests were measured by HV with loading of 200 g, which complied with the CNS 2115 Z8904 standard.

3. Results and Discussion

The microstructure of AISI H13 tool steel substrate obtained after quenching and tempering processes is shown in Fig. 2. This is the typical microstructure obtained through commercial heat treatment, which comprises the structure of tempered martensite and preeutectoid carbides. Figure 3 shows the microhardness test of AISI H13 tool steel after gas nitriding and different oxy-nitriding treatments. Gas nitriding was done with ammonia, decomposition \( \text{NH}_3 \) was used to dilute the ammonia so that it was not too aggressive. Normally, pure decomposition \( \text{NH}_3 \) is used to reduce the compound layer (white layer). Both carbon content and alloying additions raise surface hardness, thereby enhancing wear resistance. The actual hardness of the compound layer is usually substantially higher. However, a better thickness of the compound layer should be created during gas nitriding, instead of creating excessive layer and reducing it later. Due to the effects of the compound layer, gas nitriding has the highest hardness of HV 1058. The nitriding depth of oxy-nitriding 1 and gas nitriding treatments were 0.12 mm, however, oxy-nitriding 2 was only 0.1 mm, as shown in Fig. 3.

Figure 4 shows the optical microscopy of different surface treatments for AISI H13 tool steel. Gas nitriding is a typical method of producing gas nitrided surfaces that have uniform microstructures covering the whole gap depth. In most studies, a compound layer is present on the nitrided surfaces, as shown in Fig. 4(a). The compound layer is not an appropriate surface layer for die steel as many defects and cracks can be found on the surface compound layer of
the steel. The cracks of the white layer are damaged in the molds during production; most of the ruptured sources are originated from the mold and are in the compound layer.\textsuperscript{5,6} Therefore, the compound layer must be removed by grinding, polishing or sand blasting. Erosion is a progressive loss of material from a solid surface due to mechanical interaction between surface and impinging fluid stream. The result of erosion is washout of the die surface.\textsuperscript{5,10}

In the oxy-nitriding treatments with air and steam processes, the last oxidation step of the oxy-nitriding treatment is the formation of a black, porous oxide layer (0.01 mm thick), which is composed of a mixture of Fe\textsubscript{2}O\textsubscript{3} and Fe\textsubscript{3}O\textsubscript{4} oxides, as shown in Figs. 4(b) and 4(c). Generally, Fe\textsubscript{2}O\textsubscript{3} and Fe\textsubscript{3}O\textsubscript{4} structures are present in oxy-nitriding of air process, but only Fe\textsubscript{3}O\textsubscript{4} appears in the oxy-nitriding of steam process. The complex oxide layer with an Fe\textsubscript{3}O\textsubscript{4} structure is formed at the surface, improving the erosion resistance with a minimal effect on its tribological properties. At the same time, the surface assumes a dark or black appearance, which gives it an attractive aspect, desirable in many applications. There is no white layer on the surface of steel. This oxide layer can eventually be sealed with an impregnation fluid to increase corrosion resistance.\textsuperscript{2,11,12}

Figure 5 shows the appearance of untreated and different surface treatments for AISI H13 tool steel after 4 h erosion test. Most specimens are erosive by the high temperature of the aluminium alloy solution, but only a few erosions appear in the oxy-nitriding process 1. There was almost no melting phenomenon could be observed, as shown in Fig. 5(c). It showed that the oxide layer has obviously enhanced the erosion resistance of AISI H13 tool steel through the air oxidative procedure, especially in the longer erosion test. Moreover, the weight loss percentage of untreated and different surface treatments for AISI H13 tool steel after 2 and 4 h erosion tests are compared, as shown in Fig. 6. It is found that the oxy-nitriding process 1 has the best anti-melting ability. Molten aluminium has affinity for the AISI H13 tool steel surface. The diffusion of aluminium and iron atoms across the interface occurs to form Fe\textsubscript{2}Al\textsubscript{1}Si\textsubscript{2} intermetallic at the interface. Silicon changes the rate kinetics and the solubility of iron in aluminium.\textsuperscript{6} Mass loss has taken place on the steel surface. The cyclic process of dissolution continues while the steel surface continuously loses iron to the melt. In this study, the thickness and density of the oxide layer clearly affect the erosion resistance of the post-nitriding process. Complex oxide layers with Fe\textsubscript{2}O\textsubscript{3} and Fe\textsubscript{3}O\textsubscript{4} structures are formed on the surface of the steel after air oxidizing treatment. They decreased the weight loss to 97.3% of AISI H13 tool steel after 4 h erosion test.

Intermetallic layers (Al\textsubscript{1}Fe\textsubscript{7}Si\textsubscript{2}) form at the cast metal–die steel interface when the liquid metal is in intimate contact with the die steel. Their morphology depends on the composition of the cast metal and die steel, the temperature of the cast metal and the time of contact.\textsuperscript{5,12,13} Figure 7 shows the SEM micrographs of un-treated and different surface treatments for AISI H13 tool steel after 4 h erosion test. This erosive reaction with the iron-rich surface which causing intermetallic formation, and dissolution of the steel surface into the melt. Al–Fe–Si compound is observed in the boundaries of the nitride layer and A380 alloy. Deposition of the aluminium on the die surface during the solidification process leads to solid-state diffusion between iron, and the aluminium leads to chemical sorption and adhesion.\textsuperscript{6} Figure 7(a) has the more thickness of Al–Fe–Si compound layer on the surface for un-treated steel. Both nitriding and oxidiz-

![Fig. 6. Comparison of weight loss percentage of un-treated and different surface treatments for AISI H13 tool steel after 2 and 4 h erosion tests.](image)

![Fig. 5. Appearance of un-treated and different surface treatments for AISI H13 tool steel after 4h erosion test (a) untreated, (b) nitriding, (c) oxy-nitriding process 1, (d) oxy-nitriding process 2.](image)

![Fig. 7. SEM micrographs of un-treated and different surface treatments for AISI H13 tool steel after 4h erosion test (a) untreated, (b) gas nitriding, (c) oxy-nitriding 1, (d) oxy-nitriding 2.](image)
ing layers have been dissolved by the A380 alloy. This could be due to the higher affinity of nitrogen and oxygen for aluminium than iron, which causes the reduction of the two layers and their dissolution, as shown in Figs. 7(b) and 7(d). However, the oxynitriding process presented the nitriding layer still retain on the surface, only a few Al-Fe-Si compound layers appear, as shown in Fig. 7(c). These results clearly indicated that the oxynitriding process 1 has the better erosion resistance.

Figure 8 shows the EDS results of the Al-Fe-Si compound layer observed for AISI H13 tool steel after 4 h erosion test. Figure 8(a) shows the precipitation of Al, Si, and Fe elements at A380 and AISI H13 tool steel boundaries, which represent the Al, Si, Fe, and oxygen elements emerged in the boundaries, as shown in Fig. 8(b). However, the mapping analysis shows that the nitriding and oxide layers have effectively protected the erosion of aluminum alloy after 2 h test, and increased the erosion time to 4 h, as shown in Fig. 7. Al-Fe-Si compounds completely replaced the original nitriding layer, only the oxynitriding process 1 retained the nitriding layer and Al-Fe-Si compounds.

4. Conclusions

(1) Nitriding layer can effectively protect material and decrease the weight loss during erosion tests. Furthermore, both gas nitriding and oxynitriding treatments can increase the erosion resistance of AISI H13 tool steel. However, oxynitriding process could produce a thicker and complex oxide layer, which has higher hardness and optimal weight loss. It showed that the oxynitriding process has better erosion resistance than gas nitriding.

(2) The depth of the oxide layer improves the erosion resistance of AISI H13 tool steel by A380 alloy immersion test. Through an air process, oxynitriding treatment can produce thicker oxidation that can more effectively reduce the effects of Al-Fe-Si compounds.

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