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STUDIES ON THE HIGH TEMPERATURE PROPERTIES OF DUPLEX-TREATED AISI H13 STEEL

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ABSTRACT

In oder to improve the wear resistance as well as oxidation resistance at high temperature a AISI H13 steel was treated by a duplex process of calorizing followed by plasma nitriding. The surface properties of the duplex-treated AISI H13 steel was characterized and compared with those treated by single surface process of calorizing and plasma nitriding, in terms of microstruture, microhardness, wear resistance at 500℃, and the oxidation behaviours at 700℃. Duplex process on H13 steel had created duplex layer of approximately 190μm on the surface, and surface microhardness was measured to be above 1450Hv(0.1Kgf). There was considerable improvement of the high temperature wear resistance at 500℃ in the duplex-treated steel when both wear volume and weight change due to oxidation were considered. In addition the duplex-treated steel showed an improved high temperature oxidation resistance than the plasma nitrided steel at 700℃.

INTRODUCTION

Extensive researches on the development of the surface treatment processes to increase the service life of mold steels and high speed tool steels for high temperature applications have been made. Some of the processes developed include plasma enhanced CVD process, ion plating process for TiN, TiC, TiCN, CrN hard coating, and gas and plasma nitriding process.[1-4] Plasma nitriding developed by Egan in the 1930s is one of the important surface hardening processes which provides enhanced wear-, fatigue-, and corrosion-resistant surface. Details of the principles and applications of the plasma nitriding process have been well described in many literatures[5-8]. Significant improvements on plasma nitriding process were made in the past decade. However successful application of plasma nitriding on the materials used at high temperature is very limited as the nitrided layer becomes very unstable due to the dissociation of the nitrogen from the nitride and the oxidation problems when exposed to high temperature. Al diffusion coating, also known as calorizing, provides steel with not only excellent oxidation and corrosion resistance at elevated temperature up to 900℃, but reasonable scaling resistance[9]. In this study, as a part of different attempts to develop a process to enhance both the wear and oxidation resistance at high temperature at the same time, the duplex treatment of calorizing followed by plasma nitriding was studied. The oxidation properties at 700℃ as well as the wear properties at 500℃ were compared for three different processes, calorizing, nitriding, and the duplex treatment of calorizing followed by plasma nitriding.

EXPERIMENTAL PROCEDURE

AISI H13 disk samples of a diameter of 25 mm and 50mm with a thickness of 5mm were machined for microstrutural analysis and for wear test respectively. The surface of each specimens were polished with 0.3μm alumina powder, and cleaned specimens were calorized using pack cementation for 3 hours at 1000℃. Specimens was air-cooled after calorizing. The pack composition was 30wt% FeAl (+325mesh), 69wt% Al_2O_3(+250mesh), and 1wt% NH_4Cl. Calorized specimens were polished through 0.3μm alumina and cleaned for 10 minutes in acetone prior to plasma nitriding treatment. Sputtering cleaning was performed prior to plasma nitriding for 30 minutes under H_2 atmosphere and plasma nitriding was performed in a 25% N_2-75% H_2 atmosphere for 7 hours at 530℃. Specimens were cooled in the nitriding champer under N_2. The microstruture was studied using SEM and O.M. analysis. Microvickers hardness was measured using a load of 0.1Kgf and average of five reading was taken. A diffractometer using CuKα radiation was used for XRD analysis. Ball-on-disk type wear test was performed at 500℃. Applied load was 0.3Kgf and the sliding distance was 1km with a sliding velocity of 0.2m/sec. Al_2O_3 ball with 8 mm in diameter was used as counterpart. Oxidation test was performed at 700℃ for 40hours and the weight change was measured. the oxidized layer was examined using SEM.

RESULTS AND DISCUSSION

Microstructure and XRD analysis

Fig. 1a shows the cross-sectional microstructure of calorized H13 steel. Al diffusion layer of approximately 250μm was created after calorizing for 3hours at 1000℃. The

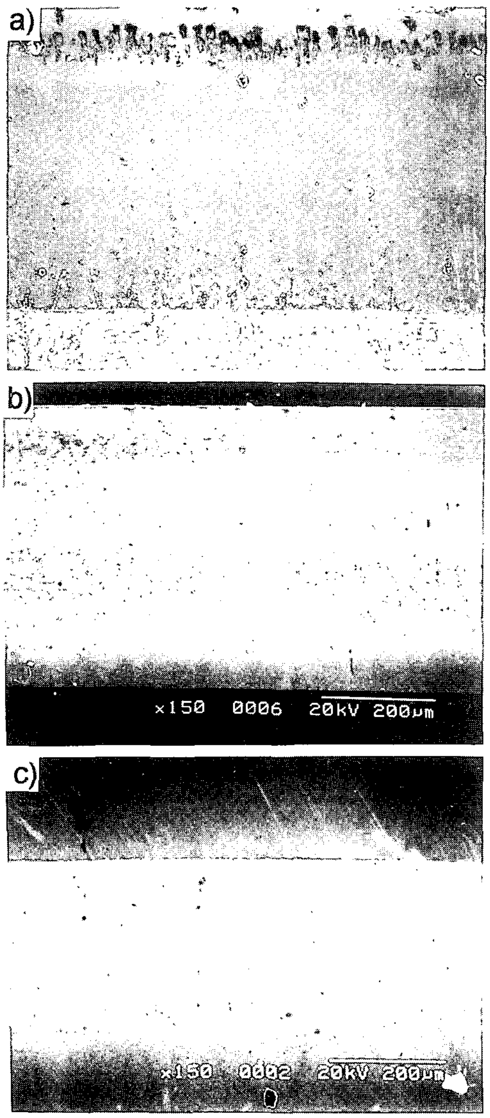

Fig. 1 Cross-sectional SEM micrographs AISI H13 steel, a) after calorizing, b) after plasma nitriding and c) after calorizing & plasma nitriding

results of the XRD analysis on the calorized surface of the H13 steel specimen as shown in Fig. 2a indicated that only bcc FeAl phase was present on the calorized surface and noother types of Al rich compounds, such as $FeAl_3$, Fe_2Al_5, etc. was detected. Generally

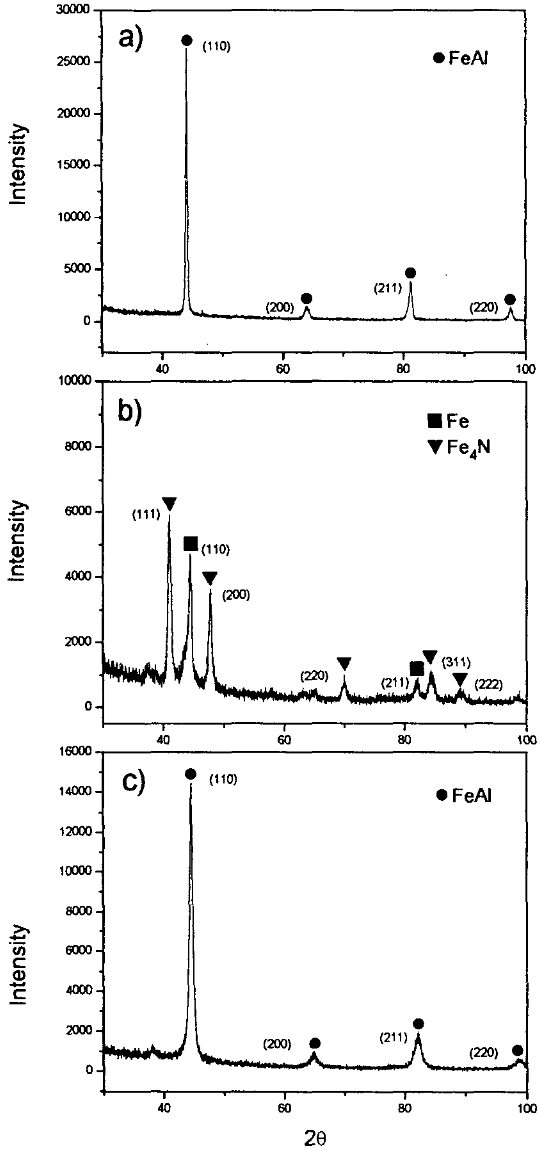

Fig. 2 XRD patterns of AISI H13 steel, a) after calorizing, b) after plasma nitriding, and c) after calorizing & plasma nitriding

the type of the Al compounds formed in the calorized steels were reported to be dependent upon the Al activity in the pack[9]. Previous work by Bahadur and Mohanty[9] on coating on mild steel using a low Al activity pack (35wt% Al powder) showed that only FeAl compound formed in the diffused layer. Our results from calorizing using a low Al activity pack (30 wt% FeAl powder) was consistent with the results from the previous work. The cross-sectional microstructure of the nitrided H13 steel was shown Fig. 1b. The thickness of the nitrided layer was of the order of 430μm. The phase formed on the nitrided surface of the H13 steel was identified to be $\gamma'(Fe_4N)$ by XRD, as shown in Fig. 2b. Duplex treatment by calorizing and plasma nitriding was done and cross-sectional microstructure is shown in Fig. 1c. The surface of the duplex-treated H13 steel was analyzed using XRD and the results are shown in Fig. 2c. It showed that the surface consisted of only FeAl compound and no nitride was present. It was noted that the diffraction peaks in Fig. 2c became broad and the 2θ values of the peaks were shifted in a small degree and peaks intensity were decreased. This could be attributed to the strain produced by interstitial nitrogen solutionized into the FeAl phase or by fine nitrides formed in the duplex treated layer[10].

Microhardness

The microhardness profiles of the H13 steel after plasma nitriding and duplex treatment are shown in Fig. 3. H13 steel showed a high surface hardness of about 1000Hv after plasma nitriding and nitriding depth was of approximately 430μm. Further improvement of the surface hardness was made by the duplex treatments and the surface hardness was measured to be about 1450Hv as shown in Fig. 3. The duplex treated H13 steel also showed a higher hardness profile up to about 160μm below the surface than the nitrided steel. This is because that FeAl compound with a large amount of interstitial nitrogen was formed on the duplex-treated surfaces without forming nitride.

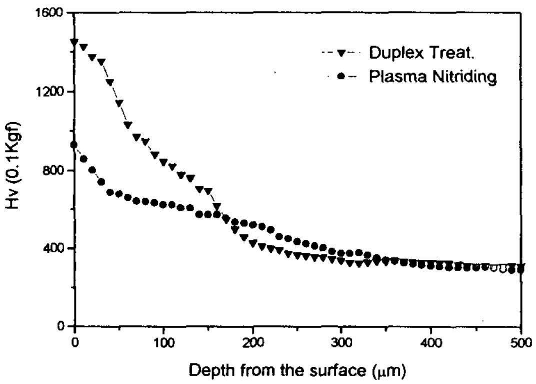

Fig. 3 Cross-sectional microvickers hardness profilesafter different treatments of AISI H13 steel

Wear test at 500℃

The results from the ball-on-disk type wear test at 500℃ are summarized in Fig. 4. The wear volume in Fig. 4a was calculated from the wear scar using a profilermeter. The calorized H13 steel showed a superior wear resistance to the untreated substrate at 500℃. This is because the Al oxide formed at the calorized surface prevented the specimen from the oxidation as previously reported in[9]. Further improvement was observed in the nitrided and duplex-treated specimens. Although a similar degree of wear resistance in terms of wear volume as shown in Fig. 4a was observed, the weight gain(see Fig. 4b) of the nitided specimen was considerably larger than that of the duplex-treated specimen due to surface oxidation, suggesting better oxidation resistance in th duplex-treated specimen.

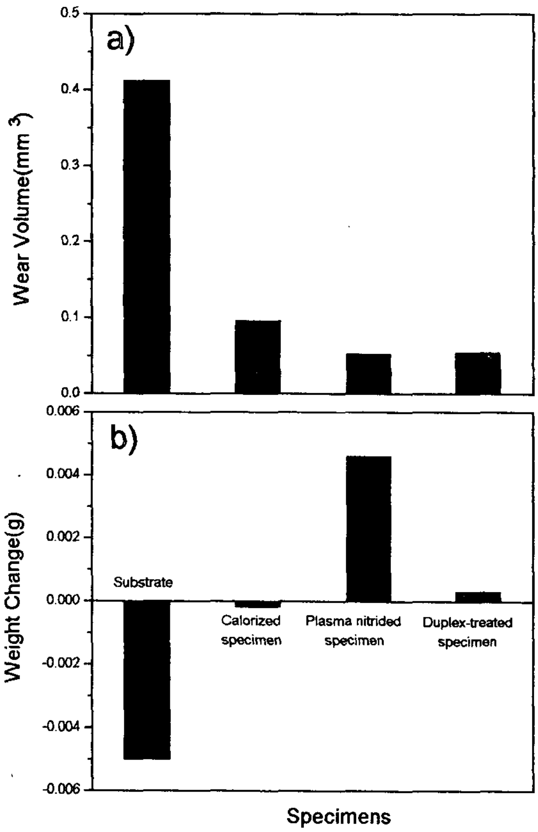

Fig. 4 Results from the wear test of AISI H13 steel at 500℃, a) wear volume, b) weight change

Oxidation Test

The weight gain and the microstructure of the oxidized layer after oxidation test at 700 ℃ are shown in Fig.5. The present results agree with the report[9] that oxidation resistance was decreased due to the dissociation of the nitrogen from the plasma nitrided specimen when exposed up 600℃. But duplex-treated specimen showed superior oxidation

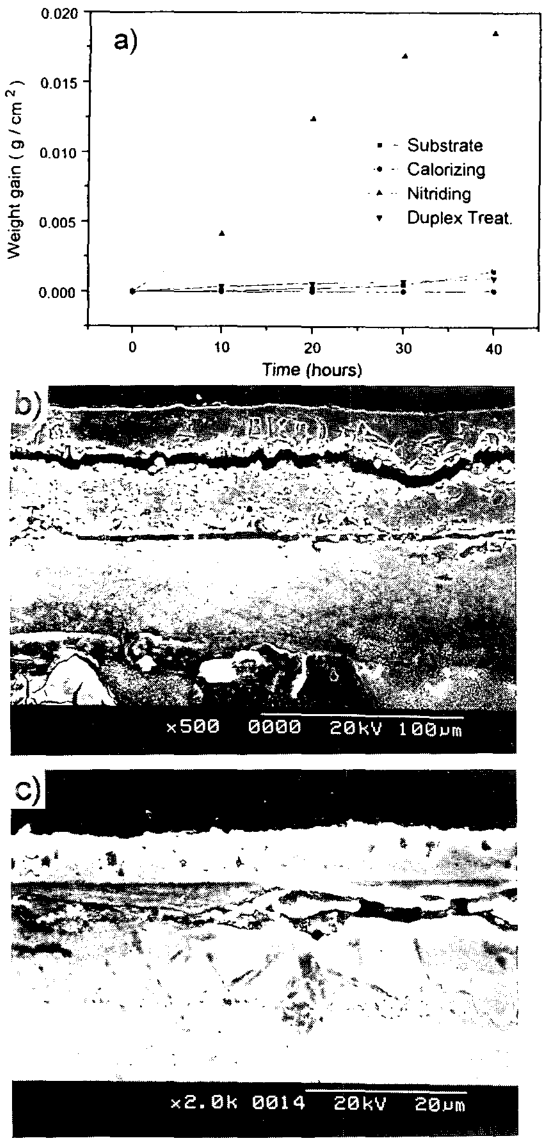

Fig. 5 Results from the oxidation test of AISI H13 steel at 700℃ for 40hours, a) weight gain after oxidation test, b) cross-sectional SEM micrograph of plasma nitrided specimen after oxidation test, c) cross-sectional SEM micrograph of duplex-treated specimen after oxidation test

resistance to nitrided specimen. This is because the Al oxide formed at calorized surface prevented the specimen from further oxidation[10].

CONCLUSIONS

The present study shows the effect of duplex treatment, calorizing and plasma nitriding, in wear and oxidation test at high temperature. The main conclusions are as follows.

1) XRD result showed FeAl compound with a large amount of interstitial nitrogen was formed on the duplex-treated surface, but no nitride was observed.

2) Surface hardness of the H13 steel was increased above 1450Hv(0.1Kgf) by duplex treatment.

3) Wear properties of duplex treated H13 steel at high temperature was better than those by plasma nitriding treatment when both wear volume and weight change due to surface oxidation are considered.

4) Oxidation resistance of duplex-treated specimen was superior to that of plasma nitrided specimen.

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