Wear characteristics of austenitic steel and martensitic steel at high temperature

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Abstract
Wear under high temperature is one of the mechanisms of die failure. Therefore, wear resistance at high temperature is an important parameter for selecting die materials. In this work, the wear resistance of SDHA austenitic steel (6Mn14Cr3Mo2Si1V2 steel) and 4Cr5Mo2V martensitic steel at 400 °C to 700 °C was investigated using a friction and wear tester. The wear behaviour and oxide type were investigated using a scanning electron microscope (SEM) and by X-ray diffraction (XRD), respectively. The results show that the oxides on the worn surface at the test temperatures are Fe2O3 and Fe3O4. With increasing test temperature, from 400 °C to 700 °C, the wear volume of the two steels initially decreases and then increases. Between these two temperatures, the wear volume of SDHA austenitic steel increased from 29.7 mm3 to 134.7 mm3, a 267.4% increase. Hence, SDHA austenitic steel has better wear resistance than 4Cr5Mo2V martensitic steel. This is attributed to excellent hardness stability at high temperature. The coarse M7C3 carbides in 4Cr5Mo2V martensitic steel cause peeling and delamination of the oxide layer, reducing wear resistance at 700 °C.

1. Introduction

Hot-working die steels are widely used in the hot-working dies of parts prepared by hot forging and hot extrusion [1, 2]. Their service temperature during hot forging and hot extrusion can exceed 500 °C–600 °C (local temperature can reach almost 700 °C) [3, 4]. Furthermore, the dies suffer dry wear and heating impact when directly contacting hot metals in an un lubricated environment. Therefore, dry wear is one of the failure mechanisms in the hot-working die industry [5, 6]. Based on previous studies, abrasive wear, adhesive wear and oxidative wear are the reasons for degradation in hot-working dies [7, 8]. According to statistics, wear failure accounts for 70% of die failures in the hot forging industry [9]. The wear failure affects die life and results in huge financial losses.

According to the characteristics of dry wear, it can be divided into adhesive wear, abrasive wear, fatigue wear and corrosion wear, etc. [10]. However, when dry wear occurs at high temperature, the oxides formed by metal oxidation have an important influence on the wear resistance. FeO, Fe3O4 and Fe2O3 will form at high temperatures during wear. An acceptable view is that Fe3O4 with spinel structure can reduce the wear rate compared with α-Fe2O3 [11]. Regarding the role of FeO in wear, some researchers have different opinions. Cornelius found that FeO protected the matrix and reduced the wear rate, but Tsuji and Cornelius [12, 13] proposed that the wear rate increased significantly when the oxides are mainly composed of FeO at 500 °C–600 °C. Some studies show that wear resistance is also related to the matrix microstructure. Wang [14] suggested that the wear resistance of microstructure in 1080 steel increased in order: martensite, bainite and lamellar pearlite. The plastic deformation of subsurface contributed to the formation of tribological layer, the wear behaviour was affected by oxides and subsurface deformation [15]. It is generally accepted that wear resistance of the different microstructures depends on thermal stability, toughness and plastic deformation resistance, especially the microstructure change and energy consumption during sliding [16–18]. By comparing the wear characteristics of austenitic stainless steel and austenitic-ferritic stainless steel, Straffelini [15] found that the low ductility can...
The samples cool to room temperature in both steps. Therefore, the test temperatures were set to 400 °C for 30 min and 10 N respectively. Hot-working dies usually work at 400 °C. However, due to the low wear resistance at high temperature, 4Cr5Mo2V martensitic steel is not suitable for use under hostile service conditions. Therefore, a new 6Mn14Cr3Mo2Si1V2 austenitic steel, known as SDHA austenitic steel, has been self-developed for execrable wear environments.

In this work, the wear behaviour of SDHA austenitic steel and 4Cr5Mo2V martensitic steel was investigated at high temperature. The purpose is to clarify the difference of wear resistance between the two steels, and it provides the theoretical support for selecting hot-working die steels. This work attempts to explain the relationship between the wear resistance, oxide and microstructure, so as to determine the important factors affecting wear resistance.

### 2. Experiment

#### 2.1. Material

According to the ASTM G133–2016 standard, SDHA austenitic steel and 4Cr5Mo2V martensitic steel were used to test wear resistance at high temperature. The chemical composition of the two steels are shown in table 1. The heat treatment process of the two steels is shown in table 2. It is worth emphasizing that 4Cr5Mo2V martensitic steel experiences tempering treatment twice. The hardness of the two steels is about 450 HV after heat treatment.

#### 2.2. Methods

The wear resistance was analysed using a wear tester with a ball-on-disk arrangement (UMT-3, America). During dry sliding, the samples were in contact with the SiC ceramic ball in an atmosphere environment. The wear tester and working principle are shown in figure 2. The sliding speed, sliding frequency, test time and load were 50 mm s⁻¹, 5 Hz, 60 min and 10 N respectively. Hot-working dies usually work at 400 °C–700 °C [31, 32]. Therefore, the test temperatures were set to 400 °C, 500 °C, 600 °C and 700 °C. The wear volume was measured with a white-light interferometer (GT-K, America). The microhardness at high temperature was measured with

| Table 1. Chemical composition of the two steels (wt%). |
|---|---|---|---|---|---|---|---|
| Steel grade | C | Si | Mn | Cr | Mo | V | Fe |
| SDHA | 0.60–0.65 | 0.80–0.90 | 10.00–11.00 | 3.00–3.10 | 1.60–1.80 | 1.80–1.90 | Bal. |
| 4Cr5Mo2V | 0.38–0.42 | 0.20–0.30 | 0.40–0.50 | 4.90–5.10 | 2.30–2.50 | 0.40–0.60 | Bal. |

| Table 2. Heat treatment methods of the two steels. |
|---|---|---|---|---|---|---|
| Steel grade | First step | Second step |
| SDHA | Solution temperature | Solution time | Cooling medium | Aging temperature | Aging time | Cooling medium |
| | 1170 °C | 45 min | oil | 720 °C | 3h | air |
| 4Cr5Mo2V | Austenitizing | Austenitizing time | Cooling medium | Tempering temperature | Tempering time | Cooling medium |
| | 1030 °C | 30 min | oil | 620 °C | 2h | air |

The samples cool to room temperature in both steps.
a high temperature hardness tester (Zone-De, China) with a load of 1 Kg and a dwell time of 5 s, the test temperature was selected as 400 °C, 500 °C, 600 °C and 700 °C. The types of oxide on the worn surfaces were analyzed by X-ray diffraction (XRD, D/MAX-2500, Japan). The microstructures and worn morphologies were characterized using a scanning electron microscope (SEM, Supra-40, Germany) and transmission electron microscope (TEM, JEM-2010F, Japan). The observation area of worn cross-section is in the center of the worn surface, as shown in figure 3.

3. Results

3.1. Wear volume
Figure 4 shows the relationship between temperature and wear volume at 400 °C–700 °C, the wear volume curves can be divided into two stages. At test temperatures from 400 °C to 500 °C, the wear volume of SDHA austenitic steel and 4Cr5Mo2V martensitic steel gradually decreased, and reached lowest values of 29.7 mm³ and 34.7 mm³ at 500 °C, respectively. During the second phase, 500 °C–700 °C, the wear volume of SDHA austenitic steel and 4Cr5Mo2V martensitic steel increased, and the wear volume reached highest values of 81.2 mm³ and 134.7 mm³ at 700 °C. The growth of wear volume, from 500 °C to 700 °C, of SDHA austenitic steel and 4Cr5Mo2V martensitic steel is 173.4% and 267.4%, respectively. The wear volume of SDHA austenitic steel was lower than that of 4Cr5Mo2V martensitic steel at experimental temperatures. Hence, SDHA austenitic steel has better wear resistance.

3.2. Worn surface morphology
Figures 5(a)–(d) presents the surface morphologies of SDHA austenitic steel that underwent wear tests between 400 °C and 700 °C. At 400 °C, there were a large number of oxide particles on the worn surface, as shown in figure 5(a). These exfoliated oxides accumulated locally during sliding to result in adhesive wear. When the temperature rose to 500 °C, a dense oxide layer with a few pits was formed during continuous wear, figure 5(b). The dense oxide layer could effectively reduce the direct contact between the matrix and the SiC ceramic ball. It
can be seen from figure 5(c) that the oxide layer seriously peeled off with the aggravation of oxidation. The exfoliated oxides were continuously squeezed to form a smooth oxide layer. Stott [33] proposed that this smooth oxide layer, which formed during wear, acts as a barrier layer to temporarily protect the matrix. Figure 5(d) is the surface morphology at 700 °C, the worn surface consists of exfoliated oxide particles and a loose oxide layer. With the increase in oxidation, the oxide layer peeled off under pressure when it grew to the critical thickness. Figures 5(e)–(h) shows the worn surface morphologies of 4Cr5Mo2V martensitic steel at 400 °C–700 °C. There were a few furrows parallel to the sliding direction on the worn surface, as shown in figure 5(e). The oxide particles are scattered on the worn surface. This phenomenon was similar to the surface morphology of SDHA austenitic steel at 400 °C. As the test temperature increased to 500 °C, the worn surface was covered by a dense oxide layer, the oxide particles changed to the oxide debris, as shown in figure 5(f). In addition, some oxides were pushed out of the oxide layer under shear stress condition, known as extruded oxides. The grooves caused by the sliding of oxide particles and the plastic deformation can be observed in figure 5(g). The oxides at the edges of grooves peeled off from the oxide layer. Due to the brittleness of the oxide layer, the reciprocating sliding on the worn surface resulted in the generation of fatigue cracks perpendicular to the sliding direction, which was a precursor to spalling. As the extent of oxidation increased, the thick oxide layer with a poor binding force could not resist the shear stress to form the loose regions, as shown in figure 5(h). In particular, cracks parallel to the sliding direction were generated due to the fatigue mechanism.

It can be seen from the wear morphologies of the two steels at 500 °C, that the formation and exfoliation of oxide layers reach an equilibrium state. In this case, the bearing capacity of the matrix is enough to support the oxide layers. Therefore, the existence of oxide layers protects the worn surface and reduces the wear volume. However, when the temperature rises to 700 °C, the bearing capacity of 4Cr5Mo2V martensitic steel declines. The exfoliation and porosity of the friction oxide layer is the dominating reason for the increase in wear volume.
3.3. Worn cross-sectional morphology

The worn cross-sectional morphologies of SDHA austenitic steel at 400 °C–700 °C are shown in figures 6(a)–(d). The existence of oxides could be found from the cross-sectional morphologies at each test temperature. Figure 6(a) illustrates that few oxides were observed due to the poor oxidation rate at 400 °C. Figure 6(b) shows cracks and plastic deformation at the experimental temperature of 500 °C. The cracks formed in two locations, the oxide layer and the matrix. The reciprocating sliding causes stress accumulation to form plastic deformation,
and when the stress exceeds the yield strength, the cracks will initiate and expand in the matrix. However, cracks in the oxide layer are attributed to the internal stress of the oxide layer. When the test temperature reached 600 °C, the oxide layer grew unevenly and the thickness increased to 5–8 μm, as shown in figure 6(c). The severe plastic deformation accelerated the diffusion of oxygen atoms, especially cracks, where oxidation reactions took place preferentially. The matrix near the oxide layer was subjected to high pressure to form the wear affected zone. The appearance of the wear affected zone meant a decrease in the wear resistance of the material. Figure 6(d) shows that the worn cross-sectional morphology contains the wear affected zone and oxides at
700 °C. The broken matrix caused by high pressure provided a poor supporting force for the oxide layer, resulting in the peeling of oxides.

Figures 6(e)–(h) show the worn cross-sectional morphologies of 4Cr5Mo2V martensitic steel at 400 °C–700 °C. A few oxides were found from the worn cross-section at 400 °C, this was similar to the result of SDHA austenitic steel, as shown in figure 6(e). As the temperature increased to 500 °C, an oxide layer with a thickness of 1–3 μm was observed, figure 6(f). Compared with the worn cross-section of SDHA austenitic steel at 500 °C, there was no obvious plastic deformation of the matrix under pressure. From figure 6(g), the maximum thickness of the oxide layer increased to 12 μm at 600 °C. Due to the existence of cracks, the binding force of the oxide layer was strictly limited. In this case, the oxide layer peeled off easily with the expansion of cracks. In addition, the diffusion rate of alloying elements increased with the increase in temperature. At the same time, accompanied by the stress inducement, a large number of carbides precipitated from the matrix. Some precipitated carbide particles grew rapidly under thermal and stress conditions, especially near the oxide layer [24, 34]. When the experimental temperature reached 700 °C, the spalling zone and the delamination of matrix and oxide layer were observed from the worn cross-section, as shown in figure 6(h). Li [34] proposed that the carbides precipitated along the interface between the matrix and the oxide layer to cause stress concentration, resulting in crack formation. These cracks acted as fast channels to promote the contact between oxygen and matrix, and then newly formed oxides grew along the cracks, leading to delamination [35]. Moreover, it is worth emphasizing that the oxide layer could not obtain sufficient support due to the matrix softening. Therefore, a large number of oxide particles peeled off from the oxide layer during reciprocating friction.

3.4. Friction coefficient
An increasingly accepted view is that wear is divided into the running-in stage, the stable stage, and the severe stage [36]. At the beginning of wear, the friction coefficient rises rapidly due to the contact between the friction pair and the surface bumps. As wear progresses, the contact area gradually increases with the disappearance of contacting bumps, creating a stable condition for wear. A stable wear stage is marked by a stable friction coefficient. As the wear time increases, the stable wear stage changes to the severe wear stage with the appearance of fatigue cracks on the wear surface. The friction coefficients of the two steels at 400 °C–700 °C are presented in figure 7. Under a certain load at 400 °C, the dispersed oxide particles slid on the worn surface to result in the wide fluctuation of friction coefficient in the two steels. With the increase of experimental temperature, a completely covered oxide layer was formed to stabilize the friction coefficient. For SDHA austenitic steel at 700 °C, the friction coefficient continued to rise after the running-in stage due to the spalling of matrix and oxide particles. Similarly, after long-term wear, oxide layer damage significantly increased the friction coefficient at 700 °C.

3.5. Phase calibration of the oxides on the worn surface
During wear at high temperature, the composition and structure of the oxide layer have an important influence on wear behaviour. The oxides on the worn surface were analyzed by XRD, the results are represented in figure 8. From the results, at 400 °C–700 °C, the oxide types on the worn surface of the two steels were identified as Fe2O3 and Fe3O4. The diffraction peak intensity of oxides increased with increasing temperature, indicating more oxides were formed at higher temperature. During reciprocating friction, the local high energy state and plastic deformation on the worn surface can greatly promote the occurrence of the oxidation reaction [37]. Some studies indicate that the oxidation reaction obeys the rule: Fe → Fe3O4 → Fe2O3 [38].
3.6. Hardness
The hardness of the material plays a significant role in the wear resistance, especially at high temperature. Archard [39] proposed an empirical formula for the dry sliding wear of metals, in which the wear volume and hardness are inversely proportional. Therefore, the hardness of the two steels at 400 °C–700 °C was measured by a high temperature hardness tester, the results are shown in figure 9. The hardness of the two steels decreased with the increase of temperature, but presented different hardness reduction rates. Compared with 4Cr5Mo2V martensitic steel, SDHA austenitic steel showed better hardness stability at the test temperature. Due to the relatively high hardness at 400 °C, the wear resistance was related to the number of oxide particles and the thickness of the oxide layer. However, the hardness of 4Cr5Mo2V martensitic steel rapidly reduced when the test temperature rose to 700 °C. The matrix, with a relatively low hardness, could not provide effective support for the oxide layer. Thus, there was a direct correlation between the oxide layer damage and wear resistance.

4. Discussion

4.1. Effect of microstructure on wear resistance
Quinn [40, 41] proposed an oxidation wear theory that the oxide layer on the worn surface could grow until it reached a critical thickness, before peeling off. In this case, the wear was related to the oxide layer rather than to the matrix. However, in this work, the wear behaviour at high temperature seemingly disagrees with this oxidation wear theory. During high temperature wear, the surface bumps oxidize quickly, due to the high
friction contact temperature. The plastic deformation of the matrix and the internal stress of the oxide layer cause the peeling of oxides. These exfoliated oxide particles slide on the worn surface and increase the wear rate.

When the test temperature increases, the strength of the steel decreases. Repeated friction leads to plastic deformation accumulation and stress concentration at the interface between the matrix and the second phase. When the stress exceeds the critical strength, it will result in crack initiation and propagation in the matrix. These cracks cause the delamination of the oxide layer at high oxidation rate, which is a bad for wear resistance. Since the matrix softens at high temperature and bears a large shear force, the oxide layer may peel off before reaching the critical thickness. Severe oxidative wear will appear under this circumstance. Thus, an excellent supporting force for the oxide layer is vital. In other words, matrix hardness plays an important role in reducing wear.

The change of hardness depends upon the microstructure transformation, especially carbide evolution. The effect of carbides on wear resistance has been widely studied. Carbides with high hardness and stability are considered beneficial to wear resistance [42, 43]. In order to clarify the influence of carbides on wear behaviour in the two steels at high temperature, samples of SDHA austenitic steel and 4Cr5Mo2V martensitic steel were heat treated to 430 HV, 410 HV and 410 HV, 250 HV, corresponding to the hardness of the wear samples at 400 °C and 700 °C. The phase calibration of SDHA austenitic steels with different hardness was analyzed in figure 10. According to the phase calibration results, VC carbides were found only in the samples with a hardness of about 410 HV and 430 HV. Some researchers have suggested that VC carbide with high hardness (about 2800 HV) can greatly improve the wear resistance [44]. Besides, the VC carbide with a stable structure was difficult to transform at high temperature [45]. Thus, the stable VC carbides give SDHA austenitic steel excellent hardness stability and wear resistance at high temperature.

Figure 11 indicates that the carbide with quadrangular structure in 4Cr5Mo2V martensitic steel was calibrated as the M2C-type carbide by SAD analysis, the size is approximately 40 nm. In addition, this carbide has the close-packed hexagonal structure and is precipitated at the intermittent subgrain boundaries produced during the recovery process.

Figure 12 shows the microstructure of 4Cr5Mo2V martensitic steel with a hardness of 250 HV, the precipitated carbide with a width of about 75 nm was calibrated as M7C3-type carbide. Furthermore, the size of M7C3-type carbide is significantly larger compared with M2C-type carbide. By comparing the hardness of the two carbides, the hardness of the M2C-type carbide (about 2230 HV) is greater than that of M7C3-type carbide (about 1500 HV) [46, 47]. Therefore, a transition of carbides from M2C to M7C3 in 4Cr5Mo2V martensitic steel reduced the wear resistance. Shi [24] proposed that the coarse carbides caused the delamination of the oxide layer, which damaged the wear resistance at high temperature. A similar phenomenon was also found in this work.

4.2. Wear mechanism

Based on the wear behaviour at different temperatures, a schematic diagram of wear characteristics of SDHA austenitic steel is suggested, figure 13. For SDHA austenitic steel, the oxide layer was first formed on the worn surfaces due to frictional heat and high temperature, especially at the contacting bumps, presenting a
discontinuous and uncompacted state. In this situation, the matrix possesses enough hardness (about 433HV). Therefore, the discontinous and uncompacted oxide layer plays a partial protective role. Meanwhile, the oxide particles are dispersed on the worn surface. Therefore, the wear mechanism is defined as abrasive and mild oxidation wear at the first step. Then, with the increase in oxidation, the worn surface is covered by an oxide layer and the plastic deformation forms in the matrix near the oxide layer. This is the second step of wear. However, the matrix provides an excellent supporting force to the oxide layer due to the stable VC carbides, so that the oxide layer can act as a protective layer to resist wear very well. In this case, mild oxidation wear prevails. Besides, some cracks appear in the oxide layer due to internal stress. As the wear affected zone appears at the third step, the wear mechanism shifts to severe oxidation wear. The oxidation on the worn surface further intensifies with the appearance of a large amount of oxides. Matrix near the oxide layer presents fragmentation tendency due to its thermal brittleness. In this case, the protective effect of the oxide layer becomes weakened, resulting in severe oxidation wear. Therefore, this is a brittleness-induced transition.

The schematic diagram of wear characteristics of 4Cr5Mo2V martensitic steel is shown in figure 14. The oxidation wear transformation at 400 °C–700 °C can be divided into three steps:

At the first step, the wear mechanism is judged as abrasive and mild oxidation wear. The typical characteristics of mild oxidation wear is that the oxides appear on the worn surface. The oxides are peeled off during sliding, and these oxide particles, regarded as abrasive particles, result in abrasive wear damage under load.

With the formation of a protective oxide layer covering the worn surface, the wear enters the second stage. The amount of oxide increases with improving oxidation rate on the worn surface. The more continuous and
compacted oxide layer exists as a protective substance to resist wear. The stable existence of the protective oxide layer depends on the strength and toughness of the matrix. According to the worn cross-sectional morphology and hardness at 500 °C, the matrix without obvious plastic deformation and cracks seems to provide strong strength to support oxide layer.

The appearance of oxide layer delamination marks third wear stage; the wear mechanism changes to severe oxidation wear. With the appearance of coarse M₇C₃-type carbides, the repeated friction results in stress concentration forming cracks in the matrix. The cracks in the matrix promote contact between oxygen atoms and matrix to oxide layer delamination. When the matrix cannot give support for the oxide layer, the oxide layer on top severely peels off. In this case, the oxide layer totally loses its protective function, and severe wear prevails. The lack of sufficient intensity triggers this state, known as intensity-induced transition.

5. Conclusions

In this work, the wear behaviour of SDHA austenitic steel and 4Cr5Mo2V martensitic steel at 400 °C–700 °C has been investigated. The results are summarized as follows:
(1) The wear mechanism transition of the two steels is abrasive wear \+ mild oxidation wear → severe oxidation wear. The oxide types on the worn surface of the two steels are identified as Fe$_2$O$_3$ and Fe$_3$O$_4$.

(2) The wear volume at first decreases and then increases. At 500°C, the wear volumes of SDHA austenitic steel and 4Cr5Mo2V martensitic steel are lowest with values of 29.7 mm$^3$ and 34.7 mm$^3$, respectively. At 700°C, the wear volumes of SDHA austenitic steel and 4Cr5Mo2V martensitic steel reaches their maximum values, increasing by 173.4% and 267.4%, respectively, compared to at 500°C. Hence, SDHA austenitic steel has better wear resistance than 4Cr5Mo2V martensitic steel.

(3) SDHA austenitic steel possesses excellent hardness stability at high temperature, due to stable VC carbides, resulting in good wear resistance.

(4) The carbides experience a transition from M$_2$C to M$_7$C$_3$ in 4Cr5Mo2V martensitic steel, and the coarse M$_7$C$_3$ carbide results in the peeling and delamination of the oxide layer.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Disclosure statement

No potential conflict of interest was reported by the author(s).

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