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Study on wear behaviour of 4Cr4Mo2NiMnSiV die steel at high temperature

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Abstract. In this paper, with reference to the composition and properties of widely used H13 hot working die steel, and the amount of alloying elements in the steel is reasonably improved, and a new steel grade of 4Cr4Mo2NiMnSiV die steel is designed. A new process was designed and the wear properties of 4Cr4Mo2NiMnSiV die steel were studied. SEM, EDS and XRD were utilized to observe the microstructure of the worn surface, and the wear products were analyzed to discuss the wear mechanism. Conclusion illustrates the coefficient of friction of cast steel 4Cr4Mo2NiMnSiV gradually increases with increasing temperature. Wear of the 4Cr4Mo2NiMnSiV die steel is mainly caused by spalling and high-temperature oxidation. The surface oxidation products of high temperature wear samples are Fe_2O_3 and Fe_3O_4, and spalling debris caused by fatigue cracks is blocky and a small amount of long flake oxide. The oxide film generated during the high temperature wear process effectively reduces the wear of the material. Through comparison experiments, it is found that the 4Cr4Mo2NiMnSiV die steel of martensite and bainite structure has the best wear resistance, followed by martensitic alloy steel, and the wear resistance of H13 steel which is involved in comparison is lower than that of alloy steel.

1. Introduction

Compared with the traditional hot forging mold, the casting mold can significantly improve the material utilization rate, shorten the manufacturing cycle, reduce the production cost, and facilitate the forming of complex molds. Moreover, cast hot work die steel has better red hardness, fatigue resistance and low notch sensitivity than forged steel. Therefore, casting forming hot work die has remarkable economy and advancement, and gradually becomes a new hot work die manufacturing technology [1-3]. However, hot die steel high content of alloying elements has a high content of alloying elements, and it tends to form a coarse structure and macrosegregation during solidification, resulting in early brittle fracture of the mold. Refining the grain by modification, reducing the segregation of alloying elements, and matching with reasonable heat treatment can significantly reduce the brittleness of the cast hot work die steel [4].

When the hot work die is working, the high temperature blank is deformed under the action of high load, and generates strong friction and wear with the surface of the cavity. High temperature wear is the main failure mode of hot work die and one of the key factors affecting the service life of hot work die. In recent years, material researchers have conducted extensive research on the high temperature wear behavior and wear mechanism of hot work molds. According to different physical processes during wear, Burwell divides wear into

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different types such as abrasive wear, adhesive wear, fatigue wear, erosion wear, corrosion wear and fretting wear, and thus established a foundation wear scientific research [5]. Quinn, as the proponent of the theory of spalling wear and oxidative wear, pointed out in his early research that the shedding of metal will accelerate the process of wear, and the shedding of the hot surface reaction layer will slow down the wear rate [6]. The reaction layer can be produced not only in the atmosphere, but also in the reaction with oil, additives and impurities to form a protective layer with protective properties [7]. Welsh pointed out in the study that the two most basic conditions for resisting severe wear are hardening and oxidation [8]. The study also shows that Fe_3O_4 protects steel better than Fe_2O_3 during wear, which results in low wear rate of steel [9]. The above research results show that, on the one hand, the microstructure and properties of the die steel affect its high temperature wear performance; on the other hand, the formation and shedding of the wear surface oxide film has an important influence on the high temperature friction and wear behavior of the hot work die steel. However, these studies are mainly for forging hot work die steel, and the high temperature wear of cast forming hot work die steel is less studied.

In this paper, 4Cr4Mo2NiMnSiV hot work die steel is used as the object to study the high temperature wear behavior of casting die steel at different temperatures, and the high temperature wear mechanism is discussed. The research results will be helpful for the optimization of casting hot work die steel and the die life prediction.

2. Experimental procedures

The experimental steel is obtained by high temperature melting of raw materials such as pig iron, electrolytic copper, low carbon ferrochrome, pure nickel, ferromolybdenum, vanadium iron ore, melting molten steel in GW medium frequency induction furnace. Table 1 shows the chemical composition of H13 steel and 4Cr4Mo2NiMnSiV steel. The heat treatment process of 4Cr4Mo2NiMnSiV steel is shown in Fig. 1. Among them, 4Cr4Mo2NiMnSiV steel, obtained by the process of (b), is the martensite structure, which is written as 4Cr4Mo2NiMnSiV (M), and the (c) is obtained by the martensite and bainite multiphase structure, which is written as 4Cr4Mo2NiMnSiV (M+B).

The abrasion test was carried out on a HT-1000 high temperature friction and wear tester. The test used a ball-and-disk type friction pair. The top of the grinding rod was a metal ball made of GCr15 bearing steel, and the grinding disc was a ϕ30 mm×10 mm disc made of the tested material. The experimental conditions are dry sliding friction and wear conditions. The experimental load was 0.5 Kg. The grinding wheel has a rotation frequency of 10 Hz. The friction time is 30 min. The total taxi distance d is approximately 3.4×102 m. The experimental temperature was set to room temperature, 200℃, 400℃, and 600℃. During the experiment, the electric resistance furnace is used for automatic heating, and the temperature is controlled by thermocouple and temperature control instrument. The friction coefficient, wear amount and wear rate are directly measured by the computer. After the abrasion test, the phase composition of the material was analyzed by X Perth pro type multifunctional X-ray diffractometer (XRD) (Cu target, scanning range 2θ was set to 10-110°, scanning speed was 3°/min). The FEI QUANTA 200F scanning electron microscope was used to observe the surface morphology after wear, and the fracture mechanism, oxidation and wear characteristics of the material were analyzed and judged. The energy dispersive X-ray spectrometer (EDAX) attached to the scanning electron microscope was used to analyze the energy spectrum of the material.

Table 1 Chemical compositions of H13 and 4Cr4Mo2NiMnSiV steel (mass fraction/%).

	C	Cr	Mo	V	Si	Mn	Ni	P, S
H13	0.38	5.15	1.45	0.90	1.04	0.43	/	<0.03
4Cr4Mo2NiMnSiV	0.40	4.00	2.30	0.60	0.40	0.60	1.00	<0.03

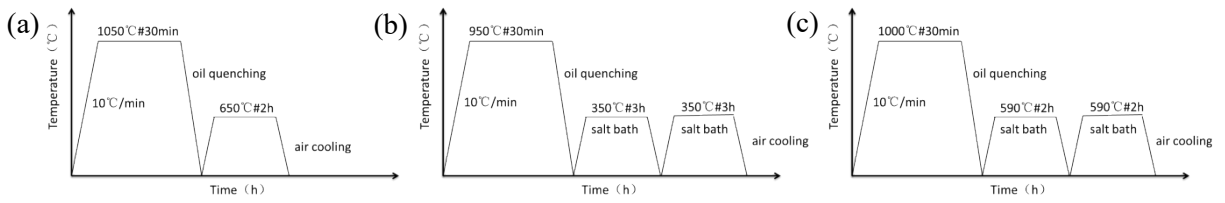


Fig.1. Heat treatment process flow chart: (a) H13 steel; (b) 4Cr4Mo2NiMnSiV (M) steel; (c) 4Cr4Mo2NiMnSiV (M+B) steel

3. Results and Discussions

3.1. *Coefficient of friction*

Fig. 2 shows the coefficient of friction of 4Cr4Mo2NiMnSiV (M) steel with time at different temperatures. The results show that the friction of each sample has no obvious running-off period at different temperatures, and the friction coefficient reaches a stable value in a short time. At room temperature, the coefficient of friction is the lowest, but the coefficient of friction at room temperature fluctuates relatively more time. Studies [7] have shown that when the wear is severely worn, the contact between the two surfaces may cause plastic transfer from one surface to the other, resulting in wear debris. Plastic flow in the transfer phase is an important factor in the large fluctuation of the friction coefficient. As the temperature increases, the coefficient of friction also increase due to the coefficient of friction of the material is related to the state of the contact surface. When the friction surface of contact has a large rigidity, and the lower roughness has no direct interaction with the grinding metal, the friction coefficient of the material is small. On the contrary, the friction coefficient of the material is relatively high, and the temperature has a great influence on these factors. However, as the temperature increases, the fluctuation of the friction coefficient decreases. This is because in the process of high temperature wear, as the ambient temperature increases, the material will temper soften, resulting in an increase in the contact area of the friction surface. As the temperature increases, the thickness of the surface oxide film gradually increases. The effect of the oxide film on the friction is reflected in the following two aspects. On the one hand, the oxide film can reduce the frictional force when the surface microprotrusions are in contact. On the other hand, the oxide is low in strength and easily deformed to reduce friction [10]. And the temperature rise increases the degree of oxidation of the surface of the 4Cr4Mo2NiMnSiV (M) steel, and the appearance of the oxide makes the friction surface rough and increases the friction coefficient.

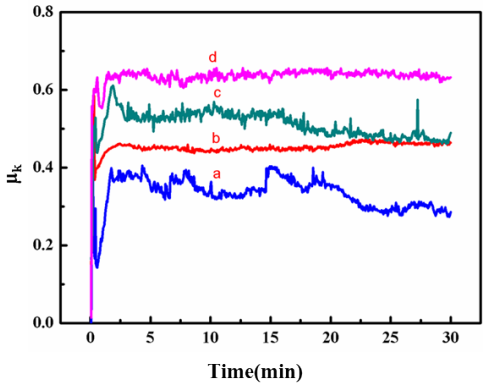


Fig. 2. 4Cr4Mo2NiMnSiV (M) friction coefficient at different ambient temperatures (μ_k: friction coefficient): (a) Room temperature; (b) 200℃; (c) 400℃, (d) 600℃

Fig. 3 shows the relationship between the average friction coefficient and the wear rate of 4Cr4Mo2NiMnSiV (M) steel. The wear rate of 4Cr4Mo2NiMnSiV (M) steel increased when the temperature was raised to 200 °C, and the wear resistance of the material decreased. When the temperature rises above 400 °C, the wear rate of 4Cr4Mo2NiMnSiV (M) steel decreases again. In the process of high temperature wear, due to the change of ambient temperature and the generation of heat in the friction engineering, an oxide layer appears on the worn surface, and the appearance of the oxide layer and covering the surface of the substrate will protect the material, which can make the wear rate reduce. However, most of the oxides are brittle, and peeling occurs under the action of friction, and the wear rate is thus increased. The rate of exfoliation of the oxide generally depends on the amount of adhesion between the oxide and the substrate and the support of the substrate to the oxide. The wear rate of 4Cr4Mo2NiMnSiV (M) steel increases from room temperature to 200 °C because the generated brittle oxide is peeled off under the action of friction. When the temperature is raised to 200 °C, the oxide formed has a strong adhesion to the substrate, which can effectively protect the substrate, and thus the wear rate is lowered.

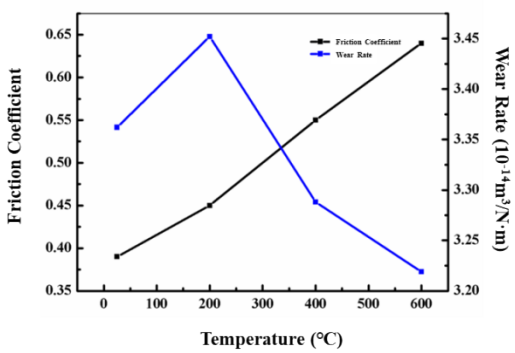


Fig. 3. Wear rate and average friction coefficient of 4Cr4Mo2NiMnSiV (M) steel at different temperatures

3.2. Wear surface

Fig. 4 shows the wear surface morphology of H13 steel, 4Cr4Mo2NiMnSiV (M) steel and 4Cr4Mo2NiMnSiV (M+B) steel at 600 °C. The wear rate measured by the experiment are 4.012, 3.589 and 3.216 (10^{-14} m^3/N·m). It can be seen from the SEM pictures in Fig. 4(a) and (d) that there are deep pits in the peeling zone of H13 steel, and the surface wear and peeling of H13 steel under the same experimental conditions is the most serious. The wear surface is oxidized to a deeper extent, with blocky and granular wear debris, fine scratches locally, less oxide around the scratches, and obvious cracks at the edges, which are characterized by adhesive wear and oxidative wear. It can also be found in Fig.4 (b) and (e) that although the surface of 4Cr4Mo2NiMnSiV (M) steel is not severely peeled off, there are obvious furrows and granular wear debris along the rubbing direction, wear depth and peeling pit. The area is large, showing plow wear and abrasive wear. Figure 4(e) also shows that the depth of the spalling zone is also deeper than that of 4Cr4Mo2NiMnSiV (M+B) steel, which is also reflected in the difference in wear rate of the three samples. From Fig. 4(c), 4Cr4Mo2NiMnSiV (M+B) steel is distributed with intermittent strip-shaped wear scars, and block-shaped abrasive chips are distributed on both sides, and a large number of cracks from the outside to the inside are generated on the surface of the wear scar. As the cracks expand, they form new wear debris. And there are a large number of fine wear oxide particles in the peeling zone of the three samples.

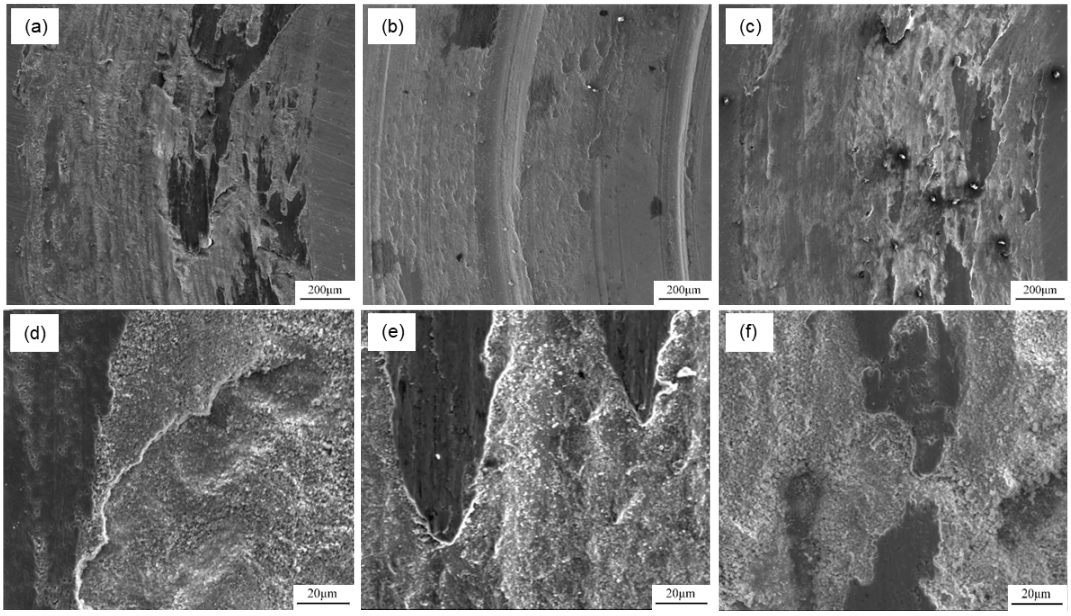


Fig. 4. High Temperature Wear Morphology of H13 and 4Cr4Mo2NiMnSiV steel at 600 °C (SEM): (a) (b) H13 steel; (c) (d) 4Cr4Mo2NiMnSiV (M) steel; (e) (f) 4Cr4Mo2NiMnSiV (M+B) steel

The morphology and composition of the wear debris of H13 steel and cast 4Cr4Mo2NiMnSiV specimens at 600℃ were observed by SEM-EDS (Fig.5). The results showed that the shape of the abrasive chips was blocky (containing a small amount of long flakes) and granular, and the average oxygen content in the wear debris was about 34.74%. Combining the analysis of the wear surface (Fig.4) and the cross section (Fig.7), the high temperature wear process of the cast 4Cr4Mo2NiMnSiV steel is known. As the friction progresses, the surface of the sample oxidizes to form an oxide layer. Due to frictional heat generation, the wear scar position is oxidized faster than both sides. Under the action of fatigue stress and internal stress of the oxide, cracks are generated on both sides of the wear scar and spread to the periphery. When the crack propagates to the surface of the substrate, the position of the contact surface between the oxide layer and the substrate gradually oxidizes, which makes the combination of the two unstable. When cracks on both sides penetrate, large-sized block-shaped abrasive chips are formed, and some of the block-shaped abrasive chips are crushed and become fine particles of abrasive debris. It is concluded that the main mechanism of high temperature wear of cast 4Cr4Mo2NiMnSiV sample steel is oxidative spalling wear and fatigue wear. Due to the lowest content of Fe in H13 steel, the loss of Fe in the matrix during wear is the most. Therefore, the 4Cr4Mo2NiMnSiV sample steel has better resistance to wear than H13 steel, which also has a good correspondence with the wear rate of the material.

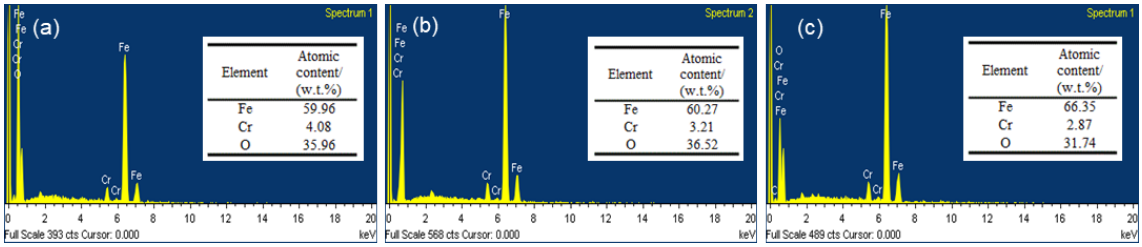


Fig. 5. EDS Analysis of Wear Surface of H13 and 4Cr4Mo2NiMnSiV steel at 600 °C: (a) H13 steel; (b) 4Cr4Mo2NiMnSiV (M) steel; (c) 4Cr4Mo2NiMnSiV (M+B) steel

3.3. Analysis of the wear surface phase

Fig. 6 shows the XRD phase analysis of the worn surface of H13 steel and cast 4Cr4Mo2NiMnSiV specimen at 600 °C. From the figure, the wear surface of the three samples after the high temperature wear at 600 °C showed the oxide of Fe, and the surface also had the presence of matrix iron, indicating that the wear surface did not completely oxidize and peel off during the wear process. The oxide of the cast 4Cr4Mo2NiMnSiV sample is mainly composed of Fe_2O_3 and Fe_3O_4, and its peak intensity is obviously higher than that of α-Fe, which indirectly indicates that a uniform oxide layer is formed on the worn surface and is consistent with the surface morphology analysis results.

Generally, Fe oxides include FeO, Fe_3O_4, and Fe_2O_3. FeO generates temperatures above 600 ℃ and Fe_2O_3 forms temperatures from 200 ℃ to 600 ℃ [10-11]. The flash point calculation formula based on Arachard [12]:

$$(\theta_x)_{max} = 1.64\ \theta_m \quad (1)$$

Where $(\theta_x)_{max}$ is the maximum temperature of the friction surface, and θ_m is the ambient temperature. When the temperature is 600 °C, the flash point temperature of the worn surface is much higher than 600 °C, but the FeO phase is not detected by XRD. Due to the high temperature friction, the surface defects of the material increase, which is favorable for the diffusion of Fe atoms and O atoms and forms high oxygen content Fe_3O_4 and Fe_2O_3. Comparing the XRD results of different samples, it can be found that the oxide film after wear at 600 °C is mainly Fe_2O_3. For the 4Cr4Mo2NiMnSiV steel, Fe_3O_4 is also present in the oxide film. Oxidation spalling is the primary cause of material wear during high temperature friction, and a stable oxide layer provides effective protection for the substrate. The existing research results show that both Fe_2O_3 and Fe_3O_4 oxides have antifriction effect, but Fe_3O_4 antifriction effect is better than Fe_2O_3 [13]. Thereby reducing the wear rate of the material, which is also an important factor in the wear resistance of alloy steel under the same conditions is higher than that of H13 steel.

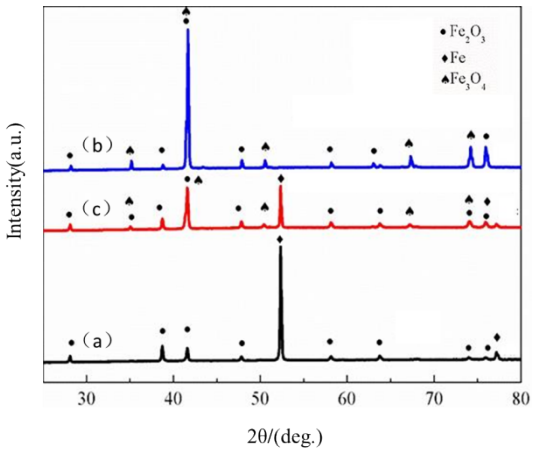


Fig. 6. XRD pattern of wear surface of H13 and 4Cr4Mo2NiMnSiV steel: (a) H13 steel; (b) 4Cr4Mo2NiMnSiV (M) steel; (c) 4Cr4Mo2NiMnSiV (M+B) steel

3.4 Wear section

Fig.7 shows the wear profile and EDS analysis of H13 steel and cast 4Cr4Mo2NiMnSiV steel after oxidation at 600 °C for 200 h. The three samples have a thick oxide layer on the surface layer, and a layered inner oxide layer and an unoxidized layer exist in the lower portion of the oxide layer. The inner layer is oxidized from inclusion and microspores. During the friction process, oxygen atoms easily enter the matrix through the micropores or microcrack defects of the material matrix, causing oxidation inside the matrix. When the surface oxide layer is detached, the inner flaky oxidized region forms an oxide layer, and a region having a low oxygen content is formed at the center of the high oxygen content oxide layer. This area has weak bonding ability and is easy to crack and peel off. Thus reducing the wear resistance of the material. After calculation, the thickness of the oxide layer of H13 steel, 4Cr4Mo2NiMnSiV (M) steel and 4Cr4Mo2NiMnSiV (M+B) steel is 58.27μm, 89.23μm and 97.62μm. H13 steel oxide layer is the thinnest, divided into two layers inside and outside, the outer layer is loose, small holes appear in some positions, the inner layer is dense, and the matrix is firmly bonded, and there is no crack in the scanned photograph. The results of EDS analysis showed that the outer oxide layer O aggregated. The Fe content was slightly lower than that of the matrix, and the internal oxide layer had more Cr elements. 4Cr4Mo2NiMnSiV (M) steel oxide layer thickness is large, also divided into two layers inside and outside. The outer layer is loose and porous, and the void is larger and larger than H13 steel. After EDS analysis, the content of Fe and O is higher than that of the matrix, and the main component is presumed to be the oxidation of Fe. The content of the inner Cr and Ni elements is enriched with respect to the matrix, so that a relatively dense Cr-Ni oxide layer is formed, which can effectively prevent the oxidation reaction from continuing. The 4Cr4Mo2NiMnSiV (M+B) steel oxide layer is also clearly divided into a tight inner oxide layer and a loose outer oxide layer, but it can be seen that the oxide layer thickness is the largest among the three samples. The results of EDS show that the content of Fe and O in the outer layer is higher than that in the matrix, and the inner layer Cr and O are segregated. It is speculated that the outer layer is the oxide of Fe and the inner layer is the oxide of Cr. During repeated enthalpy of external force on the surface of the sample, the source of the crack is reduced and the tendency of oxidation inside the substrate is suppressed. The thick oxide layer produced by the 4Cr4Mo2NiMnSiV die steel during the wear process provides stable support and resistance to plastic deformation and thermal softening. Therefore, the 4Cr4Mo2NiMnSiV die steel has better wear resistance at high temperature.

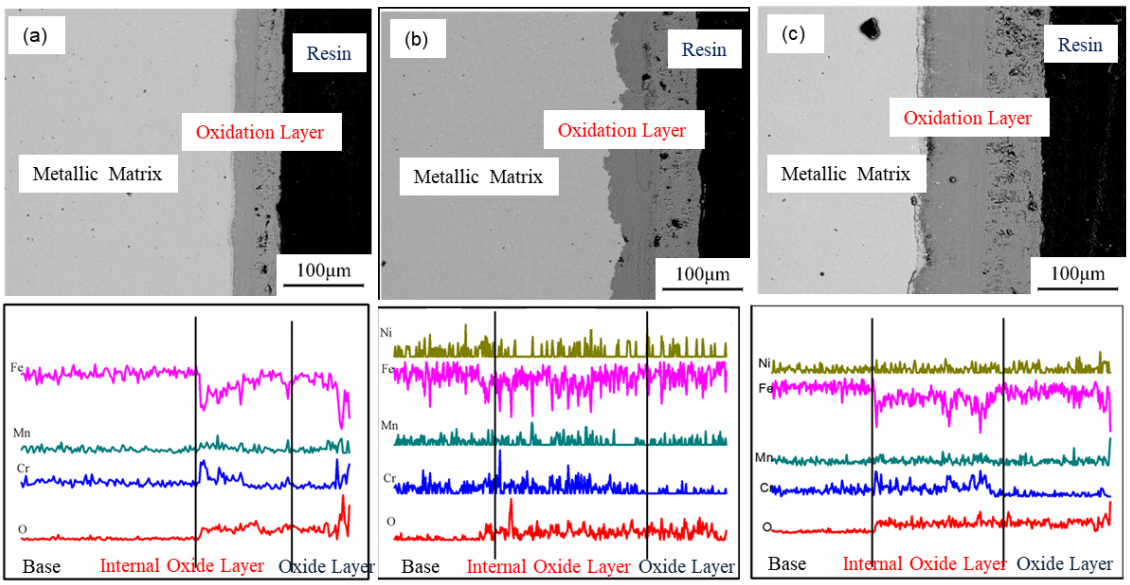


Fig. 7. Comparison between the simulation and experimental true stress-strain curves at different temperatures and strain rates: (a) 0.01 s^{-1}, (b) 0.1 s^{-1}, (c) 1 s^{-1}.

4. Conclusions

In this paper, the properties of the high temperature wear mechanism of 4Cr4Mo2NiMnSiV hot work die steel were studied. Through experiments and analysis, the following conclusions were obtained:

1. The friction coefficient of cast 4Cr4Mo2NiMnSiV steel increases with the increase of temperature, and the main mechanism of high temperature wear is oxidative spalling wear and fatigue wear.
2. Surface oxidations of cast 4Cr4Mo2NiMnSiV steel are Fe_2O_3 and Fe_3O_4. The oxide film generated during the high temperature wear process effectively reduces the wear of the material.
3. The 4Cr4Mo2NiMnSiV (M+B) steel has the best wear resistance, followed by 4Cr4Mo2NiMnSiV (M) steel, and the wear resistance of H13 steel is lower than that of 4Cr4Mo2NiMnSiV steel.

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