Effect of Fe on the high-temperature tribological behavior of NiAl/WC-Fe\textsubscript{x} self-lubricating composites produced by thermal explosion

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Abstracts
In order to improve the self-lubricating properties of NiAl-WC composite, a novel solid-lubricating material, NiAl/WC-Fe\textsubscript{x} (x = 5, 10, 15 and 20 in weight percent) composites were synthesized by thermal explosion using Ni-Al-WC-Fe mixed powders. The microstructure, phase constituent, and tribological behavior of NiAl/WC-Fe\textsubscript{x} composites at 800 °C were studied. According to the results, the addition of Fe could reduce the cracks of the composites. Compared with NiAl/WC, the increased Fe content could reduce the friction coefficient and high-temperature wear rate due to its enhanced ductility, oxidation, and low shearing strength. In particular, composite with 10 wt% Fe content had the lowest friction coefficient and wear rate because of the self-lubricating behavior of Fe at elevated temperature. The result also showed that the wear mechanism of NiAl/WC-Fe\textsubscript{x} is related to Fe content at high-temperature.

1. Introduction

In recent years, NiAl-based composites have attracted attention from many researchers because of their stable physical and chemical properties and excellent high-temperature friction and wear characteristics [1–4]. In addition, due to their excellent tribological property, NiAl-based composites are also regarded as one of the promising materials in the harsh wear field at high-temperature [5, 6].

A lot of studies on NiAl-based materials have shown that the addition of some oxides and ceramics to NiAl alloy can significantly improve the wear resistance due to their excellent hardness, high thermal stability and grain refinement [7–10]. During the high-temperature wear process, oxides and ceramic particles acting as solid lubricants can reduce the friction coefficient and wear rate [11–13].

In our earlier study, we successfully synthesized NiAl-WC composites by thermal explosion, and investigated its high-temperature wear behavior at 800 °C. The results show that the addition of WC can evidently decrease high-temperature wear rate [14, 15]. Based on earlier literature, WC particles contribute to the excellent wear resistance and tribological properties of NiAl/WC due to their higher hardness and stable physical property [16, 17].

However, the different thermal expansivity between NiAl and ceramic particles as well as the grain spalling and cracks on the worn surface pose a great challenge to the improvement of high-temperature wear resistance [18, 19].

Many studies have confirmed some soft metal, such as Ag, Au, Pb, Cu, Mo, exhibits lubricating effect due to their excellent ductility and lower shearing strength during wear process at high temperature [20, 21]. Soft metals can deformation and can be oxidized to form tribofilm during sliding and accommodate both interacting surfaces, which can result in reducing friction and wear [22, 23].
However, little literature have been reported about the lubricating effect of Fe on tribological property of NiAl-based composites at high temperature. Compared with other soft metals, Fe has fine ductility, lower price and is widely used in the industry. As for the NiAl-based composites, the ideal state of worn surface is covered by a dense and self-lubricating layer formed in high-temperature wear test \[24, 25\]. Therefore, it is a beneficial attempt to add Fe into NiAl-based composites and utilize its physical and chemical properties to improve wear resistance at high temperature.

Based on the above-mentioned work, this study focused on the tribological properties of NiAl/WC composite with different Fe content (NiAl/WC-Fe\(_x\), \(x = 5, 10, 15\) and 20 in weight percent) at 800 °C. The NiAl/WC-Fe\(_x\) composites are synthesized by thermal explosion, in which WC is used as a grain refinement and Fe can be provided as a solid lubricant because of its oxidation reaction. It is a meaningful research on investigating the influence of Fe content of the high-temperature tribological behavior for the NiAl/WC-Fe\(_x\) composites.

2. Experimental

2.1. Preparation of materials

In this work, NiAl/WC and NiAl/WC-Fe\(_x\) (\(x = 5, 10, 15,\) and 20 in weight percent) composites were made of Ni-Al-WC-Fe mixed powders subjected to thermal explosion. The size of powder did not exceed 200 mesh. The SEM images of the commercial powders are shown in figure 1. Firstly, 5 wt%, 10 wt%, 15 wt%, 20 wt%, and 25 wt% Fe powders were added into NiAl-20 wt% WC mixed powders respectively. Secondly, the mixed powders were mixed for 2 h using a planetary ball mill under Ar environment which can protect the samples from oxidation. Then the mixed powders were pressed into a cylindrical compact under pressure of 200 MPa. The diameter and height of the compact are 45 mm and 5 mm respectively. The photograph and morphology of the mixed powder sample is shown in figure 2. It can be seen from figure 2(a) that the samples exhibited dense and metallic luster. Figure 2(b) shows the mixed powders were deformed and agglomerated after ball milling. As the EDS result is shown, The WC particles are bonded on the surface of mixed powders and the main element of the mixed samples is Fe, Ni, and Al. The result indicates soft metal particles are bonded with each other during ball milling. Finally, the compacts were put into resistance furnace with the preheating temperature of 700 °C under air environment till the thermal explosion was ignited.
2.2. Tribological testing

Based on our previous work, the tribological behavior of NiAl/WC composite with different WC contents at 800 °C was investigated, and the main wear mechanism of the composites is oxidation wear. In order to further study the effect of Fe on NiAl/WC composite at high temperature, in this study, the dry wear test was still adopted at the temperature of 800 °C using a UMT-2 tribometer (made in Center for Tribology Inc., USA). The test equipment and the schematic diagram of wear test is shown in figure 3. The test surface was polished and buffed to make sure its roughness parameter Ra ≤ 0.1 μm before the wear test. In the present study, the counterpart used a Si₃N₄ ball with diameter of 2 mm. At the same time, the value of rotating radius of Si₃N₄ ball, the contact load, and test-time were 5 mm, 20 N, and 30 min, respectively.

The friction coefficient data were recorded by a sofware from supplied by the manufacturer. Wear scratches and wear volume was measured by a confocal scanning laser microscopy (OLYMPUS LEXT OLS 4000).

The wear rate was calculated using the following formula:

\[
\text{Specific wear rate} = \frac{V}{SP}
\]

where V, P and S are worn volume in mm³, normal load in N and total sliding distance in mm, respectively.

All the wear tests were measured at least five times under the same conditions, and the average results were calculated.
2.3. Characterization
The microstructure and worn surface of NiAl/WC-Fe$_x$ composites were examined through FESEM (Model JSM-5310, Japan) and energy-dispersive spectrometry. The phase constituents were characterized with a SIMMENS D500 x-ray diffractometer using Cu K$_\alpha$.

3. Result and discussion
3.1. Phase constituent and microstructure
The NiAl/WC and NiAl/WC-Fe$_x$ composites, except the sample with 25 wt% Fe content, were successfully produced by thermal explosion and their photographs are shown in figure 4. Thermal explosion does not occur in the sample with 25 wt% Fe content due to the dilution of WC powders and Fe powders. It is clear to see that the surfaces of the samples are oxidized in the thermal explosion and there are several cracks and pores existed on the surface of NiAl/WC composite, but for NiAl/WC-Fe$_x$ composites, the surfaces become smoother and denser with the increase of Fe content. The result proves that the addition of Fe can benefit to decrease the cracks and improve the density.

The XRD pattern of the final product fabricated by thermal explosion is shown in figure 5. There are three main phases in the composites including NiAl, WC, and Fe. The XRD result also indicates that NiAl/WC-Fe$_x$ composites were successfully synthesized while WC and Fe are not involved in the thermal explosion. The addition of WC particles and Fe particles can reduce the reaction temperature and inhibit the thermal explosion reaction, but the pressure during the thermal explosion can make the mixed reactive powders easy to contact. The pressure plays a key role in the synthesis process and can improve the density of the final product [26, 27].

The microstructure of the NiAl/WC-Fe$_{15}$ is displayed in figure 6, showing that there are no apparent cracks and pores in the composites. As presented in distribution of elements, Ni element and Al element have similar distribution, indicating that the basic microstructure of the composite is mainly composed of NiAl, and the similar distribution of W element and C element proves the white particles in the microstructure are WC ceramics. As can be seen from figure 6(d), Fe element presents a homogeneous distribution, indicating Fe is remelt due to the exothermic reaction in the thermal explosion. It is known that the expansion coefficient between NiAl and WC has a greater gap, which may easily cause cracks in the final product. The SEM image results confirm the addition of Fe can serve as a bridging function in the bonding mechanism between NiAl and WC, contributing to the density of the final product [28]. The microstructure result is in accordance with that of XRD of the final samples.
3.2. Tribological behavior at high temperatures
The friction coefficients of NiAl/WC and NiAl/WC-Fe\textsubscript{x} composites are shown in figure 7. It can be seen from figure 7(a) that the friction coefficient of NiAl/WC composite is unsteady at the beginning and fluctuates significantly when the running-in period is shorter than 15 min. The main reason for this variation is that a high-performance tribofilm is not formed during the running-in period and WC particles are falling off the worn surface [29, 30]. It is worth noting that the friction coefficient of NiAl/WC-Fe\textsubscript{x} (x = 5, 10) exhibits numerical stability even at the overall wear rate. For the samples with the higher Fe content (15 wt% and 20 wt%), the friction coefficient exhibits an apparent decline in the initial stage, and then a significant fluctuation in the wear test. The results indicate the different addition of Fe plays a key role in the friction coefficient. As shown by the XRD result and microstructure of NiAl/WC-Fe\textsubscript{x}, Fe does not take part in the thermal explosion and the increasing Fe content can decrease the hardness of the composites.

As is shown in figure 7(b), the adding Fe can decrease the friction coefficient of NiAl/WC-Fe\textsubscript{x}. It is worth noting that the friction coefficient of NiAl/WC-Fe\textsubscript{x} decreases remarkably in the range of Fe content from 0 to 10 wt%, and increases slightly when the Fe content exceeds 10 wt%. The minimum friction coefficient of composites containing 10wt % Fe is 0.27 and the maximum value presenting in NiAl/WC, which is 0.42.

The wear rates of NiAl/WC and NiAl/WC-Fe\textsubscript{x} composites are shown in figure 8. It can be seen that Fe content affects the wear rate of composites. When Fe content is less than 10wt %, the wear rate decreases with the increase of Fe content. However, once the Fe content is over 10 wt%, the wear rate rises with the increase of Fe.
NiAl/WC-Fe₁₀ has the lowest wear rate of $4.02 \times 10^{-5} \text{mm}^3\text{N}^{-1}\text{m}^{-1}$, while NiAl/WC has the highest wear rate of $5.10 \times 10^{-5} \text{mm}^3\text{N}^{-1}\text{m}^{-1}$.

The 3D-profile micro-graphs of NiAl/WC composite and NiAl/WC-Fex composites are presented in figure 9. Figure 9(a) shows NiAl/WC composite exhibits rough surface and some irregular pits, revealing some small WC particles spalling from the worn surface. This might be attributed to the shearing by the hard asperities of Si₃N₄ ball. It can be seen from figures 9(b) and (c) that the scratch of NiAl/WC-Fe₅ and NiAl/WC-Fe₁₀ has a smooth surface, especially for NiAl/WC-Fe₁₀, the composite has the smoothest and narrowest worn surfaces. With the increase of the Fe content, it can be observed from figures 9(d) and (e) that he scratches and scrapes become severer. The reason is that the addition of Fe can reduce the microhardness of the NiAl/WC composite, and the Si₃N₄ ball can plow into the NiAl/WC-Fex composites, resulting in the striping of the composites. Additionally, the results also suggest that the NiAl/WC-Fex composites are dominated by abrasive wear in Fe samples of 15 wt% and 20 wt% Fe.

The worn surfaces of NiAl/WC and NiAl/WC-Fex composites are shown in figure 10. The dark areas of the worn surface reveal oxidation reactions occurring during the wear test at high-temperature. Figure 10(a) shows some white smooth WC particles, which are shattered. Furthermore, brittle micro-peeling exists on worn surface of NiAl/WC, indicating that abrasive attrition is one of the wear mechanisms [31]. There are some cracks and pull-out grains on the worn surface. Due to the different expansion coefficients between NiAl and WC, some fragile WC particles are easy to fall off the NiAl substrate in the high-temperature wear test [32, 33]. As shown in figures 10(b)–(e), for NiAl/WC-Fex, the worn surface is free of obviously visible shattered particles, furrows and cracks. The worn surfaces of NiAl/WC-Fe₅ and NiAl/WC-Fe₁₀ are shown in figures 10(b) and (c), it can be seen that their worn surfaces show uniform colors without significant scratches. The continuous and
Figure 9. 3D-profile micro-graphs of NiAl/WC (a) and NiAl/WC-Fe\textsubscript{x} composites with different Fe content: (b) 5\%wt Fe, (c) 10\%wt Fe, (d) 15\%wt Fe, and (e) 20\%wt Fe.

Figure 10. Worn surface of NiAl/WC and NiAl/WC-Fe\textsubscript{x} composites with different Fe content: (a) NiAl/WC, (b) NiAl/WC-Fe\textsubscript{5}, (c) NiAl/WC-Fe\textsubscript{10}, (d) NiAl/WC-Fe\textsubscript{15}, and (e) NiAl/WC-Fe\textsubscript{20}.
smooth glaze layers are formed on their worn surfaces. The resulting smooth glaze layers help to reduce the friction coefficients of the composites [34, 35]. The results are consistent with those of the 3D-profile micrographs. With the increase of the Fe content, it can be observed from figures 10(e), (f) that the glaze layer on the worn surface is partly broken down, while some flakes and delamination pits exist in the worn surface of NiAl/WC-Fe15 composite and NiAl/WC-Fe20 composite, which indicates that plastic yield occurs during the wear process. Therefore, for the NiAl/WC-Fex with higher Fe content, the wear mechanisms are manifested as multi-plastic deformation wear and oxidative wear. NiAl/WC-Fex with high Fe content has certain adhesive wear characteristics. The softening of Fe results in the decrease of hardness of composite at high-temperature, and the NiAl and Fe are distributed around the hard WC particles under the effect of adhesion.

A typical SEM image of cross-section of wear scar on NiAl/WC-Fe10 obtained at 800 °C is shown in figure 11. It can be seen that there are two different morphologies exhibited in the cross-section. The top surface is a dark and continuous tribofilm, offering further evidence that oxidation occur during wear test. Meanwhile, the oxide lubrication film can decrease the coefficient and wear rate of the composites. The internal area is not oxidized due to protection effect of dense metal oxide layer. The results indicate that the worn surface can also prevent further intrusion of O ions and can improve the high-temperature oxidation resistance.

In order to further study the composition distribution of the worn surface and wear mechanism, the mapping of the EDS elements of Al, Fe, O, Si, and W is shown in figure 12. Obviously, almost all the major elements have a homogeneous distribution. Furthermore, the presence of a large of O elements reveals that oxides are developed in the wear test at 800 °C and form the glaze layer. As demonstrated by some previous works, oxides in the NiAl-based or TiAl-based composites can act as solid lubricants [36]. At the same time, the self-lubricating layer is conducive to improving the tribological properties at high temperature [37, 38]. The results of elemental distribution confirm that the oxidation wear is the main wear mechanism of the NiAl/WC-Fe composite. The Si elements and W elements also have homogeneous distribution, indicating that hard Si3N4 and WC ceramics transfer occur during the high-temperature wear test. Based on the EDS analysis, we can speculate the self-lubricating films are mainly composed of aluminum oxide, ferric oxide, nickel oxide, and WC.

According to the previous studies, the schematic diagram of the NiAl/WC-Fe self-lubricating layer is shown in figure 13. In the schematic diagram, white particles present WC, blue particles present Fe, and yellow particles present mixed metal oxide. Firstly, the active elements from the NiAl/WC-Fe composite, such as Ni, Al and Fe, are oxidized and form oxide layers on the worn surfaces during the high-temperature tribological process. It is known that in the NiAl-based composites, continues oxides can play a role in solid lubricants during the sliding process at high temperature. Meanwhile, the low enhanced ductility and low shearing strength of Fe further enhance self-lubricating of the composite. Afterwards, under the thermal pressure of Si3N4 ball, WC particles are embedded on the worn surface and can protect the matrix from scraping due to their high stiffness. which further enhance the wear resistance of the composites. Meanwhile, the pressure at high temperature can promote the production of continuous and dense self-lubricating films.
4. Conclusions

In this paper, NiAl/WC, and NiAl/WC-Fe<sub>x</sub> composites with different Fe content were successfully produced by thermal explosion. The high-temperature tribological behavior of NiAl/WC-Fe<sub>x</sub> was investigated at 800 °C. The main conclusions can be drawn as follows:

1. NiAl/WC-Fe<sub>x</sub> (x = 5, 10, 15 and 20 in weight percent) composites can be produced by thermal explosion. Compared with NiAl/WC, the NiAl/WC-Fe<sub>x</sub> with addition of Fe can reduce the cracks of the composites.

Figure 12. Element distribution of the NiAl/WC-Fe<sub>10</sub> surface: (b)–(d) versus Al, Fe, O, Si, and W element distribution.

Figure 13. A schematic illustration which showed the wear mechanisms of NiAl/WC-Fe<sub>x</sub> composites during the sliding progress at 800 °C.
(2) The addition of Fe can noticeably affect the friction coefficient and the wear rate of the NiAl/WC-Fe<sub>x</sub>. In particular, NiAl/WC-Fe<sub>10</sub> has the lowest friction coefficient and the wear rate, which are 0.31, and 4.02 × 10<sup>-5</sup> mm<sup>2</sup>N<sup>-1</sup>m<sup>-1</sup>, respectively.

(3) NiAl/WC-Fe<sub>x</sub> composites exhibit excellent tribological properties at 800 °C, which is attributed to WC reinforcement and oxides layer generated on the worn surface.

(4) The wear mechanism of NiAl/WC-Fe<sub>x</sub> composites is related to the Fe content. The main characteristics of composites with lower Fe content are oxidation wear and continuous self-lubricating film on the worn surface. With the increase of Fe content, the wear mechanisms of the composite exhibit multi- plastic deformation wear and oxidative wear.

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