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Improvement of High Temperature Wear Behavior of In-Situ Cr$_3$C$_2$-20 wt. % Ni Cermet by Adding Mo

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Abstract: Cr$_3$C$_2$-Ni cermet is a kind of promising material especially for wear applications due to its excellent wear resistance. However, researches were mainly concentrated on the experiment condition of room temperature, besides high-temperature wear mechanism of the cermet would be utilized much potential applications and also lack of consideration. In present paper, the influence of Mo content on the high-temperature wear behavior of in-situ Cr$_3$C$_2$-20 wt. % Ni cermet was investigated systematically. The friction-wear experiment was carried out range from room temperature to 800 $^\circ$C, while Al$_2$O$_3$ ceramic was set as the counterpart. According to experimental results, it is indicated that the coefficient of friction (COF) of friction pairs risen at the beginning of friction stage and then declined to constant, while the wear rate of Cr$_3$C$_2$-20 wt. % Ni cermet risen continuously along with temperature increased, which attributes to the converted wear mechanism generally from typical abrasive wear to severe oxidation and adhesive wear. Generally, the result of wear resistance was enhanced for 13.4% (at 400 $^\circ$C) and 31.5% (at 800 $^\circ$C) by adding 1 wt. % Mo. The in-situ newly formed (Cr, Mo)$_7$C$_3$ ceramic particle and the lubrication phase of MoO$_3$ can effectively improve the wear resistance of Cr$_3$C$_2$-20 wt. % Ni cermet.

Keywords: Cr$_3$C$_2$–Ni cermet; molybdenum; high temperature; wear

1. Introduction

Metal-ceramic composite (cermet) simultaneously possesses the excellent strength of metals and high hardness of ceramics, which has attracted widely attention in thermal spraying, cutting operation and aerospace fields [1,2]. Ordinarily, chromium carbide ceramic particle served as an additive to increase the physical and abrasion resisting properties of the metal-ceramic composite [3]. By consider the multiple crystal structures of binary chromium carbides, two of them appearing, in which Cr$_3$C$_2$ behaves the highest melting temperature and thermal mechanism stable structure, while the hardness and toughness of Cr$_7$C$_3$ is superior to Cr$_3$C$_2$ particle [4,5].

In the past decades, Cr$_3$C$_2$-Ni cermet was traditionally fabricated by reactive sintering. Hussainova et al. [6] found that microstructure and physical properties of the cermet after reactive sintering were condensed and superior to the traditional sintering technology. Juhani et al. [7] researched the impact wear behavior of Cr$_3$C$_2$-Ni cermet and concluded that the wear resistance was enhanced by the Ni content additive. Pirso et al. [8] fabricated the Cr$_3$C$_2$-Ni cermet by reactive sintering and the specimen was carried out on the block-on-ring abrasion testing machine (two body wear resistance). The results were shown the volumetric wear was attributed to the crushed and removed Cr$_3$C$_2$ ceramic particle in the subsurface. Sarjas et al. [9] synthesized and sprayed the Cr$_3$C$_2$-Ni powder on the surface of steel C45. The wear resistance of steel C45 was improved obviously due to high
hardness Cr$_3$C$_2$-Ni coating protection. Generally, various researches were focused on the experiment condition of room temperature, besides the influence of the content of Mo on the high temperature wear behavior of Cr$_3$C$_2$-Ni cermet was lack of investigation.

Basically, Cr$_3$C$_2$-Ni cermet exhibited high hardness and melting temperature, excellent high temperature oxidation and wear resistance. It had been widely applied in the wear applications such as coating protection and cutting tools [9]. The preparation method and application between monolithic (sintered) materials and thick coatings are evidently different.

The bulk Cr$_3$C$_2$-Ni cermet [10–12] is prepared by high energy ball milling, compression molding and vacuum sintering. After ball milling processing, the initial powders are mixed uniformly. During the vacuum sintering process, Cr$_3$C$_2$ ceramic is in-situ generated by the chemical reaction of single Cr and C elements. The low heating rate and long enough holding time are sufficient for the interface bonding. The microstructure of bulk Cr$_3$C$_2$-Ni cermet is also uniform and the porosity is lower. Therefore, the bulk Cr$_3$C$_2$-Ni cermet exhibits better mechanical properties and wear resistance. The bulk Cr$_3$C$_2$-Ni cermet can be used as the structural material.

However, the thick Cr$_3$C$_2$-Ni coating [13–16] is prepared by the supersonic thermal spraying. Cr$_3$C$_2$ and Ni powders are used as the raw materials. The Cr$_3$C$_2$ is not in-situ generated by the chemical reaction of Cr and C. The preparation time is shorter than bulk Cr$_3$C$_2$-Ni cermet. The matrix can be protected from corrosion by the Cr$_3$C$_2$-Ni coating. But the interface bonding between the matrix and Cr$_3$C$_2$-Ni coating is not enough, which is not suitable for the wear-resistant material. The thick Cr$_3$C$_2$-Ni coating can be used as the functional material.

Generally, metals were chosen as the counter materials at room temperature. Besides, metals were easily oxidized at high temperature. Thus, Al$_2$O$_3$ ceramic material was chosen as the counter material in this study. On the one hand, the experimental temperature (room temperature to 800 °C) was below the oxidation temperature of Al$_2$O$_3$ ceramic material. One the other hand, the generation of the oxidative products and Al$_2$O$_3$ ceramic belonged to the typical two-body wear condition. As we known, the changes of the sliding speed and load had an important influence on the metallic materials. In this study, the effects of the hardness difference of Cr$_3$C$_2$-Ni cermet and Al$_2$O$_3$ ceramic were more important than the sliding speed and load under two-body wear condition. The sliding speed of 50 r/min at the normal load of 50 N were chosen as the constants in this work.

In our previous researches [17–19], Cr$_3$C$_2$-Ni cermet had been prepared by reactive sintering. Cr, Ni, and C powders were chosen as the raw materials. The optimal Ni content and sintering temperature were confirmed as 20 wt. % and 1300 °C, respectively. In addition, the influence of the content of Mo on the microstructure, mechanical properties and high temperature oxidation resistance of Cr$_3$C$_2$-20 wt. % Ni cermet had been also studied systematically. Based on these basic studies, the influence of the content of Mo on the high temperature wear behavior of in-situ Cr$_3$C$_2$-20 wt. % Ni cermet was investigated in this paper.

2. Experimental Method

2.1. Preparation of In-Situ Cr$_3$C$_2$-20 wt. % Ni Cermet

In this work, Cr$_3$C$_2$-20 wt. % Ni cermet were prepared by vacuum sintering. Cr, Ni, Mo, and C powders were used as the raw materials. The grain sizes of Cr, Ni, Mo, and C powders were 1–5 µm (Purity of 99.9%, XSF Technology Co.Ltd, Shenzhen, China). The atomic ratio of Cr and C powders was 3:2. The content of Ni powder was 20 wt. %. The contents of Mo powder were 0.5, 1.0 and 1.5 wt. %. The raw powders were milled by the planetary ball mill (QM-3SP4), which was manufactured by the Nanjing University, Nanjing 210020, Jiangsu Province, China. The mixed powders were pressed by the cold isostatic pressing equipment (LDJ6000) under 200 MPa pressure. The equipment was manufactured by the Hengli Technology Co. LTD, Taiyuan 030000, Shanxi Province, China. The pressed powders were sintered in a vacuum sintering furnace (PT-V220LW-G) at 1300 °C for 1 h. The vacuum sintering furnace was manufactured by the Nuotai Technology Co. LTD, Zhengzhou 450000, Henan
Province, China. The detailed fabrication technology and experimental parameters had been reported in references [17–19].

2.2. Wear Resistance Test of In-Situ Cr$_3$C$_2$-20 wt. % Ni Cermet

The tribological tests were carried out on the multifunctional friction and wear testing machine (MMU-10G) shown in Figure 1. Cr$_3$C$_2$-20 wt. % Ni cermet and Al$_2$O$_3$ ceramic material were chosen as the pin and disc specimens, respectively. Pin specimen was set on the disc specimen to collect the wear debris. The dimensions of them were $5 \times 5 \times 12.5\ mm^3$ and $44\ mm \times 6\ mm$, respectively. In order to research the effect of Mo content on the high temperature wear behavior of Cr$_3$C$_2$-20 wt. % Ni cermet, the experimental temperature was confirmed from room temperature to $800\ ^\circ\ C$. The tribological tests were carried out in the air atmosphere to confirm the wear mechanism of the cermet at different temperatures. The sliding speed and the normal load of the friction and wear testing machine were $50\ r/min$ and $50\ N$, respectively. The sliding distance and the linear speed were $100\ m$ and $3.97\ m/min$, respectively. Therefore, the testing time was about $25\ min$ in this study. The abrasive resistance of Cr$_3$C$_2$-20 wt. % Ni cermet was evaluated by testing the coefficients of friction (COF) and calculating the volume abrasion rate [20]. In this study, one specimen can be used for one time. The experiment was repeated for three times under identical conditions to obtain the scatter bars.

Besides, the coefficients of friction (COF) can be obtained and the volume abrasion rate was calculated by the following equation:

$$ I = \Delta W \cdot \rho^{-1} \cdot S^{-1} \cdot P^{-1} \quad (1) $$

where $I$ was the volume abrasion rate, $\Delta W$ was the loss of the mass before and after wear, $\rho$ was the density, $S$ was the wear distance and $P$ was the normal load.

![Figure 1. The schematic diagram of the multifunctional friction and wear testing machine.](image)

2.3. Materials Characterization

The microstructure and chemical component of Cr$_3$C$_2$-20 wt. % Ni cermet before and after wear tests were studied by field emission scanning electron microscopy (FE-SEM, FEI Quanta600) equipped with Energy Disperse Spectroscopy (EDS). The chemical component of the wear product was researched by X-ray photoelectron spectroscopy (XPS, AXIS ULtrabld). 3D laser scanning microscope (3D-LSM, VK-9700K) was applied to analyze the depth of the wear surface. The wear mechanism of Cr$_3$C$_2$-20 wt. % Ni cermet was confirmed based on the experimental results of the inclined cross section and vertical cross section specimens.
3. Results and Discussion

The coefficients of friction (COF) and wear rates of in-situ Cr$_3$C$_2$-20 wt. % Ni cermet with different Mo content and temperature are shown in Figure 2a,b. Obviously, the COF rises at the first stage and then declines, while the wear rate of the cermet rises continuously with the temperature increasing. The detailed reasons will be explained below according to the SEM and 3D-LSM results. Accordingly, the specimen with 1.0 wt. % Mo exhibits the best wear resistance. The wear rates are $2.17 \times 10^{-5}$ mm$^3$ N$^{-1}$ m$^{-1}$ at 400 °C, 9.32 and $6.38 \times 10^{-5}$ mm$^3$ N$^{-1}$ m$^{-1}$ at 800 °C for the specimens without Mo and with 1.0 wt. % Mo addition. It can be concluded that the wear resistance of Cr$_3$C$_2$-20 wt. % Ni cermet is enhanced for 13.4% (at 400 °C) and 31.5% (at 800 °C) by adding 1 wt. % Mo.

![Figure 2](image_url)

**Figure 2.** The variation trends of the friction coefficient and wear rate with the Mo content at different temperature: (a) friction coefficient and (b) wear rate.

In our previous research [11], it was found that the (Cr, Mo)$_7$C$_3$, except (Cr, Mo)$_3$C$_2$ and Ni, was formed in the microstructure after adding Mo element. The influence of the newly formed (Cr, Mo)$_7$C$_3$ ceramic particle on the wear resistance of Cr$_3$C$_2$-20 wt. % Ni cermet is mainly important for the application. The bulk modulus, shear modulus and elasticity modulus of Cr$_7$C$_3$ is lower than Cr$_3$C$_2$, indicating that the Cr$_7$C$_3$ possesses the better toughness. Moreover, the hardness of Cr$_3$C$_3$ is also superior to Cr$_3$C$_2$ [4]. It can be concluded that the improvement of physical properties of Cr$_3$C$_2$-20 wt. % Ni cermet is attributed to the superior hardness and toughness of the newly formed (Cr, Mo)$_7$C$_3$ ceramic particle.

The SEM and 3D laser scanning morphologies of the wear surface for the specimens without Mo and with 1.0 wt. % Mo addition at different temperature are shown in Figure 3. As the wear temperature below 400 °C, the Cr$_3$C$_2$-20 wt. % Ni cermet is scratched by the hard Al$_2$O$_3$ ceramic material. The wear scratches can be clearly observed in the wear surface shown in Figure 3a1,a2,c1,c2. Some of the Cr$_3$C$_2$ ceramic particles peel off from the matrix because of the continuous wear condition, leading to the formation of the micro-cracks and peeling pits. The exfoliated Cr$_3$C$_2$ ceramic particles will enter into the wear particle system and further damage the matrix, resulting in the increase of the coefficients of friction (COF) and wear rates. Some of oxidative products can be also observed due to the local tribological oxidation. Comparing with the specimen without Mo, the wear surface and wear depth are improved obviously by adding 1.0 wt. % Mo. As shown in Figure 3b1,b2,d1,d2, the maximal wear depths decrease from 18.2 µm to 13.8 µm at 200 °C and form 21.9 µm to 21.3 µm at 400 °C, respectively.

As the wear temperature exceed 400 °C, the wear scratches decrease obviously and the matrix is covered by the oxidative products, as shown in Figure 3a3,a4,c3,c4. In this case, the oxidative wear is the dominate wear type. The oxidative products are soft and lubricative, which can effectively decrease the coefficients of friction (COF). However, the large area of oxidative products results in the increase of the wear rates. Comparing with the specimen without Mo, the maximal wear depths decrease from 33.7 µm to 22.5 µm at 600 °C and form 53.7 µm to 26.0 µm at 800 °C (as shown in Figure 3b3,b4,d3,d4),
respectively. The wear mechanism is abrasive wear at 200–400 °C and changes to severe oxidation and adhesive wear at 600–800 °C. 

![SEM and 3D laser scanning morphologies of the wear surface for the specimens without Mo and with 1.0 wt. % Mo addition at different temperature: (a1–b4) the specimen without Mo, (c1–d4) the specimen with 1.0 wt. % Mo addition.](image)

Figure 3. SEM and 3D laser scanning morphologies of the wear surface for the specimens without Mo and with 1.0 wt. % Mo addition at different temperature: (a1–b4) the specimen without Mo, (c1–d4) the specimen with 1.0 wt. % Mo addition.

In Figure 4a1–b4, it is shown the SEM morphologies of the inclined cross section for the specimens without Mo and with 1.0 wt. % Mo addition at different temperature. The cracks and broken Cr$_3$C$_2$ ceramic particles can be clearly observed in the interface between the wear surface and matrix at 200–400 °C, which well matches with the wear surface results. The exfoliated Cr$_3$C$_2$ ceramic particles are filled by the oxidative products, as shown in the black areas in Figure 4a2. When the wear temperature exceed 400 °C, the mechanical mixture and oxidation layer can be clearly observed in the interface between the wear surface and matrix at 600–800 °C, as shown in Figure 4a3,a4. Comparing with the specimen without Mo, the cracks and broken Cr$_3$C$_2$ ceramic particles decrease distinctly after adding 1 wt. % Mo element. Besides, the thickness of the mechanical mixture and oxidation layer are also decreased. Therefore, it can be concluded that Mo element is benefit for the improvement of the high temperature wear resistance of Cr$_3$C$_2$-20 wt. % Ni cermet.

According the XPS results in Figure 5a–c, the oxidative products are Cr$_2$O$_3$, NiO and MoO$_3$, respectively. MoO$_3$ possesses the lubricating property and the oxidative products are benefit for the decrease of the COF. The wear resistance of Cr$_3$C$_2$-20 wt. % Ni cermet is improved distinctly by adding 1 wt. % Mo, which is attributed to the superior hardness of the newly formed (Cr, Mo)$_7$C$_3$ ceramic particle and the lubrication phase of MoO$_3$.

As shown in references [21,22], the oxygen deficiency is accommodated by homologous series (Magnéli-type phases) of ordered structures. Molybdenum is a tribological active element in thick coatings and hardmetal industry. Molybdenum and tungsten oxides can form similar homologous series of Mo$_n$O$_{3n-1}$. According to the XPS results, MoO$_3$ is detected in this study and it has high melting points without any tendency to sublimate. Besides, M. N. Gardos [23,24] used the term “lubricious oxides” to describe expected low coefficients of friction (COF) and wear rates in unlubricated dry conditions.
sliding conditions. The tribo-oxidatively on carbides formed sub-oxides results in lower friction forces. The sub-oxides (MoO$_3$) have a contribution to the tribological behavior of carbides and cermets. Therefore, tribo-oxidatively formed MoO$_3$ can effectively improve the wear resistance of Cr$_3$C$_2$-20 wt. % Ni cermet by adding some amount of Mo element.

Figure 4. SEM morphologies of the inclined cross section for the specimens without Mo and with 1.0 wt. % Mo addition at different temperature: (a1–a4) the specimen without Mo; (b1–b4) the specimen with 1.0 wt. % Mo addition.

Figure 5. XPS results of the wear surface for the 1.0 wt. %Mo specimen: (a) Cr$_2$O$_3$; (b) NiO; and (c) MoO$_3$.

The SEM morphology and EDS results of the wear vertical cross section for the specimen without Mo and the schematic illustration of the wear mechanism are shown in Figure 6a,b1–b5. The mechanical mixture layer is the compounds of oxidative products, the broken Cr$_3$C$_2$ and Ni. At the initial stage, the micro-cracks and partial oxidative products form at room temperature and 200 °C. The degree
of wear aggravates and the mechanical mixture layer generates as the temperature increasing to 400 and 600 °C. The dense oxidative layer forms in the sub-surface of the cermet with further increasing the temperature to 800 °C, as shown in Figure 6b5. Therefore, the wear mechanism transforms from abrasive wear to severe oxidation and adhesive wear with the increase of temperature.

Figure 6. (a) SEM morphology and EDS results of the wear vertical cross section for the specimen without Mo; and (b1–b5) Schematic illustration of the wear mechanism.

4. Conclusions

In present paper, the influence of the content of Mo on the high temperature wear behavior of in-situ Cr3C2-20 wt. % Ni cermet was investigated systematically. The important conclusions were as following:

1. The wear rates are 2.17 and 1.88 × 10^{-5} \text{mm}^3\cdot\text{N}^{-1}\cdot\text{m}^{-1} at 400 °C, 9.32 and 6.38 × 10^{-5} \text{mm}^3\cdot\text{N}^{-1}\cdot\text{m}^{-1} at 800 °C for the specimens without Mo and with 1.0 wt. % Mo addition. It can be concluded that the wear resistance of Cr3C2-20 wt. % Ni cermet is enhanced for 13.4% (at 400 °C) and 31.5% (at 800 °C) by 1 wt. % Mo additive.

2. The wear mechanism of the cermet is abrasive wear at 200–400 °C and changes to severe oxidation and adhesive wear at 600–800 °C.

3. The wear resistance of Cr3C2-20 wt. % Ni cermet is improved distinctly by adding 1 wt. % Mo, which attributes to the superior hardness of the newly formed (Cr, Mo)7C3 ceramic particle and the lubrication phase of MoO3.

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