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Tribological Properties of Ni_3Al Matrix Composite Sliding Against Si_3N_4, SiC and Al_2O_3 at Elevated Temperatures

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The Ni_3Al matrix self-lubricating composite was fabricated by powder metallurgy technique. The tribological behavior of the composite sliding against commercial Si_3N_4, SiC and Al_2O_3 ceramic balls was investigated from 20 to 1000 °C. It was found that the composite demonstrated excellent lubricating properties with different friction pairs at a wide temperature range, which can be attributed to the synergetic effect of Ag, fluorides, and molybdates formed by oxidations. The Ni_3Al matrix self-lubricating composite/Si_3N_4 couple possessed the stable friction coefficient and wear rate.

Keywords ceramic balls, Ni_3Al matrix composites, self-lubricating, tribological properties

1. Introduction

The tribological properties of materials are different from their mechanical properties, that is to say, they are not inherent characteristics but the combination properties of the practical tribological system of the friction couple. The tribological properties of a definite material rely on the physical and chemical natures of this material itself and the material it coupled, the surface morphology of the couple, the environment conditions (including humility, temperature and the concentration of oxygen), as well as the operation conditions (the contact stress and the sliding speed) (Ref 1-3).

In the last 30 years, the Ni_3Al intermetallic materials have been extensively investigated due to their potential for high-temperature applications. A great deal of work has been done to study their mechanical properties, oxidation, corrosion and tribological properties. However, the tribological properties of Ni_3Al intermetallic materials have not been systematically investigated. Only some work is related to the effect of friction pair on their tribological properties and the lubrication mechanism in different tribo-systems (Ref 4-21, 25-27, 37, 38).

Recently, a series of Ni_3Al matrix high-temperature self-lubricating composites with low friction coefficient and wear rate over a wide temperature range were developed in Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences. In previous work, the effects of solid lubricants (Ag, fluorides, oxides), reinforcement phases (Cr, Mo, W) and technological parameter on tribological behavior of Ni_3Al high-temperature self-lubricating composites were investigated intensively (Ref 4-6, 21, 25-27, 32). To further promote the industrial application, study on the matching principle between Ni_3Al and friction pair is necessary, because different tribo-systems lead to different tribological properties usually.

In the present paper, the influence of tribo-pair on Ni_3Al matrix self-lubricating composite was investigated. One advanced self-lubricating composite Ni_3Al-BaF_2-CaF_2-Ag-Mo (abbreviate to "NBCAM") was revealed by Bi et al. (Ref 21), which is comprised of Ni_3Al matrix phase, Ag and BaF_2/CaF_2 as solid lubricants, and Mo as reinforcement phase (Ref 21, 25-27, 32). At high temperatures, especially at 800 or 1000 °C, ceramics like Si_3N_4, SiC and Al_2O_3 with good high-temperature strength and anti-oxidation ability are the promising materials; thus, they were chosen as the counterpart in the present tribological test. The objective of this paper is to find out the best tribo-coupler with NBCAM self-lubricating composite and clarify the wear mechanisms of NBCAM self-lubricating composite sliding against three kinds of balls in tribological properties test. Therefore, the tribological properties of NBCAM self-lubricating composite sliding against commercial Si_3N_4, SiC and Al_2O_3 ceramic balls from 20 to 1000 °C were studied.

2. Experimental Procedure

The NBCAM self-lubricating composite was fabricated by powder metallurgy technique. Commercially available Ag, Mo, BaF_2, CaF_2 powders and Ni_3Al powders (produced by self-propagating high temperature synthesis in our laboratory) with mean particle size of 30–70 μm were used as the starting materials. The composition of the composite in mass is: Ni_3Al (50%), BaF_2/CaF_2 (20%), Ag (20%) and Mo (10%). The milling operation was carried out in a Fritsch Pulverisette 5 planetary high-energy ball milling system in argon at room temperature. The milling time is 8 h, the ball-to-powder ratio is 2.5:1 (in weight), and the milling speed is 300 rpm. The as-milled powders were put into an hBN-coated graphite die and then pressed at 30 MPa to remove the air in the powders. The furnace was heated at a rate of 10 °C/min when it was

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evacuated to a dynamic vacuum of about 10^{-2} Pa. The powders were pressed at 700 °C and held for 15 min under 70 MPa until heated to 900 °C and then heated to 1100 °C and held for 15 min.

A conventional Vickers indentation tester (Shang Hai Heng Yi Technology Company, China) was used to evaluate the hardness of the NBCAM composite. The hardness is 380 HV with a relative error of about 5%. At the same time, the density was tested using Archimedes' method, and the value is 7.1 g/cm^3.

The tribological tests were conducted on an HT-1000 ball-on-disk high-temperature tribometer. The disk was made of the sintered sample with a size of 18.5 × 18.5 × 5 mm. Before the test, the surfaces of the disk were polished by sandpaper and polishing machine until the roughness (Ra) was about 0.3–0.5 μm, then cleaned with acetone and dried in hot air. The

Table 1 Properties of ceramic balls and NBCAM composite

Material name	Density, g/cm^3	Hardness, HV
Si_3N_4	3.23	1500
SiC	3.12	2800
Al_2O_3	3.92	1650
NBCAM	7.1	380

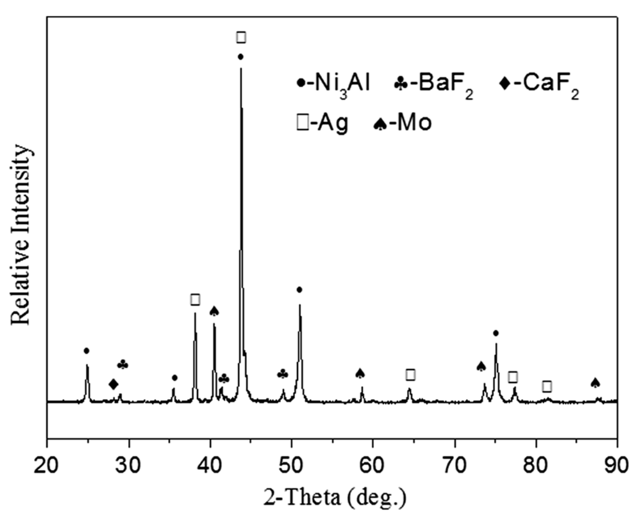

Fig. 1 XRD pattern of the NBCAM composite

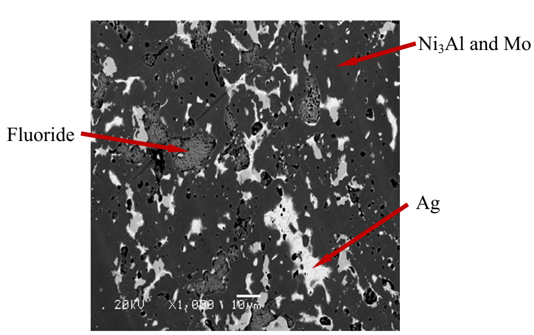

Fig. 2 Distribution of the different phases in the NBCAM composite

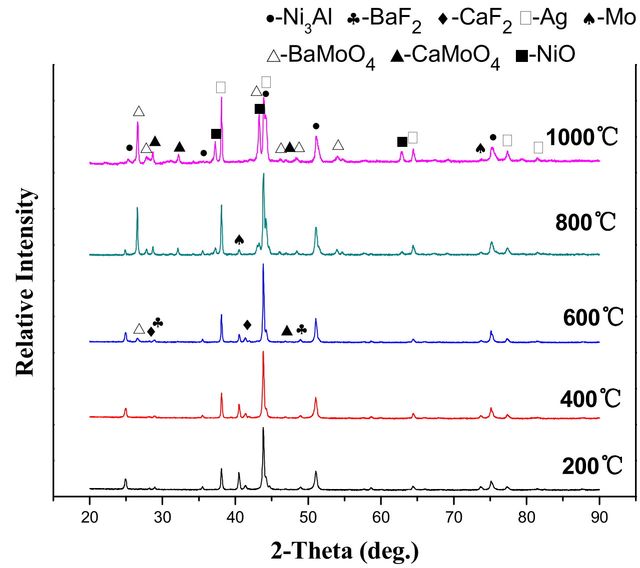

Fig. 3 XRD patterns of worn surfaces of NBCAM composite at different temperatures

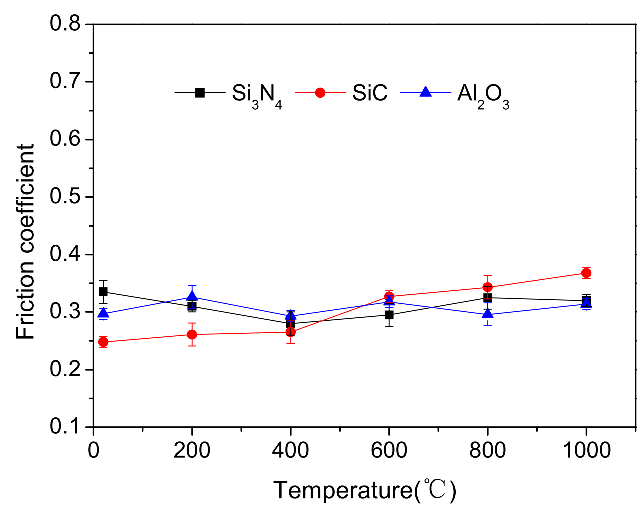

Fig. 4 Variation of friction coefficient of the NBCAM composite at different tested temperatures

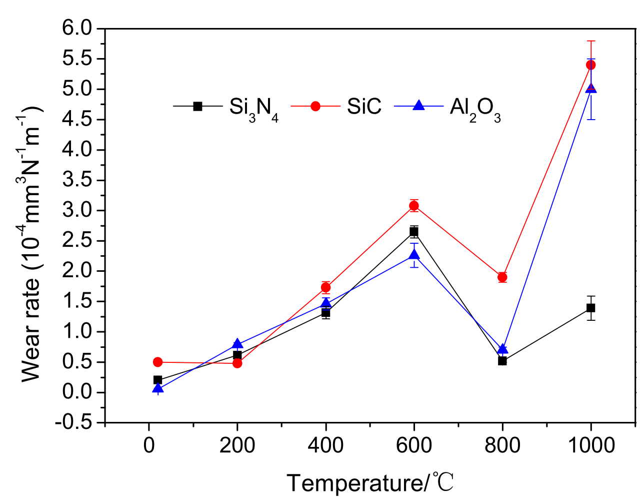

Fig. 5 Variation of wear rates of the NBCAM composite at different tested temperatures

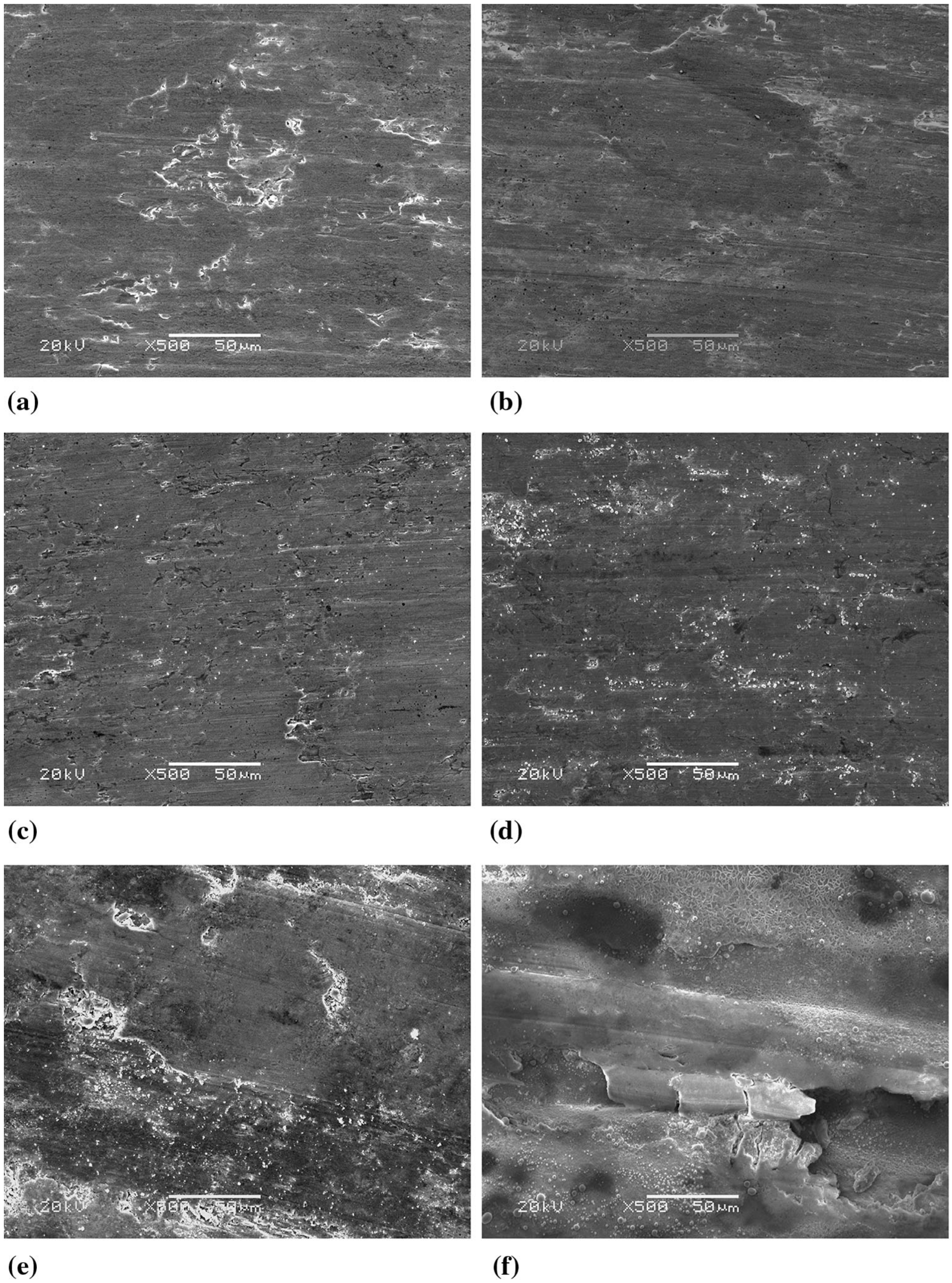

Fig. 6 Worn surfaces of NBCAM in NBCAM/Si_3N_4 tribo-system at different temperatures: 20 °C (a), 200 °C (b), 400 °C (c), 600 °C (d), 800 °C (e) and 1000 °C (f)

counterpart Si_3N_4, SiC and Al_2O_3 balls were commercial ceramic (G 20, Jie Naier Hard Alloy Co. Ltd) with a diameter of 6 mm and the roughness (Ra) of about 0.02 μm, and their properties are presented in Table 1. The selected test temperatures were 20, 200, 400, 600, 800 and 1000 °C. The sliding speed was 0.2 m/s, the applied load was 20 N, and the testing time was 30 min.

The cross-sectional profile of worn surface was measured by a surface profilometer. The wear volume was determined as $V = AL$ and the wear rate as $W = V/SN$, where A was the cross-sectional area of wear track, and for each wear track, four locations were measured to determine the cross-sectional area A, and L was the circumference of the wear track, S was the total sliding distance, and N was the applied load. All the tribological tests were carried out at least three times to make sure the reproducibility of the experimental results under the same condition, and the average results were reported.

The microstructures, morphologies of the worn surfaces and phase structures were examined by JSM-5600LV scanning electron microscope (SEM), energy-dispersive spectroscopy (EDS, Kevex, USA) and x-ray diffraction (XRD, Philips X' Pert-MRD x-ray diffractometer, 40 kV, 30 mA, CuKa radia-

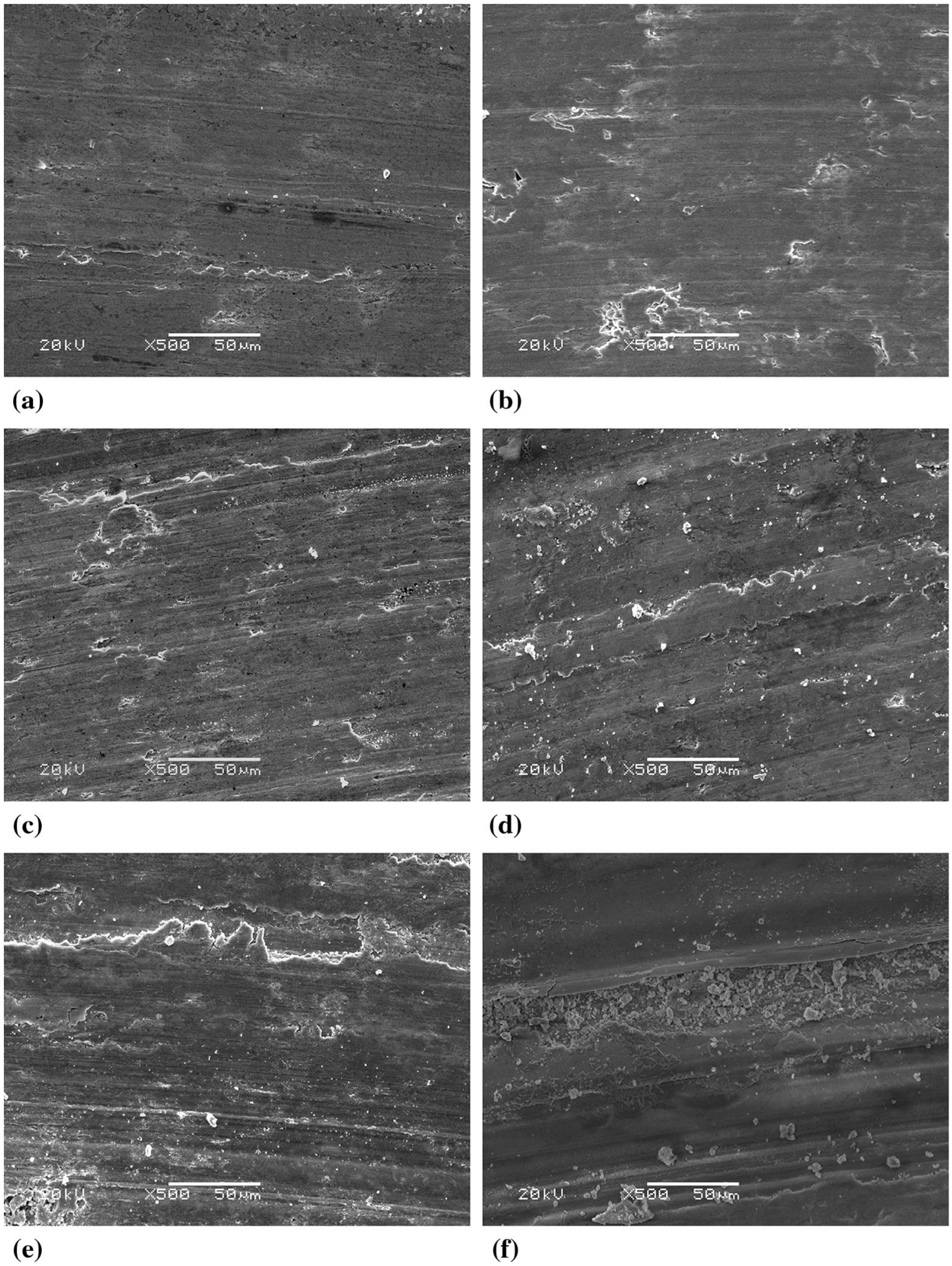

Fig. 7 Worn surfaces of NBCAM in NBCAM/SiC tribo-system at different temperatures: 20 °C (a), 200 °C (b), 400 °C (c), 600 °C (d), 800 °C (e) and 1000 °C (f)

tion). Before SEM observations, samples were cleaned with acetone and then dried in hot air.

3. Results

3.1 Properties, Compositions and Microstructure

From the XRD pattern results (Fig. 1), it can be found that the compositions of NBCAM composite did not change after hot-press sintering. The distribution of the different phases in the NBCAM composite is shown in Fig. 2. EDS analysis indicates that the gray area is the continuous bulk Ni_3Al phase with uniformly dispersed Mo element; the white area is Ag-rich phase, and the deep gray area is fluoride-rich phase. It can be observed that the isolated Ag phase and fluorides phase are surrounded by the continuous bulk Ni_3Al phase.

The XRD patterns of worn surfaces of NBCAM composite at different temperatures are given in Fig. 3. Compared with Fig. 1, it can be found that the compositions of the worn surfaces did not change at 200 and 400 °C. At 600 °C, the peaks of new phases $BaMoO_4$ and $CaMoO_4$ appear in XRD results. As the temperature increase, the peaks of BaF_2 and CaF_2 disappear, but the peaks of $BaMoO_4$ and $CaMoO_4$ are getting stronger and a new phase NiO appears. The presence of $BaMoO_4$, $CaMoO_4$ and NiO on the worn surface is attributed to complex reactions, including high-temperature and tribo-chemical reactions.

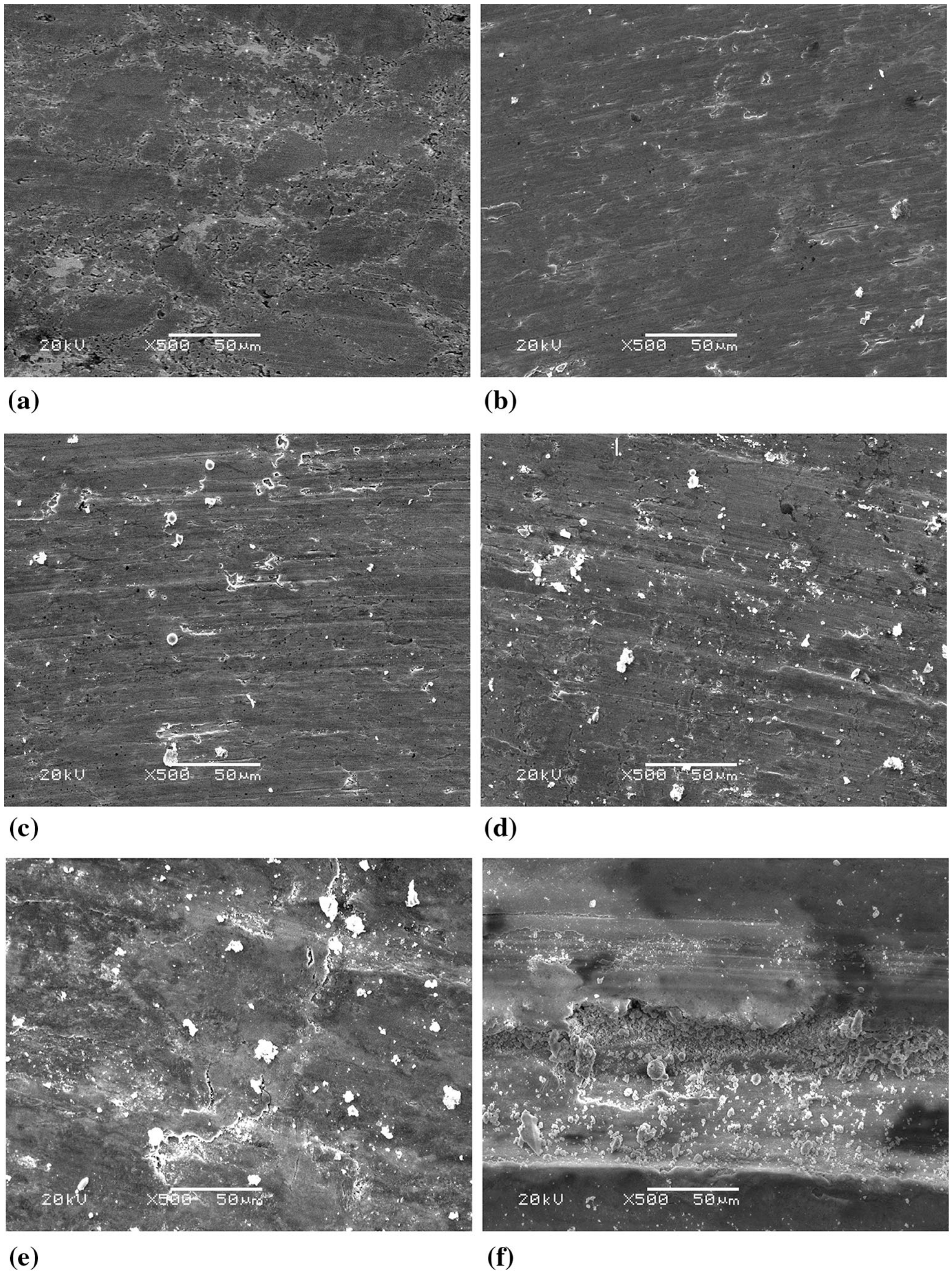

Fig. 8 Worn surfaces of NBCAM in NBCAM/Al_2O_3 tribo-system at different temperatures: 20 °C (a), 200 °C (b), 400 °C (c), 600 °C (d), 800 °C (e) and 1000 °C (f)

3.2 Tribological Behavior

The friction coefficients of NBCAM composite against all the three ceramic balls are given in Fig. 4. It can be observed that NBCAM composite sliding against all the three ceramic balls showed low friction coefficients from 20 to 1000 °C. In addition, the friction coefficients of NBCAM/Si_3N_4 and NBCAM/Al_2O_3 tribo-systems are 0.31, which is more stable than that of NBCAM/SiC tribo-system in the whole temperature range. The friction coefficients of NBCAM/SiC tribo-system increased along with the rise of the temperature, the lowest friction coefficient is 0.25 at 20 °C, and the highest friction coefficient is 0.37 at 1000 °C.

The wear rates of NBCAM composite sliding against the three ceramic balls are given in Fig. 5. It can be seen that the composite has similar development trend of wear rate from 20 to 1000 °C. Below 600 °C, NBCAM composite has the lowest wear rate [0.07×10^{-4} $mm^3(N^{-1}\ m^{-1})$] at 20 °C, and the wear rate tends to increase as the temperature increases. At the same time, the wear rate of NBCAM composite shows obvious differences in three tribo-systems when the temperature is >600 °C (especially at 800 and 1000 °C), and NBCAM has the lowest wear rates in NBCAM/Si_3N_4 tribo-system at 800 and 1000 °C (0.52×10^{-4} and 1.39×10^{-4} $mm^3(N^{-1}\ m^{-1})$, respectively).

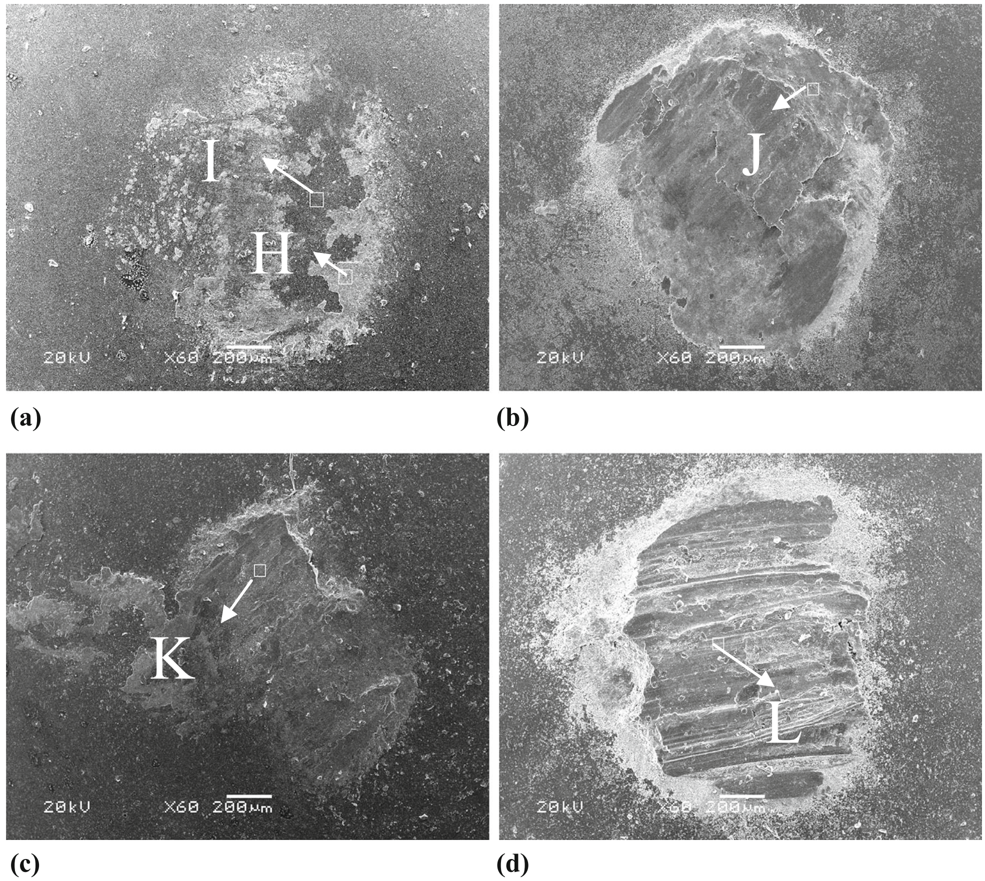

Fig. 9 Worn surfaces of ceramic balls: Si_3N_4 balls at 800 °C (a) and 1000 °C (b), SiC balls at 800 °C (c) and 1000 °C (d)

Figure 6 shows the worn surfaces of NBCAM in NBCAM/Si_3N_4 tribo-system at different temperatures. Some fine grooves and flakes on the smooth worn surface in Fig. 6(a) suggest that the wear mechanism is microcutting at 20 °C, and obviously, it has slight differences compared with NBCAM/SiC (Fig. 7a) and NBCAM/Al_2O_3 (Fig. 8a) tribo-systems. When the temperature increases to 200 and 400 °C in each system, local deformations in some spots on the worn surface suggest the wear mechanisms are microploughing and stripping in three tribo-systems, and NBCAM has the most smooth worn surface after sliding against Si_3N_4 ceramic ball (see Fig. 6b and c). When the temperature rises to 600 °C, although wear rates have increased, the fine and loose wear debris on the worn surface illustrates that the wear mechanisms are still microploughing and stripping in NBCAM/Si_3N_4 (Fig. 6d) tribo-system. In addition, larger wear debris and traces can be found on the worn surfaces in NBCAM/SiC (Fig. 7d) and NBCAM/Al_2O_3 (Fig. 8d) tribo-systems. At 800 °C, the deformed surface and grooves are present on the worn surfaces in three tribo-systems, and glaze film is formed on the worn surface, which is composed of NiO, $BaMoO_4$ and $CaMoO_4$ by XRD analysis, and the dominant wear mechanism is surface deformation. When the temperature further rises to 1000 °C, the coarse grooves break down on the smooth surface of NBCAM sliding against Si_3N_4 (Fig. 6f), whereas severe plow grooves and delaminated pits are found on the worn surfaces of NBCAM sliding against SiC (Fig. 7f) and Al_2O_3 (Fig. 8f). And the XRD results showed that large numbers of oxides consisted of $BaMoO_4$, $CaMoO_4$ and NiO appeared on the worn surfaces, and under this condition, the wear mechanisms are mainly microploughing and oxidative wear.

4. Discussion

The above-mentioned results showed that the NBCAM composite can provide excellent lubricating properties with different friction pairs over a wide temperature range and friction pairs have some influence on friction coefficient and wear rate. By this token, the tribological property of NBCAM composite is closely related to its friction pair for sliding against Si_3N_4, SiC and Al_2O_3 ceramic balls from 20 to 1000 °C.

The NBCAM self-lubricating composite shows good lubricating property from 20 to 1000 °C in different tribo-systems. It could be attributed to the favorable high-temperature properties of Ni_3Al matrix and the co-work action of Ag (act as lubricant at relative low temperature below 450 °C) (Ref 21-23), fluorides (act as lubricant above 400 °C) (Ref 24) and molybdates formed by the complex reaction (as high-temperature solid lubricants) (Ref 25, 26). In addition, some different friction phenomenon happened after tribological test in three tribo-systems. The reason is briefly showed as following.

Figure 3 shows that the friction coefficients in NBCAM/Si_3N_4 and NBCAM/Al_2O_3 tribo-systems are more stable with excellent performances than that in NBCAM/SiC tribo-system from 20 to 1000 °C, although NBCAM/SiC tribo-system exhibits slightly lower friction coefficients under 400 °C. At

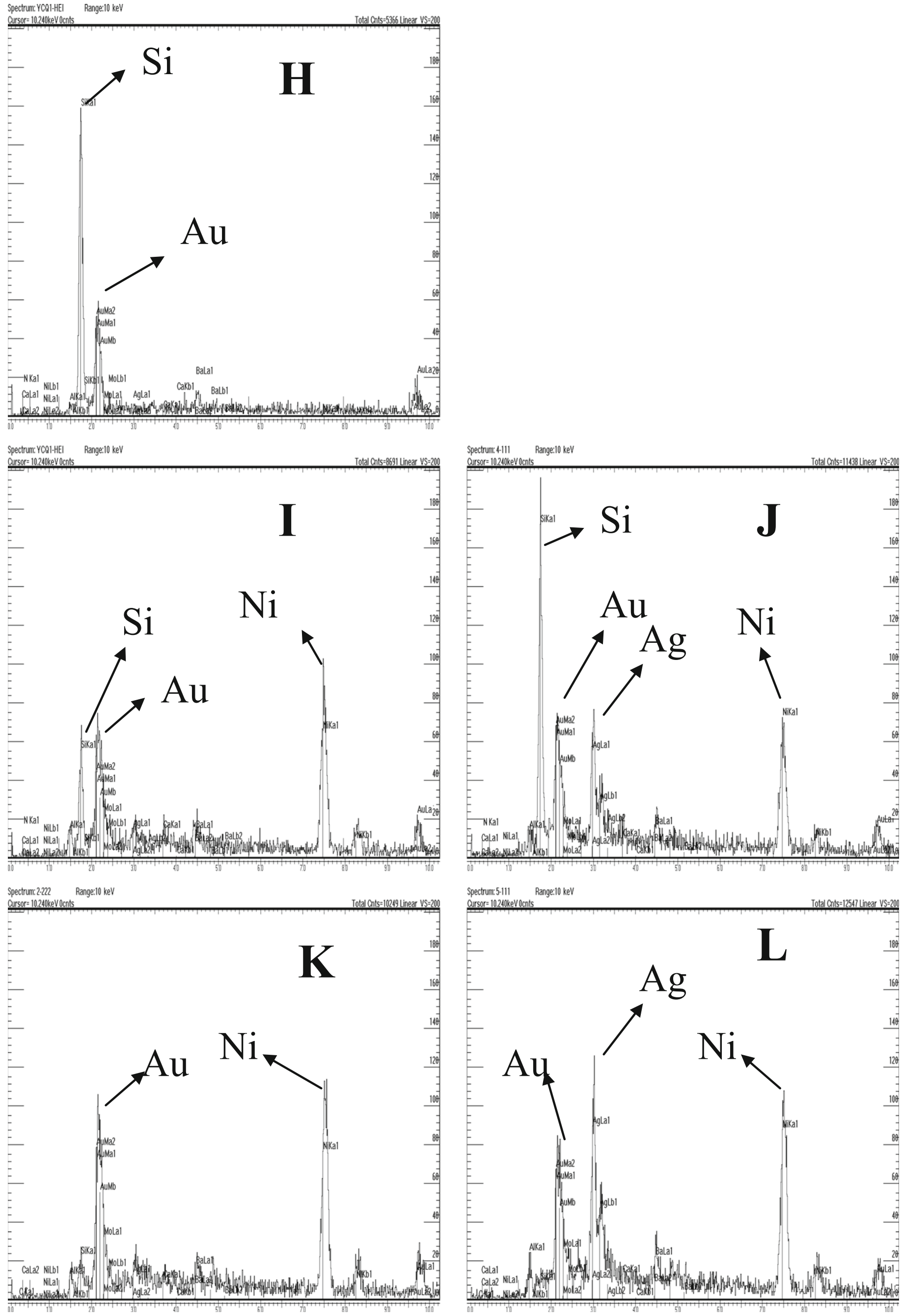

Fig. 10 EDS results of the marked areas on the worn surfaces

low temperatures (from 20 to 400 °C), the soft and coarse Ag particles on the sliding surface of NBCAM were scratched and squeezed gradually by the ceramic balls and then spread on the sliding surface and formed a Ag-rich film on the worn surface to provide effective lubrication (Ref 27-29). In addition, continuous Ag-rich film attached on the worn surface in NBCAM/SiC tribo-system than other tribo-systems, this is because SiC ball has the highest hardness (see Table 1), which could separate out Ag effectively to reduce friction coefficient slightly during 20 to 400 °C (Ref 36). At high temperatures (above 600 °C), the high-temperature lubricant of fluorides and molybdates (formed during the sliding process) provide lubrication and make friction coefficient lower. And as the temperature rose further, a large number of molybdates and oxides formed as the glaze film to further reduce the friction coefficient at 800 and 1000 °C (Ref 30-32).

With respect to wear behavior, the following explanations are given to clarify the obtained results. NBCAM has the same wear behavior trend in three tribo-systems. Below 600 °C, strength of the monolithic Ni_3Al increased with the increase in the temperature (Ref 33). In addition, Ag-rich and Mo-rich phases gathered to be lumps which is different from fluorides scattered on Ni_3Al (see Fig. 2), so the Ag and molybdates in the Ni_3Al framework are not well precipitated as the temperature rose to 600 °C (Ref 34-38). However, the fluorides appeared and showed the important role of lubrication with the destruction of the matrix (the hard particles on the worn surface (see Fig. 6d, 7d, 8d), so the friction coefficient is stable after the temperature increased to 600 °C. But the strength of NBCAM decreases with the increase in the temperature, so the wear rate increases and it has the highest wear rate at 600 °C.

At 800 °C, the formation of the glaze film by NiO, $BaMoO_4$ and $CaMoO_4$ composition effectively reduces wear rates in three tribo-systems. As shown in Fig. 9(a) and (c), 10(H), (I) and (K), Ni element appeared on the Si_3N_4 and SiC balls' worn surface, which suggests that some NBCAM transferred on ceramic balls during the friction process. As shown in Fig. 10(K), the Si element disappearing on SiC ball indicates that more content of transfer film appeared on the worn surface of SiC ball, as can be seen in Fig. 10(I) and smaller content of transfer film appeared on the worn surface of Si_3N_4 ball, so NBCAM composite has lower wear rate in NBCAM/Si_3N_4 tribo-system. As the temperature rose to 1000 °C, the strength of the NBCAM decreased leading to the degradation of the wear resistance. Smaller content transfer film appeared on the worn surface of Si_3N_4 ball, which is similar to that at 800 °C (see Fig. 9(b) and (d), 10(J) and (L)), so NBCAM composite has the lowest wear rate in NBCAM/Si_3N_4 tribo-system at 1000 °C.

5. Conclusions

(1) At a wide temperature range from 20 to 1000 °C, the Ni_3Al matrix self-lubricating composites demonstrate good lubricating properties with different friction pairs, which can be attributed to the co-work action of Ag, fluorides and molybdates formed by the complex reaction and oxides.
(2) The Ni_3Al matrix self-lubricating composite shows the lowest friction coefficient in the range of 20 °C–400 °C when sliding against SiC ceramic ball.
(3) The Ni_3Al matrix self-lubricating composite shows the lowest wear rate at 800 and 1000 °C when sliding against Si_3N_4 ceramic ball.

Acknowledgments

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