

# Friction and Wear Properties of Spark Plasma Sintering NiCr–SrSO<sub>4</sub> Composites at Elevated Temperatures in Sliding Against Alumina Ball

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**Abstract** Nickel-based self-lubricating composites with SrSO<sub>4</sub> as a high-temperature solid lubricant have been prepared by spark plasma sintering method. Their friction and wear properties were evaluated using a high-temperature friction and wear tester in dry sliding against alumina ball up to 600 °C. NiCr–10 wt% SrSO<sub>4</sub> composite exhibits satisfactory mechanical properties and tribological properties from room temperature to 600 °C (except for 200 °C) as contrasted with unmodified Ni–Cr alloy. The friction coefficients of NiCr–10 wt% SrSO<sub>4</sub> at 400 and 600 °C are 0.32 and 0.20, respectively. The corresponding wear rates are  $1.44 \times 10^{-5}$  and  $2.4 \times 10^{-5}$  mm<sup>3</sup>/(Nm). The unmodified Ni–Cr alloy exhibits friction coefficients of 0.41 and wear rates in order of  $10^{-4}$  mm<sup>3</sup>/(Nm) in a temperature range of 400–600 °C. At elevated temperatures, a dense and protective glaze layer is generated on the worn surface of composites. The glaze layer was then investigated by means of scanning electron microscopy and XPS. Tribo-chemical reaction during high-temperature wear tests is responsible for the formation of the protective glaze layer. The newly formed oxides (NiO and Cr<sub>2</sub>O<sub>3</sub>) and some self-lubricating oxysalts (SrSO<sub>4</sub> and SrCrO<sub>4</sub>) can promote the densification of oxide glaze layer, and finally generate a dense lubricating film to effectively reduce friction and wear at elevated temperatures.

**Keywords** NiCr alloy · SrSO<sub>4</sub> · Self-lubricating composites · High temperature

## 1 Introduction

With the advent of science and technology, the demand for developing high-temperature structural materials to meet the severe conditions of mechanical systems such as higher temperatures and higher applied load is ever increasing [1]. Ni-based alloys have been widely used in some industrial applications like power generation, material processing and other components suitable for high-temperature structural materials in the harsh environment due to their high strength, excellent oxidation resistance and corrosion resistance ability at elevated temperatures [2]. Ni–Nb–Fe–Se and Ni–Mo–S high-temperature alloys with excellent tribological properties at room temperature were prepared by incorporating S(Se) and Nb, Mo, W, etc., into nickel-based alloy, but their high-temperature tribological properties are very poor [3, 4]. Ni–Cu–Re alloy has been proved to be very promising high-temperature material over 500–800 °C [5]. A layer of oxide film composed of soft-phase oxides such as Re<sub>2</sub>O<sub>7</sub> and complex oxides such as CuO·Re<sub>2</sub>O<sub>7</sub> was found to form on the contact surface to improve the tribological properties of this material. However, as the adhesion between the oxide films formed on the contact surfaces and the matrix is very poor, which would cause the films to delaminate from the matrix and could not work effectively at elevated temperatures. Though many enforcement elements (Cr, W, Mo, Al, etc.) and enforcement phases (rare earth compound La<sub>2</sub>O<sub>3</sub>, etc.) are often added into nickel-based alloys to obtain materials with excellent overall properties, their tribological

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properties at elevated temperatures are still very poor, which has restrained their applications to a large extent.

Recently, research on self-lubricating Ni-based high-temperature composites has been initiated as a response to the requirements for improving its poor tribological properties at extreme temperatures [6]. Ni-based high-temperature composites not only possess high strength, high hardness and excellent oxidation resistance ability of the matrix, but also exhibit good self-lubricating properties. In order to achieve and maintain low friction coefficients and wear rates at high temperatures, different kinds of solid lubricants were incorporated into Ni-based alloy to develop Ni-based high-temperature composites, but their applications were limited by the temperature and oxidation at high temperatures. Conventional solid lubricants like MoS<sub>2</sub> and graphite are easily oxidized, while some soft metals such as Ag will easily melt and lose lubrication at high temperatures [7–10]. In order to find effective high-temperature solid lubricants, a large volume of work was done on various materials like soft metals, metal oxides, fluorides, oxythiomolybdates and some oxysalts [11–13]. The high-temperature lubricious behavior of some oxysalts like sulfates (BaSO<sub>4</sub> and SrSO<sub>4</sub>) and chromates (BaCr<sub>2</sub>O<sub>4</sub> and BaCrO<sub>4</sub>) was discussed and the results indicated that they were all promising high-temperature solid lubricants. Among all these oxysalts, nanoscale SrSO<sub>4</sub> tends to be thermally stable during high temperatures and can significantly improve the tribological properties of the composites over a wide temperature range. Ceramic matrix composites such as Al<sub>2</sub>O<sub>3</sub>–SrSO<sub>4</sub> and TZ3Y20A–SrSO<sub>4</sub> presented outstanding tribological properties at elevated temperatures. The self-lubricating film mainly consisting of SrSO<sub>4</sub> on the worn surface was responsible for reducing friction and wear above 400 °C [14–19].

In this paper, Nickel-based self-lubricating composites incorporated with SrSO<sub>4</sub> were fabricated by powder metallurgy. Further studies were carried out to provide a comprehensive research of the mechanical properties and tribological properties of Ni-based composites with addition of SrSO<sub>4</sub> from room temperature to 600 °C, as well as to explore the self-lubricating mechanism of the composites at elevated temperatures.

## 2 Experimental Procedure

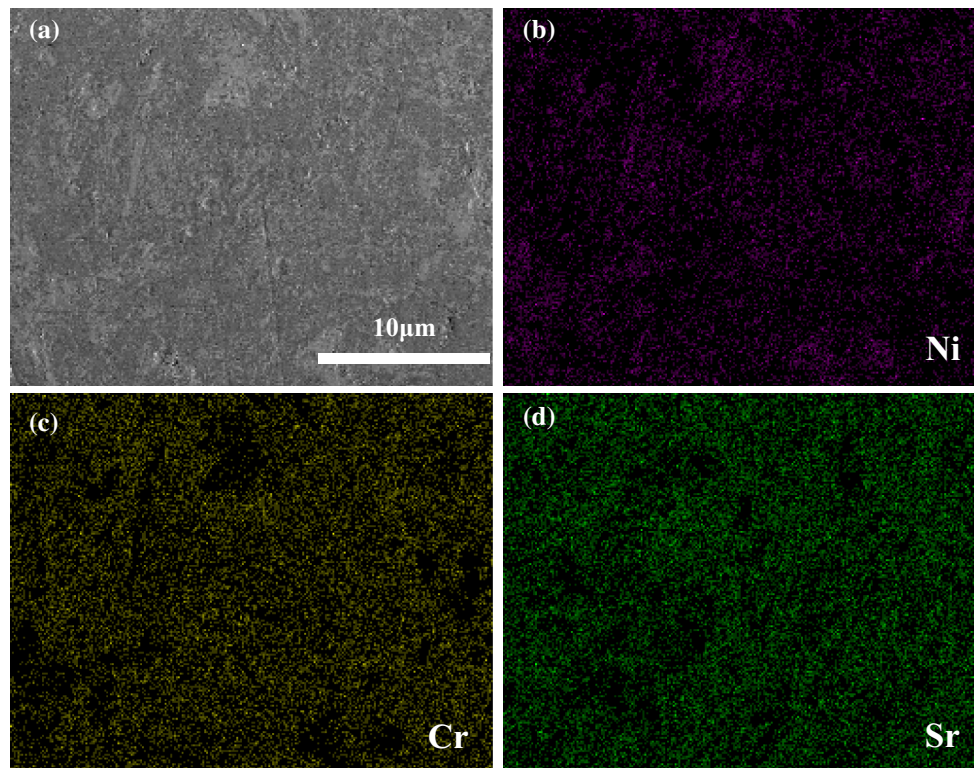
In this research, nanoscale SrSO<sub>4</sub> powders with average size of 50–100 nm were prepared by chemical precipitation process using analytically pure SrCl<sub>2</sub>·6H<sub>2</sub>O, Na<sub>2</sub>SO<sub>4</sub> and ethanol at the condition of room temperature and atmospheric pressure [20]. Commercially, Ni (1–5 μm) and Cr (50–100 μm) metallic powders were used as the starting materials. The Ni-based self-lubricating composites with addition of different amount of SrSO<sub>4</sub> were fabricated by spark plasma sintering (SPS) method. The compositions of the as-sintered samples were listed in Table 1. The Ni, Cr and SrSO<sub>4</sub> powders were primarily mixed in setting ratios by weight and then continuously ball-milled for 20 h in a planetary ball mill device. The ratio of ball to powders in weight was 10:1. Afterward, the mixed powders were enclosed and cold compacted in a graphite mold (inner diameter of 20 mm) at a pressure of 30 MPa. Then, the pressed specimens were sintered at 1050 °C for 5 min under the pressure of 30 MPa in a SPS furnace at a dynamic vacuum of 10<sup>−2</sup> Pa. The as-sintered specimens were machined and polished for further analyses and tests.

The mechanical properties of the composites were examined after surface treatment. The densities of the sintered specimens were measured using JA5003B electronic balance (Shanghai Jingke Techcomp Instrument Corporation, China) with density measurement component. The hardness measurement was carried out on a HV-5 Vickers hardness tester (Lai Zhou Testing Machine General Factory Corporation, China) with a normal load of 29.4 N and a dwell time of 15 s. Each test was repeated at least ten times and the mean value of different composites was given in Table 1.

The friction and wear tests of the composites were performed on a HT-1000 ball-on-disk friction and wear tester (Zhong Ke Kai Hua Corporation, China) in dry sliding against Al<sub>2</sub>O<sub>3</sub> ball with a diameter of 10 mm. The applied Al<sub>2</sub>O<sub>3</sub> ball has a surface roughness of 0.02 μm (*Ra*), a hardness of 18 GPa and a density of 3.95 g/cm<sup>3</sup>. Each sample was cleaned in a mixture of 50 vol% acetone and 50 vol% petroleum benzene for 10 min using an ultrasonic cleaning device before wear tests. According to our preliminary research and the Refs. [15, 17–19], the

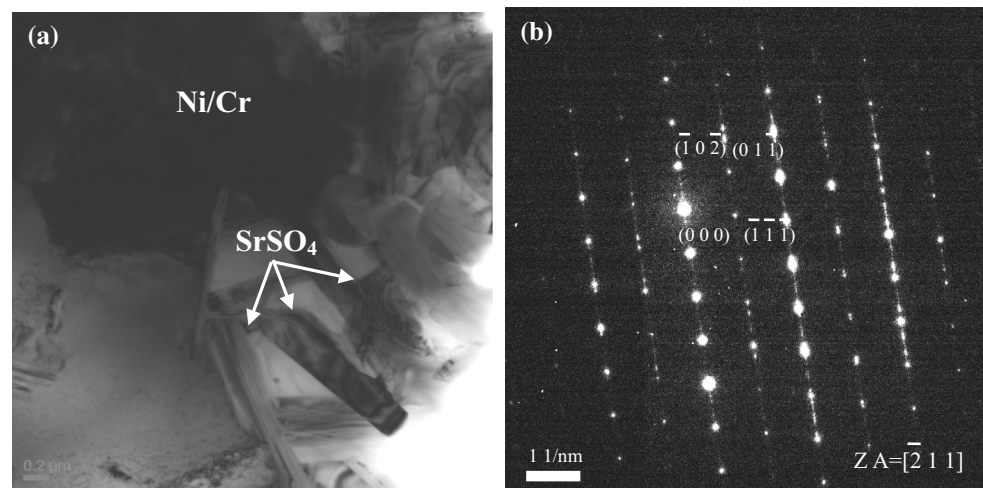
**Table 1** Compositions, densities and hardness of as-sintered samples

Sample	Compositions (wt%)	Densities (g/cm <sup>3</sup> )	Hardness (GPa)
NC	80Ni + 20Cr	8.39	2.61
NC-10S	72Ni + 18Cr + 10SrSO <sub>4</sub>	7.47	2.74
NC-20S	64Ni + 16Cr + 20SrSO <sub>4</sub>	6.44	2.56
NC-30S	56Ni + 14Cr + 30SrSO <sub>4</sub>	5.71	2.05



**Fig. 1** SEM micrograph and typical element distribution mappings of Ni, Cr and Sr in the NC-30S composite: **a** SEM micrograph, **b** Ni element distribution, **c** Cr element distribution, **d** Sr element distribution

**Fig. 2** TEM micrograph of the NC-30S composite and the corresponding  $[-2\ 1\ 1]$  zone axis electron diffraction pattern of  $\text{SrSO}_4$ : **a** TEM micrograph, **b** SAED pattern at the  $[-2\ 1\ 1]$  zone axis

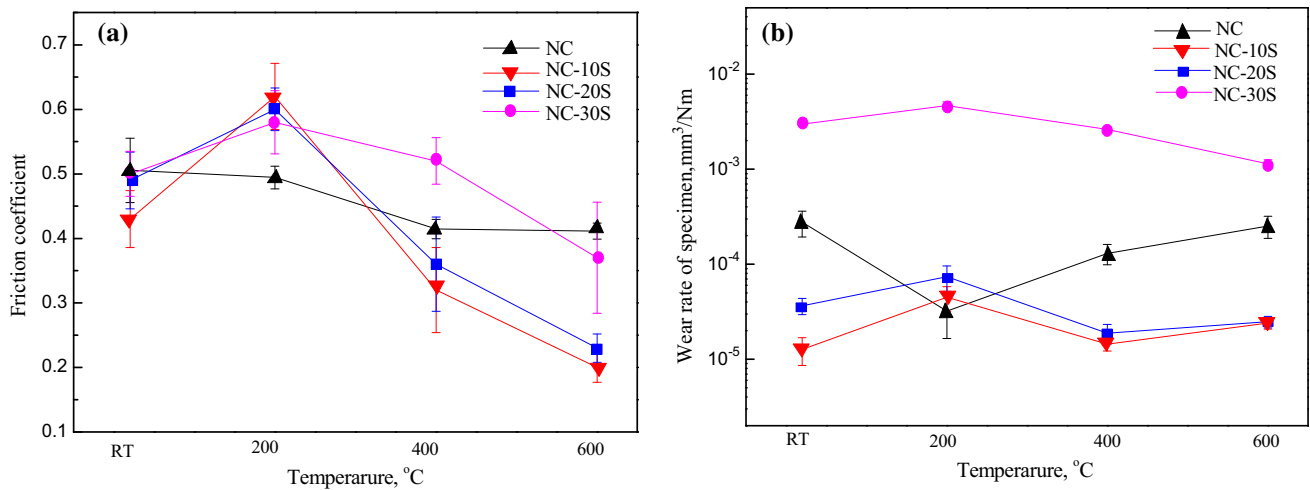


$\text{SrSO}_4$  plays well lubrication at low load and speed. Thus, the applied load and the sliding velocity were fixed at 5 N and 0.08 m/s, and there were four testing temperature in air, namely, room temperature, 200, 400 and 600 °C. It was noted that the produced wear track after 60 min test had a diameter of 6 mm in the diameter. The tests at room temperature were carried out in laboratory air with a relative humidity of 40 %. In respect of high-temperature wear tests, each specimen was mounted in a holder which was heated by resistance heating to the testing temperature.

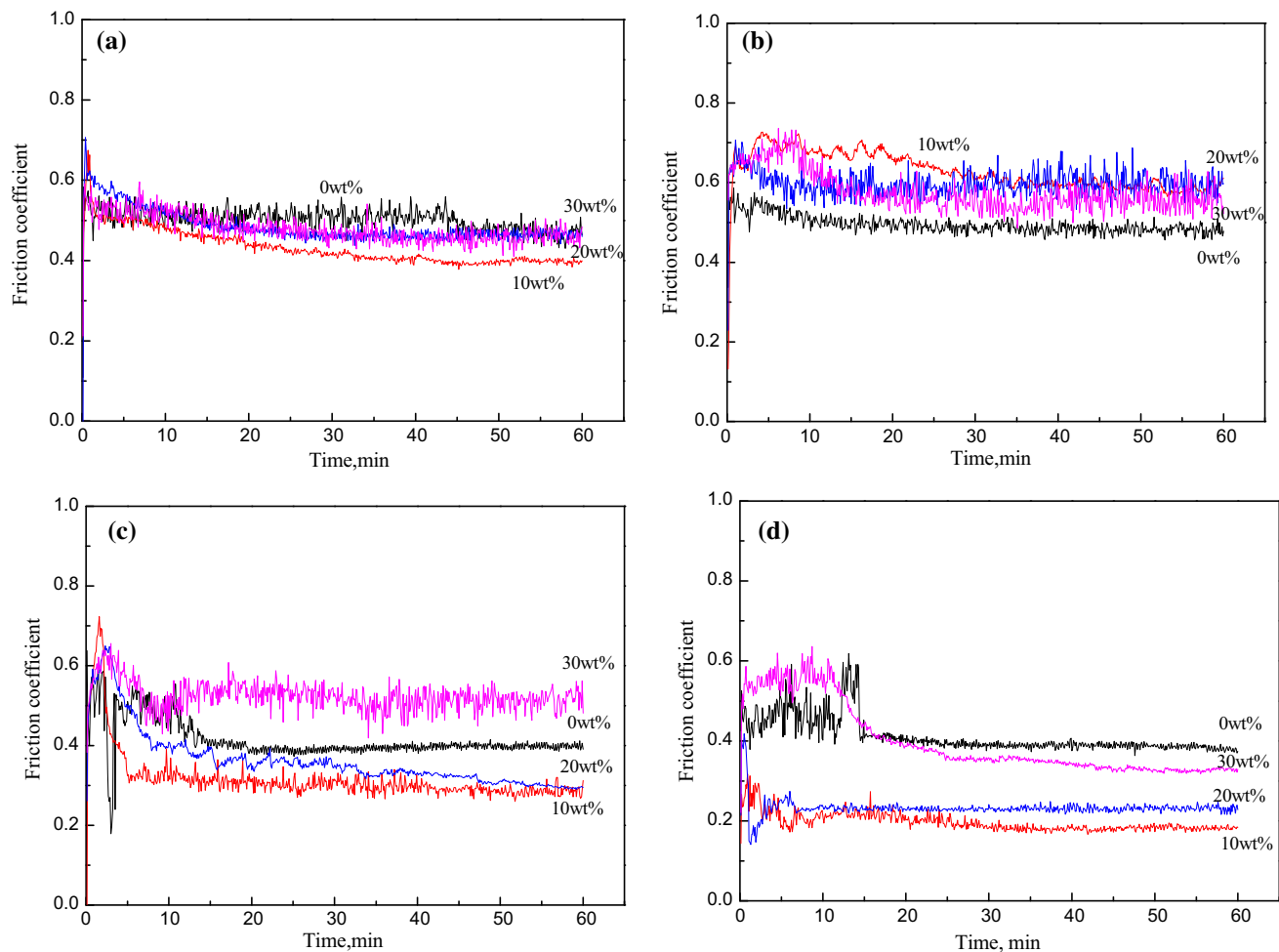
After wear testing, the cross-sectional profiles of all the wear tracks were examined by a surface profilometer to measure the wear volume of the composites. Therefore, the wear rate ( $W$ ) was calculated as:

$$W = AL/SN \quad (1)$$

where  $A$  was the cross-sectional area of the wear track,  $L$  was the length of the wear track,  $S$  was the sliding distance and  $N$  was the applied load. Each test had been repeated three times to make sure the reliability of the data,



**Fig. 3** Tribological properties of the sintered Ni–Cr alloy incorporated with or without different SrSO<sub>4</sub> contents as a function of test temperature: **a** friction coefficient, **b** wear rate



**Fig. 4** Friction coefficients of the sintered Ni–Cr alloy incorporated with or without different SrSO<sub>4</sub> contents as a function of testing time at various test temperatures: **a** room temperature, **b** 200 °C, **c** 400 °C, **d** 600 °C

and the friction coefficient and wear rate reported in this paper were the average value of the three times.

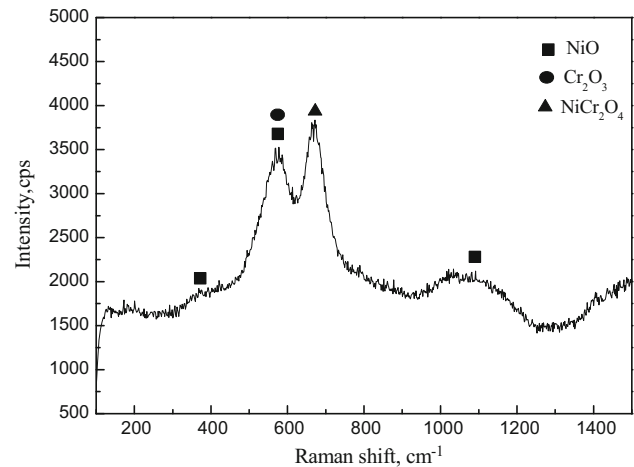
The morphology and the chemical compositions of the worn surfaces on each sample after wear tests were investigated by Hitachi S-4700 scanning electron microscope (SEM) equipped with energy-dispersive X-ray spectrometers (EDS) analyzer. The wear tracks were analyzed using a laser Raman spectroscopy and a X-ray photoelectron spectroscopy to identify the variations of phase compositions on the worn surfaces after wear tests.

### 3 Results

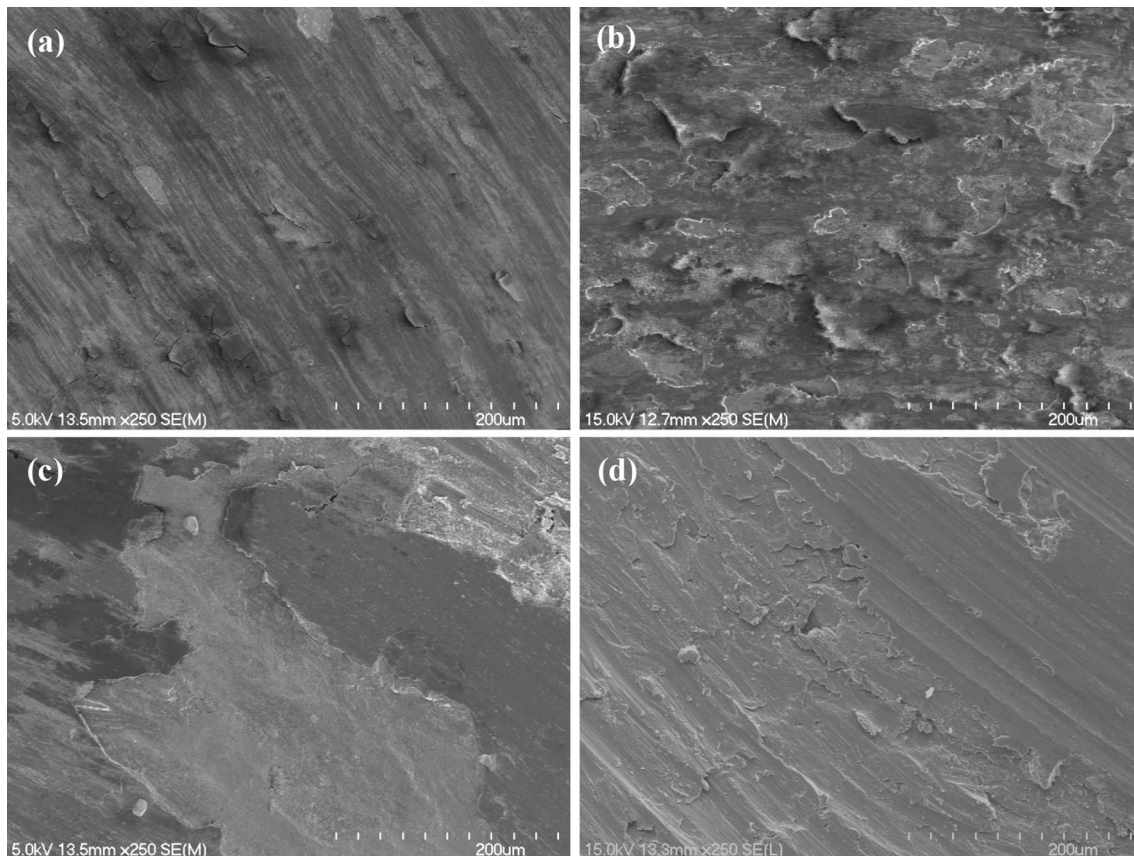
Table 1 shows the compositions and mechanical properties of the sintered Ni–Cr alloy and NiCr–SrSO<sub>4</sub> composites. The Ni–Cr alloy has a density of 8.39 g/cm<sup>3</sup> and an average hardness of 2.61 GPa. It can be seen that the densities and average hardness of the composites would deteriorate with the addition of SrSO<sub>4</sub> which is a kind of soft phase with low density and hardness as compared with the Ni–Cr alloy. When the adding amount of SrSO<sub>4</sub> is 30 wt%, the density

and hardness of the composite decrease into 5.71 g/cm<sup>3</sup> and 2.05 GPa, relatively.

Figure 1 shows the SEM micrograph and typical element distribution mappings of Ni, Cr and Sr in the NC-30S



**Fig. 6** Raman spectrum of the worn surface of Ni–Cr alloy after wear tests at 600 °C



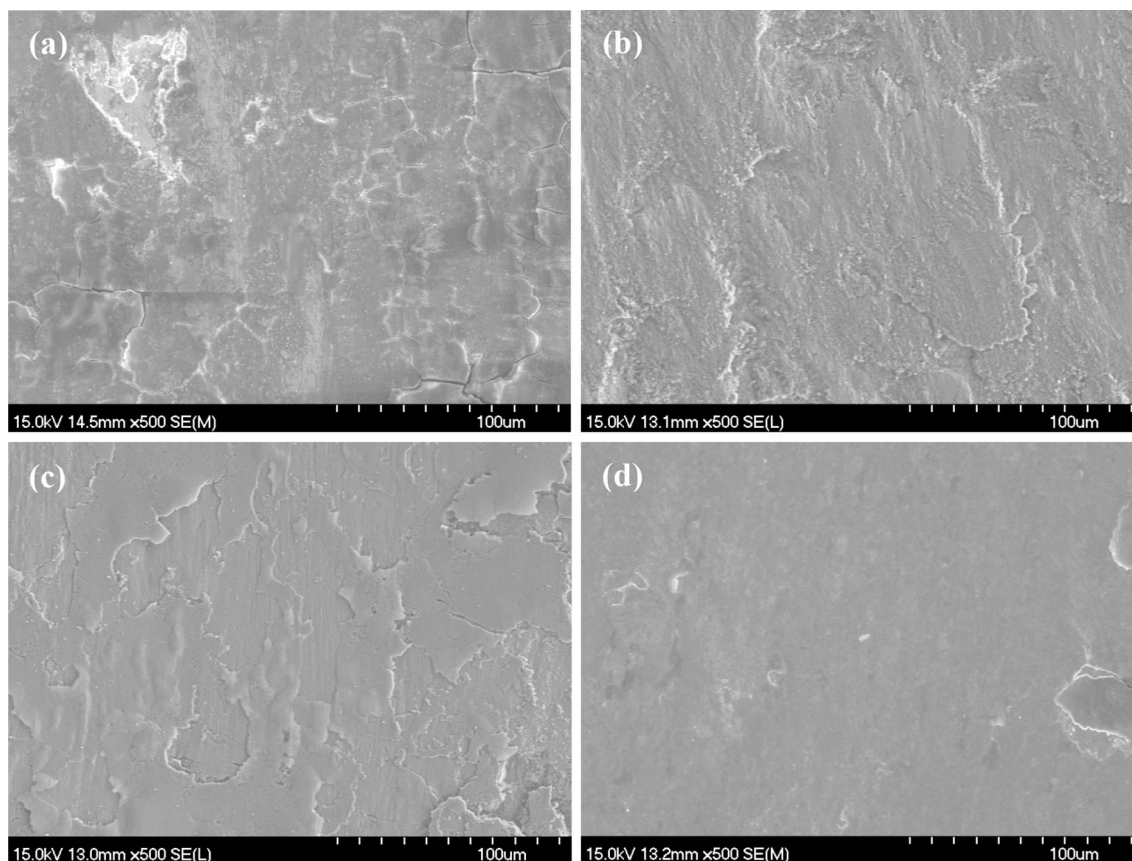
**Fig. 5** Surface morphology of worn surfaces of Ni–Cr alloy after wear tests at different temperatures: **a** room temperature, **b** 200 °C, **c** 400 °C, **d** 600 °C

composite. From Fig. 1d, the result indicates that  $\text{SrSO}_4$  particles distribute uniformly in the Ni–Cr metallic matrix. In order to observe the structure of  $\text{SrSO}_4$  in the composites, the Ni-based composite with addition of 30 wt%  $\text{SrSO}_4$  was characterized by transmission electron microscopy (TEM) and selected area electron diffraction (SAED) as shown in Fig. 2. The presence of  $\text{SrSO}_4$  in the composite was confirmed through the typical  $[-2\ 1\ 1]$  zone axis of  $\text{SrSO}_4$  in the SAED pattern taken from the marked region in Fig. 2a.

The tribological properties of the sintered Ni–Cr alloy incorporated with or without different  $\text{SrSO}_4$  contents as a function of test temperature are shown in Fig. 3. From Fig. 3a, the friction coefficient of Ni–Cr alloy is 0.51 at room temperature and decreases to 0.41 at 600 °C. The wear rates of Ni–Cr alloy, as shown in Fig. 3b, exhibit very high value in order of  $10^{-4}$   $\text{mm}^3/(\text{Nm})$  at room temperature, 400 and 600 °C. Clearly, the friction and wear properties of Ni–Cr alloy at a wide temperature range are poor, from room temperature to 600 °C. When 10 wt%  $\text{SrSO}_4$  is added into the Ni–Cr metallic matrix, the improvement of tribological properties is the most significant. The friction coefficients of NC-10S composite at

room temperature, 400 and 600 °C decrease to 0.43, 0.32 and 0.20, respectively. The corresponding wear rates are  $1.27 \times 10^{-5}$ ,  $1.44 \times 10^{-5}$  and  $2.4 \times 10^{-5}$   $\text{mm}^3/(\text{Nm})$ . It can be seen that the NC-10S composite exhibits great lower friction coefficients and wear rates from room temperature to 600 °C (except for 200 °C) as contrasted with unmodified Ni–Cr alloy. However, the increasing adding amount of  $\text{SrSO}_4$  would deteriorate the tribological properties of Ni–Cr– $\text{SrSO}_4$  composites as shown in Fig. 3. The friction coefficients of NC-30S composite at 200 and 400 °C are 0.58 and 0.53, obviously higher than those of Ni–Cr alloy. Meanwhile, it is evident that when the adding amount of  $\text{SrSO}_4$  is 30 wt%, the wear rates of the composite distinctively increase, reaching the order of  $10^{-3}$   $\text{mm}^3/(\text{Nm})$  at a wide temperature range from room temperature to 600 °C.

Figure 4 shows the friction coefficients of the sintered Ni–Cr alloy incorporated with or without different  $\text{SrSO}_4$  contents as a function of testing time at various test temperatures. It can be clearly seen that the variation of friction coefficient of each material with testing time corresponds with the varying trend of its average friction coefficient. The variation in the friction coefficient of Ni–Cr alloy



**Fig. 7** Surface morphology of worn surfaces of NC-10S composite after wear tests at different temperatures: **a** room temperature, **b** 200 °C, **c** 400 °C, **d** 600 °C

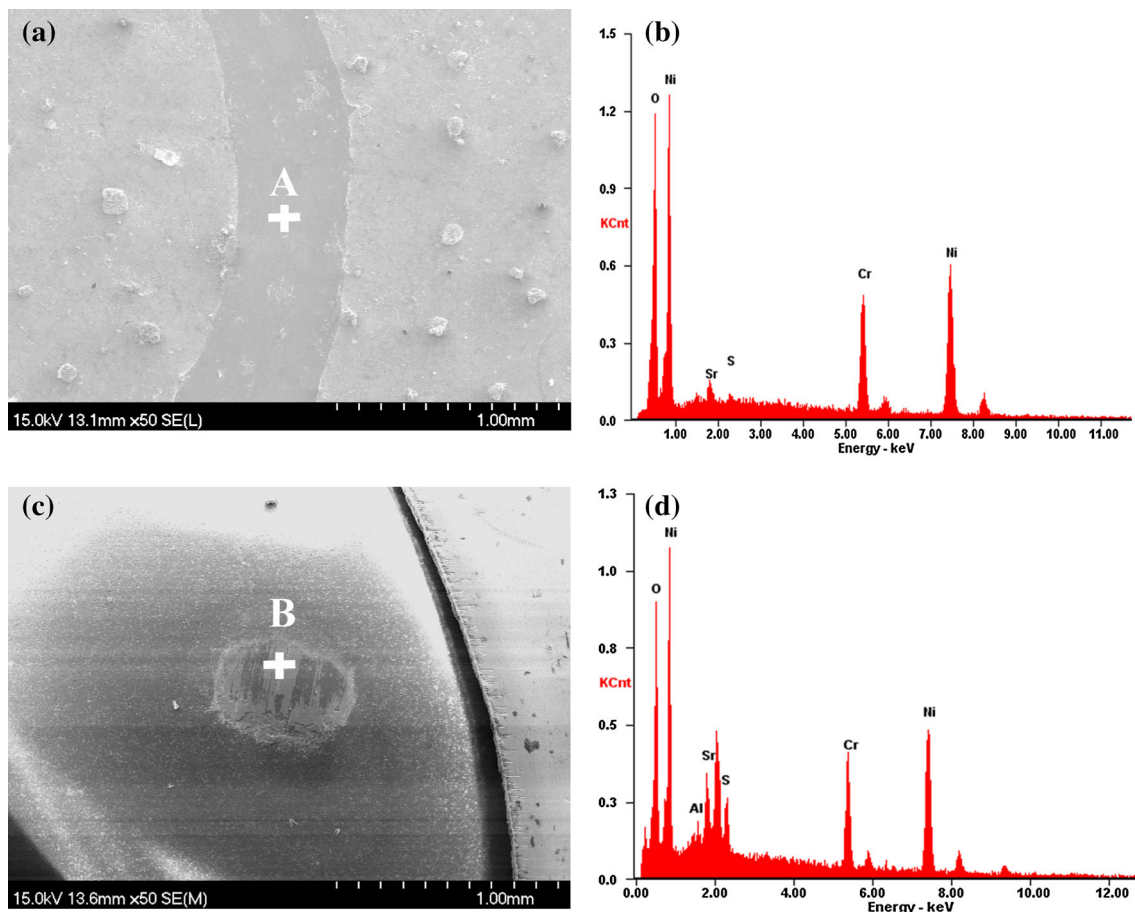
tends to be more and more stable with the increase in testing temperature, while that of NiCr–SrSO<sub>4</sub> composites at 200 °C becomes unsteady and presents large variation and then tends to be stable when temperature increase to 400 and 600 °C. From Fig. 4c, d, the friction coefficients of NC-10S composites are relatively low and stable during sliding at elevated temperatures, although they exhibit relatively larger variations at 200 °C. It is also obvious that the fluctuation of friction coefficients of NC-30S composite is much bigger as compared with other materials, which is corresponding with its high friction coefficients and wear rates.

Figure 5 shows the worn surfaces of Ni–Cr alloy after wear tests at different temperatures. Some microcracks and delamination can be seen on the worn surface as shown in Fig. 5a, which suggests that the main wear mechanism at room temperature is plastic deformation and brittle fracture. At 200 °C, the worn surface is then covered by a continuous and compacted surface film with some delamination. When the temperature increases to 400 and 600 °C, the films generated on the worn surfaces become more and more compacted but with more and more severe

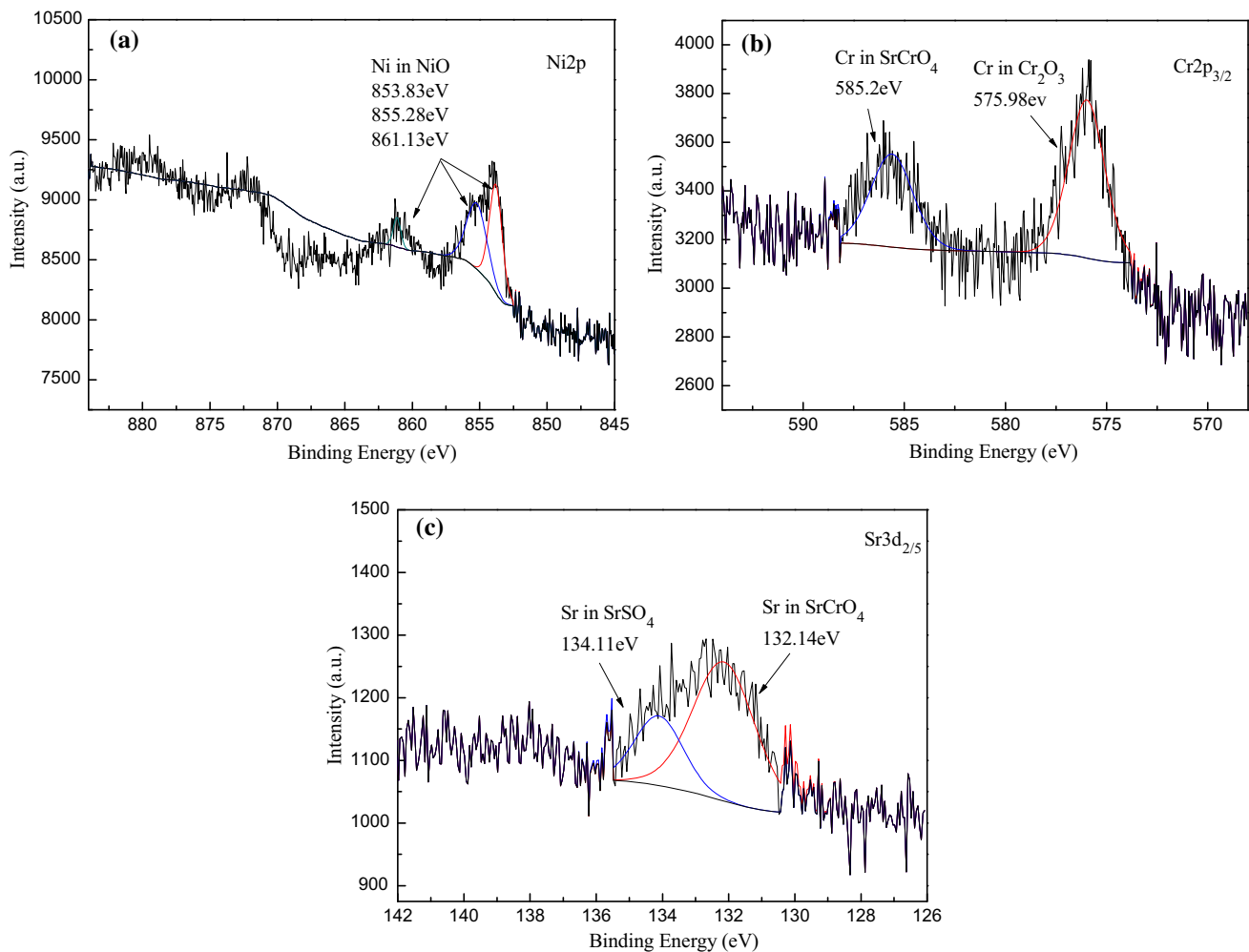
delamination which deteriorates the wear rates of Ni–Cr alloy at the same time.

Figure 6 shows the Raman spectrum of the worn surface of Ni–Cr alloy after wear tests at 600 °C. As shown in Fig. 6, the surface film generated on the worn surface of Ni–Cr alloy is mainly composed of oxides, including NiO, Cr<sub>2</sub>O<sub>3</sub> and NiCr<sub>2</sub>O<sub>4</sub>, which indicates that the worn surface undergoes a radical oxidation reaction during the rubbing process at elevated temperatures and a protective and compact layer of oxides is formed on the worn surface.

Figure 7 shows the worn surfaces of NC-10S composite after wear tests at different temperatures. As shown in Fig. 7c, d, the worn surfaces at 400 and 600 °C appear to be covered with more continuous and smoother lubricating films than that of Ni–Cr alloy. When the temperature increases to 600 °C, the lubricating film turns to be very smooth and spreads out onto the whole wear track after wear tests. In contrast, there are some microcracks and delamination on the worn surfaces at room temperature and 200 °C. In particular, more severe delamination wear can be found on the worn surface at 200 °C. Based on our previous research and the Refs. [15, 17–19], the SrSO<sub>4</sub>



**Fig. 8** Surface morphology of worn surface of NC-10S composite and the grinding Al<sub>2</sub>O<sub>3</sub> ball after wear tests at 600 °C: **a** surface morphology of worn surface, **b** EDS analysis of the marked region A in (a), **c** surface morphology of Al<sub>2</sub>O<sub>3</sub> ball, **d** EDS analysis of the marked region B in (c)



**Fig. 9** XPS spectra of elements on the worn surface of NiCr-10S composite after wear tests at 600 °C: **a** Ni 2p, **b** Cr 2p<sub>3/2</sub>, **c** Sr 3d<sub>5/2</sub>

only can be used as effective solid lubricant at high temperature, which results in the composites have severe wear and poor tribological properties at 200 °C.

Figure 8 shows the surface morphology of worn surface of NC-10S composite and the grinding Al<sub>2</sub>O<sub>3</sub> ball after wear tests at 600 °C. EDS results in Fig. 8b, d reveal that the lubricating film generated on the worn surface of NC-10S composite mainly consists of Ni, Cr, Sr, S and O elements and would somehow transfer to the surface of counterpart Al<sub>2</sub>O<sub>3</sub> ball during the rubbing process. In order to identify the phase structure of the lubricating film on the worn surface at 600 °C, further study was carried out by XPS spectroscopy. The XPS analysis results in Fig. 9 exhibit the variation of chemical states of Ni, Cr and Sr on the worn surface of the NC-10S composite after wear tests at 600 °C. It is found that the lubricating film on the worn surface at 600 °C is mainly composed of oxides NiO and Cr<sub>2</sub>O<sub>3</sub>, and oxysalts SrSO<sub>4</sub> and SrCrO<sub>4</sub>. As combined with the results of tribological properties and worn surfaces of as-obtained composites, it demonstrates that the formation

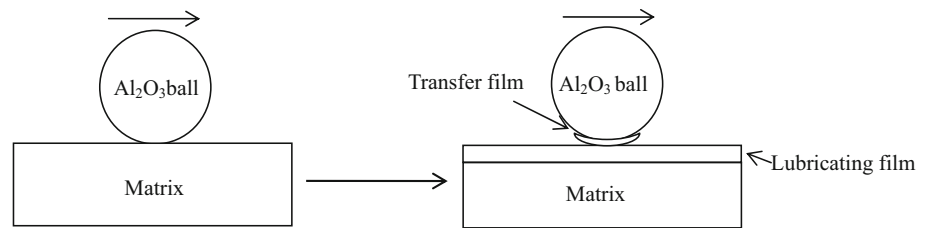
of lubricating films on the worn surfaces play an important role in the reduction of the friction and wear at elevated temperatures.

#### 4 Discussion

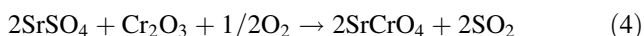
The Ni-Cr alloy has a very high friction coefficient of 0.41 at 400 °C and 600 °C and the wear rates at elevated temperatures are in the order of 10<sup>-4</sup> mm<sup>3</sup>/(Nm). Though a layer of surface film comprised of oxides (NiO, Cr<sub>2</sub>O<sub>3</sub> and NiCr<sub>2</sub>O<sub>4</sub>) was found to form on the worn surface at elevated temperatures, it would easily get softened and the adhesion between the matrix and the film became very poor, which would result in very severe delamination on the worn surface. From the SEM observation, it is also identified that the oxide films become more and more compacted with the increase in temperature, which may account for the reduction of friction coefficients at elevated temperatures.



**Fig. 10** Schematic diagram showing the high-temperature self-lubricating mechanism of NiCr–SrSO<sub>4</sub> composites



As compared to the Ni–Cr alloy, the addition of SrSO<sub>4</sub> obviously improves high-temperature tribological properties of the composites. Of the three Ni-based composites incorporated with addition of different contents of SrSO<sub>4</sub>, the NC-10S composite exhibits very low and stable friction coefficients of 0.30 and 0.2 and wear rates in the order of 10<sup>−5</sup> mm<sup>3</sup>/(Nm) at 400 and 600 °C. It is inferred that the spark plasma-sintered NiCr–SrSO<sub>4</sub> composites show great potential in some high-temperature applications like high-temperature structural components in turbines or engines. With respect to some alkali-earth metallic sulfates, it was reported that SrSO<sub>4</sub> was easily softened, and formed lubricating film on the worn surface to effectively reduce friction and wear at elevated temperatures. As the surface morphology of worn surfaces at 600 °C, it was also identified that a layer of more continuous and smooth self-lubricating film formed, and further spread out onto the whole worn surface due to friction stress. With respect to self-lubricating NiCr–SrSO<sub>4</sub> composites, the formation and effective spreading of lubricating films composed of oxides NiO and Cr<sub>2</sub>O<sub>3</sub>, and oxysalts SrSO<sub>4</sub> and SrCrO<sub>4</sub> are considered as the most important factor in reducing friction and wear at elevated temperatures. Based on the XPS results above, it is reasonable to assume that the following simplified tribo-chemical reaction occurred at 600 °C:



Consequently, the high-temperature self-lubricating mechanism of NiCr–SrSO<sub>4</sub> composites is shown as Fig. 10. At the early stage of wear tests at high temperature, the composites containing SrSO<sub>4</sub> undergo a running-in process with high friction coefficient and wear rate. With the friction process goes on, a layer of surface film containing oxides (NiO, Cr<sub>2</sub>O<sub>3</sub>) and self-lubricating oxysalts (SrSO<sub>4</sub> and SrCrO<sub>4</sub>) with a low shear strength is formed on the worn surface and would transfer to the surface of counterpart Al<sub>2</sub>O<sub>3</sub> ball. The direct contact between the matrix and Al<sub>2</sub>O<sub>3</sub> ball in the rubbing process turns into three body wear process, which effectively reduces friction and wear of the composites. Therefore, SrSO<sub>4</sub> is considered as very promising high-temperature solid lubricant in composite materials for tribological sliding applications.

## 5 Conclusions

1. The Ni–Cr alloy has a high friction coefficient of 0.41 at 400 and 600 °C and the wear rates at elevated temperatures are in the order of 10<sup>−4</sup> mm<sup>3</sup>/(Nm). The dominating wear mechanism of unmodified Ni–Cr alloy is delamination at elevated temperature.
2. Though the addition of SrSO<sub>4</sub> would decrease the relative density and the average hardness of the NiCr–SrSO<sub>4</sub> composites, it can significantly improve their high-temperature tribological properties. 10 wt% SrSO<sub>4</sub> is the most suitable adding amount into the Ni–Cr matrix. NC-10S composite that possesses excellent high-temperature tribological properties exhibits very low friction coefficients of 0.32 and 0.20 and low wear rate of 1.44 × 10<sup>−5</sup> and 2.4 × 10<sup>−5</sup> mm<sup>3</sup>/(Nm) at 400 and 600 °C, relatively.
3. Delamination and brittle fracture are considered as the main wear mechanism of the Ni-based composites at room temperature. The low friction coefficients and wear rates of self-lubricating NiCr–SrSO<sub>4</sub> composites at elevated temperatures are attributed to the formation and effective spreading of a layer of lubricating film mainly composed of oxides NiO and Cr<sub>2</sub>O<sub>3</sub>, and oxysalts SrSO<sub>4</sub> and SrCrO<sub>4</sub>.

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