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ORIGINAL PAPER

Friction and Wear Properties of MoS_2-Overcoated Laser Surface-Textured Silver-Containing Nickel-Based Alloy at Elevated Temperatures

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Abstract The solid lubricant that is coated on a flat surface is easily removed during friction. Surface texture dimples, which act as reservoirs of solid lubricant, can prolong the wear life of solid lubricant films. We textured silver-containing nickel-based alloys by a pulse laser and filled the micro-dimples with molybdenum disulfide powders. The tribological properties of the alloys were tested by rubbing against alloyed steel on a ring-on-disk tribometer at temperatures ranging from room temperature to 600°C . After laser surface texturing, the friction coefficients of the silver-containing nickel-based alloy smeared with molybdenum disulfide powders were reduced at temperatures ranging from room temperature to 400°C. With increasing dimple density, the wear life of the MoS_2 film increased while the wear rate of the nickel-based alloy decreased. The wear life of the textured surface with a dimple density of 11.2% exceeded 10,000 m at room temperature. We conclude that molybdenum disulfide and its oxides stored in the micro-dimples play a role in lubrication at room temperature and high temperatures, respectively.

Keywords Self lubrication · Solid lubricants · Molybdenum disulfide · Laser surface texturing

1 Introduction

Lubrication over a wide temperature range has been a challenge for several decades and has not yet been completely overcome. Commonly used lubricants, such as silver, graphite, and molybdenum disulfide (MoS_2), are only effective within a limited temperature range. A number of methods have been developed that involve combining two or more than two solid lubricants to achieve lubrication over a wide temperature range. Muratore et al. [1] prepared composite coatings containing silver (Ag) and molybdenum as lubricant elements that had low friction coefficients and wear rate at temperatures ranging from room temperature to 700°C. Aouadi et al. [2] studied the lubrication effect of $Mo_2N/MoS_2/Ag$ at elevated temperatures and found that the compound oxide products of MoS_2 and silver were good lubricants at high temperatures. In a previous study, we investigated composites containing several lubricants and their synthesis for their lubricating effects, such as combinations of graphite/MoS_2 [3], Ag/Mo [4], Ag/CeO_2 [5], and Ag/BN [6]. The lubricating effects of these combinations are good over a wide temperature range due to fact that the lubricants complement each other. However, the MoS_2 reacted with the chromium (Cr) element in the matrix during the hot pressing of the Ag- and MoS_2-containing nickel-based composite by powder metallurgy (PM) and the intrinsic lubricating character of MoS_2 was lost [7, 8]. A method was therefore introduced in which silver was added as the interior lubricant in the matrix during the hot pressing and coating of MoS_2 on the laser-textured surface as the external lubricant.

Laser surface texturing is a method used for fabricating an artificial topography on the surface of a material by laser scanning. This topography improves the elastic–hydrodynamic lubrication between frictional surfaces [9–14]. Surface texturing through the storage of MoS_2 was reported by Rapoport and Moshkovich [15, 16] in a method that was also combined with hard coatings [17, 18]. The tribological properties were significantly improved by texturing and the

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subsequent application of a solid lubricant on the frictional surfaces. Voevodin et al. [18] prepared textures on a Ti–TiC–TiC/DLC coating, and the grooves were fabricated along the wear trace as storage sites for solid lubricant. Compared with the untextured surface, the wear life of the MoS_2 film cover on the textured surface was extended by more than one grade of magnitude [19]. Basnyat et al. [20] studied the Mo/MoS_2/Ag lubricant on the textured TiAlCN hard layer and found that the friction coefficient and wear rate at temperatures from 25 to 570°C were significantly decreased.

Silver-containing nickel-based alloys show excellent friction and wear properties at moderate temperatures due to the lubrication of silver [5, 6]. With the aim of achieving lubrication over a wide temperature range and extending the working life of the external lubricant, we applied laser surface texturing (LST) with different dimple densities onto the silver-containing nickel-based alloy (Ni–Cr–W–Al–Ti–Ag). The effects of laser surface texturing and textured dimple density on the tribological properties of solid lubricant materials were subsequently investigated.

2 Experiment Details

2.1 Laser Surface Texturing

The silver-containing nickel-based alloy (Ni–Cr–W–Al–Ti–Ag, Ф 45 × 6 mm) was prepared by powder metallurgy (PM). Its composition is shown in Table 1. The laser surface texturing was carried out on the silver-containing nickel-based alloy surface using a Nd:YAG pulse laser (wavelength 1064 nm, pulse energy 30 μJ/pulse, period 450 ns, frequency 1–100 Hz). Dimples with diameters ranging from 120 to 150 μm and depths of 30–40 μm (Fig. 1) were fabricated on the alloy surface in the form of formula arrays. The distances between dimples were 400–1000 μm in the circular direction and 400–1000 μm in the radial direction.

The dimples are used as the storage for solid lubricant. The dimple density, which was varied to evaluate the effectiveness of storage for solid lubricant, can be defined as $D_{\text{area}} = \pi d^2/4l_1l_2 \times 100\%$ [13], where d is the diameter of dimple, and l_1 and l_2 are the distances between dimples along the circuit and radical directions, respectively. The parameters of a sample with a dimple density of 1.8, 7.1, and 11.2%, respectively, are shown in Table 2.

2.2 Friction and Wear Test

Friction and wear tests were carried out on a MG2000 high temperature tribometer (Beilun Corp, China). The friction properties of textured and untextured surfaces were tested under the lubrication of MoS_2 powders (particle size 20 μm) which were smeared on the textured and untextured surfaces. The textured and untextured silver-containing nickel-based alloys (Ф 45 × 6 mm, hardness 42 HRC) were used as the lower samples, while a GH1131 alloyed steel ring with an inner diameter of 36 mm, outer diameter of 44 mm, hardness of 23 HRC, and roughness (Ra) of 0.1 μm was used as the upper sample. The composition of GH1131 steel is shown in Table 3. The upper sample was kept still while the lower one was rotated. The flat face of the ring was in contact with the disk surface. The entire system was surrounded by an electrical furnace. The diameter of the wear track was 40 mm. The sliding friction tests were conducted at a speed of 0.4 m/s and the applied load was 20 N; the trials were run at temperatures ranging from room temperature to 600°C. The rings and disks were cleaned by ethanol before and after each trial. The wear mass loss was weighted by an analytical balance with a precision of 0.1 mg. The wear rate was calculated by dividing the weighted mass loss by the density, load, and sliding distance. Oxidization of both MoS_2 and the alloy matrix at 600°C would increase the total weight. The weight increase by oxidization was about one grade of magnitude lower than the weight loss by wear at the high temperature. The morphology of the worn tracks was observed by white light optical interferometry, optical microscopy (BMX Olympus, Japan), and scanning electron microscopy (JSM-6380 LV; JOEL, Tokyo, Japan) attached with energy-dispersive spectrum (EDS).

3 Results and Discussion

3.1 Topography of Textured Surface

The surface topography of micro-dimples and the cross-sectional profile are shown in Fig. 1. The micro-dimples

Table 1 Composition of raw materials and resulting mechanical properties

Composite	Composition (wt%)						Density (g/cm^3)	Hardness (HRC)
	Ni-20Cr	W	Al	Ti	Ag	CeO_2		
Ni-based alloy	Bal.	10	3.8	5.5	5	2	7.50	42

Ni, Nickel; W, tungsten; Al, aluminum; Ti, titanium; Ag, silver

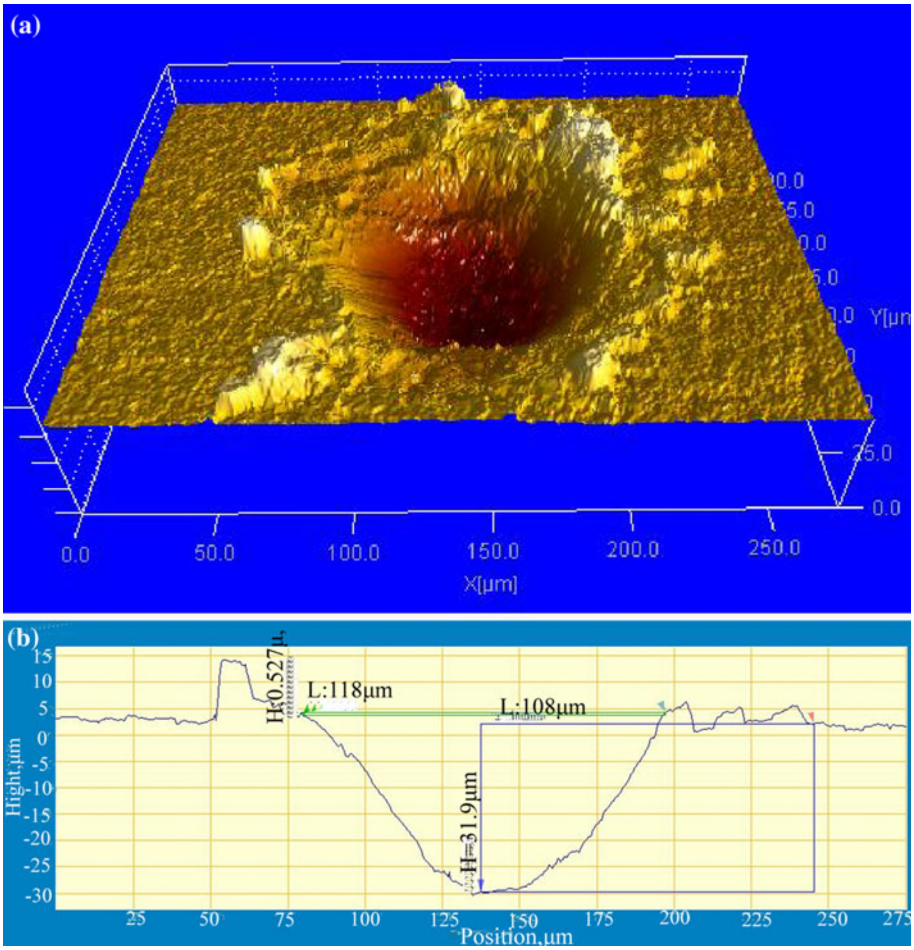

Fig. 1 Optical topography of the laser surface texturing (LST) (**a**) and its profile (**b**) by white light optical interferometry

Table 2 The distribution of micro-dimples

Parameter	Diameter (μm)	Distance in circular direction (μm)	Distance in radial direction (μm)	Dimple area density (%)
High density	150	400	400	11.2
Middle density	150	500	500	7.1
Low density	150	1,000	1,000	1.8

are about 120–150 μm in diameter and more than 30 μm in depth. It can be seen from Fig. 1b that the brims of the dimples are a little higher than the plane areas of the matrix. In laser processing, expansion of the volume of heated material results in a patterned surface. The materials ejected from the dimple are stacked on the brim of dimple, which leads to a high surface roughness after laser texturing [14].

Figure 2 shows the surface morphology of the textured and untextured silver-containing nickel-based alloy. The high-energy action of the pulse laser resulted in the heated metal being ejected from the matrix and partly deposited on the brim of the dimples. Prior to the wear test, the surface should be polished to remove the asperity on the brim. Figure 2b shows that the embedded lubricants of the silver islands are dispersed in the matrix.

3.2 Effect of LST on the Friction and Wear Properties

Figure 3 shows the friction coefficients of textured and untextured silver-containing nickel-based alloy after being smeared with MoS_2 against rotating cycles when rubbing

Table 3 Composition of alloyed steel (GH1131)

Composition	Fe	Ni	Cr	Mo	W	Nb	Mn	C	Si	N
Wt%	Bal.	15.0–30.0	19.0–22.0	2.80–3.50	4.80–6.00	0.70–1.30	0–1.20	0–0.10	0–1.20	0.15–0.30

Fe, Iron; Cr, chromium; Mo, molybdenum; Nb, niobium; Mn, manganese; Si, silicon; N, nitrogen

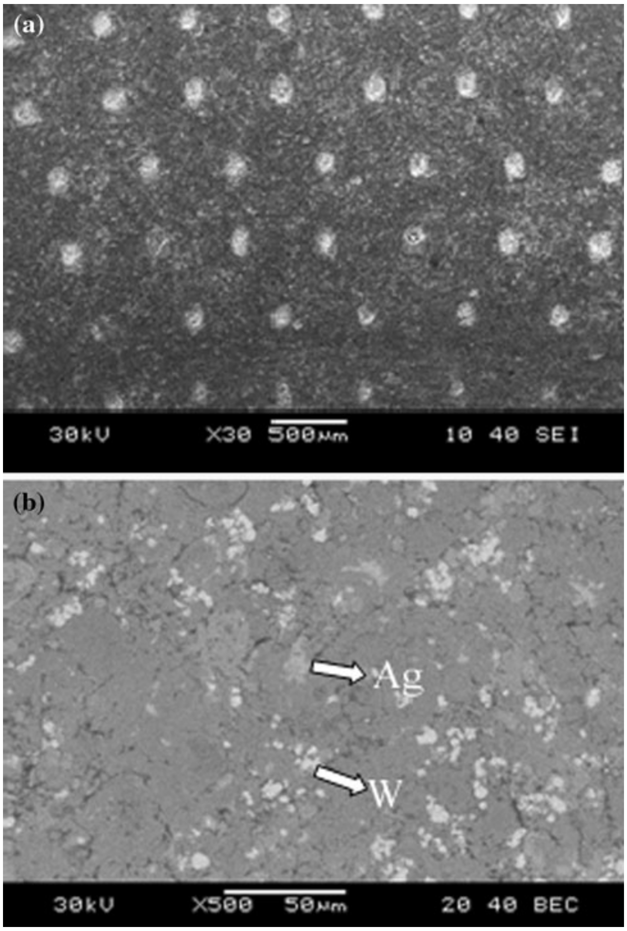

Fig. 2 Scanning electron microscopy (*SEM*) of the laser-textured surface with a dimple density of 7.1% (**a**) and the untextured surface (**b**). *W* Tungsten, *Ag* silver

against GH1131 steel; the trials were run at temperatures ranging from room temperature to 600°C. The friction coefficient of the textured nickel-based alloy was a little lower and fluctuated less than that of the untextured sample between room temperature and 600°C. The friction coefficients of both the textured and untextured alloy were low at temperatures ranging from room temperature to 400°C due to the effective lubrication of MoS_2. The friction coefficient of the textured surface was a little lower than that of the untextured surface due to the lubricant storage effect of the textures. At temperatures >400°C, the friction coefficient of the untextured was observed to increase, whereas the textured sample retained its low-temperature value (0.2), indicating that the effective lubricating temperature of MoS_2 can be elevated by LST.

The MoS_2 film on the frictional surface of the untextured Ni-based alloy was easily scratched under the action of frictional force at the elevated temperatures. For the textured Ni-based alloy, the solid lubricants stored in the dimples provided fresh lubricant on the frictional surface when the lubricant films on the plane area were scratched or oxidized under high temperature.

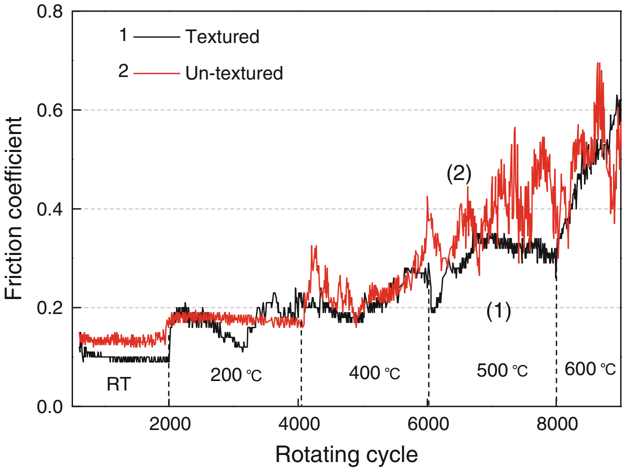

Fig. 3 Friction coefficient of textured and untextured nickel-based alloy coated with MoS_2 rubbing against GH1131 steel (dimple density 7.1%). *RT* Room temperature

3.3 Effect of Dimple Density on the Friction and Wear Properties

Figure 4 shows the curves of friction coefficients and wear rates of the textured silver-containing nickel-based alloy coated with MoS_2 varied with dimple density at elevated temperatures. The friction coefficients represent the average values before the MoS_2 coating failed. The friction coefficient was only slightly affected by the dimple density, although it did increase a little with increasing dimple density at 600°C (Fig. 4a). At room temperature and at 200°C, the MoS_2 powder was not oxidized and the friction coefficients fell below 0.1; this is caused by the layer structure of MoS_2 and is less influenced by the dimple density. From room temperature to 400°C, the friction coefficients of the textured sample with a dimple density of 1.8–11.2% were in the range of 0.07–0.23.

With the increase of dimple density, the wear rates of textured alloy show the decrease tendency at 200 and 400 °C (Fig. 4b). The wear rate of textured sample with dimple density of 11.7% is much lower than the sample with other dimple density at 400 °C. The MoS_2 powders are seriously oxidized at 600 °C, which leads to the mass gain, so the wear rate at 600 °C are not presented. The textures with high dimple density provide more reservoirs for the lubricant, so the higher dimple density leads to good lubrication and lower wear rates.

Figure 5 shows the wear life of textured nickel-based alloy smeared with MoS_2 varied with dimple density. The wear life of MoS_2 film is defined as the sliding distance until the friction coefficient exceeds 0.3 suddenly [21]. The

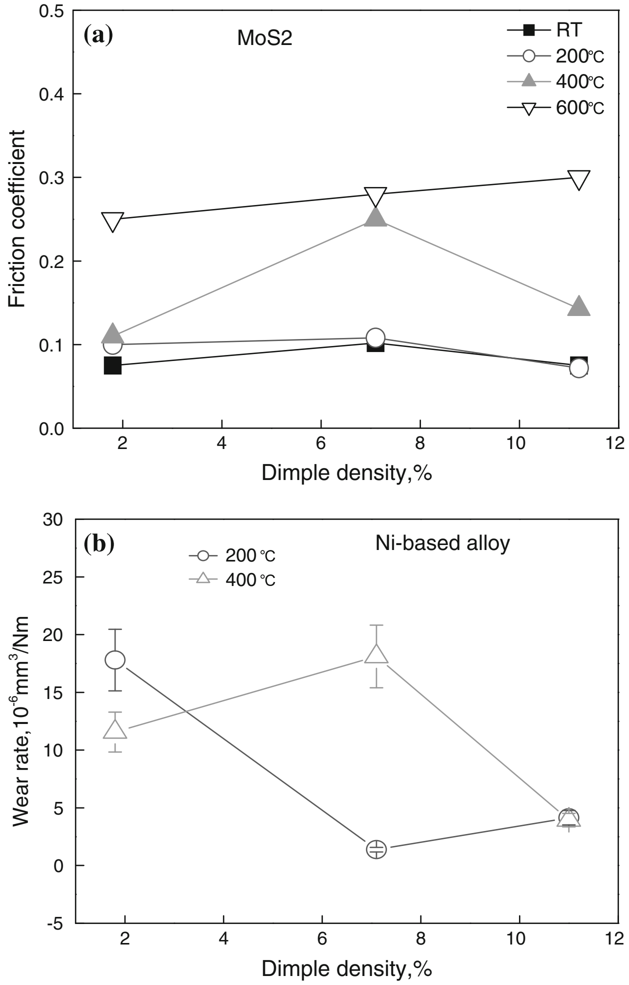

Fig. 4 Friction coefficient (**a**) and wear rates (**b**) of GH1131/Ni-based alloy versus dimple density (applied load 20 N, sliding velocity 0.4 m/s, MoS_2)

wear life increases with the increase of dimple density in the temperature range of room temperature to 400 °C when the dimple density is in the range of 1.8–11.2%. The texture with high dimple density stores more lubricant, which can continuously supply fresh lubricant on the frictional surface and prolong the wear life.

The friction coefficient of MoS_2 film on textured surface increased with increasing temperature. The friction coefficients at 600°C were >0.3 due to the degradation of MoS_2. The MoS_2 film on the textured silver-containing nickel-based alloy with a dimple density of 11.2% had the longest wear life from room temperature to 600°C and the lowest wear rate at moderate temperatures.

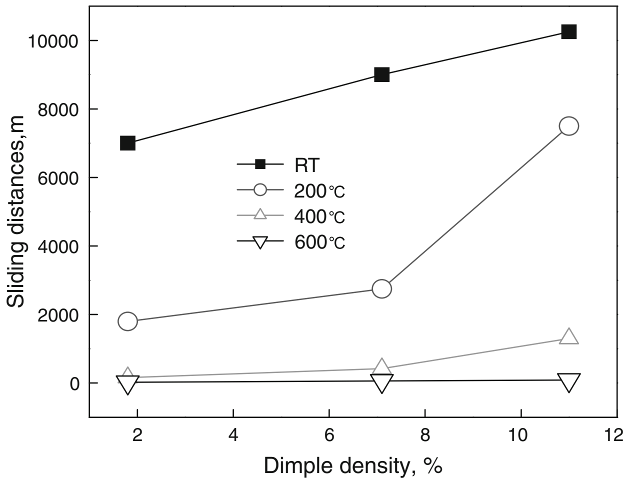

Fig. 5 Wear life of MoS_2 films on textured nickel-based alloy versus dimple density rubbing against GH1131 steel

When there is a low density of dimple area, the dimple is not sufficiently filled with solid lubricant. With increasing dimple density, the amount of solid lubricant in the dimples increases and the wear life of lubricant film is subsequently extended. Consequently, a high density of dimple area means a high percentage of lubricant storage, resulting in a longer wear life during friction. However, the high dimple density will inevitably lead to high surface roughness; however, this is less harmful to the wear life of MoS_2 films under the constant contact in the ring-on-disk configure. The optimum dimple density should provide sufficient lubricant storage and have a relatively lower surface roughness [22].

3.4 Effect of Temperature on the Friction Properties of Textured Surface

Figure 6 shows the curves of the friction coefficients of the textured nickel-based alloy smeared with MoS_2 in relation to the sliding distance. At room temperature, the MoS_2 film on the textured surface had a wear life for >10,000 m. The wear life of MoS_2 film at 200°C was slightly less than that at room temperature. With increasing temperature, the

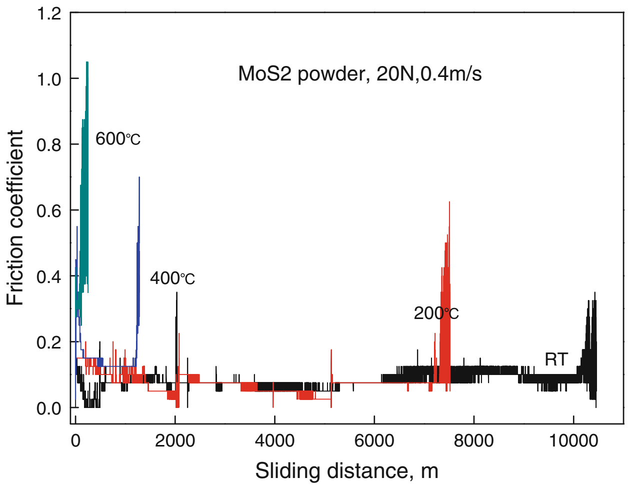

Fig. 6 Friction coefficient of the textured nickel-based alloy with a dimple density of 11.2% smeared with MoS_2 rubbing against GH1131 steel (applied load 20 N, speed 0.4 m/s)

wear life of the MoS_2 film decreased significantly: The MoS_2 film sustained only 1, 000 m at 400°C and <50 m at 600°C. The wear life of the MoS_2 film was observed to be determined by temperature, even on the textured surface. The lubricant of MoS_2 showed stable lubrication properties from room temperature to 400°C; above 400°C, the MoS_2 powders are oxidized before the wear test. Therefore, the friction coefficient will be higher initially and the wear life lower at high temperatures than at a moderate temperature. However, the oxidizing products of MoS_2 in the dimples play some lubricating so the friction coefficient retains a relatively low value even after the MoS_2 has failed.

3.5 Worn Surface Analysis

Figure 7a and b shows the worn surface morphology of the MoS_2-coated and -uncoated surface texture after rubbing against the GH1131 steel ring at 600°C under a load of 20 N. The worn surface of the textured nickel-based alloy smeared with MoS_2 was smooth and the dimple less damaged (Fig. 7a). The black lubricating films were covered along the wear trace of the dimpled area. The surface texture with no MoS_2 as lubricant comprised severely deformed plastic, and wear particles were stacked above the dimples (Fig. 7b).

The laser surface-textured dimples act as storage areas of solid lubrication from room temperature to 400°C, which favors the formation of lubricating film on the frictional surface [20]. After the lubricants on the surface are oxidized, solid lubricants released from the dimples and fresh lubricants are released on the frictional surface, which is helpful for the reduction of friction at high temperature. After the complete degradation of MoS_2, the dimples function to trap wear particles [23].

Figure 8 shows the worn morphology of untextured and textured alloy smeared with MoS_2 after rubbing against GH1131 steel at 600°C under a load of 20 N. The worn surface of the untextured Ni-based alloy can be seen to be relatively smooth (Fig. 8a), while some wear particles are trapped in the dimples of the textured surface after high temperature friction (Fig. 8b). The dimples are able to trap wear particles and relieve the damage of lubricating film,

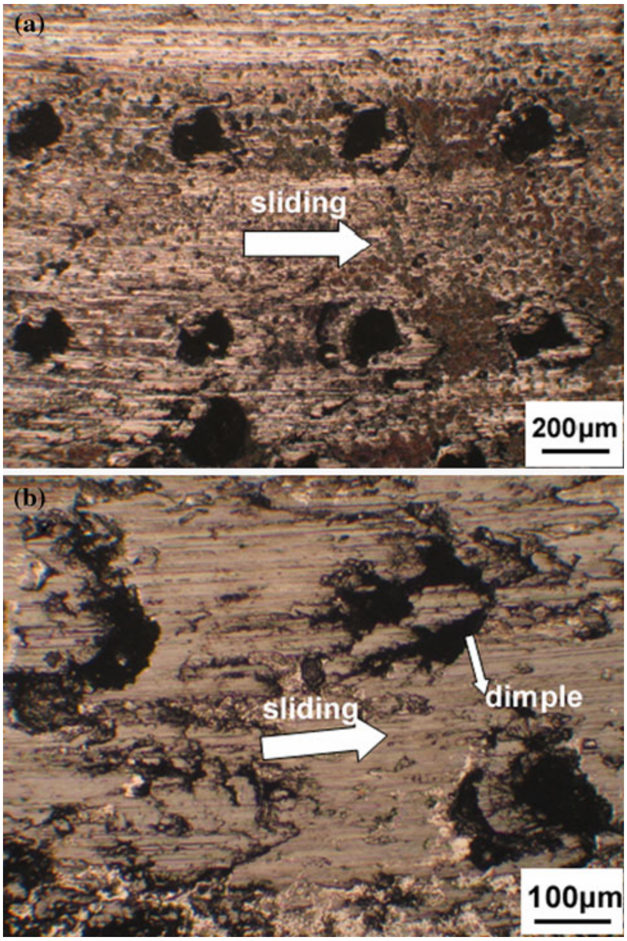

Fig. 7 Worn morphology of the textured alloy smeared with MoS_2 (**a**) and dry friction (**b**) after rubbing against GH1131 steel at 600°C

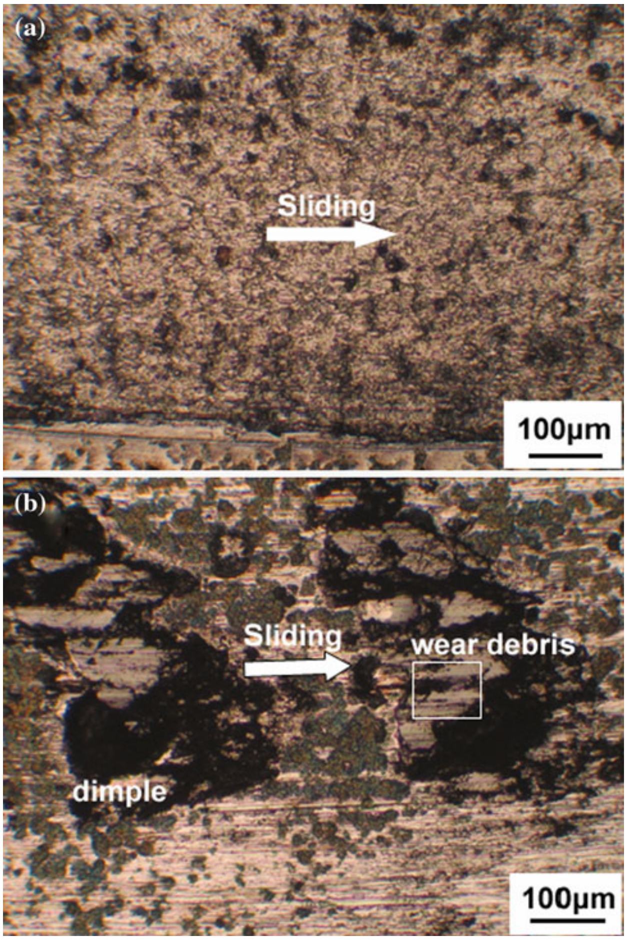

Fig. 8 Worn surface morphology of untextured (**a**) and textured (**b**) surface rubbing against a nickel-based alloy at high temperature

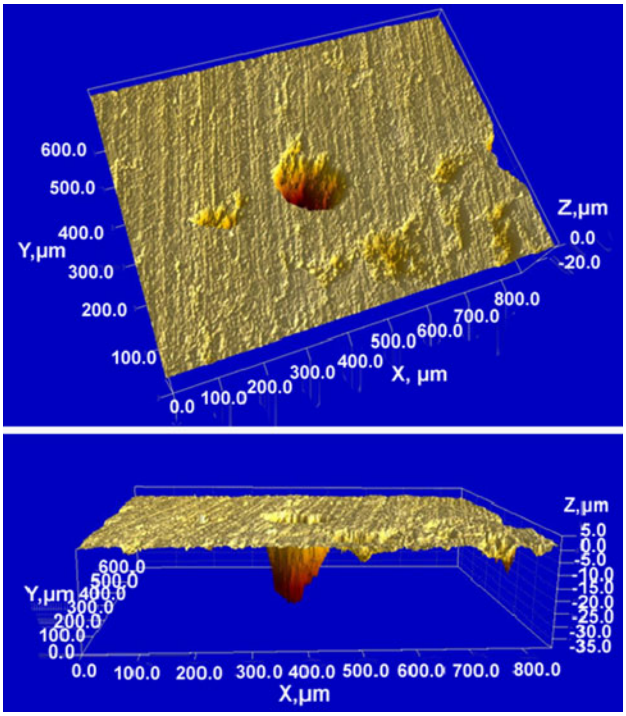

Fig. 9 Worn surface topography of textured alloy after wear at 600°C by white light optical interferometery. The MoS_2 was removed from the worn surface during friction

such as the oxide film of molybdenum, tungsten, and silver on the surface [11].

Figure 9 shows the worn surface topography of the textured alloy and its profiles after rubbing against the GH1131 ring at high temperature. The dimple kept its original shape after being worn at high temperature and its depth decreased only a little. A few plough grooves appeared on the worn surface and the grooves were uniform and fine, indicating there was no serious abrasive wear. In Fig. 9, the MoS_2 can be seen to be oxidized after exposure to high temperature and to be removed from the plane surface during long-term friction. However, few oxides of tungsten and molybdenum were in the dimples.

Figure 10 shows the worn surface morphology of the textured silver-containing nickel-based alloy in the EDS elemental analysis in the dimples and plane area after wear for about 200 m at 600°C. A number of wear particles were observed to be stacked in the dimples, thereby reducing the depth of the dimples. The element analysis of the wear particles in the dimples indicated that nickel, chromium, tungsten, molybdenum and oxygen form the main part of the dimples. It can be seen from Table 4, there was more silver, sulfur and tungsten in the dimple. However, the molybdenum content was also a little lower. The oxides, such as NiO, WO_3, and MoO_3 stored in the dimples, which is combined with embedded silver, function as lubricants at high temperature [24].

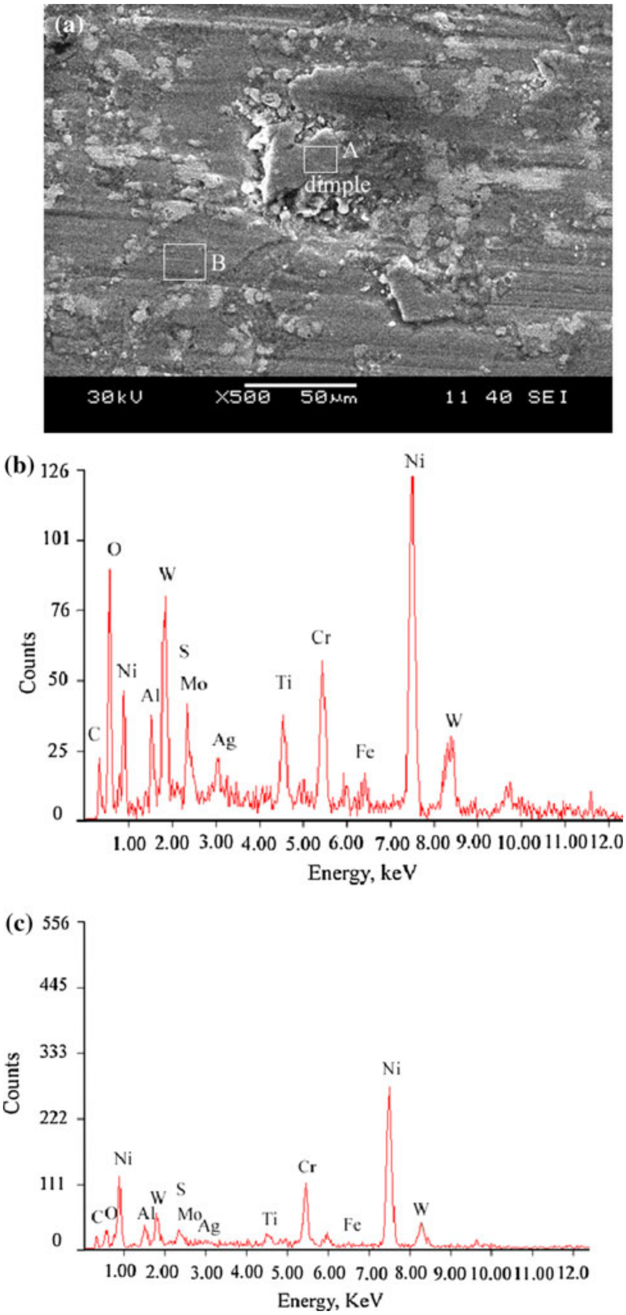

Fig. 10 Worn surface morphology of textured silver-containing nickel-based alloy (**a**) after the wear test at 600°C and energy-dispersive spectrum analysis in dimples marked *A* (**b**) and plane area marked *B* (**c**)

4 Conclusions

1) Lower friction coefficients from room temperature to 400°C are obtained by laser surface texturing of a silver-containing nickel-based alloy filled with MoS_2 powder.
2) The textured sample with a dimple density of 11.2% had the lowest wear rate and the longest wear life.

Table 4 EDS analysis of element in worn dimples

Element (Wt%)	Ni	Cr	W	Mo	Al	Ti	Ag	Fe	S	O	C
Worn dimples (A)	23	6	14	1	4	3	3	2	2	24	18
Plane area (B)	45	10	8	5	5	2	1	–	–	8	16

With increasing temperature, the friction coefficients of textured surface also increased.

3) The surface texture stored MoS_2 powders and trapped wear particles; thus the wear life of lubricant films at elevated temperatures will be extended by laser surface texturing.

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