Original Article

Microstructure and tribological property of CoCrFeMoNi High entropy alloy treated by ion sulfurization

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\begin{abstract}

The microstructures and tribological properties of CoCrFeMoNi high entropy alloy (HEA) before and after ion sulfuration were compared and discussed in the present study. The results showed that the CoCrFeMoNi HEA was mainly composed of FCC solid solution. After being sulfurized at 260°C for 2h, the FeS/MoS\textsubscript{2} multisulfides solid lubricant films with a thickness of approximately 5.0\,\mu m were successfully fabricated and well combined with the HEA. The stable friction coefficients and weight losses of the sulfurized HEA (0.15 and 4.25\,mg) were decreased obviously than the original HEA (0.47 and 15.1\,mg). At the beginning of the friction, the friction coefficient of the sulfurized HEA was easier to reach into stable stage. Because of the synergistic work of FeS and MoS\textsubscript{2} lubricant phases, the sulfides boundary lubricant films were formed in the later stage of friction under the wearing condition, which could help the friction system work for longer time under lower friction coefficient.

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\end{abstract}

1. Introduction

The high entropy alloy (HEA) is a recently discovered materials system. Unlike the traditional alloys, which are mainly composed of one or two principle elements, the HEAs are composed of five or more principle elements with equal or nearly equal atomic ratio [1]. Because of the high entropy, the Gibbs Phase rule would no longer works and the HEAs are usually composed of simple solid solutions (i.e. FCC, BCC and HCP) and sometimes accompanied by some intermetallics [2]. Because of the high entropy and severe-lattice-distortion effects, the HEAs always perform good mechanical properties no matter at high or low temperatures [3–5]. Which have shown great potential for tribological applications [6]. So far as the recent research was concerned, the ways to improve the tribological properties of HEAs were mainly focused on the following three aspects: Addition of alloy elements (Mo, W, Al, Ta et al) [2,7–11], hard phases (WC) [12–14] as well as rear earth [15], annealing heat treatment [16] and nitriding treatment [17,18]. Through the research, it was found that the improvement of tribological properties was mainly attributed to the improvement of hardness, however, which sometimes would cause excessive wear of the counter-
parts [11,19]. Thus, the key to solve this issue is to improve the anti-friction performance of the HEAs without decreasing their own properties.

Ion sulfurization is a novel surface lubrication technology that could prepare solid lubricant films composed of sulfides with inter-lamellar hexagonal structure. Compared with other sulfurizing methods (such as solid sulfurization, salt bath sulfurization, plasma spraying and so on), the low temperature gas ion sulfurization performs irreplaceable advantages on safety, economy, environmental protection and easy operations [20–24]. Furthermore, the sulfides solid lubricant films could be applied in severe conditions that the lubricating oil and grease are limited (such as liquids, high temperature and vacuum, et al.) [25–27]. Thus, the ion sulfurizing technology has been becoming effective and widely used in industrial fields [28]. The well-known sulfides as FeS, MoS2 and WS2 are the most commonly used solid lubricants because of good friction-reducing performance [26,29,30]. However, the sulfurizing layers were soft and always easily deteriorated during rubbing [31–33]. In order to maintain the sulfides films for longer time, the ion sulfurizing technique was always applied on the metals with high hardness [31,32,34,35]. Han Bin [19,36,37] has carried out the ion sulfurization on laser cladding Ni/WC coatings and fabricated the FeS/WS2 lubricant films, which could obviously improve the anti-friction performance of the original coatings. Zhu lian [26] has combined the magnetron sputtering W/Mo and low temperature ion sulfurizing techniques and fabricated the WS2/MoS2 multilayers films on 1045 steels, which showed that the WS2/MoS2 lubricant films exhibited lower friction coefficient and better wear-resistance. Thus, it would be worth believing that when the hard HEAs were treated by ion sulfurizing technique, the HEAs could not only give sulfurizing films good support, which is significant to the fabrication of ideal friction system [29,36,38]; But also provide various sulfides forming elements for the fabrication of lubricant films. However, there was few research on this has been openly published yet.

CoCrFeNi HEA was one of the most simple alloy systems and has been extensively studied [3,39,40]. In this paper, the Mo was added in the CoCrFeNi HEA as a sulfide forming element, which is helpful to the fabrication of lubricant phases during sulfurization. The ion sulfurizing technique was carried out on CoCrFeMoNi HEA prepared by laser cladding. The microstructures, phase constituents, and tribological properties of the HEA before and after being sulfurized were tested and discussed, which would be significant to further investigation of friction mechanism and industrial application of laser cladding and ion sulfurizing techniques.

2. Experimental details

2.1. Samples preparation

The CoCrFeMoNi HEA was fabricated by laser cladding technique on medium carbon steel (wt. % C = 0.45) substrate, which owns yield strength of 350 MPa and hardness of HRC23. The cladding powders were synthesized by Co, Cr, Fe, Mo, Ni powders with equally atomic ratio. The purity and particle size of the raw powders were 99.9 % and 75 μm, respectively. The raw powders were blended and put in the QM-3SP2 ball mill at 200 r/min for 2.5 h. The laser cladding was carried out by DL-HL-5 kW type cross-current CO2 laser in an argon protecting atmosphere. The cladding powders were preset on surface of carbon steels with 1 mm thickness. The protective gas flow was advisable not blowing the preset powders. The schematic diagram of laser cladding processing is shown in Fig. 1(a).

Before laser cladded, the substrate were pre-heated for 2 h at 200°. The laser cladding parameters were listed in Table 1, which were determined by lots of preliminary experiments. After laser cladded, the CoCrFeMoNi HEA with the hardness of 640HV0.2 were polished to Ra 0.4 μm and ultrasonically cleaned in acetone for 10 min. Then the HEA were treated by ion sulfurization with LDME-15A type multifunctional ion chemical heat treatment furnace (Fig. 1(b)). The H2S was the sulfurizing gas, Ar and H2 were added as auxiliary gases. The gas volume ratio was H2:H2S: Ar = 400:30:10. The sulfurizing parameters were listed in Table 1.

2.2. Microstructures and constituent phases

The microstructures of HEA as well as wear scars before and after sulfurization were observed and analyzed by JEOL JMS-6380LA Scanning Electron Microscope (SEM) equipped with Oxford Energy Dispersive Spectrometer (EDS). The 3D mor-

Fig. 1 – Schematic diagram of (a) laser cladding processing and (b) ion chemical heat treatment furnace.


<table>
<thead>
<tr>
<th>Laser cladding</th>
<th>Parameters</th>
<th>Power</th>
<th>Scanning speed</th>
<th>Overlapping rate</th>
<th>Powder thickness</th>
</tr>
</thead>
<tbody>
<tr>
<td>3000W</td>
<td>Voltage</td>
<td>180 mm/min</td>
<td>30 %</td>
<td>Temperature</td>
<td>1 mm</td>
</tr>
<tr>
<td>600-620V</td>
<td>Current</td>
<td>0.7A</td>
<td>Temperature</td>
<td>260°</td>
<td>Sulfurized time</td>
</tr>
</tbody>
</table>

The phase constituents of HEA before and after sulfurization were investigated by X-pert PRO MPD type X-Ray Diffraction (XRD). The XRD was carried out with a Cu target, scanning from 20 to 90 degrees in 2θ with a scanning rate of 4°/min. The valence state of the typical elements in sulfurizing layers were analyzed by MICROLAB MK II type X-ray photoelectron spectroscope (XPS). The binding energy was calibrated with C1s = 284.8 eV.

**2.3. Tribological properties**

The bonding strength of the sulfurizing layers was tested using WS - 2005 scratch tester. The load was ranged from 0N to 50N, loading speed was 50N/min and scratch length was 5 mm. The dry friction property of HEA before and after being sulfurized were tested under room environment (23-27°C, air humidity 40 %-60 %) using a WTM-2E pin-on-disk tester. The upper counterpart was a GCr15 ball with a diameter of 6 mm and hardness of HRC65, the lower was the specimen to be tested [38]. The ASTM G 99 standard was applied in friction tests [27,30]. The carried load, sliding velocity and friction time were 10 N, 0.2 m/s and 60 min, respectively. The friction coefficients were obtained synchronously by the test system. The weights before and after friction tests were measured using an FB224 electronic balance with an accuracy of ±0.1 mg. In order to assure the reliability and accuracy of the tests, the specimens were ultrasonically cleaned in acetone for 10 min before and after friction tests and the results of weight losses were the average of three experiments.

**3. Results**

**3.1. Microstructures and constituent phases**

Fig. 2 (a) shows the overall cross section morphologies of laser cladding CoCrFeMoNi HEA coating. It indicated that the uniform CoCrFeMoNi HEA coating without cracks or pores was fabricated by laser cladding. Fig. 2 (b) shows the bond zone between the cladding coating and substrate. The metallurgical binding was helpful to extend its service life under harsh wear.
condition. And the cellular and dendrite structures were the main components of the bottom region. Fig. 2(c) and Fig. 2(d) shows the typical morphologies of middle and top regions of the cladding coatings. Because of the different degrees of constitutional supercooling, the middle and top regions were mainly composed of ultrafine dendrites and equiaxed structures, respectively.

Fig. 3 shows the surface morphologies of CoCrFeMoNi HEA before and after ion sputtering. It shows that the CoCrFeMoNi HEA with no pores or cracks was successfully fabricated by laser cladding. The elementary components detected by EDS were tabulated in Table 3 (Area W). It showed that the elemental components of the laser cladding coatings were different from the original powders. It could be explained by the followings: During laser cladding, the preset powders and surface metals of substrates were both melted by laser beam and the molten pool was formed. The substrate metal was mainly composed of Fe element, so the Fe content was higher than the other elements in the molten pool. The different elements of the molten pool diffused and mixed during solidification. In addition, because of the high temperature caused by high energy laser beam, the elements in molten pool were born out in different degrees. Thus, the elemental compositions of laser cladding coatings were not consistent with the original cladding powders and performed higher Fe content than the original powders. According to the SEM morphologies, the HEA was mainly composed of two distinct structures, the interdendrite phase with eutectic structure (labeled as Q) was distributed on the basic structure of dendrite phase (labeled as P). The chemical compositions of different regions were tested by EDS, which tabulated in Table 2. It indicates that the dendrite phase has higher Fe and Ni contents whereas the interdendrite phase has higher Mo content. After sputtering, a film characterized granular and porous was deposited on the HEA. The EDS results (Table 2) revealed that the element S was appeared and accounted for a certain amount, which could be demonstrated that the sulfurizing layers have been successfully fabricated on the HEA. In addition, the constituent elements of original HEA (such as Cr, Ni, and so on) were also detected on the sulfurized HEA. It could be explained by two factors: Firstly, the metal atoms of HEA surface were sputtered out under the condition of ion bombardment and sulfurizing temperature, so the elements were mixed into the sulfurizing layers. Secondly, the sulfurizing layer was thin and porous, some X ray of EDS could penetrate through and reach the HEA. Thus, the constituent elements of original HEA could be detected. In order to investigate the phase constituents, the XRD patterns were analyzed and shown in Fig. 4.

<table>
<thead>
<tr>
<th>Area</th>
<th>Composition (at. %)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Co</td>
</tr>
<tr>
<td>W</td>
<td>19.33</td>
</tr>
<tr>
<td>P</td>
<td>19.07</td>
</tr>
<tr>
<td>Q</td>
<td>19.31</td>
</tr>
<tr>
<td>R</td>
<td>4.23</td>
</tr>
<tr>
<td>S</td>
<td>4.10</td>
</tr>
</tbody>
</table>

Fig. 3 – Surface morphologies of (a) CoCrFeMoNi HEA and (b) sulfurized HEA.

Fig. 4 – XRD patterns of CoCrFeMoNi HEA before and after sulfurization.
Fig. 4 shows the XRD patterns of CoCrFeMoNi HEA before and after sulfurization. According to the XRD patterns, the CoCrFeMoNi HEA before being sulfurized was mainly composed FCC solid solutions. The Fe_{0.64}Ni_{0.36} (PDF#47-1405, 2θ = 43.60°, 50.79°, 74.67°) and Ni-Cr-Co-Mo (PDF#35-1489, 2θ = 43.45°, 50.49°, 74.33°) were the main FCC solid solutions. In addition, the compounds Co_{2}Mo_{2} (PDF#29-0489) and CoMoNi_{2} (PDF#09-0298) were also detected in the CoCrFeMoNi HEA. Combining the surface morphologies of Fig. 2 and the corresponding EDS tests, it could be inferred that the basic structure of dendrite phase (labeled as P) was dominated by Fe_{0.64}Ni_{0.36} phase and the interdendrite phase with eutectic structure (labeled as Q) was dominated by Ni-Cr-Co-Mo phase. According to the distribution of Mo element, the compounds (Co_{2}Mo_{2} and CoMoNi_{2}) were mainly concentrated in the interdendrite eutectic structure (labeled as Q). After sulfurization, the MoS_{2} (PDF#17-0477, 2θ = 38.37°, 44.50°, 69.17°, 76.15°), FeS (PDF#37-0477, 2θ = 49.55°, 53.04°, 59.09°, 76.80°) and FeS_{2} (PDF#37-0475, 2θ = 33.24°, 37.23°, 57.75°, 64.86°) were identified on the HEA according to the XRD pattern. The FeS and MoS_{2} were ideal lubricant phases owning to the layered hexagonal structure [19,26,33], while FeS_{2} was not because of its cubic structure. From the XRD pattern of the sulfurized HEA, it could be found that the constituent phases of the original HEA could be still detected. It could be explained by that the sulfurizing layer was thin and porous, some X ray could penetrate through the sulfurizing layer to the HEA. Thus, the sulfurizing layer would be further confirmed by the following XPS test.

Fig. 5 shows the valence states of S, Fe and Mo elements in the sulfurizing layer. The Fe2p spectrum in Fig. 5(a) shows that the peaks were high when the binding energies were 710.3 eV, 712.1 eV, 713.6 eV, respectively, which demonstrated the existence of FeS [41]. The high peaks around 720.4 eV and 724.0 eV were mainly attributed to the iron and iron oxides [41,42]. In addition, the peaks were low when the binding energies ranged from 704.0 eV to 708.6 eV, which indicated that the FeS_{2} phase was hardly detected in the sulfurizing layers [37,38]. The Mo3d spectrum in Fig. 5(b) shows that the peaks were high when the binding energies were 232.3 eV and 232.8 eV, respectively, which demonstrated the existence of MoS_{2} [43]. The high peaks around 235.75 eV were demonstrated the existence of MoO_{2} [30,43]. The S2p spectrum is shown in Fig. 5(c), which was fitted into peaks of 160.8 eV, 161.5 eV, 162.0 eV, 162.6 eV and 164.2 eV. It indicated that the S element was mainly existed in the forms of FeS and MoS_{2} [28,38,42,43]. In addition, the peaks around 162.9 eV were low, which indicated that the FeS_{2} phase was hardly detected in the sulfurizing layers [33], which was consistent with the analysis of Fe2p spectrum. Therefore, combining the results of XRD and XPS, it could be concluded that the FeS/MoS_{2} multisulfides lubricant films have been successfully fabricated on CoCrFeMoNi HEA.
Fig. 6 – Cross-section morphology of sulfurizing layer and correspondingly elemental distributions (a) cross-section morphology (b) enlarged SEM morphology of the cross-section morphology (c) distribution of S, Fe, Mo elements.

Table 3 – Chemical compositions of the marked areas in Fig. 6.

<table>
<thead>
<tr>
<th>Area</th>
<th>Co</th>
<th>Cr</th>
<th>Fe</th>
<th>Mo</th>
<th>Ni</th>
<th>S</th>
<th>O</th>
</tr>
</thead>
<tbody>
<tr>
<td>M</td>
<td>5.10</td>
<td>5.29</td>
<td>31.28</td>
<td>18.57</td>
<td>6.17</td>
<td>18.02</td>
<td>3.21</td>
</tr>
<tr>
<td>N</td>
<td>18.18</td>
<td>17.58</td>
<td>28.38</td>
<td>17.83</td>
<td>18.03</td>
<td>19.32</td>
<td>3.21</td>
</tr>
<tr>
<td>O</td>
<td>17.20</td>
<td>18.26</td>
<td>26.32</td>
<td>17.98</td>
<td>19.32</td>
<td>19.32</td>
<td>3.21</td>
</tr>
</tbody>
</table>

Fig. 6 (a) shows the cross-section morphology of sulfurized HEA. As shown in the figure, there was an obvious boundary being existed between the outmost and subsurface. The EDS tests of areas M, N and O (Table 3) indicated that the outmost layer was regarded as sulfurizing layer and the subsurface was regarded as CoCrFeMoNi HEA. Fig. 6(b) shows the enlarged SEM morphology of the cross section of the sulfurizing layer. It shows that the sulfurizing layer was mainly composed of some grains and the grains in the cross section were more compact than the surface. The distributions of S, Fe, Mo elements along the scanning line (Fig. 6a) were shown in Fig. 6 (c). It shows that there was an S rich layer being existed on the outmost layer, the Fe and Mo also existed in this area. According to the surface analysis results stated above, the S rich layer was regarded as the sulfurizing layer. Thus, it could be shown that the sulfurizing layer with mean thickness of 5.0 um was successfully fabricated on CoCrFeMoNi HEA. Compared with the laser cladding Co and Ni based coatings [19,33,38] that obtained superior performances on wear and corrosion resistance, the HEA could provide enough and various sulfides forming elements, so the sulfurizing layers generated on the CoCrFeMoNi HEA surface performed larger thickness. In addition, according to the EDS results of areas O and W, it was found that the elemental compositions of the coatings changed little before and after sulfurization, which could be inferred that the ion sulfurizing treatment has not changed the phase constituent of the original HEA.

3.2. Tribological properties

The results of scratch tests of sulfurizing layers are shown in Fig. 7. It shows that the average bonding strength was about 40.06 N. According to the research of Wang HD [31] and Han B [19], the sulfurizing layer was well combined with CoCrFeMoNi HEA.
rubbing. The discontinuous black masses that formed on the scars were clarified to be the oxides according to EDS tests (Table 4). As wearing time went on, because of the friction force and heat, the amount of oxides were increased and accumulated into a continuous film as Fig. 9(b) shows (wearing time was 60 min). The oxides film could avoid the direct contact of the HEA and counterparts and decrease the friction coefficient in some terms [36,44]. Thus, in the later stage of friction, the friction coefficient of HEA began to decrease gradually until the continuous oxides films were formed (Fig. 8(a)).

Fig. 10(a, b) shows wear morphologies of the sulfurized HEA after wearing of 30 min and 60 min, respectively. It shows that the debris and furrows were both generated and there were more debris on the wear scars of 30 min, the EDS tests indicated that the debris (area G and E) were mainly composed of sulfides (Table 4). In addition, there were no obvious peeling pits on the worn surface. Thus, it could be concluded that slight adhesive wear and abrasion wear were the main wear mechanisms during rubbing. The EDS tests of wear scars (area F and H) shows the S element was decreased as the wearing time increased (Table 4). So it could be indicated that as wearing went on, the sulfurizing layers were gradually worn away. But there was still amounts of sulfurizing layer remains on the worn surface and worked. Combining the stable friction coefficient curves, it could be seen that the sulfurizing layers left could still work as superior lubricant.

Fig. 11 shows the 3D morphologies and cross-sectional profiles of wear scars on the CoCrFeMoNi HEA before and after being sulfurized. Because of generation of debris and oxides, the profiles showed some bulges. In addition, because of the friction force, some sulfides were piled up at two edges of wear scars (Fig. 11(c, d)). From the figures, it could be found that the widths and heights of wear scars on sulfurized HEA were obviously lower than those without being sulfurized, which could be explained by the superior friction reducing performance of the sulfurizing layers.

Fig. 11(a, a’, b, b’) shows wear scars of the original HEA after wearing of 30 min and 60 min, respectively. It shows that there was almost no increase in the widths of the wear scars while the depths were increased by 70 % from 1086.03um to 1720.26um. Fig. 11(c, c’, d, d’) shows the wear scars of sulfurized HEA after 30 min and 60 min wearing,
respectively. There was hardly wear loss in the depth of sulfurizing layers expect some fluctuations caused by rubbing. In addition, because of the sulfurizing layers were soft and easily deformed, the widths of wear scars were increased from 560.48µm to 664.08µm, which was still lower than those on original HEA. According to the results, it could be further demonstrated that the sulfurizing layers could help the HEA experience less damage at the same condition, which was helpful to extend the service life span of the friction system.

### 4. Discussions

According to the results, it could be demonstrated that the FeS and MoS₂ phases were successfully fabricated in the sulfurizing layers. The FeS and MoS₂ were typical inter-lamellar hexagonal structure and easy to slip along the inert basal planes [19, 33]. Thus, they could both act as superior solid lubricants. According to the tribology theory [45], the friction force between the contact surfaces was mainly composed of two
Fig. 11 – 3D morphologies of the original HEA after wearing of (a) 30 min and (b) 60 min and the sulfurized HEA after wearing of (c) 30 min and (d) 60 min, cross-sectional profiles of the original HEA after wearing of (a’) 30 min and (b’) 60 min and the sulfurized HEA after wearing of (c’) 30 min and (d’) 60 min.
components: Adhesive component and plough component. The existence of sulfurizing layers could avoid direct contact of the original surfaces and reduce the possibility of "sticky point". Which was helpful to reduce the adhesive component of the friction force. The sulfurizing layers were soft and obtain low shear strength. When the sulfurized HEA were slid against the counterparts, the sulfurizing layers were easy to be deformed and slid. Which was helpful to reduce the plough component of friction force. Thus, the sulfurized CoCrFeMoNi HEA exhibited lower friction coefficient than the original HEA during rubbing.

Combining the friction coefficients and wear morphologies of different rubbing time, the wear processes of HEA and corresponding sulfurizing layers could be classified into several stages. When the grinding ball came into contact with the HEA and slid, due to the existence of irregular bulges on contact surfaces, the friction coefficient increased firstly and got stable while the bulges were worn down. Then the wearing reached into steady stage. The debris was formed because of the friction heat and pressure during rubbing. On the one hand, the debris formed at the interface could lead to unsteady friction, so the friction coefficient curve performed relative fluctuations in this stage (as shown in Fig. 8); On the other hand, the debris was attached on the surface due to the pressure and formed oxides [28], the oxides could decrease the friction coefficient in some terms [40,46], so the friction coefficient curve of the HEA in Fig. 8(a) decreased slightly after 36 min wearing. As wearing went on, the amount of oxides increased and accumulated into a continuous film, which as shown in Fig. 9(b). The continuous oxides film could avoid the direct contact of the metals and could improve the anti-friction performance to some extent. Then the friction coefficient got stable again and the wearing went into the last stage.

When the grinding ball came into contact with the sulfurized HEA and slid, because of the existence of sulfurizing layers, the friction was easier to reach into stable stage [45]. In the stable stage, the sulfurizing layers were crushed and adhered to the surface of HEA, which could effectively prevent direct contact of the metals. FeS and MoS$_2$ were the main lubricant phases of the sulfurizing layers, which owns their own respective function. The FeS lubricant phase is hard and could work in large temperature range, while the MoS$_2$ phase is soft and obtains superior patience and stickness [25,26,47]. During the wearing process, the FeS and MoS$_2$ phases worked in an effective synergistic way, which could overcome its own weakness by acquiring other’s strong point. Thus, the sulfurizing layers could play a continuous role of friction reduction. The wear morphology and EDS tests after 90 min were shown in Fig. 12 and Table 4. It shows that the wear morphology was similar with that after 60 min, and there was little reduction on S content compared with the scars after 60 min. Which could be concluded that the stable boundary lubricant film had been formed on the interface under the synergistic work of FeS and MoS$_2$ lubricant phases. The boundary lubricant film would play a continuous role in reducing friction in the long time if not broken. Compared with the oxides film on original HEA, the sulfurizing layers could not only avoid the direct contact of metals, but also the FeS and MoS$_2$ lubricant phases in the sulfurizing layers played an important role in reducing friction. Thus, the friction coefficients and weight losses of the sulfurized HEA were both lower than the original HEA.

5. Conclusions

(1) The CoCrFeMoNi HEA with no pores or cracks was successfully fabricated by laser cladding. The HEA was mainly composed of FCC solid solution and a small amount of Fe$_2$Mo$_y$ and CoMo$_2$Ni compounds.

(2) The FeS/MoS$_2$ multiphase solid lubricant films were successfully fabricated on CoCrFeMoNi HEA by ion sulfurizing technique. The sulfurizing layers exhibited a porous morphology and owned a thickness of approximately 5.0 $\mu$m.

(3) The tribological properties of the sulfurized HEA were improved greatly compared with the original HEA. The synergistic work of FeS and MoS$_2$ lubricant phases as well as sulfides boundary lubricant films were the main factors that guarantee the tribological properties of the sulfurized HEA.

Conflict of interest

The authors declared that they have no conflicts of interest to this work. We declare that we do not have any commercial or associative interest that represents a conflict of interest in connection with the work submitted.

Acknowledgements

This work was supported by National Natural Science Foundation of China (51771228); Natural Science Foundation of Shandong Province (ZR201702200455); State Key Laboratory of Solid Lubrication (LSL1609); Fundamental Research Funds for the Central Universities (18CX06052A).
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