

The Tribological Properties of Cu-Ni₃Al-MoS₂ Composite Coating Deposited by Magnetron Sputtering

Mahdi Mirzaaghaei^{1,*}, Mohammad Hossein Enayati², Mahdi Ahmadi³

¹ Student, Department of Materials Engineering, Naghsh-e Jahan University, Isfahan, Iran

² Department of Materials Engineering, Isfahan University of Technology, Isfahan 84156-83111, Iran

³ Master of science in Coverage, Iran Aircraft Company

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ABSTRACT

In industrial applications, most materials are exposed to wear and friction because multiple conditions are used. However, the tribological properties of these materials can be improved with different techniques. One such technique that improves the frictional property of a surface is the use of self-lubricating coatings. In this study, multicomponent coatings of nominal composition Cu-Ni₃Al-MoS₂ have been sputter from the target using the best sputtering condition to get a good morphology and microstructure of the coating. The morphology and micro structure of the coatings were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy depressive spectroscopy (EDS). The measured microhardness of this coating was 380 HV under a load of 25 g. Tribological properties of Cu-Ni₃Al-MoS₂ coatings were investigated using ball-on-disk (BOD) tribometer at room temperature. These composite coatings showed a good morphology and adhesion of the coating to the substrate. Also, it had acceptable friction coefficient and higher wear resistance due to its hard matrix containing Cu-Ni₃Al and MoS₂ Nano particles.

1. Introduction

Cu-matrix composite coatings have a high hardness, friction coefficient; wear resistance, thermal and electrical conductivity for several engineering applications. Metal-matrix composites can be improved by the incorporation of reinforcing elements to develop wear resistance and structural applications [1, 2]. In Cu-Ni₃Al-MoS₂ composite coating, Ni₃Al is used to reinforce and increase wear resistance, because it has a good physical/mechanical properties, melting point, thermal conductivity, and strength [3, 4].

A very large variety of self-lubricating coatings have been developed, including soft metal coatings [5], transition metal

dichalcogenide coatings [6], carbon based coating [7], oxide [8], fluoride [9] and sulfate [10] coatings, and polymer coatings [11].

MoS₂ particles help to improve the rolling and sliding properties of tribocomponents encountered in micro/nano electromechanical systems [1, 2]. It has a lamellar structure. The bonding among atoms is covalent and strong, but the layers of the structure are quite weak. The low friction occurs because of the lamellar structure and weak Van der Waals bonding of MoS₂ [12, 13]. To benefit from the friction properties, the layers of MoS₂ have to be parallel to the substrate surface [12]. The friction coefficient increases and the lifetime decreases when MoS₂ is used in humid air; therefore, this

* Corresponding author:

E-mail: mahdymirzaaghaei@yahoo.com

material is only used in vacuum and in water vapor-free environments [14].

In recent years, the attainment of composite coatings has become relatively easy using physical vapor deposition and CVD technologies [15-17]. These coating deposition techniques offer a wide variety of means and flexibility for depositing solid lubricant or self-lubricating coatings on substrates of various shapes and materials [18]. In this study, Cu-Ni₃Al-MoS₂ composite coatings were deposited onto 4340 steel using the magnetron sputter system. Pin-on-disk friction experiments were conducted by Si₃N₄ balls in sliding contact with the composite coating at room temperature. The composite coating and their wear surface were examined by SEM and EDS. These techniques were used to determine the structural morphology and elemental composition of the worn surface and some wear debris.

2. Materials and methods

2.1. Deposition

Cu-Ni₃Al-MoS₂ coating was deposited by Dc magnetron sputtering on 4340 steel substrates. Generally, the resulting coating properties are highly dependent on the preparation parameters due to the non equilibrium nature of the sputter-deposition process. The main process parameters are: the substrate temperature, the substrate bias voltage, the sputters gas pressure, the deposition rate and back ground pressure [3]. Deposition parameters are given in Table 1.

The magnetrons within the coating chamber were arranged so that one pure Cu target containing 6 tablets of Ni₃Al-30%MoS₂ was used. Figure 1 presents a schematic picture of Cu-Ni₃Al-30%MoS₂ target designed and Figure 2 displays a schematic diagram of the magnetron sputtering process with Cu-Ni₃Al-MoS₂ target. Ni₃Al-30%MoS₂ tablets were fabricated by press milling the mixture of pure Ni₃Al and MoS₂ powder, followed by pressing the mixture under the pressure of 350 Mpa. Afterwards, the composite coatings with a thickness of 4 μm were obtained by sputtering the composite targets.

2.2. Chemical composition analysis

The phases of Cu-Ni₃Al-MoS₂ coatings were characterized by X-ray diffraction (XRD,

Philips X'PERT MPD) with Cu-Kα radiation ($\lambda=1.54056\text{\AA}$) and the scan step size was 0.05°.

2.3. Micro structure examination

The morphology of composite coatings was observed by means of LEO-440i Oxford scanning electron microscopy (SEM) at an acceleration voltage of 20 kV.

2.4. Mechanical and tribological properties measurements

Micro hardness of the coating was measured by using the WILSON tester. A load weight of 25 gr was applied to perform the indentation and maintained for 10 S. The tribological properties of the coating were characterized on a ball-on-disk at room temperature tribometer and tested at the sliding speed of 0.10 m/s, under a load of 5 N and duration of 1 h. The wear and friction tests were conducted at room temperature. The counterpart ball was the Si₃N₄ with a diameter of 5mm.

2.5. Adhesion strength

This technique does not give any absolute measurement of adhesion but with the same load for all samples comparative results can be obtained. Figure 3 gives a qualitative adhesion property considering the crack network from the indentation spot. This test also gives a qualitative measure of the toughness of the coating. The adhesion value is reported from HF1 to HF6 (HF is the German short form of adhesion strength) (Figure 3). HF1 shows excellent adhesion property with a few crack networks while HF6 shows the poorest adhesion properties showing complete delamination of the coating [19].

3. Results and discussion

3.1. Chemical composition analysis

The XRD pattern of the Cu-Ni₃Al-MoS₂ coating is presented in Figure 4, where two diffraction peaks can be found. The diffraction peaks are in accordance with the standard diffraction peak of Ni₃Al and Cu (111), (002). The diffraction pattern of the coating did not show any evidence of MoS₂, but the EDS results indicated the presence of this material in the coating. Figure 5 shows the EDS analysis: the Cu and Ni₃Al and MoS₂ phases have an approximately uniform distribution in the coating. The important point

Table 1. Sputtering parameters and details of the coating deposition

| Substrate | target | Substrate to target distance | Applied voltage to substrate | Applied current to target | Applied current to substrate | Deposition time | Base pressure | Working pressure |
|------------|--------------------------|------------------------------|------------------------------|---------------------------|------------------------------|-----------------|-----------------------|-------------------------|
| 4340 steel | Cu-NiAl-MoS ₂ | 15 cm | 300 V | 1.2-2 A | 0.5 A | 27 min | 10 ⁻⁷ mbar | 5×10 ⁻³ mbar |

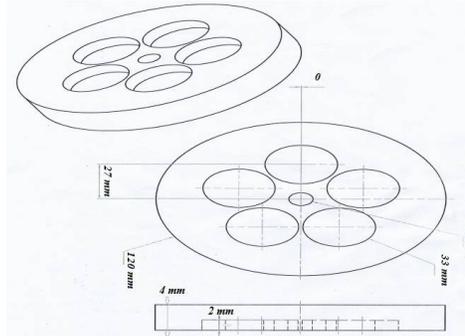


Fig. 1. Schematic picture of Cu-Ni₃Al-30%MoS₂ designed target.

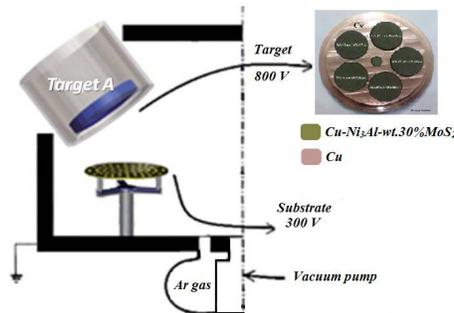


Fig. 2. Schematic diagram of the magnetron sputtering process.

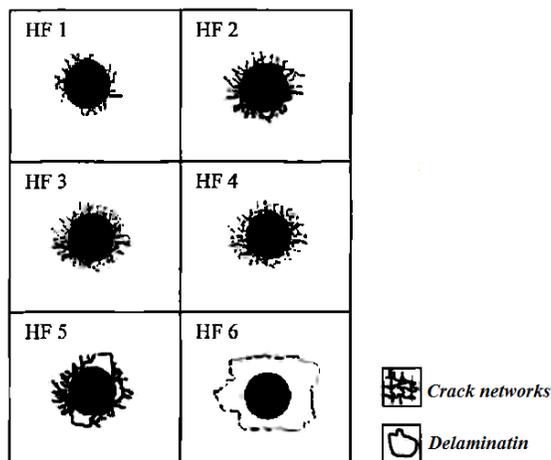


Fig. 3. Adhesion strength quality based on Rockwell C indentation test [19]

growth in all crystalline directions, diffraction fails to occur in all crystalline pages or may have low intensity. So, they do not show a crystal X-

ray diffraction from all pages. Figure 6 is a schematic illustration of the formation and growth of Fe-Mn oriented to the (111) plane [20].

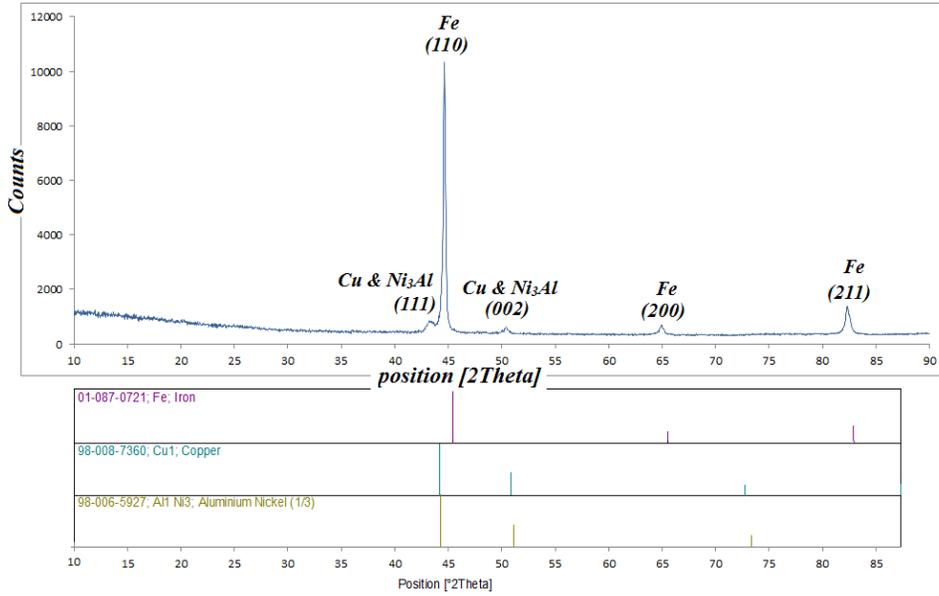


Fig.4. XRD pattern of Cu-Ni₃Al-MoS₂

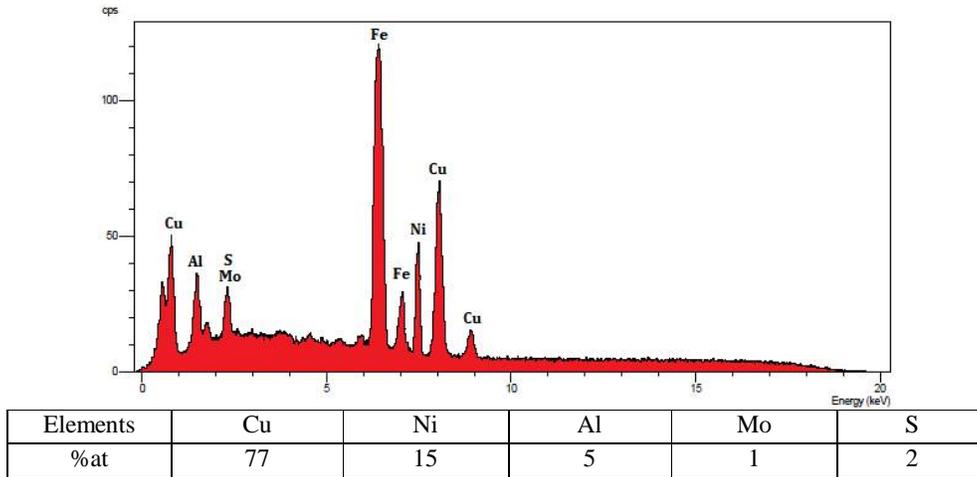


Fig. 5. About quantitative values of Cu-Ni₃Al-MoS₂

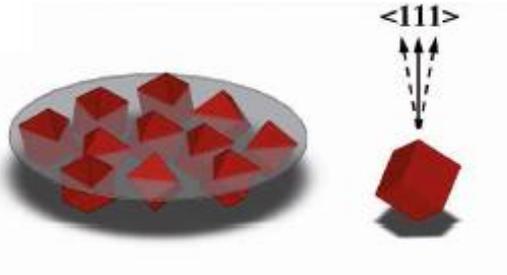


Fig. 6. Schematic illustration of in the structure covering the physical vapor deposition method [20]

3.2. Adhesion strength

Figure 7 shows the optical micrographs of Rockwell-C indentation on the Cu-Ni₃Al-MoS₂ coating. The Rockwell-C indentation test shows an acceptable adhesion. According to the standards presented in the previous section, the coating adhesion strength was evaluated to be HF1, which represents good strength of adhesion to the substrate. Few crack networks were observed around the indentation spot, as displayed in figure 7.

3.3. Microstructure and microhardness of the composite coatings

The cross-section of the as-deposited coating on glass is shown in Figure 8. The thickness of the coating at the highest target current of 2A was 4μm. The measured microhardness of this coating was 320HV. The morphology of the Cu-Ni₃Al-MoS₂ composite coatings illustrated in Figure 9 indicates that the coating has a good flatness and dense surface.

3.4. Tribological properties of the composite coating

Figure 10 illustrates the evolution of the friction coefficient of the composite coating under dry sliding for the duration of 1 h. The friction coefficient changes as a function of the sliding time. The friction force was continuously monitored during the friction experiments. The

sliding wears life for the coating in this investigation was determined in terms of the number of passes at which the initial coefficient of friction was between 0.4-0.5 at the end of 600 S sliding times. As the sliding time increases to 3600 S, the friction coefficient remains a low, constant value (μ : 0.5) maintained until the end of the test (Figure 10). A thin and uniform transfer film on the Si₃N ball is observed at the test conditions. Figure 11 shows the contacting area on the ball.

As can be seen in Figure 12, the EDS data were taken from two positions in the wear track where the worn surface from the composite indicates two worn zones. The EDS analysis was performed to determine the surface composition in each of the two zones. A qualitative analysis by EDS confirmed the presence of Cu, Ni, Al, Mo and S within the composite coating (see Figure 5). The worn area on the film counter body is smooth with patchy and powdery debris. Some worn debris piled up at both sides of the wear track with no abrasive wear effect. In the frictional process, the coating was crushed and adhered to the substrate, preventing the direct contact between the 4340 steel and Si₃N ball. Therefore, its friction-reduction property is very good. This is the reason why the friction coefficient remained very stable during 1 h sliding test. The overall aspects of this worn surface image are typical of all the multicomponent systems that have been studied. It is well known that the main reason for low coefficient of friction seems to be pressure

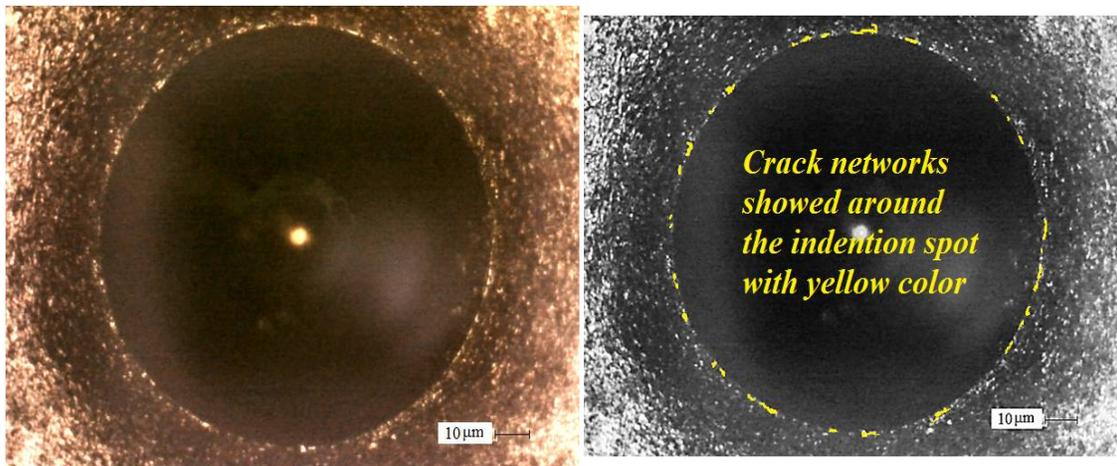


Fig.7. Rockwell C adhesion test of Cu-Ni₃Al-MoS₂ coating.

composite structure of Cu-Ni₃Al with MoS₂ composite in the coating.

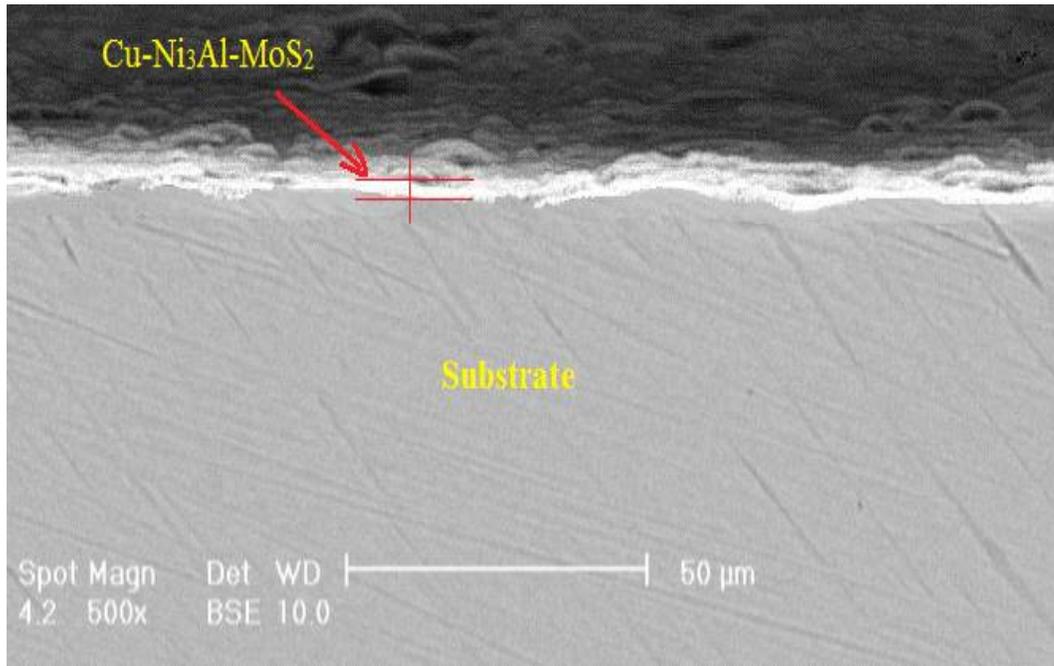


Fig. 8. Cross section of the sample

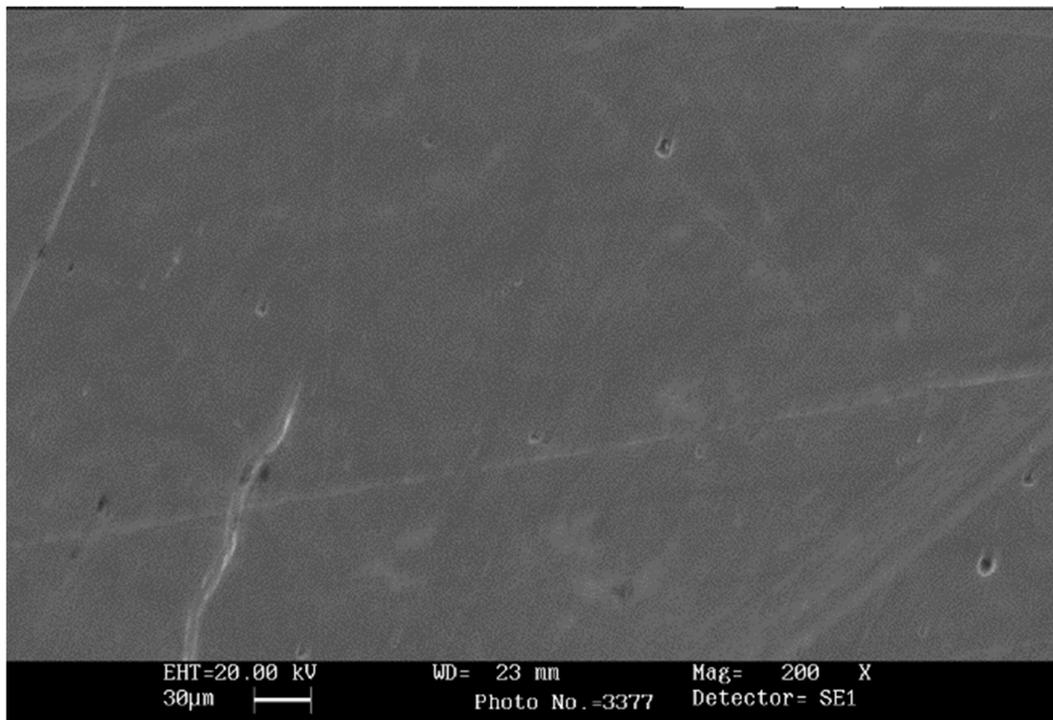


Fig. 9. SEM image of the coating.

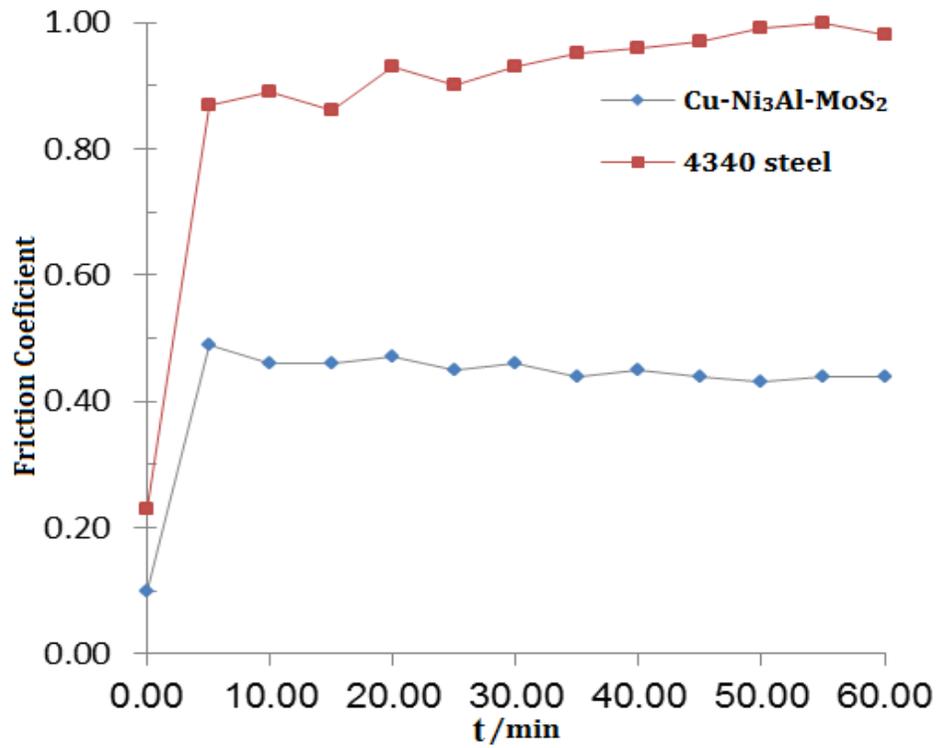


Fig. 10. Friction coefficients of the composite coating.

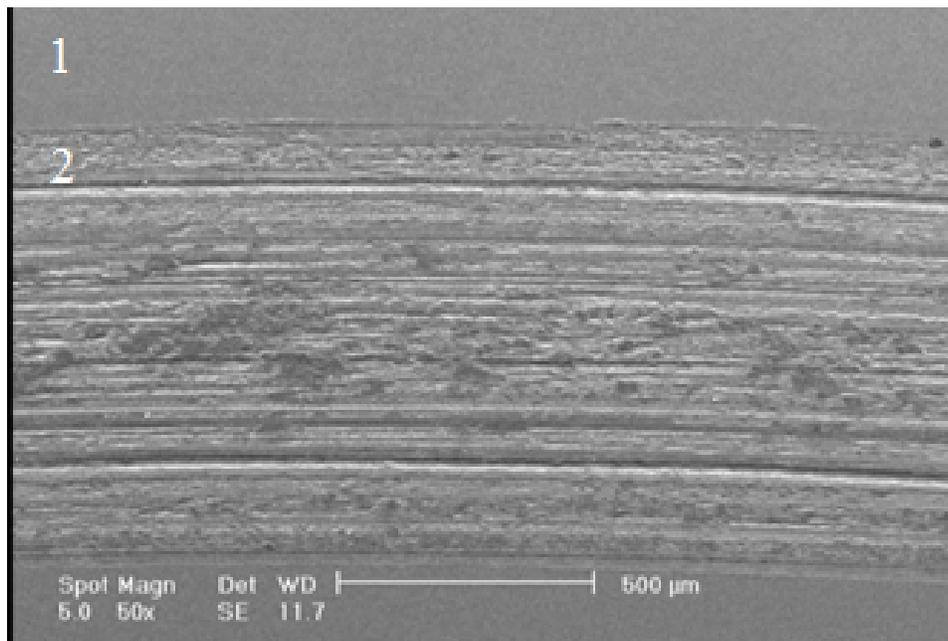


Fig. 11. SEM picture from the contacting area on the ball.

revealed that the friction reduction and wear improved at room temperature.

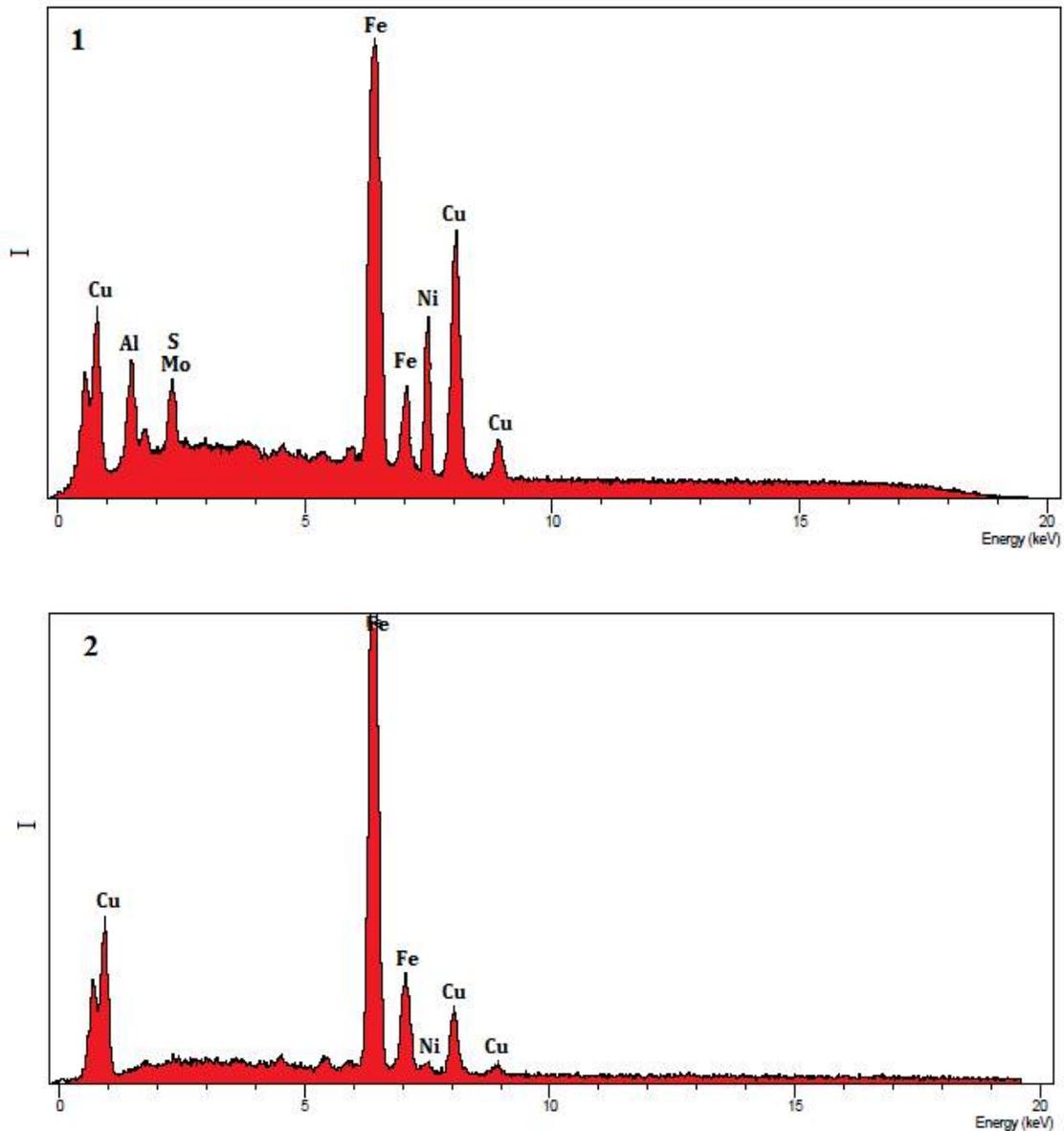


Fig. 12. EDS analysis from the wear scars

4. Conclusions

The Cu-Ni₃Al-MoS₂ composite coating was successfully prepared by magnetron sputtering. Different test results indicated a quite smooth surface of the coating with good strength of adhesion to the substrate. In addition, it has been

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