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Article

Application of Nano High-Entropy Alloys to Reduce Energy Consumption and Wear of Copper Oxide and High-Grade Iron Ores in Heavy Mining Industries—A Case Study

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Abstract: Problems relating to the abrasion of equipment is one of the most important issues in mining and associated industries. Hardening is a method of protecting metal equipment, metal tools, or important components against erosion, corrosion, and abrasion. This can be achieved by welding a thin layer of abrasion-resistant metal onto the surface of the work piece. The useful life of a piece of equipment or parts can be significantly increased by applying abrasion-resistant coatings, thereby reducing repair or replacement costs associated with damaged parts. This process is inexpensive in the production of parts and is often economically justifiable. This study focuses on measuring the abrasion resistance of a nano high-entropy alloy against copper oxide and high-grade iron ores. When a base alloy was coated with the nano high-entropy alloy, the abrasion indexes of iron and copper ores decreased from 0.0001647 kg to 0.0000908 kg and 0.0001472 kg to 0.0000803 kg, respectively. The standard deviation, repeatability, and reproducibility were calculated for the alloy steel blade covered with nano high entropy alloy (N-HEA), producing values of 0.00016, 0.00047, and 0.00040, respectively, while a standard alloy steel blade exhibited values of 0.0003, 0.00047, and 0.00042, respectively. High-entropy alloys and high-entropy nano-alloys have not been used as practical coatings in the mineral industry in any form to date. Utilizing high-entropy nano-alloys in this industry would introduce innovative alternatives for customers, thereby increasing competitive advantages and providing international markets and customers = with the most efficient choices of operational materials.

Keywords: nano high-entropy alloys (N-HEAs); hard coatings; iron and copper ores; abrasion index; ball mills; rods; lining material

1. Introduction

Minimizing energy consumption is one of the most important issues for industries. In the world of economy and industry, saving energy by applying a balanced set of price and non-price measures to continually reduce energy has always been the most important goal to keep the industry wheel turning [1]. Various types of materials are used in each sector of the mining industry. In alloying, various elements are added to a main element to improve its properties. However, a new class of alloys with at least five basic elements of equal or similar atomic compositions has recently been created made [2]. Usually, the atomic percentage of each element is more than 5%. The entropy of

incorporation of these alloys is maximal, resulting in high-entropy alloys, also known as multi-element alloys. Unlike conventional alloys, the mechanical properties of high-entropy alloys are independent on their constituent elements. In fact, the type of structure is the determining parameter ultimately controlling the strength or hardness of the high-entropy alloy. High-entropy alloys have excellent properties [3], including high levels of hardness, fatigue resistance, and strength at ambient and high temperatures, as well as dramatic tensile properties [4]. The microstructure type and properties of high-entropy alloys depend on the types and amounts of constituent elements; careful selection of compounds and elements allow for a proportion of body-centered cubic (BCC) or face-centered cubic (FCC) solid solutions to be obtained, therefore giving the best mechanical and physical properties to the alloy.

Hard-facing is a method of protecting metal equipment, metal tools, or other important components to prevent wear [5], stress, and abrasion so that a thin layer of engineered (strong metal) metal that is wear-resistant is added onto the work piece surface (weak metal) [6]. In many industrial applications, parts with hard surfaces, but are still tough and impact-resistant, are essential. Some examples include camshafts, crankshafts, cogwheels, and other parts used in mineral processing plant equipment (wearing and crushing circuits). These components require very hard and wear-resistant surfaces [7–12], as well as a high level of toughness and impact-resistance in mineral processing circuits during operation.

Welding is the deposition of a filler on the surface of a work piece to obtain desired properties or dimensions that are typically used to extend the work life of a piece or to replace metal that has been worn or corroded. Some common welding methods employed for this purpose include arc welding with a hood electrode [13], submerged arc welding [14], arc welding with a powdered electrode [15], laser hard-facing [15], gas welding (flame) [13,15], metal arc welding with protective gas [13], flame welding with powder [15,16], gas tungsten arc welding [16], and plasma arc welding [17]. Some coatings are precipitated by gas or arc welding processes or by thermal spraying processes, and manual, semi-automatic, or automatic methods can be applied to any of these processes. Suitable fillers are available in various shapes and types, such as welding rods, coating electrodes, pastes, and powders.

Choosing a suitable alloy and a suitable hard coating to crush and scrub mineral equipment has always been a challenging issue in the process of economic production of alloying. Today, casting and molding of alloys such as FeCoNi, FeNiMo, SS304 [18], and D2 [19] are utilized in the manufacture of most mining equipment parts. By choosing a suitable coating and coating method, the base alloy of parts can be made of much cheaper materials.

High-entropy alloys [3,19–22] and high-entropy nano-alloys [23,24] have not been used as a practical coating in the mineral industry in any form so far. The extraordinary characteristics of high-entropy alloys in can be made us of in heavy industries [25–37] to produce unique compositions [38–45] and their behaviors and properties [46–48] can be manipulated to introduce innovative alternatives, such as filler metals [49], which are hard-facing [50–52] and wear resistant [53] to increase to shear failure, high cavitation erosion, and corrosion [54,55]. In general, the impact strength of hardened alloys decreases with increasing carbide content. In applications where impact resistance is very important, manganese austenitic steels can be used to repair parts [17]. The higher the number of alloying elements in the intermetallic compounds, the more complex the structure. Due to the limited solubility of the elements in the alloy, brittleness is increased [55,56].

High-entropy alloys are a group of materials that combine between 5 and 13 different elements with atomic percentages of 5–35% [20,23,57]. The essence of high-entropy alloys is that it combines maximum static entropy with equal atomic ratios according to the Boltzmann entropy hypothesis [58]. The high strength of the solid solution allows for industrial applications of these high-strength alloys as high-temperature tools, molds, and components with high wear and oxidation resistance [17]. Mechanical alloying [59,60] is a solid state-of-the-art method of producing nanocrystals [23,24] with potential strategic applications [17]. In applications where impact resistance is very important, manganese austenitic steels can be used for component reconstruction [61]. Some coatings are used to resist abrasion, cavitation, corrosion, and friction and include a wide range of metals and their

alloys, ceramics, cermets, carbides, and even low-friction plastics. The usual hardness of coatings varies from 20–70 Rockwell hardness (HRC) [16,62,63]. Metal surfaces are hardened by rapid freezing, where hard phases, such as chromium carbides, tungsten, titanium or tantalum, and hard oxide impurities, are added separately [64–66].

The choice of hardening alloy is based on two main factors, namely, wear rate and cost. However, other important factors, such as base metal type, deposition process, impact, corrosion, oxidation, etc., must also be considered. Hardened alloys are typically used as powders or coated rods, flax-coated rods, solid welds, and long powder-coated welds. The different methods of surface treatment are divided into several groups, including building up, buttering, cladding, and hard-facing. This project may be the first of its kind in terms of using high-entropy nano-alloys as coatings in the above-mentioned industry.

The statistical studies of standard deviation, repeatability, and reproducibility were performed to evaluate the accuracy of the bond abrasion index results [67,68]. Experimental data of each alloy were fitted and examined by calculation of the correlation factor (R^2). Measurements regarding the standard alloy steel blade and alloy steel blade covered by N-HEA were calculated according to important statistical factors, such as standard deviation (s), test deviation (d), average standard deviation tests ($s_{\bar{x}}$), standard deviation of repeatability (S_r), and standard deviation of reproducibility (S_R), among others.

To date, very few studies have been conducted on HEA containing Hf [50], and none on HEA containing Ba. This investigation explores the properties of HEA containing these two elements.

2. Materials and Methods

2.1. High-Entropy Nano-Alloy Coating Method

In this paper, hardening operations were performed using welding. The manufactured powder of the high-entropy FeMoCrNiBaHf nano-alloy was formed into a flux-cored electrode and used as a coating on a sample of mild steel using the Oxyfuel powder spray (OFP) method. After welding, the thickness of the coating was 4 mm, as measured with a caliper, model IP 54 from the G.A.L Gage company (Bridgman, MI, USA). The most common hard-facing processes associated with the type of consumables appropriate to them are presented in Table 1.

Table 1. Common hard-facing processes with different types of materials used in them [12].

Hardfacing Process	Consumable
Oxyfuel/Oxyacetylene (OFW/OAW)	Bare cast or tubular rod
Shielded metal arc (SMAW)	Coated solid or tubular rod (stick electrode)
Oxyfuel powder spray (OFP)	Powder, tubular wire (flux-cored)
Gas-tungsten arc (GTAW)	Bare cast or tubular rod
Gas-metal arc (GMAW)	Tubular or solid wire
Flux-cored open arc	Tubular wire (flux-cored)
Submerged arc (SAW)	Tubular or solid wire
Plasma transferred arc (PTA)	Powder
Laser beam	Powder

2.2. Characteristics of High-Entropy Nano-Alloy

The nanoscale properties and X-ray diffraction data of this experimental trial are presented herein. According to the diffraction pattern of the FeMoCrNiBaHf advanced alloy sample, the peak bandwidth indicated a fine-grained and ultimately homogeneous solid network. In fact, the formation of a nano-crystalline structure and high lattice strain were the main factors of peak flattening, and the increase in lattice strain was due to the different atomic sizes of the intergranular components (Figure 1).

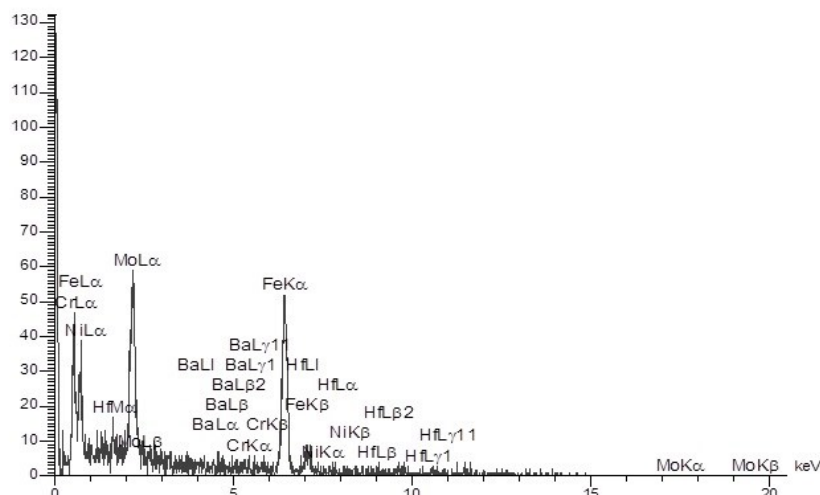


Figure 1. X-ray diffraction of high-entropy nano-alloy samples.

The morphology of the high-entropy FeMoCrNiBaHf nano-alloy magnified by a Fe-SEM (Field Emission Scanning Electron Microscopes) instrument (Fe-SEM-MIRA3, TESCAN-XMU) is shown in Figure 2. The smaller particles tended to boil and produce larger particles, while the larger particles themselves became smaller particles during the manufacturing process; consequently, the particle size distribution was very small. As can be seen, the particle dimensions were less than 100 nm (Figure 2).

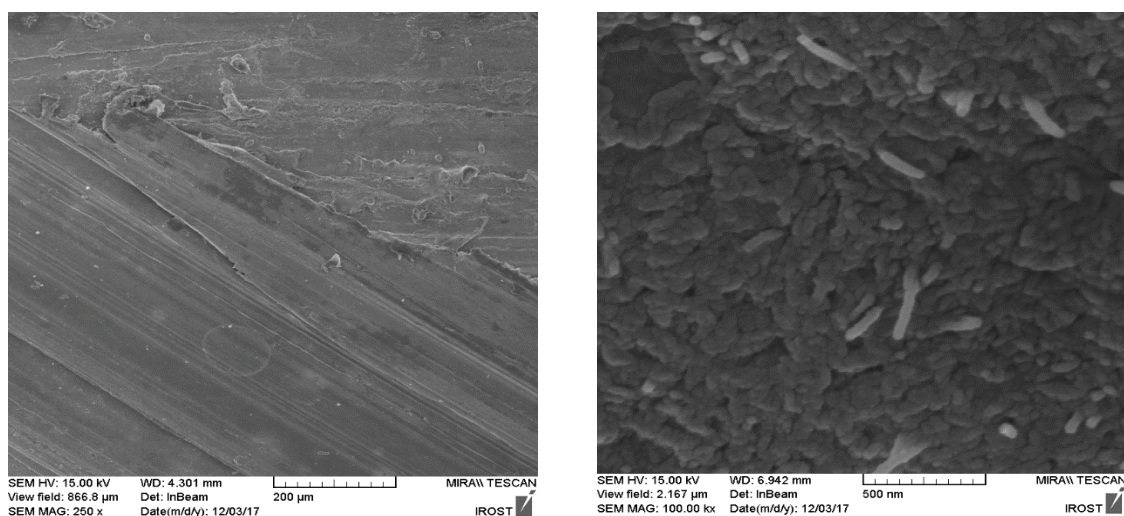


Figure 2. FE-SEM microscopy image of high-entropy nano-alloy, with dimensions less than 100 nm (current 298 μA; voltage 15.0 kV).

2.3. Preparation of Mineral Sample for the High-Entropy Alloy Wear Test

Iron ore (hematite) with 94% Fe_2O_3 from Afghanistan (Figure 3a) and copper oxide (malachite and azurite) samples were selected (Figure 3b) for use in this investigation. These specimens, which were required to be completely dry, consisted of 1.6 kg with particle sizes of $-0.019 + 0.0125 \text{ m}$ ($-\frac{3}{4} + \frac{1}{2} \text{ in}$), which were obtained by crushing larger particles and sorting using a screen. Usually, the initial value required for this experiment consisted of taking 5 kg of ore with a particle range of $-0.019 + 0.0125 \text{ m}$. The results of the XRF analysis are listed in Table 2.



Figure 3. (a) Hematite iron ore sample from Afghanistan. (b) Copper oxide ore sample from Iran.

Table 2. XRF test results for titration of iron and copper oxide samples.

Ore	Element (%)									
Copper Ore	Cu	TiO ₂	SO ₃	MnO	MgO	Fer	CaO	Al ₂ O ₃	SiO ₂	LOI
	4.88	1.32	0.25	0.82	2.4	22.88	27.93	7.72	29.44	2.36
Iron Ore	Fe ₂ O ₃	TiO ₂	SO ₃	MnO	MgO	K ₂ O	CaO	Al ₂ O ₃	SiO ₂	LOI
	93.96	-	0.33	-	-	0.12	0.62	0.83	1.77	2.37

2.4. Preparation and Testing of Mineral Abrasion Measurement Using Blades Coated with High-Entropy FeMoCrNiBaHf Nano-Alloy

Typically, the device consisted of 0.076 m × 0.013 m blades of a metal substrate and a base of hard alloy steel, which included some nickel and molybdenum (500 Brinell ~51 Rockwell). In this project, we tested the wear index once with a standard blade, and then again with the blade covered with high-entropy FeMoCrNiBaHf nano-alloy to a depth of 0.004 m. The test sample was obtained from Nano Mine Tech Co. The dimensions of both blades were selected using the same test. The blade was attached to a shaft that rotated at a speed of 624 rpm. The rotor was in the center of a cylinder, 0.305 m in diameter and 0.114 m high. The cylinder also rotated in the direction of the blade at a speed of 51 rpm (Figure 4).

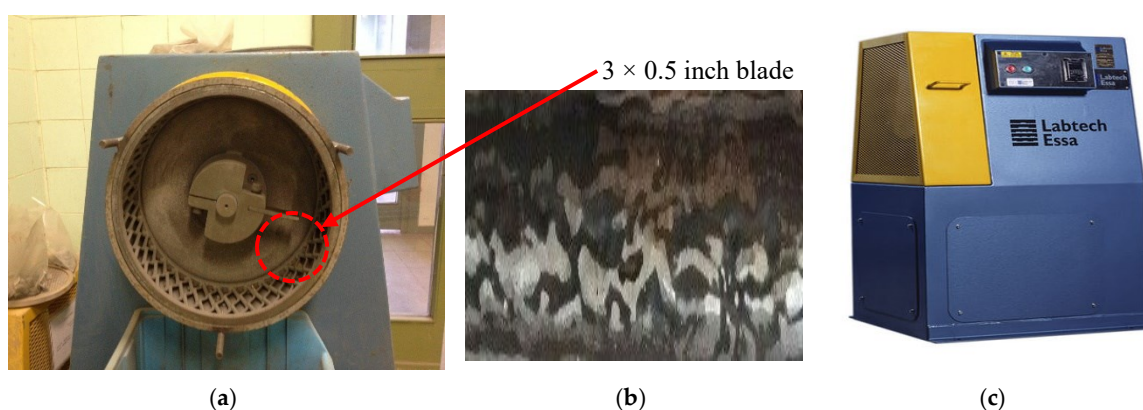


Figure 4. Mineral wear test of the band index, rotor, and mineral chamber (a); a blade coated with the high-entropy nano-alloy (b); full view of the mineral abrasive band index (Labtech Essa) (c).

This test did not require any special working conditions and was performed at ambient pressure and temperature to determine the mineral wear index of the equipment for mineral processing.

Wear studies were conducted using laboratory and pilot scale industrial samples according to the ASTM-G65 standard by exposing the metallic materials to scratching abrasions via the dry

sand/rubber wheel test. The abrasive used was 100% silica (SiO_2). The test conditions for the equipment included a wheel diameter of 235 mm, a sand flow of 320 g/min, a test load of 130 rpm, a wheel width of 12.7 mm, a wheel speed of 5610 revolutions/min, and a wheel hardness of Durometer A-60. The results of the abrasion test were reported as volume loss per cubic millimeter.

2.5. Measurement Procedure, Repeatability, and Reproducibility of Reduced Metal by Bond Abrasion Index

As shown by the tests for the first day and second day structures in Table 3, the mean weight loss due to abrasion was measured for 10 standard alloy steel blade samples and 10 alloy steel blades covered with N-HEA samples. Three measurements for each test (A and B) for samples 1–10 were performed; the total mean for the standard alloy steel blade was 0.0803×10^{-3} kg and the total mean for the alloy steel blade covered by N-HEA was 0.0908×10^{-3} kg. The standard deviation, repeatability, and reproducibility values based on 95% confidence levels were performed for the alloy steel blade covered by N-HEA, yielding figures of 0.00016, 0.00047, and 0.00040, and 0.0003, 0.00047, and 0.00042 for the standard alloy steel blade. The experimental data for each of the alloys were fitted and examined by calculation of the correlation coefficient (R^2). It was found that the alloy steel blade covered by N-HEA had an R^2 of 0.9423 and the standard alloy steel blade had an R^2 of 0.9552, which were both close to 1.0.

Table 3. The mean weight loss (kg) due to Bond abrasion process.

First Day (A)						Second Day (B)					
Abrasion Rate (AR) of Alloy Steel Blade Covered by N-HEA for Copper Ore						Abrasion Rate (AR) of Alloy Steel Blade Covered by N-HEA for Iron Ore					
x	$n = 30$	$\bar{x}, n = 10$	d	$S_{\bar{x}}$	\bar{X}	x	$n = 30$	$\bar{x}, n = 10$	d	$S_{\bar{x}}$	\bar{X}
1	0.0793×10^{-3}				0.0803×10^{-3}	1	0.0903×10^{-3}				0.0908×10^{-3}
2	0.0803×10^{-3}	0.0799×10^{-3}	-0.4×10^{-3}	0.52915×10^{-3}		2	0.0905×10^{-3}	0.0905×10^{-3}	-0.2×10^{-3}	0.2×10^{-3}	
3	0.0801×10^{-3}					3	0.0907×10^{-3}				
4	0.0801×10^{-3}					4	0.0905×10^{-3}				
5	0.0802×10^{-3}	0.0800×10^{-3}	-0.3×10^{-3}	0.20817×10^{-3}		5	0.0905×10^{-3}	0.0906×10^{-3}	-0.2×10^{-3}	0.11547×10^{-3}	
6	0.0798×10^{-3}					6	0.0907×10^{-3}				
7	0.0805×10^{-3}					7	0.0905×10^{-3}				
8	0.0800×10^{-3}	0.0801×10^{-3}	-0.2×10^{-3}	0.36055×10^{-3}		8	0.0908×10^{-3}	0.0908×10^{-3}	0.1×10^{-3}	0.35119×10^{-3}	
9	0.0798×10^{-3}					9	0.0912×10^{-3}				
10	0.0799×10^{-3}					10	0.0907×10^{-3}				
11	0.0804×10^{-3}	0.0804×10^{-3}	0.0×10^{-3}	0.45092×10^{-3}		11	0.0908×10^{-3}	0.0909×10^{-3}	0.1×10^{-3}	0.20817×10^{-3}	
12	0.0808×10^{-3}					12	0.0911×10^{-3}				
13	0.0798×10^{-3}					13	0.0909×10^{-3}				
14	0.0812×10^{-3}	0.0806×10^{-3}	0.2×10^{-3}	0.70946×10^{-3}		14	0.0910×10^{-3}	0.0910×10^{-3}	0.3×10^{-3}	0.1×10^{-3}	
15	0.0807×10^{-3}					15	0.0911×10^{-3}				
16	0.0797×10^{-3}					16	0.0904×10^{-3}				
17	0.0808×10^{-3}	0.0802×10^{-3}	-0.1×10^{-3}	0.55677×10^{-3}		17	0.0906×10^{-3}	0.0906×10^{-3}	-0.2×10^{-3}	0.15275×10^{-3}	
18	0.0801×10^{-3}					18	0.0907×10^{-3}				
19	0.0803×10^{-3}					19	0.0907×10^{-3}				
20	0.0806×10^{-3}	0.0803×10^{-3}	-0.1×10^{-3}	0.35119×10^{-3}		20	0.0907×10^{-3}	0.0907×10^{-3}	0.0×10^{-3}	5.7735×10^{-5}	
21	0.0799×10^{-3}					21	0.0908×10^{-3}				
22	0.0805×10^{-3}					22	0.0906×10^{-3}				
23	0.0807×10^{-3}	0.0804×10^{-3}	0.1×10^{-3}	0.36056×10^{-3}		23	0.0907×10^{-3}	0.0907×10^{-3}	0.0×10^{-3}	0.15275×10^{-3}	
24	0.0800×10^{-3}					24	0.0909×10^{-3}				
25	0.0804×10^{-3}					25	0.0907×10^{-3}				
26	0.0807×10^{-3}	0.0807×10^{-3}	0.4×10^{-3}	0.3×10^{-3}		26	0.0908×10^{-3}	0.0908×10^{-3}	0.1×10^{-3}	0.15275×10^{-3}	
27	0.0810×10^{-3}					27	0.091×10^{-3}				
28	0.0805×10^{-3}					28	0.0908×10^{-3}				
29	0.0811×10^{-3}	0.0808×10^{-3}	0.5×10^{-3}	0.3×10^{-3}		29	0.0909×10^{-3}	0.0909×10^{-3}	0.1×10^{-3}	1×10^{-4}	
30	0.0808×10^{-3}					30	0.0910×10^{-3}				

3. Results

We first divided the 1.6 kg sample from each ore into four 0.4 kg samples with a separator to measure wear resistance. This test was used to check the wear resistance of hard coatings. Both standard blades and blades coated with high-entropy nano-alloy were weighed with a precision of 1×10^{-7} kg using balancing. The blade was inserted into the rotor and secured by a clamp. The first sample (0.4 kg) was poured into the apparatus and sealed. The machine was turned on for 900 seconds using an automatic timer. This was repeated for all four samples of iron ore and copper ore and the product was kept separately for each step. The blade was then removed from the enclosure and thoroughly cleaned and dried with alcohol and acetone. Then it was weighed again. Screening for the test materials was performed in four steps up to about 0.000075 m. The amount of wear was measured, weighed, and expressed in standard units, as shown in the corresponding tables. The calculated values of the wear rate and index are displayed in Table 4. As can be seen, using the high-entropy alloy sample, the amount of metal was reduced significantly by pounds per kilowatt per hour of energy consumed.

Table 4. Calculated rates and wear indexes for both blades and the copper and iron ore samples.

The Bond Abrasion Index (A_i) and Abrasion Rate (AR)				Ore Type	
				Iron Ore	Copper Ore
Standard alloy steel blade, A_i (kg) (hardness 500 Brinell ~52 Rockwell–HRC)				0.1647×10^{-3}	0.1472×10^{-3}
Alloy steel blade covered by FeMoCrNiBaHf, A_i (kg) (hardness 746 Brinell ~67 Rockwell–HRC)				0.0908×10^{-3}	0.0803×10^{-3}
Standard alloy steel blade Abrasion rate (AR)	Dry ball mill (lb/kWh)	Balls	$0.05A_i^{0.5}$	0.0203×10^{-3}	0.0192×10^{-3}
		Liners	$0.005A_i^{0.5}$	0.0020×10^{-3}	0.0019×10^{-3}
	Crushers (gyratory, jaw, cone) (lb/kWh)	Liners	$(A_i + 0.22)/11$	0.0350×10^{-3}	0.0334×10^{-3}
Alloy steel blade covered by Nano high-entropy alloy Abrasion rate (AR)	Dry ball mill (lb/kWh)	Balls	$0.05A_i^{0.5}$	0.0151×10^{-3}	0.0142×10^{-3}
		Liners	$0.005A_i^{0.5}$	0.00151×10^{-3}	0.0014×10^{-3}
	Crushers (gyratory, jaw, cone) (lb/kWh)	Liners	$(A_i + 0.22)/11$	0.0283×10^{-3}	0.0273×10^{-3}

The hardness test results for the alloy sample were calculated as 67 Rockwell based on ASTM-G65 analysis. According to the abrasion rate parameters for the two samples of the hard alloy steel (including nickel and molybdenum) and the nano high-entropy alloy, the amount of alloy lost was reduced by 50%. For instance, for the dry ball grinder and crusher (gyratory, jaw and cone) the rate of abrasion in the balls and liners was different for the standard alloy. However, the rate was decreased using a nano high-entropy alloy. Alloy use is not only important from an economical standpoint, but also shows a direct effect on the breakdown time of equipment for repair and maintenance. Reducing the amount of the alloy lost by half could have a great effect on the mining economy.

As can be seen from the SEM micrographs, the wear mechanisms for the metal matrix of the high-entropy nano-alloy were a mixture of impact, abrasion, and scratching wear (Figure 5).

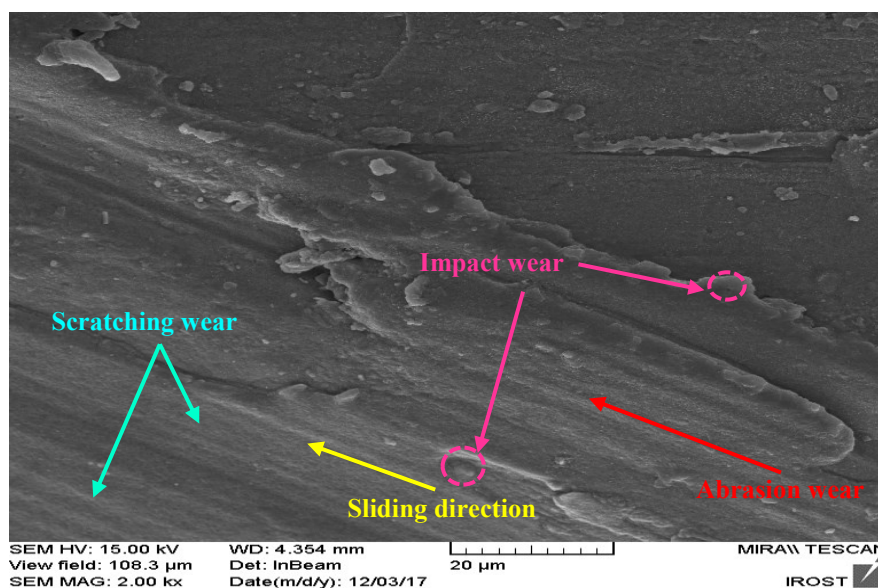


Figure 5. FE-SEM micrograph of wear of the metal matrix high-entropy nano-alloy, which were a combination of impact, abrasion, and scratching wear (current 298 μ A; voltage 15.0 kV).

4. Conclusions

Evaluation of the wear effect of natural and inorganic iron and copper samples on a high-entropy nano-alloy and a parent alloy in wear tests showed different weight losses of the blade. The experimental wear causing loss of mass due to the use of the iron ore and copper rock ranged from 0.0001647 kg to 0.0000908 kg and 0.0001472 kg to 0.0000803 kg, respectively, for the uncovered and HEA covered materials. A substantial amount of hard-wearing coatings is required to meet the mining industry's annual needs. By creating a hard and accessible nano-coating, wear and tear of materials can be significantly reduced, which is both cost-effective and, with the application of this technical innovation, could save energy. The use of high-entropy alloys in heavy industries is a new innovational option to allow producers to gain a competitive advantage in the market place.

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Conflicts of Interest: The authors declare no conflict of interest.

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