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Effect of Copper Addition on the Properties of TiO_2 Coatings Deposited by Air Plasma Spray Process

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ABSTRACT

Mild steel is a crucial engineering material with numerous engineering uses. Still, wear frequently causes mechanical parts made of mild steel to fail. Coatings prepared using an air plasma spray process may be a good choice for improving the surface properties of mild steel. Composite materials, such as Cu-TiO_2, can potentially improve the properties of mild steel, such as hardness, wear, and corrosion resistance. This study aims to investigate the effect of copper (Cu) addition on the microstructure, surface roughness, and wear properties (coefficient of friction (COF)) of the titanium dioxide (TiO_2) coating prepared by the air plasma spray (APS) method. The TiO_2 powder and 30 wt% Cu powder in the micron-sized range were mixed by the ball milling prior to the coating process. The particle size distribution and microstructure of the TiO_2 and Cu feedstock powders were studied using particle size analyzer (PSA) and field emission scanning electron microscopy (FESEM). Then, the properties of the as-prepared TiO_2 and Cu-TiO_2 coatings were characterized using FESEM, X-ray diffractometer (XRD), 3D non-contact profilometer and pin-on-disc tester. The TiO_2 and Cu-TiO_2 coatings were composed of well-melted and partially melted particles. The presence of Cu in the TiO_2 coating led to the deposition of a thicker coating of about 242 µm, which is 28.7% thicker compared to the TiO_2 coating alone. Compared to pure TiO_2 coating, Cu-TiO_2 coating showed lower surface roughness with less pores and microcracks, resulting in a reduced coefficient of friction. The finding of this study suggested that Cu addition to the TiO_2 coating on the mild steel substrate may produce better protective coating for wear and corrosion application.

1. Introduction

Mild steel is a typical structural material used in the aerospace industry, automotive, construction of pipelines, buildings, machinery parts and oil refineries [1, 2]. However, this particular steel is highly

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prone to wear and corrosion [3, 4]. The cost of maintaining mild steel-based parts increased due to this problem, driving up expenses for the business. Since corrosion and wear cause numerous failures in mechanical design, corrosion and wear-resistant coating is becoming a growing trend to extend the life of the metal surface components [5].

Attempts have been made to address this issue by modifying the surfaces of mild steels using various techniques, including applying surface coating on the metal surface. The coating protects mild steel from corrosion and chemical deterioration, which also helps improve wear resistance and corrosion [5-7]. Unlike stainless steel, mild steel is more susceptible to rusting, corrosion, and wear. Additionally, coating can be used as an option to reduce friction-related failures of devices or damage when machine components are sliding.

One of the coating materials that can be used is the composite coating of copper-titanium dioxide (Cu-TiO_2). It is considered a potential material to protect the surface of metals from wear and corrosion [8]; hence, it is widely used in the aerospace industry to produce turbine blades, which require high strength and wear resistance [1]. This material can also be employed in the biomedical field to produce medical implants [9]. Yin *et al.*, [10] reported the application of Cu-TiO_2 coating deposited on stainless steel surfaces for superhydrophobic surfaces for metal corrosion protection. The Cu-TiO_2 coating also can be used as a self-cleaning surface [11]. Several studies reported on the Cu-TiO_2 coating for photocatalytic applications [1, 12]. Khalid *et al.,* [13] conducted studies on the Cu-TiO_2 for visible light photocatalysis. Adding Cu into the TiO_2 was reported to have increased the photocatalytic efficiency of the TiO_2 as the catalyst. In addition, Raghav *et al.,* [14] prepared Cu-TiO_2 coating on a mild steel substrate for corrosion protection using a sputtering method. The study revealed that the Cu-TiO_2 coatings have better corrosion resistance than uncoated steel. Ashok *et al.,* [15] discussed the Cu-TiO_2 composite coating deposited by electro-co-deposition on the copper substrate. The result reveals that coatings prepared using the Cu-TiO_2 have increased wear properties where less wear loss was obtained. They concluded that the presence of composite materials composed of Cu and TiO_2 is believed to be the major contributor to better wear performance. This argument is supported by a study conducted by Ramalingam *et al.,* [16], which prepared Cu-TiO_2 coatings by electrodeposition on the copper substrate. The study concluded that Cu-TiO_2 coating has higher wear resistance and better corrosion resistance than the copper substrate. The study deduced that the characteristics of the starting particles, Cu-TiO_2, which have been investigated, have a major impact on the coating's improved qualities.

Even though several studies were reported on Cu-TiO_2 coatings, limited studies have been discussed on the properties of Cu-TiO_2 coatings deposited using the air plasma spray (APS) method. Regarding coating preparation, APS is highly flexible and effective compared to other techniques like vapour deposition, electrophoretic deposition, and sol-gel. This coating technique can produce large, thick, active coatings with the best flexibility and adhesion to complex-shaped substrates [17, 18].

Therefore, a comparative study is conducted between as-prepared plasma-sprayed TiO_2 and Cu-TiO_2 coatings. This study investigates the effect of Cu addition on properties such as the microstructure, surface roughness, and wear properties of the plasma-sprayed TiO_2 coating prepared by the APS method. The characteristics analysis of this study confirmed that the Cu-TiO_2 coating prepared by the APS method has better surface roughness and coefficient of friction, which is good for wear applications.

2. Methodology
2.1 Preparation of Coating

The starting powders used in this study were TiO_2 powders, namely Metco 6233C (Oerlikon Metco, Germany) and Cu powder, Diamalloy 1007 (Oerlikon Metco, Germany). Meanwhile, the Cu-TiO_2 powders were prepared in the laboratory. Firstly, 30 wt% of Cu powder was mixed into the TiO_2 powder using ball milling. Then, 100 g of powder was mixed with 50 ml of de-ionized (DI) water during the mixing stage for 2 hours. Finally, the powder mixture was dried at 80°C for 24 hours before being sieved through 80-mesh sieves.

A mild steel plate was used as the substrate material. The substrates were sandblasted with 24-mesh aluminum grit and then cleaned with acetone before spraying. An air plasma spray (SG-100 torch, Praxair, USA) was used to prepare the coatings. Table 1 displays the parameters used to prepare the TiO_2 and Cu-TiO_2 coating using the APS method.

Table 1
Process parameters used to prepare TiO_2 and Cu-TiO_2 coating

Parameter	Value
Arc current (A)	600
Primary gas argon (psi)	80
Secondary gas helium (psi)	40
Carrier gas argon (psi)	30
Powder feed rate (rpm)	4
Spraying distance (mm)	80
Robot speed (mm/s)	250
Preheat (cycle)	2

2.2 Characterization of Powder and Coating

The particle size analyzer (PSA, Mastersizer Malvern Instrument) was used to analyze the particle size distribution of the feedstock materials. A field emission scanning electron microscope (FESEM, Hitachi SU 5000) was used to observe the morphology of the powder, the obtained fractured cross-sections and the surface of the coating. The phase analyses of the feedstock powders and the deposited coatings were conducted using XRD (Rigaku Miniflex 600-C) with Cu-Kα radiation (λ = 1.54056 Å) over the 2θ angle from 20° up to 80°. A 3D non-contact profilometer (Shodensha) was used to measure the surface roughness of the as-prepared plasma sprayed coatings. A micro pin-on-disc tribometer (Model CM9109) performed the sliding wear test on the coatings. The tests were conducted for 30 minutes in a dry condition with a load of 10N. Then, the coefficient of friction of the coatings was measured using the instrument.

3. Results
3.1 Feedstock Powder Characterization

Figure 1 presents FESEM (Field Emission Scanning Electron Microscope) images of the TiO_2 and Cu powders in their as-received state. In both cases, the feedstock powders exhibited a spherical-like shape. It is evident from the FESEM analysis that both types of powders fell within the micron-sized range. Furthermore, upon closer examination at higher magnification, as illustrated in the insets of Figure 1(a & b), it becomes apparent that these powders resulted from smaller individual particles agglomerating through the spray drying process. Figure 1(c & d) displays the particle size distribution for TiO_2 and Cu, respectively. These results confirm that the TiO_2 and Cu powders possess diameters within the range of 39.31-118.41 µm and 33.33-65.00 µm, respectively.

The morphology and uniformity of the feedstock materials are known to be parameters that can influence APS coating deposition [19, 20]. These criteria will result in good flowability, which will contribute to a more consistent feed rate and result in better coating deposition on the substrate. Furthermore, particle size distribution in the micron range is preferable for the APS method to avoid powder clogging during the injection system [21]. Therefore, the feedstock powders used in this study are suitable for the APS process, and a uniform and thick coating can be anticipated.

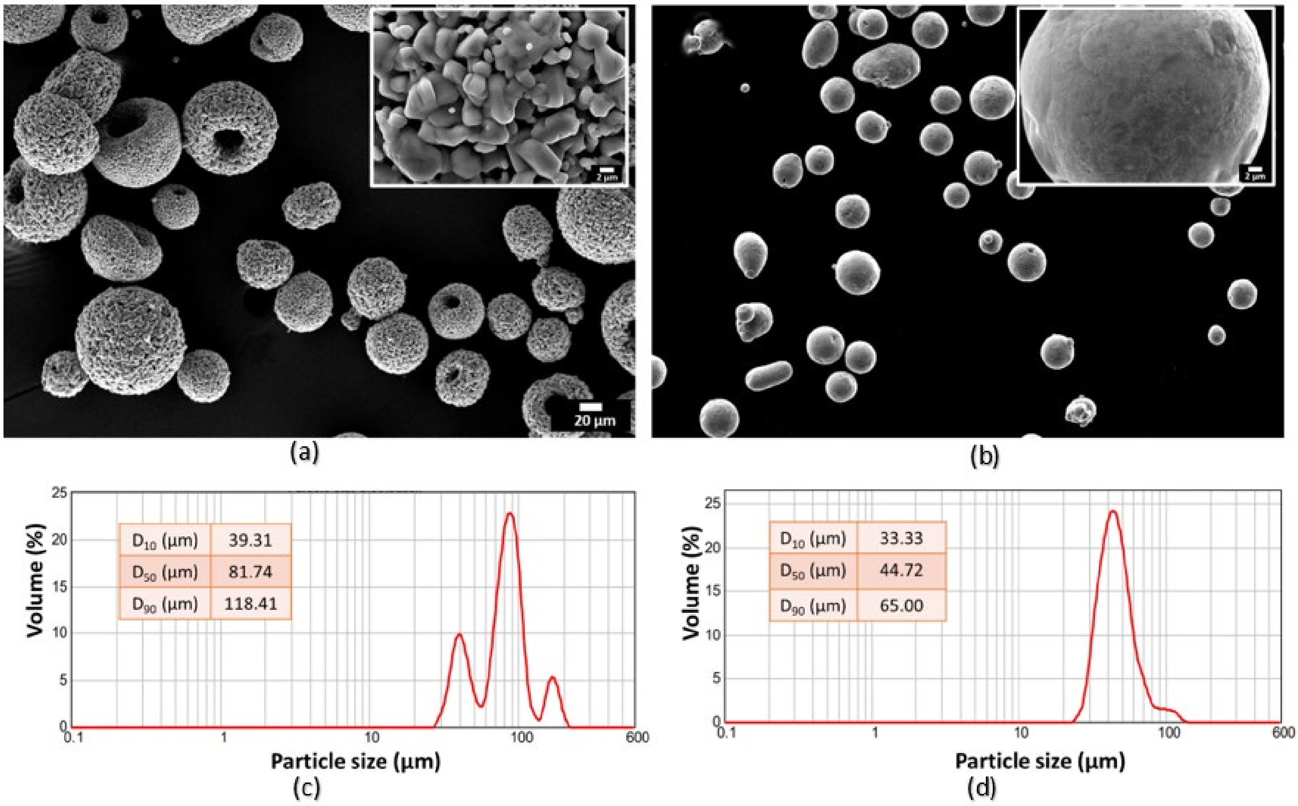


Fig. 1. Morphologies and particle size of the feedstock powders, respectively (a & c) TiO_2 (b & d) Cu

3.2 Coatings Characterization

Figure 2 shows FESEM images of the cross-section of the coatings. A pure TiO_2 coating has a thickness of about 188 µm, as shown in Figure 2(a). Meanwhile, Figure 2(d) illustrates that the Cu-TiO_2 coating is slightly thicker than the pure TiO_2 coating, which is approximately 242 µm thick. The detailed cross-section of the pure TiO_2 coating is shown in Figure 2(b & c). The results show that the coating prepared using only TiO_2 has more pores than the coating sprayed with Cu-TiO_2 as feedstock materials, resulting in an uneven surface at high magnification image as in Figure 2(c). It is suggested that the entrapment of unmelted particles within completely melted particles resulted in large and fine pores [5]. In contrast, Figure 2(d-f) shows a cross-section of a Cu-TiO_2 coating, which has fewer pores than a pure TiO_2 coating, possibly because Cu is present in the composition.

This study's finding suggested that adding Cu to TiO_2 increases deposition efficiency, as evidenced by the 28.7% increase in thickness of the as-sprayed Cu-TiO_2 coating. The as-sprayed coating has a different coating thickness and number of pores due to the different melting temperatures of the Cu and TiO_2 powders. The presence of Cu, which has a lower melting point of 1083°C compared to TiO_2, which has a higher melting point of 1843°C, led to a lower melting point temperature of composite Cu-TiO_2, contributing to the deposition of thicker coating.

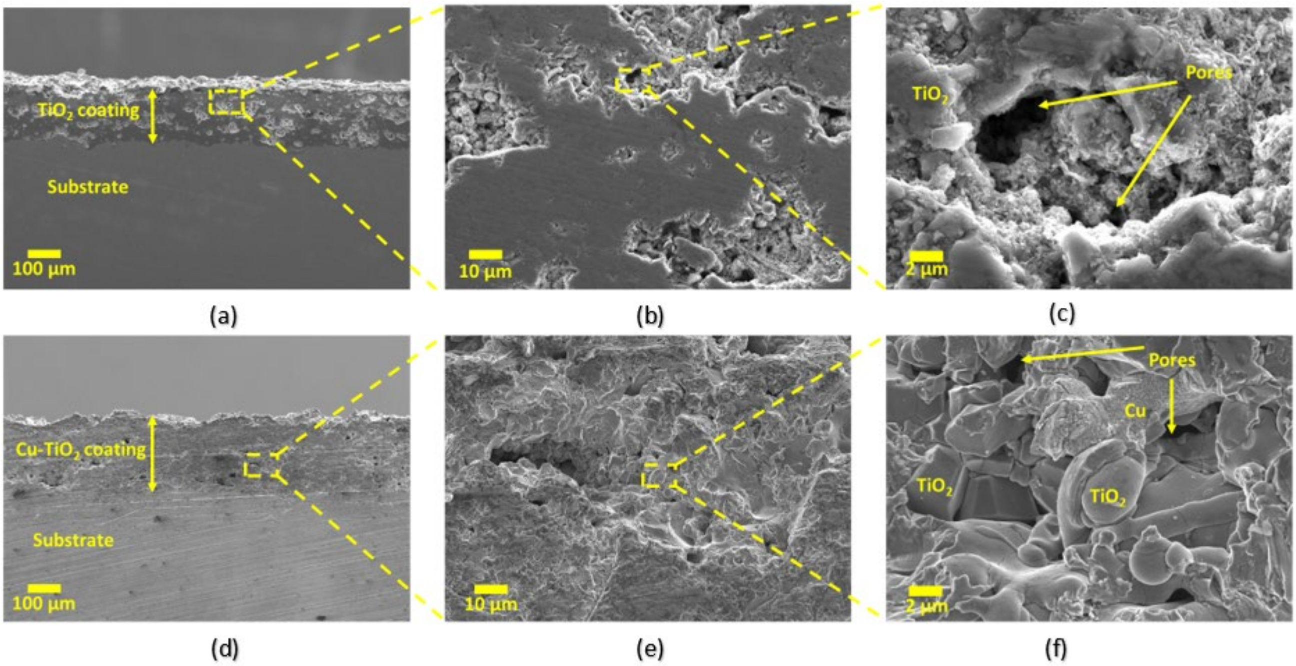


Fig. 2. Cross-sectional micrographs and magnified detail of coating of (a-c) TiO_2 coating and (d-f) Cu-TiO_2 coating

The surface morphology of the coatings is shown in Figure 3. Meanwhile, the type of element the morphology represents is confirmed by the SEM-EDX analysis in Figure 4. From the SEM images, the coatings have a bi-modal structure composed of fully and partially melted regions within splats. The presence of distinct regions in the bi-modal structure makes it more wear-resistant than a single-structure coating [6, 22]. When comparing the surface morphology of the two coatings, the pure TiO_2 coating has fewer flattened splats and more partially melted particles. In contrast, more splatting was observed on the Cu-TiO_2 coating due to Cu addition. Figure 3(d) shows that spherical-like Cu particles were most likely melted and re-solidified before impacting the substrate or previously deposited particles during their flight in the plasma jet. Furthermore, microcracks were discovered on both types of coatings. Shi *et al.*, [23] mentioned microcracks are always present in ceramic coatings caused by stored strain during the thermal cycling APS process.

Figure 4 shows the SEM-EDX analysis of Cu-TiO_2 coating at various points. The analysis reveals large Cu peaks on the white regions, as shown in Figure 4(a), indicating Cu. This region of Cu is considered completely melted. Meanwhile, as shown in Figure 4(b & c), large Ti and O peaks are observed in grey regions, indicating TiO_2. However, for TiO_2, two distinct areas were observed: fully melted regions as shown in Figure 4(b), and partially melted areas as presented in Figure 4(c). The current result confirms the presence of Cu on the surface of the Cu-TiO_2 coating. Furthermore, the results indicate that TiO_2 and Cu were distributed uniformly on the composite coating, which is due to both powders' good flowability in the APS process as a result of the proper mixing methods used to produce Cu-TiO_2 powder.

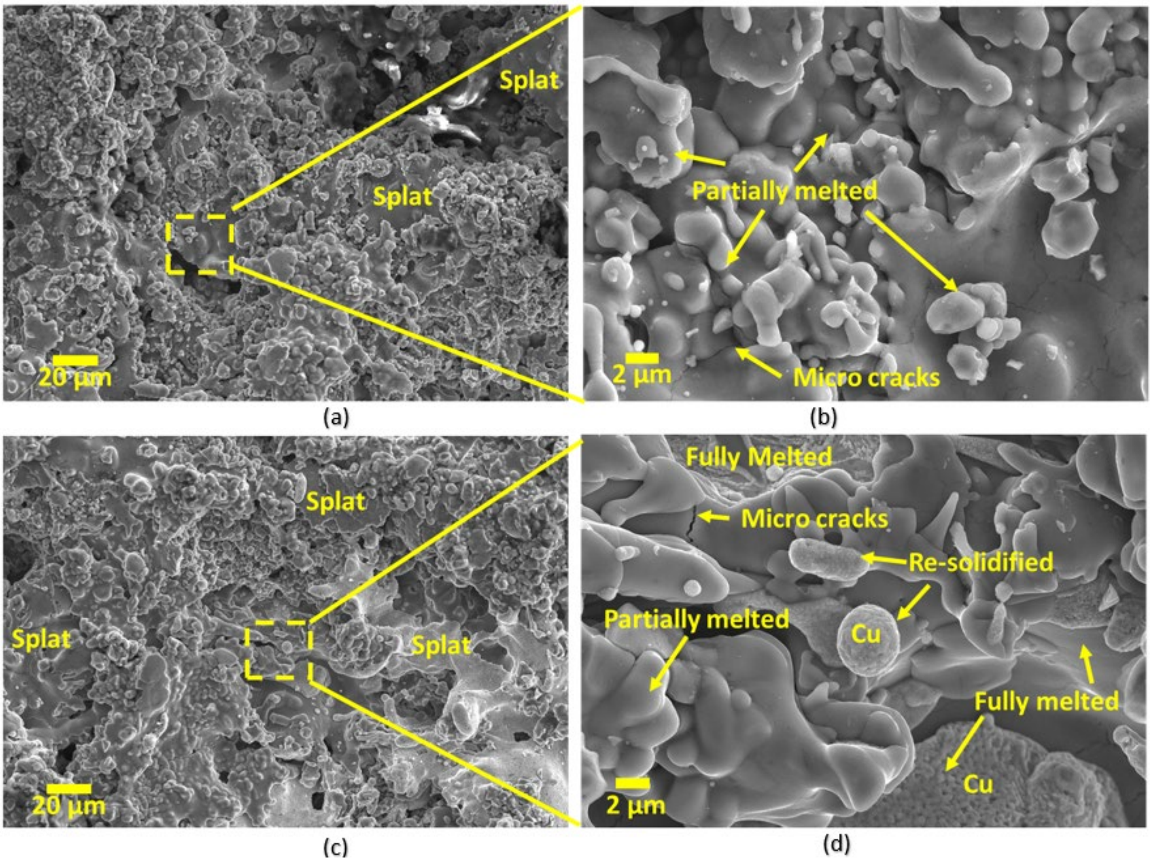
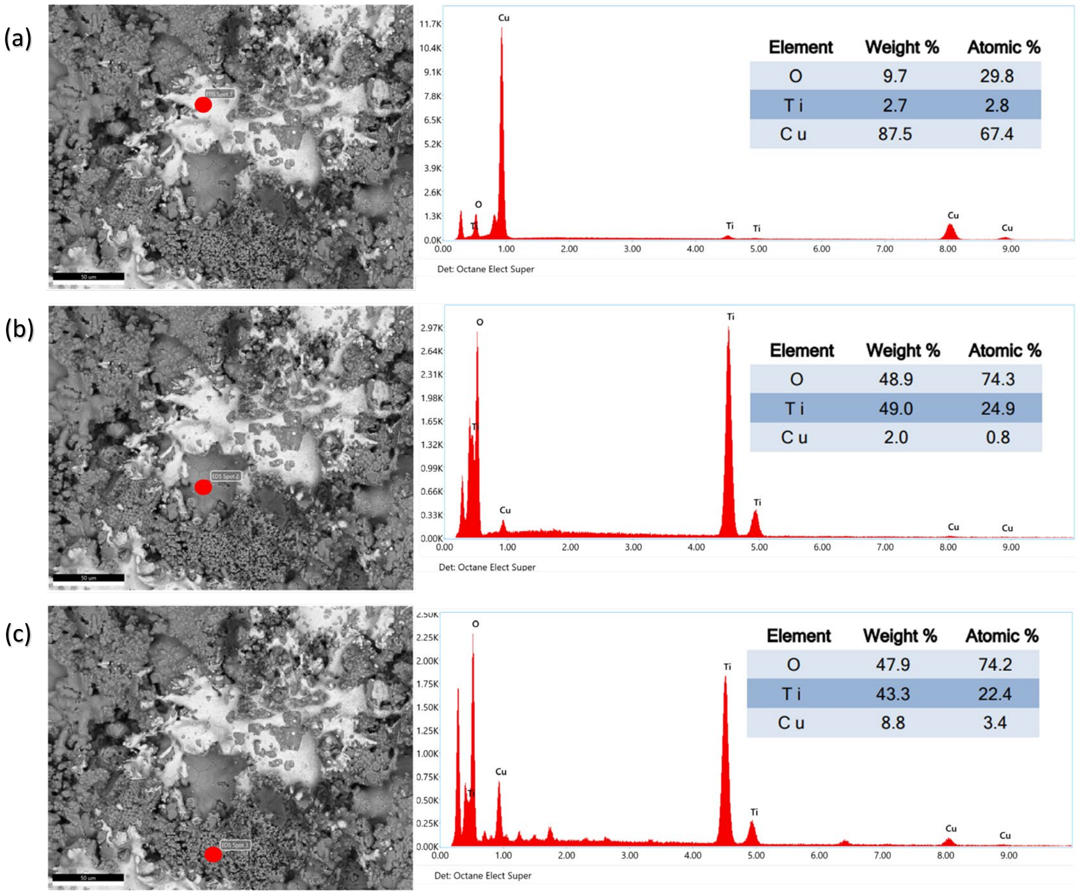


Fig. 3. FESEM surface morphology of (a-b) TiO_2 coating and (c-d) Cu-TiO_2 coating



(a)

Element	Weight %	Atomic %
O	9.7	29.8
Ti	2.7	2.8
Cu	87.5	67.4

(b)

Element	Weight %	Atomic %
O	48.9	74.3
Ti	49.0	24.9
Cu	2.0	0.8

(c)

Element	Weight %	Atomic %
O	47.9	74.2
Ti	43.3	22.4
Cu	8.8	3.4

Fig. 4. EDX analysis on different area of Cu-TiO_2 coating prepared by APS

Figure 5 shows the XRD patterns of TiO_2 powder, Cu powder, TiO_2 coating, and Cu-TiO_2 coating. According to the XRD pattern as depicted in Figure 5, TiO_2 powder is composed of rutile as the major phase (JCPDS 00-21-1276) and anatase as the minor phase (JCPDS 00-025-1164). In all temperature and pressure conditions, the rutile phase is the most stable among the titania phases, including the anatase phase. The XRD pattern of Cu powder shows the typical peaks of (1 1 1), (0 0 2), and (0 2 2), at 2 theta (degree) of 43.3°, 50.4°, and 74.1°, respectively. These peaks are very well coordinated with the standard data (JCPDS 98-005-2256) and provide further evidence of the existence of the crystalline cubic phase.

Changes in the phase composition were observed as a result of thermal spraying. Both coatings seemed to be mainly composed of the rutile phase of TiO_2 with no appearance of anatase phase. A significant increase in crystallinity is observed due to high temperature during the APS. The XRD analysis reveals that cubic Cu has been retained in the Cu-TiO_2 coating, indicating Cu's presence in the as-prepared Cu-TiO_2 coating.

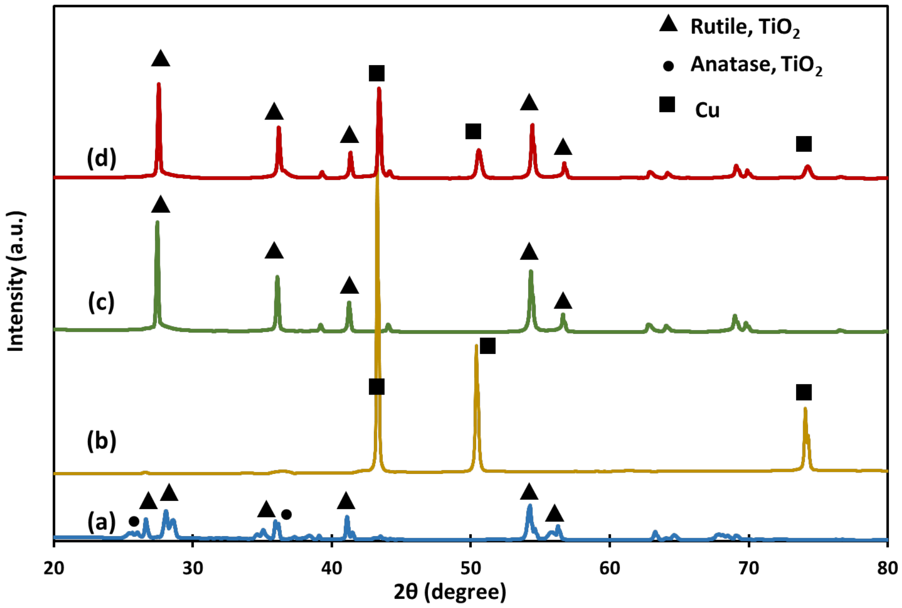


Fig. 5. XRD patterns of (a) TiO_2 powder, (b) Copper powder, (c) TiO_2 coating and (d) Cu-TiO_2 coating

The surface roughness Ra of the TiO_2 and Cu-TiO_2 coatings calculated using an average of five readings is shown in Figure 6. It is observed that the Cu-TiO_2 coating had a lower Ra than the TiO_2. The Ra of TiO_2 and Cu-TiO_2 was 2.12 and 1.82 µm, respectively. Therefore, adding Cu to TiO_2 resulted in a smoother surface finish. The observation is frequently observed in the deposition of composite materials using APS [5, 19, 23]. When feedstock powders are made up of different materials, their fusing or melting temperatures vary during the APS. Because of the temperature difference, different materials will react at different time frames during the melt, cool, deform, solidify, and contract stages before forming a deposit. In this case, the feedstock powders of Cu and TiO_2 had different fusing points of 1083°C and 1843°C, respectively. Hence, the particles went through a liquid sintering phase between 1083°C and 1843°C and a solid sintering phase when the temperature fell below 1083°C, resulting in partially melted regions. The Cu powder has a higher degree of powder melting than TiO_2 due to its lower melting temperature.

Consequently, only Cu powders will melt, forming a fully molten splat on the coating during the liquid sintering phase. The molten Cu would act as the binder and fill the pores between the Cu and

TiO_2 particles during the adhesion process while forming the Cu-TiO_2 coating. In this case, the Cu powder is a joining agent for TiO_2 particles. This mechanism will improve the density of the Cu-TiO_2 coating by reducing the space between the splats [6]. As a result, it reduces the porosity of the coating and produces a smoother surface, improving surface roughness.

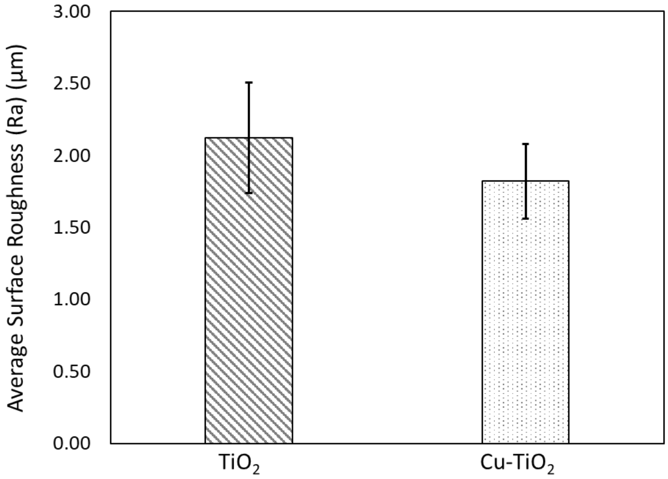


Fig. 6. Comparison between surface roughness of TiO_2 and Cu-TiO_2 coating

The wear properties of the coating are measured from their coefficient of friction (COF). The COF of TiO_2 and Cu-TiO_2 coatings is plotted against the sliding time in Figure 7. It is discovered that the TiO_2 coating has a higher COF than the Cu-TiO_2 coating. The outcome is contributed by the surface roughness of the coatings, where Cu-TiO_2 coating had a smoother surface than the TiO_2 coating. It is widely accepted that the surface roughness of the coating influences its wear properties. Few studies [8, 17] show that increasing the coefficient of friction increases the wear rate. In theory, as the roughness decreases, so does the wear rate. Smoother surfaces typically exhibit lower friction coefficients because there are fewer asperities or irregularities to generate friction. It is important to note that the wear properties of TiO_2 and Cu-TiO_2 coatings in this work agree with the theory and other reported findings. Hence, adding Cu into TiO_2 would decrease friction and improve the wear properties of the Cu-TiO_2 coating.

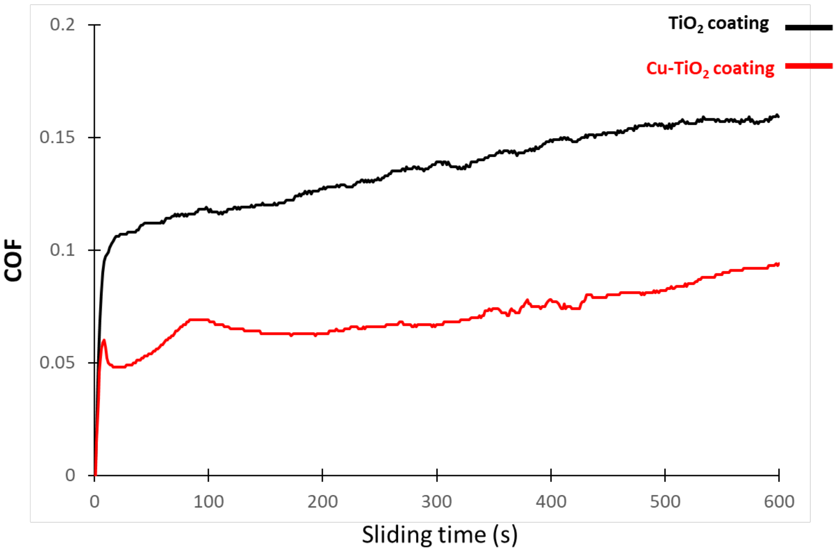


Fig. 7. Coefficient of friction (COF) of TiO_2 and Cu-TiO_2 coating

4. Conclusions

In conclusion, TiO_2 and Cu-TiO_2 coatings were successfully deposited on mild steel substrates as protective coatings by the APS method. The coating prepared using Cu-TiO_2 powder was thicker by about 28.7% than pure TiO_2. A typical bi-modal microstructure consisting of fully melted and partially melted materials was observed on both coatings. The phase composition of the coating, composed mainly of rutile TiO_2 with Cu, was observed in the Cu-TiO_2 coating. Adding Cu to the TiO_2 led to a coating with lower surface roughness, which resulted in better wear properties, as shown by the low COF value.

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