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Evaluation of Fracture Durability and Wear Resistance Properties for Orthopaedic Implant Application

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Metallic substrates coated with hydroxyapatite (HA) are commonly utilised for orthopaedic implants. Titanium grade 2 and Titanium grade 5 samples were individually plasma spray-coated with HA and HA reinforced with 5% (by wt.) multiwalled carbon nanotubes (MCNT). The wear and tear behaviour of these coated substrates was investigated using simulated bodily fluid (SBF) with a pH ranging from 7.20 to 7.40. The fracture durability and wear resistance of the HA-MCNT composite coat were designed to be superior to that of pure HA coating. This improvement is due to the basic mechanical features of MCNT as well as the crack restricted connection effect. The inclusion of MCNT also aided in fracture confinement in the coatings. As evidenced by SEM pictures, abrasive and persistent wear.

Keywords: hydroxyapatite; titanium grade 2; titanium grade 5; plasma sprayed technique; nanoindentation; tribo-test

I. INTRODUCTION

Chemically similar to human bones, hydroxyapatite (HA) is a bioactive substance that promotes bone growth and osseointegration. Its exceptional compatibility with living tissues and enhancement of tissue growth on the metallic implant surface has led to its use in orthopaedic load-bearing applications. HA bio-ceramic powder is applied to these metal surfaces (Solanke *et al.*, 2022; Solanke *et al.*, 2020). In the medical field, metallic materials coated with HA are typically utilised for implants. Artificial implant-bone fractures early due to rapid wear caused by brittleness and low fracture toughness of HA (Eliaz & Metoki, 2017). Numerous studies have documented that wear is the cause of the deterioration and eventual failure of orthopaedic implants. After 12 to 16 years of use, knee and hip implants start to degrade (Schwartz *et al.*, 2020; Aksakal *et al.*, 2004). The fracture toughness of bio-ceramic HA coatings is in the range of 0.5- 1 ($MPa.m^{0.5}$) as reported in various research studies and the corresponding value of natural compact or lamellar bone is in the range of 2- 6 ($MPa.m^{0.5}$) (Kumar *et al.*, 2013; Tan *et al.*, 2011). So, improvement in the wear properties and fracture toughness of the HA coatings used in bioimplants is required.

Recently many researchers have tried various reinforcements with HA to improve the mechanical properties of bio-metal substrates. Solanke *et al.* (2020) investigated effect of varying stand-off distance on the mechanical and tribological properties of plasma-sprayed HA coatings on metallic substrates. It has been concluded that 230mm stand-off distance is optimum for better mechanical and tribological properties. The various bio-ceramic reinforcements are aluminium oxide (Al_2O_3), titanium oxide (TiO_2), Carbon nanotubes (CNTs), and zirconia oxide (ZrO_2). Many researchers have tried carbon nanotube (CNT)

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reinforced in HA and have found advancement in the wear resistance of the composite coating (Kwok *et al.*, 2009; Catauro *et al.*, 2017; Balani *et al.*, 2007) observed a threefold improvement in the fracture strength of plasma sprayed HA coating on the titanium grade 5 (TIGR5) metallic implant with the addition of 4 wt. % CNT. Tercero *et al.* (2009) worked on the hybrid composites by reinforcing varying % (by wt.) of Al_2O_3 and CNT with HA bio-ceramic coated on TIGR5 substrate. Improvement in fracture toughness was observed in the composite coatings. Similarly, Khanal *et al.* (2016) also worked on the hybrid composites using functionalised CNT and nylon in HA bio-ceramic. The physical properties of HA bio-ceramic were found to be improved in this study. Varying % (by wt.) of multi-walled carbon nanotube (MCNT) reinforced in nano-hydroxyapatite (nHA) using microwave irradiation process resulted in improved compressive strength as reported by Khan *et al.* (2017) research group. CNT has a high potential to strengthen the HA bio-ceramic due to its excellent mechanical, structural, electrical, and thermal properties coating without compromising its bioactivity and biocompatibility. Crystallinity in the composite coating increases due to the higher speed of heat transmitted through MCNTs (~3000 W/mk) compared to HA (1.30 W/mK) (Singh *et al.*, 2020). Very few studies reported the production of thin HA coating with the help of different thermal processes on Titanium grade 2 (TIGR2) substrate. As per the literature survey, TIGR2 may be a suitable material as metallic bioimplants in medical industry owing to its surface structure stability (Carlos & Alejandro, 2012). Many research papers dealt with composite HA-coated metallic substrates, but very limited literature is available pertaining to titanium grade 2 and titanium grade 5 coated with MCNT reinforced HA. No research study has been carried out using 5 % (by wt.) MCNT reinforced HA coating on titanium grade 2 substrate. Therefore, in this work, HA, and HA- MCNT coatings were fabricated on TIGR5 and Titanium grade 2 (TIGR2) metallic substrates using the plasma spray coating method. Both the coatings were plasma sprayed on the substrates using optimum process parameters. The coated substrates were then tested for wear properties and fracture toughness using standard procedure. SEM images were taken for all the coated substrates before and after the wear test.

II. EXPERIMENTAL PROCEDURE

A. Preparation of Substrate Samples

Titanium grade 5 (TIGR5) and TIGR2 metallic substrates in solid form were obtained from Mumbai, India. The samples having 40 mm diameter and 6 mm thickness for in vitro wear test were prepared by using laser technology. The reported values of TIGR5 and TIGR2 densities are 4.43 g/cc, 4.52 g/cc respectively, (Elias *et al.*, 2019; Solanke & Gaval, 2020). Initially polishing of metallic substrates was carried out followed by cleaning with acetone ultrasonically. The drying of cleaned samples has been carried out in an air-circulated oven at 75^{o}C for 10 hours. The standard ASTM G99-95a was followed for sample preparation. The fatigue strength along with biocompatibility is desired for knee and hip bioimplants. At present titanium and its alloys are becoming popular for orthopaedic implants.

B. Coating Preparation

The plasma spray coating technique was used to create the HA and HA-MCNT coatings on the TIGR5 and TIGR2 substrates. Pure white alumina (Al_2O_3- 200 to 250 µm) was used to grit blast metallic samples at a high velocity and 4- 5 bar blasting pressure. To strengthen the bond between the coating and the metallic samples, this is done before the coating. After grit blasting, the sample's surface roughness ranged from 2.5 to 3.5 µm. The degree of surface roughness has an impact on how well the coating adheres to the metallic substrate. The embedded alumina grit particles were then removed from the grit-blasted samples by ultrasonography, which took five to ten minutes. Grease or other contaminants present on the substrate. Table 1 shows process parameters of plasma spray coating method.

Table 1. Process parameters of the Plasma spray coating method

Sr. No.	Process Parameters	Value with unit
1	Plasma power	25-28 kW
2	Primary gas	Argon
3	Secondary gas	Argon
4	Flow rate	08-10 g/min
5	Stand-off Distance (SOD)	230 mm
6	Traverse Velocity	38-46 mm/s

C. Experiment Details

1. Microstructural properties of the coatings

The HA and HA-MCNT coated metallic samples were characterised for microstructure and worn-out surfaces using FE-SEM at MMMF lab, IIT Bombay. All the coatings were analysed to find out % of open porosity by using Image J software. The average of five readings at different locations on the coating was reported. The phase composition of HA, HA-MCNT powders and fabricated HA, HA- MCNT coatings was determined by performing XRD analysis using EMPYREAN diffractometer system. XRD analysis was done by keeping the 2θ value between 20°- 80° with an increment of 0.02°. Weight percentage of elements in the coating was found out by carrying EDS analysis of coated samples.

2. Microstructural properties of the coatings

Bonding Strength Measurement: ASTM- C633 standard was followed for the measurement of coating bonding strength. The coated samples were held between the two cylindrical jigs made of stainless-steel using FM 1000 polyamide-epoxy resin film. The tensile load was applied to the coated substrates in perpendicular direction until initiation of fracture at the interface between coating and substrate. The load applied was then gradually increased till the failure occurs. The adhesive strength was calculated using load and area at which the sample failed. The average of five readings for each sample was computed and considered as final adhesive strength. The fracture occurred at the interface in most of the samples.

Nanoindentation measurement: Nanoindentation test was conducted to find nano-hardness (H) and elastic modulus (E). Nano-hardness is calculated from the ratio of maximum applied load to the area of contact, while reduced elastic modulus is calculated from the load versus depth of penetration plots using the recognised Oliver and Pharr (O-P) method. A constant load of 10 mN was kept. Ten indents at different locations of the samples were made for each coated sample. The experiments were performed ten times on each HA-coated sample, and an average value was recorded. For the nanoindentation experiments, Berkovich indenter was used with 65.30 semi-apex angle and a small tip radius around ~200 nm. The nanoindentation experiments were performed as per DIN 50359-1 standard.

3. Tribological testing

A ball-on-disc machine was used to conduct a unidirectional sliding wear test at room temperature with Simulated Body Fluid (SBF). Following Kokubo's recipe (Kokubo *et al.*, 1990), simulated bodily fluid with a pH in the range of 7.2 to 7.4, like human blood plasma, was created. For the wear test, indenters in the shape of 10 mm-diameter steel balls with an 875 HV hardness were employed. The AISI 52100 grade steel used to make the balls was acquired from Ducom Instruments in Bengaluru, India. Before the wear test, the samples' surfaces were cleaned using ethanol. The steel ball is kept stationary throughout the test, and a flat disc is rotated against it at a predetermined load value. The LVDT sensor on the machine recorded wear occurred between ball and substrates. This machine is attached with a computer. The computer has WINDUCOM 2010 software, which displays the results in graphical form. The wear experiments were conducted as per the ASTM G99 standard. During the test, the wear loss and COF values were noted and examined.

D. Selection of Wear Parameters

Based on the wear circumstances between the acetabular cup and the femoral ball head, the wear parameters were selected. Walking typically causes hip joint stress that ranges from 0.7 to 4 MPa (Ipavec *et al.*, 1996). Frictional forces are anticipated to be experienced by HA-coated hip prostheses for a period of 18 to 25 years (Neumann *et al.*, 1994). Since it is impractical to carry out these studies over an extended period of time, 10 N, a load that is thought to be higher in wear tests, is applied. Ten repeat experiments were conducted, and the average of five readings was used to determine the average wear weight loss in the tested samples.

III. RESULT AND DISCUSSION

Sinter granulated Hydroxyapatite (HA) powder with good flowability is used as feedstock powder. The average particle size of HA powder is in the range of 40 to 60 µm, Crystallinity> 95 %, Phase purity > 95 % and Ca / P ratio-1.67. This HA powder is produced and used specifically for thermal spray applications. Figure 1 shows SEM image of the sintered HA powder.

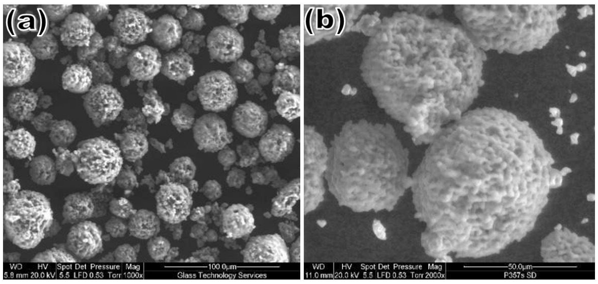

Figure 1. SEM image of HA powder

Sintered HA powder (particle size 40-60µm) was mixed with 5 wt. % of MCNT (95% purity, 35-55 nm OD, 0.6-2.1 µm in length, density- 2.2g.cm^{-3}, BARC, Mumbai) in ball mill to obtain composite powder/ powder precursor. Figure 2 and 3 shows the SEM and TEM images of MCNT powder used in this research work.

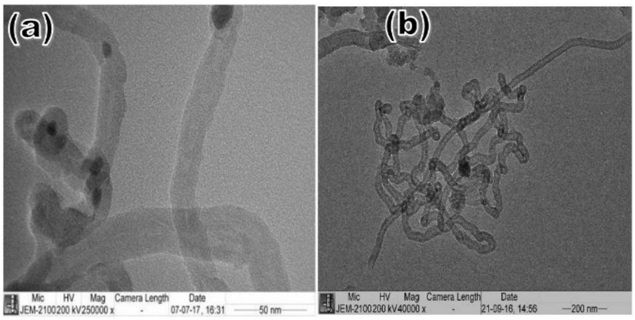

Figure 2. TEM images of the MCNT powder

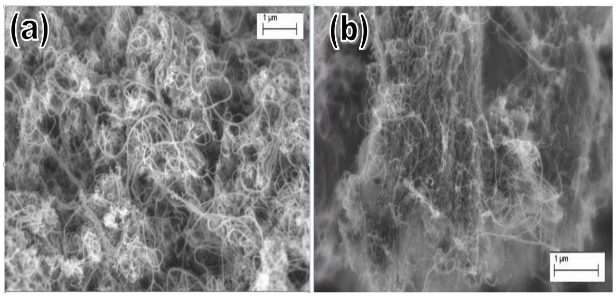

Figure 3. SEM images of the MCNT powder

The typical HA and HA-MCNT coating thickness was observed to be around 167 µm on both the metallic substrate as seen from the SEM micrograph.

Figure 4(a), 4(b), 4(c), and 4(d), 4(e), shows the plan-sectional microstructures of the plasma sprayed HA and HA-MCNT coatings. In Figure 4(c), the EDS data shows that the HA coatings developed by plasma spraying had stoichiometric Ca/P of 1.67 that carries the signature of the pure HA phase formation. Uniform distribution of MCNTs is seen in the HA precipitate in the powder form. As evident from SEM images, HA coating is comprised of irregular splats shaped, nano inter-splat crack, unmelted semi-melted HA powder particle, as well as a significant amount of nano, meso, and micro porosities. It is indicated by dotted circle and ellipse in Figure 4. SEM images of MCNT reinforced HA coating show the well formation of flattened splats. Table 2 showed the microstructural properties of plasma-sprayed HA and HA-MCNT coatings.

A. Mechanical Characteristics

The average radial crack length in HA coatings was observed around 37 µm for TIGR5 and 41 µm for TIGR2 substrate. However, in the case of HA-MCNT coatings, the average radial crack length was found to be around ~16 µm for TIGR5 and ~18 µm for TIGR2 substrate. The decrease in average radial crack length resulted in significant improvement in the fracture toughness of coatings. Crack deflection and MCNT pullout energy are one of the toughening mechanisms in improving the fracture toughness of the HA-MCNT coatings. The fracture toughness of coatings was calculated using the Anstis equation (Anstis *et al.*, 1981).

$$k_{IC} = 0.016 \left(\frac{E}{H}\right)^{0.5} \frac{P}{C^{1.5}} \qquad (1)$$

Where k_{IC} - Fracture toughness, E - Elastic modulus, H - Hardness of coating, P - Applied load and c - Radial crack length.

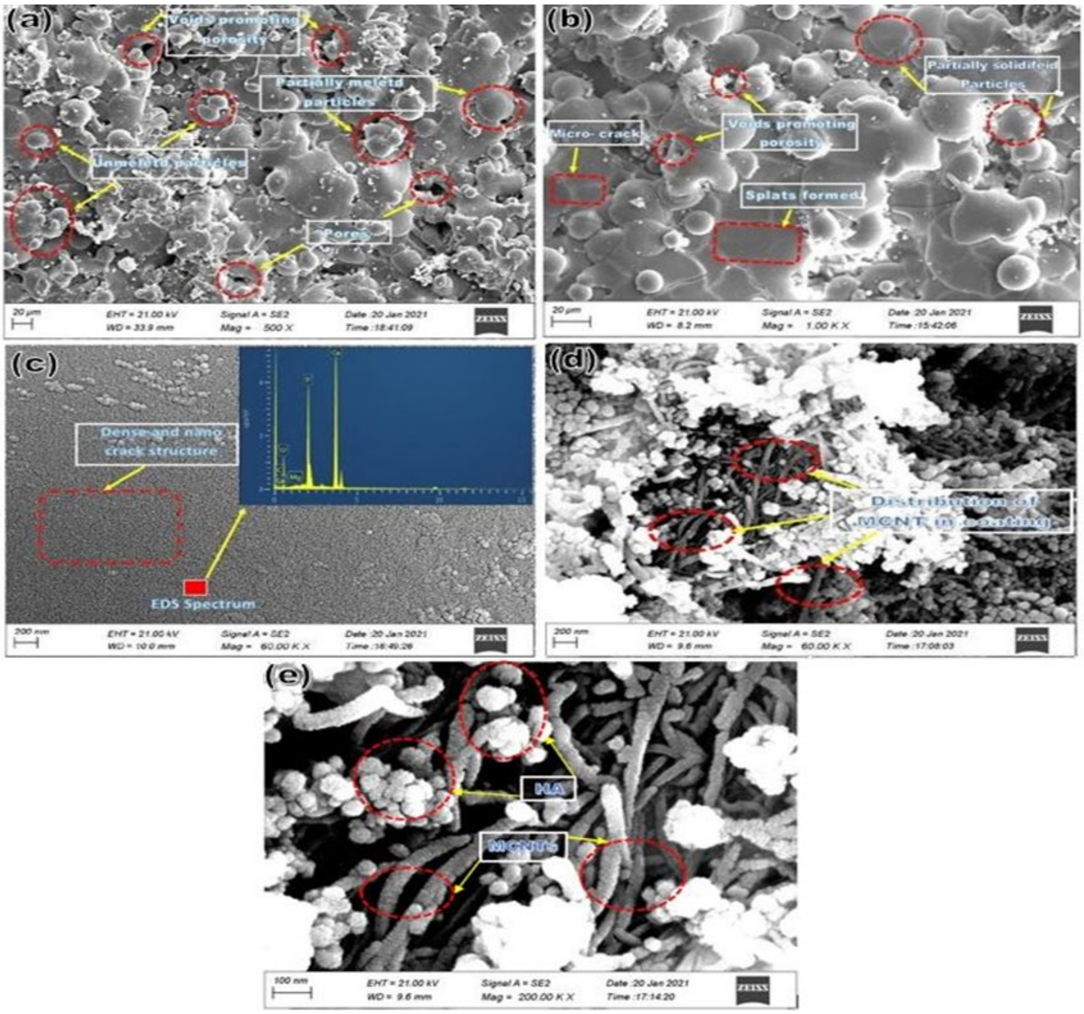

Figure 4. (a-c) Micrographs of plasma HA coatings, (d-e) Micrographs of plasma HA-MCNT coatings

Table 2. Microstructural properties of plasma-sprayed HA and HA-MCNT coatings

Coating	**Avg. Hardness (Gpa)**		**Avg. Elastic modulus (Gpa)**		**Avg. Porosity (%) of coating**		**Avg. Crack length (μm)**		**Fracture toughness (MPa.m$^{0.5}$)**	
	TIGR5	TIGR2	TIGR5	TIGR2	TIGR5	TIGR2	TIGR5	TIGR2	TIGR5	TIGR2
HA	1.6	1.8	21	21	21	19	50	53	0.59	0.56
HA-MCNT	4.3	4.1	120	116	8	7	16	18	3.35	2.90

The average fracture toughness of 3.35 and 2.90 ($MPa.m^{0.5}$) was found for MCNT-reinforced HA-coated TIGR5 and TIGR2 substrates, respectively. These fracture toughness values are in the range fracture toughness values of natural cortical bone (Nalla *et al.*, 2005). This improvement in fracture toughness is attributed to the inherent mechanical properties of MCNT and the crack-bridging effect offered by MCNT. The addition of MCNT to HA powder helps in restricting crack propagation in the coatings. The modulus of elasticity, hardness, and fracture toughness of the plasma sprayed coatings are found to be increasing with the addition of MCNT to HA powder.

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B. Friction and Wear Characteristics

To measure wear weight loss of materials, the weight of HA and MCNT-reinforced HA-coated substrates were recorded prior to and after the wear test by Electronic Weighing machine. The average wear weight loss of coated substrate materials in grams is depicted in Table 3. Significant reduction is observed in average wear weight loss and COF with the addition of 5% (by wt.) MCNT to HA powder. The lower average COF and wear weight loss in HA-MCNT coatings indicates its better wear resistance properties in SBF environment conditions. Figure 5 shows the images after the wear test on HA and HA-MCNT coated samples. This may be attributed to the good dispersion of the peeled-off graphene sheets in the coatings. This peeled of graphene layers of MCNT provides adequate lubrication. Fracture toughness of HA coating increases by the addition of MCNT, which makes the removal of mass difficult.

Table 3. Wear weight loss and COF (Gram)

Substrate sample	Avg. wear weight loss (gram)	COF
TIGR5(HA)	0.0059	0.48
TIGR2(HA)	0.0063	0.57
TIGR5(HA-MCNT)	0.0019	0.35
TIGR2(HA-MCNT)	0.0022	0.37

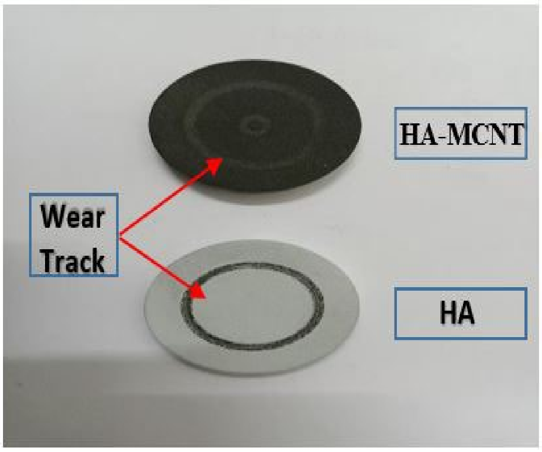

Figure 5. Wear Test on HA and HA+ MCNT coated samples

Figures 6 and 7 show the average value of wear loss and COF for coated titanium substrates. The addition of MCNT reduces wear weight loss volume of HA coatings. Higher wear rate is indicative of shorter life of an orthopaedic component (Geringer *et al.*, 2006). The retention of generated wear debris from the coatings may cause body infection and produce more toxins in the human body (Brodin, 2020; Kostarelos, 2010). Improvement in the wear resistance will generate lesser volume of wear debris, which may reduce chances of disturbance in the actual biological environment around the implant. The improvement in the wear resistance offered by MCNT-reinforced HA coating may be a combined effect of increase in elastic modulus and hardness due to presence of MCNT in the coating. Higher values of mechanical properties offer more wear resistance for the same applied normal load during the tribo-test under wet conditions. The mechanical and wear properties of MCNT reinforced HA coated TIGR2 substrate are observed to be slightly inferior as compared to MCNT reinforced HA coated TIGR5 substrate. This may be attributed to mechanical anchorage, good atomic coherence property, and physical-chemical interaction as per the literature (Prasad *et al.*, 2017). Reduced atomic coherence leads to a weakened chemical bond of the coating.

Figure 6. COF for plasma-sprayed HA and HA-MCNT coatings

Figure 7. Average wear weight loss for plasma-sprayed HA and HA-MCNT coatings

B. Worn Surface Morphology

The wear behaviour of HA and MCNT reinforced HA coatings has been studied with the help of morphology of wear track obtained through SEM images. The wear mechanism of HA-coated TIGR5 and TIGR2 substrates are observed to be dominantly affected by adhesion and abrasion such as brittle cracking, brittle fracturing/ rupture, plastic deformation, abrasive grooving as evident from SEM.

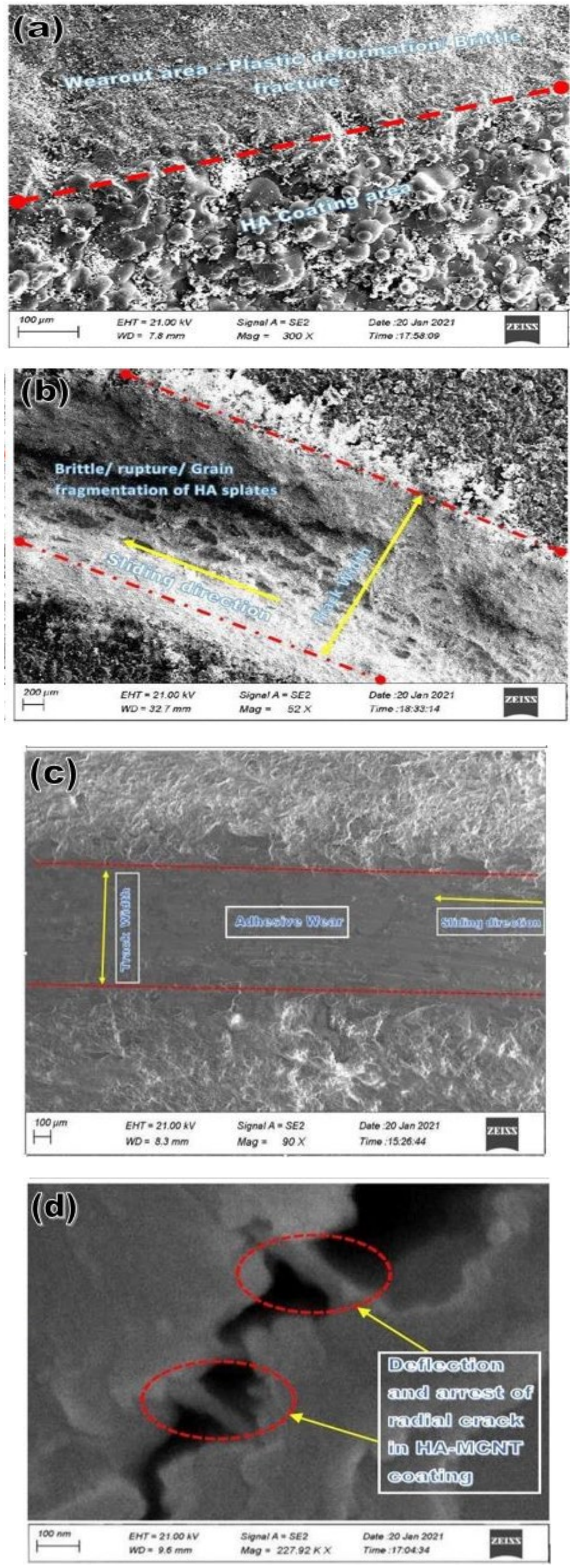

Figure 8. Micrographs of worn-out plasma-sprayed HA and HA-MCNT coatings after tribo-test

Figure 8 shows the HA-coated surface prior to tribo-testing and worn-out surface post tribo-testing. The wear track generated on HA coatings were observed to be more profound and broader than HA-MCNT coatings. It has been reported by various researchers that MCNTs offer the lubricating condition during severe abrasive wear (Schwartz *et al.*, 2020; Aksakal *et al.*, 2004). The SEM images clearly shows brittle fracturing/rupture phenomenon anchored by the addition of MCNTs in the coatings, and thereby allowing limited wear damage to HA-MCNT coatings via abrasive. Worn morphology of HA-MCNT coatings reveals the transformation of brittle fracture/rupturing (abrasive fracture) in HA coating to minor abrasive wear. MCNTs greatly removed the abrasive wear mechanism due to MCNT bridging of mass on wear track of HA-MCNT coatings. Microstructural investigations and worn morphology of HA-MCNT composites have proven the role of the inherent characteristics of MCNT reinforcement in improving the mechanical and tribological behaviour of MCNT-reinforced HA coatings.

IV. CONCLUSION

The addition of MCNT to both Titanium substrates has resulted in a significant improvement in the tribo-mechanical properties of HA coatings. The addition of MCNT to HA has also demonstrated a significant improvement in the elastic modulus and average hardness. When MCNT is added to HA, it is discovered that the porosity and average crack length are significantly decreased. The peeled-off graphite layers from the MCNT surface enhance lubrication in the wear track when MCNT is added to HA coatings. The worn morphology of HA-MCNT coatings demonstrates how minor abrasive wear and plastic deformation have replaced brittle fracture in the hydroxyapatite coating. The potential of MCNT reinforcement in the advancement of HA-MCNT fabricated coatings is revealed by microstructural investigations.

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