

Experimental Investigation of Biocompatibility in Laser Metal Deposited Titanium-Molybdenum Composite

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Abstract— Composites are materials acquired from the combination of two or more materials with the aim of having an improved property from the two or more original base materials combined. The combination of Titanium and Molybdenum are found to be a good biomaterial because of its significant corrosion resistance and mechanical biocompatibility. This paper reports on the experimental investigation of laser metal deposition of Titanium and Molybdenum composite using Laser Metal Deposition technique of additive manufacturing. The biocompatibility of Titanium/Molybdenum Composite as a biomaterial. The study show that the addition of Molybdenum to Titanium alloy through the results obtained from the Vickers hardness, biocompatibility and corrosion resistance were improved.

Keywords— biocompatibility, bio-material, laser metal depositions.

I. Introduction

Biomaterials are “nonviable materials used in medical devices with the intention of interacting with the biological system” [1]. They are commonly used in the repair, replacement or argumentation of diseased or damaged parts of the musculoskeletal system such as bones, joints and teeth It’s in very important that biomaterials meets the minimum requirement which is ensuring that both material and the tissue environment of the body coexist without having any detrimental or unsuitable effect on each other. In view of this biocompatibility is an essential requirement and non-negotiable for a biomedical application, which is the ability of the material to perform efficiently with an appropriate host response for the desired application [2].

Laser Metal Deposition (LMD), a unique manufacturing technique employed in the build-up of components most especially components with intricate parts, layer by layer according to the sequence defined by the geometry of the computer design model data of the component to be built [3]. It is important to highlight that the building of complex intricate parts layer by layer is one of the outstanding benefits

of the LMD process over the traditional manufacturing methods. Building of complex component in a single process as a unit, that need not to be coupled or assembled after the process remained a revolution to the manufacturing industry to date.

Traditional approach on the other hand, may involve assembling or joining of manufactured parts together to form the desired component, which are always time consuming and has associated wastages at different stages of the manufacturing of the individual parts. At the long run most traditional method of manufacturing are energy intensive considering the different stages before the eventual manufactured of the desired component. The benefits of LMD process is not only in the building of new components but in the repair and refurbishment of worn out components to extend its useful service life instead of discarding such component [4]-[7].

Some medical devices made from metallic biomaterials are commonly used for orthopedic implants mounted on to the skeleton of human body to aid healing, correct deformities and restore lost function of the original part. It includes bone plates, screws, total hip joints, knee joints, elbow joints, shoulder joints and reattachments for tendons or ligaments [2]. The use of metals such as iron, silver and gold for biomedical implants has been from time immemorial [8]-[9]. These metals were the candidate materials used for surgical implants at some points, however, the improvement and new technology leading to generation of new materials renders the continues use of these materials not fit for purpose as bioimplants.

In view of this, the demand for metallic implant began to increase [10]-[11] and the failures of not fit for purpose materials made it possible for the applications of the new materials. Some of the common metals uses for bioimplants include stainless steel 316L [12]-[15], Titanium and its alloys [16]-[18], Cobalt based alloy [19]-[22], Tantalum [23]-[24] etc. The development of pitting and crevice corrosion identified as a drawback in stainless steel as bioimplants opened up opportunities for Titanium and it alloys as an effective material for bio-medical applications because of its high specific strength to weight ratio and good corrosion resistance [2]. Titanium alloy grade 5 in particular is the most used biomaterial in the dental, orthodontic applications and most bioimplants. The identified drawback in Titanium and its alloy necessitated the surface modification with Molybdenum to improve the corrosion and wear property for an improved biocompatibility. This investigation aimed at depositing

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Molybdenum on Titanium alloy grade 5 for an enhanced hardness, biocompatibility and corrosion resistance.

II. Materials and Methods

A. Sample Preparation

The materials employed in the investigation are the substrate (6 mm Ti6Al4V plates), the powders for deposition are the Ti6Al4V and Molybdenum and with morphology showing particle sizes of 150-200 μ m and 45-120 μ m respectively (Fig. 1). The Ti6Al4V powder is characterised particles spherical in shape while the molybdenum powder has an irregular shape morphology.

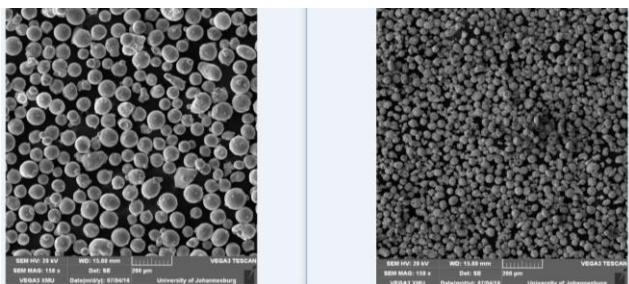


Figure 1. Morphology of Ti6Al4V and Mo

The substrates were sandblasting to remove all dirt's and roughen the surface for good bonding of the deposits on the Ti6Al4V and cleaned with acetone before deposition.

B. Deposition Process

A KUKA robot with 4.4 Nd: YAG laser system were employed for the deposition process with an improvised protective covering enclosed with Argon to protect the deposit from atmospheric contamination of oxygen and Nitrogen, the level; of Oxygen and Nitrogen were kept below 10 ppm. The sandblasted and cleaned substrates are positioned on a fixed table while the robot move along the defined path to perform the deposition while the two powders were placed in different hoppers.

The deposition were achieved with the flowing of both Ti6Al4V (3.6 rpm) and Mo (0.4 rpm) powders from the hoppers through the nozzles onto the substrate where the laser beam is focused creating a melted pool of the deposit on the substrate forming tracks of Ti6Al4V-Mo along the defined path of the robot, as shown in the schematic (Fig. 2). Several combination of both powders were mixed but 90% Ti6Al4V and 10% Mo is reported, with constant laser power of 1.7kW, scan speed range of 0.050 – 1.5 m/min, beam size of 2mm, and focal distance of 195 mm. The deposit achieved multiple of six tracks over lapping each other by 50% with scan speed increment of 0.25 m/min (Fig. 2).

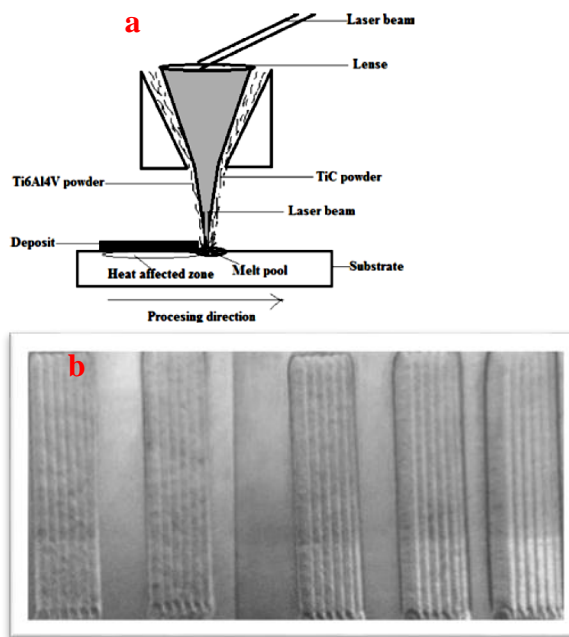


Figure 2. (a) Schematic of Laser Metal Deposition [3] (b) Deposited Ti6Al4V-Mo Samples

C. Characterization Methods

After achieving the deposition, the samples were cut to reveal the cross-sections. The cut samples were mounted, ground, polished, and etched using Kroll's reagent. The etched samples were observed under both optical microscope and Scan Electron Microscope (SEM), the samples were also subjected to Energy Dispersion Spectrometry (EDS). The micro hardness profiling was conducted on the samples using Vickers indenter with a load of 350 g at a dwell time of 15 seconds. Lastly, the corrosion property was conducted by immersing the samples into Hanks balanced salt solution in a cylindrical jar, the jar is kept in water bath with temperature kept at 37°C.

III. Results and Discussion

The results and the discussion of the investigation are enumerated in the following sections.

A. Microstructural Evaluation

The microstructural evaluations were conducted on the etched samples through both optical microscope and Scan Electron microscope. The micrograph of the deposit (Fig. 3) shows different microstructural zone formations - (i) the deposit zone- which is made up of Ti6Al4V-Mo composite and some un-melted Molybdenum, (ii) the interfacial zone – point of fusion of the deposit and the substrate whereby mixing both to form metallurgical bonding; (iii) Heat affected zone – part of the substrate affected by the heat during the deposition process; and (iv) Substrate – part of the material not affected by the heat from deposition.

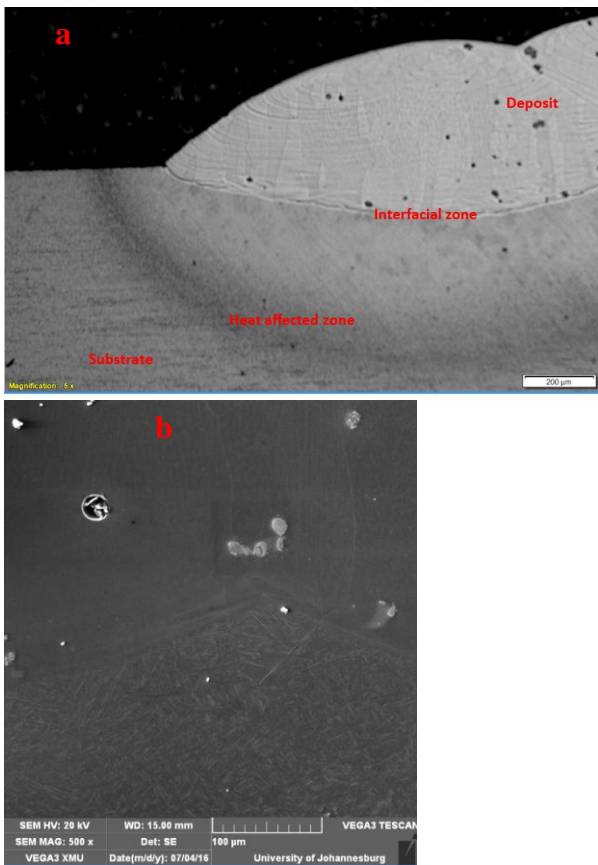


Figure 3. (a) Optical micrograph of deposit showing the zones and (b) SEM micrograph showing grain boundaries, columnar grains and un-melted Molybdenum

The composite deposit of Titanium and Molybdenum is characterized with Columnar grains formation with smaller grains at the top due to the effective cooling at the top surface when compared to other parts away from the surface. Deposit formed at high scan speed (1.5 m/min) are characterized with un-melted Molybdenum, which was due to the lack of sufficient heat to melt the molybdenum.

B. Grain Deformation

The deposition process generate high temperature in the process of formation of the melt pool, hence, most of the residua heat sink directly into the substrate where the melt pool is formed. The grains formed are function of the heat dissipated and the cooling rate, grains far from the interface region received less heat from melt pool resulting in smaller grain growth. The fusion zones are characterised with elongated grains. The grain sizes distribution show that the fifth deposit had the least grain size (Fig. 4).

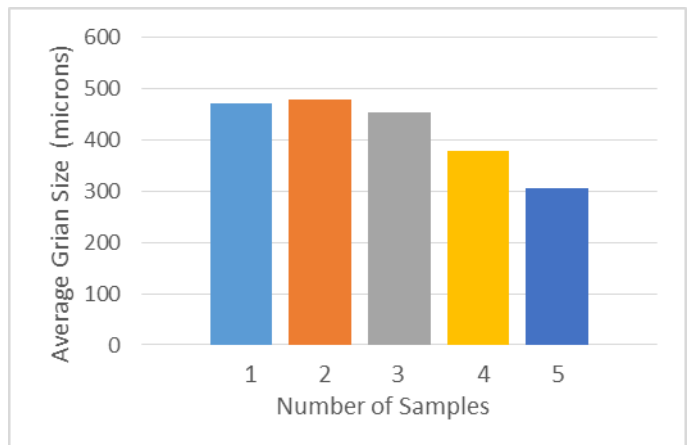


Figure 4. Plots of average grains formation across the five deposited composite

This was attributed to the high scan speed employed (1.5 m/min) in the deposition where by reducing the laser material interaction and inter mixing of the composite. This revealed an inverse relationship between the grain sizes and the scan speed.

C. Vickers Micro hardness

It is important to strike a balance between high and low Vickers hardness values because a too high value may lead to metallurgical notch which is undesirable for most industrial applications. While a too low HV value may affect the ability to retain a desired shape when a desired shape is made. The plot of Vickers hardness across the three zones of the five deposited composite (Fig. 5) shows that hardness is highest at the heat affected zone.

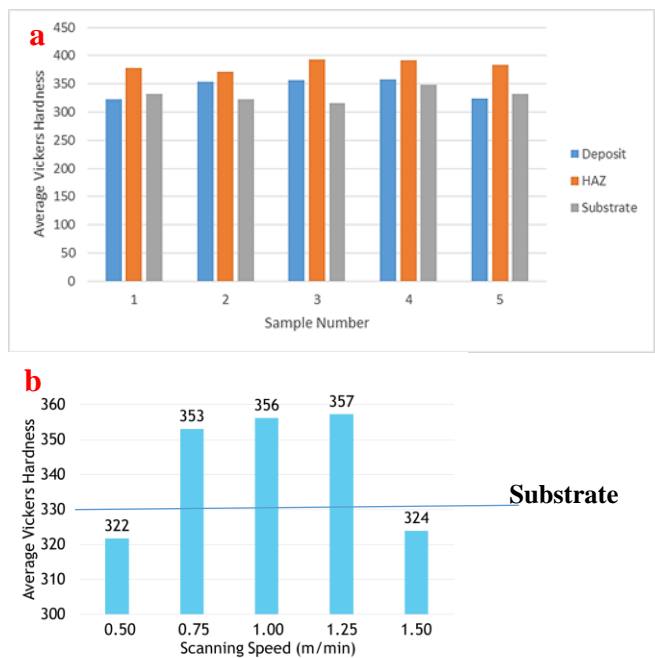
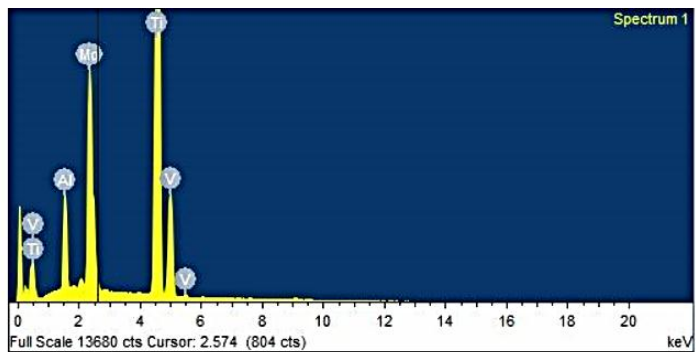
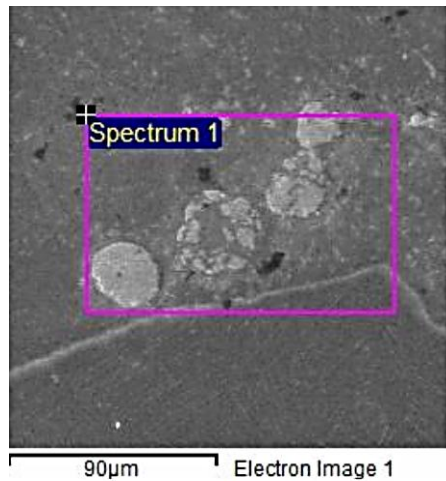


Figure 5. (a) Average hardness profile, (b) average hardness against scanning speed

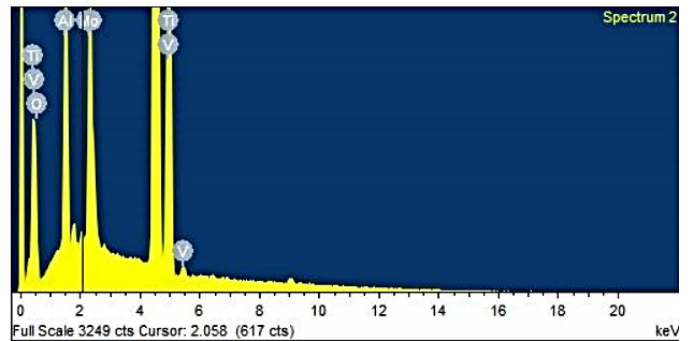
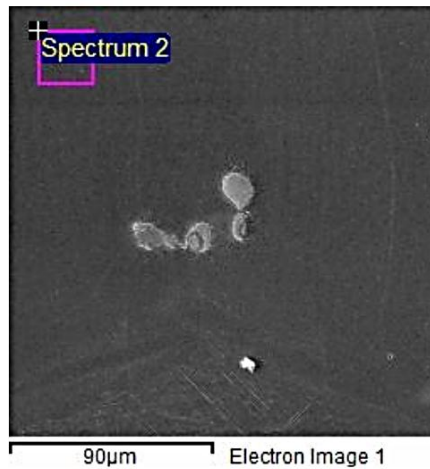
However, a closer look at the hardness in relation to the scanning speed reveals that deposit made at 0.50 and 1.50 m/min had hardness values of 322 and 324HV respectively, which may be considered for biomedical application because it is believed to be softer than other deposits and may allow easy integration with the body fluid.

D. Energy Dispersive Spectroscopy(DES)

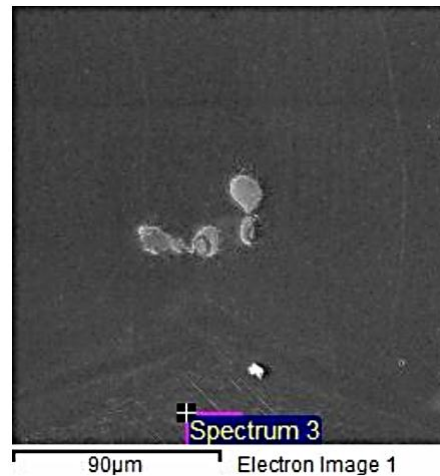
The EDS was conducted on all deposited campsites to determine and confirm the deposited zones. Spectrum 1 is a portion of the deposit containing whitish particles believed to be un-melted Molybdenum. The EDS plot revealed the elemental composition of the mapped spectrum1 with % weight of Titanium and Molybdenum highest which confirm that there was a mixture but with un-melted Molybdenum.

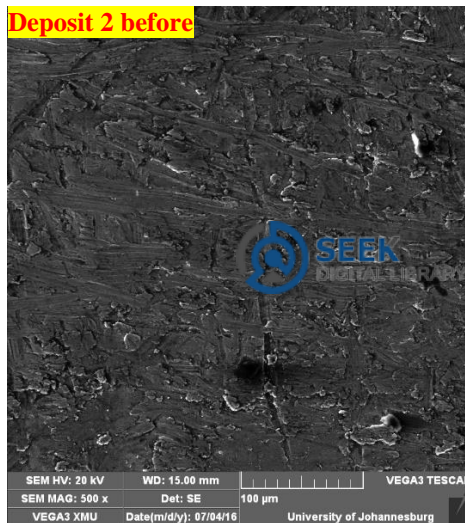
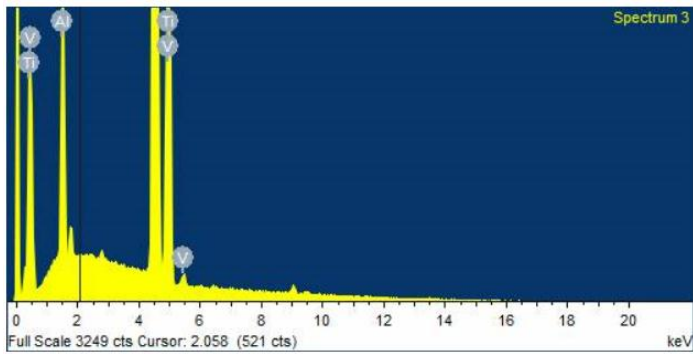


Spectrum 2 is another portion of the deposit mapped to analyze the composition. The plots show full peaks of Titanium (Ti), Molybdenum (Mo) and Vanadium (V) which are the three main elements. The full peaks of the element is an indication of full mixture within the deposit.



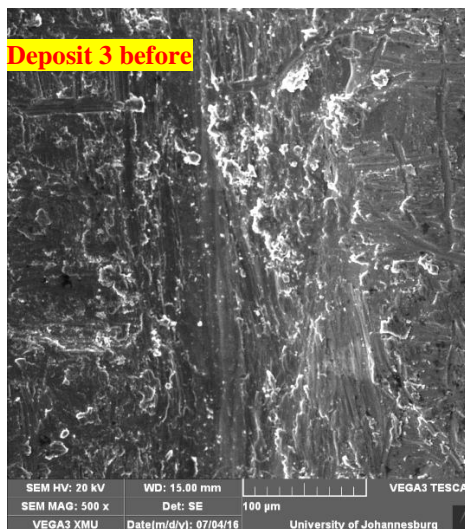
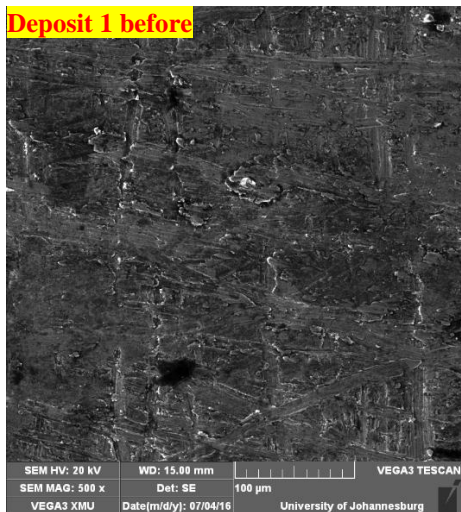
Spectrum 3 is a portion mapped in the substrate, the plots show high peaks Titanium (Ti), Molybdenum (Mo) and Vanadium (V) which are the elemental composition of the Titanium alloy.





E. Corrosion Using Hank's Solution

The result of the test of the five deposited Titanium/Molybdenum composite in Hank's solution revealed darker colouration on the deposited samples immersed in the Hank's solution when compared to the controlled deposited sample (Fig. 6).



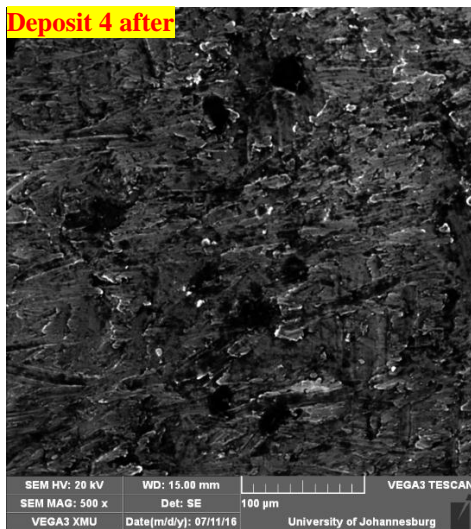
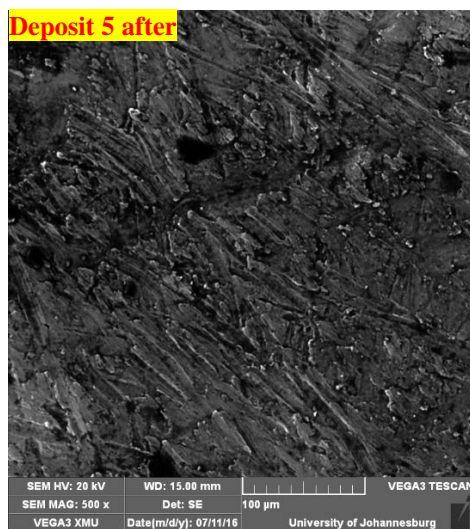
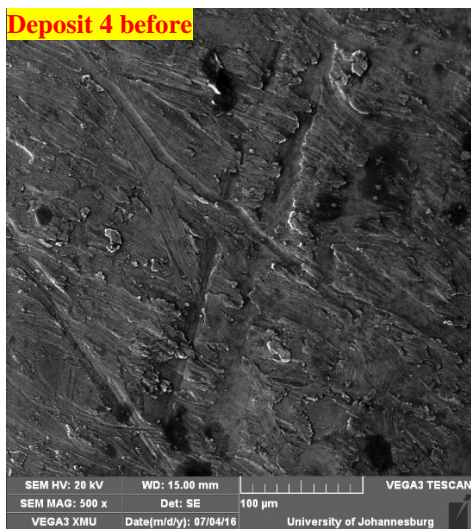
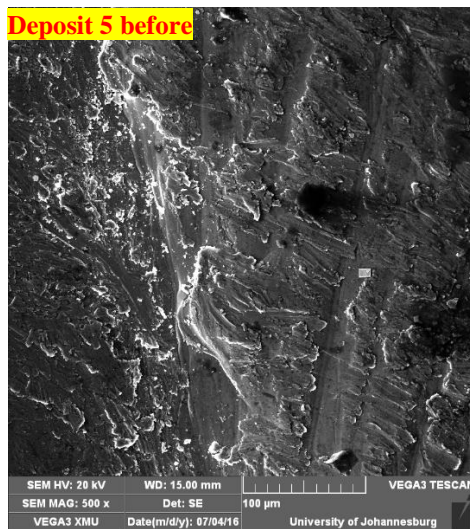
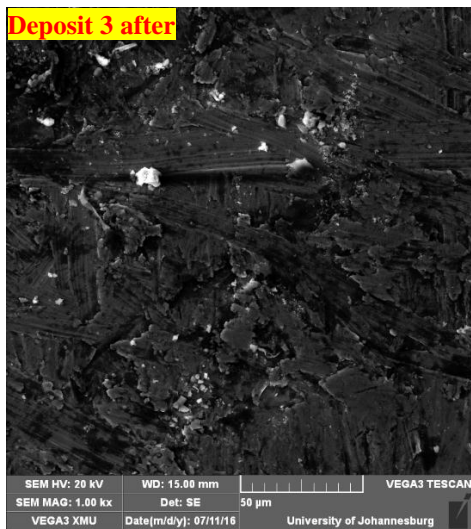


Figure 6. SEM micrographs of deposited Titanium/Molybdenum composites

Dark colorations suspected to be a form of corrosion was observed across all the five deposited composite samples, with a rough and spongy surfaces. The roughness on all the deposited composite allow the body cells to be easily attached and grow onto the implants.

iv. Conclusion

Lase Metal Deposited Titanium/ Molybdenum composite was produced at fixed laser power ad varying scanning speeds and characterised. The result revealed that the grain size is elongated, however, the scanning speed has an inverse relationship on the grain sizes. The Vickers hardness for deposited composite at 0.5 and 1.5 m/min were 322 and 324 HV respectively. These hardness values were considered suitable as a biomaterials compared to other deposited composites with higher Vickers hardness values, making is very undesirable. The corrosion test conducted with the immersion of the deposited composites in Hank's solution reveal dark colourations across all deposited composites, rough sponge surfaces, this confirm a potential corrosion when further immersed over a longer period.

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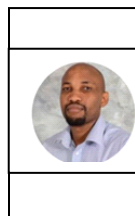
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