Functionally graded coating (silver/yttria) multi layers by pulsed laser deposition technique on 316L stainless steel substrate

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Abstract: Biomaterial surface modifications are crucial for matching the dynamics of the biological system and improving bioimplant efficiency. Surface modifications that are tailored to the material's biocompatibility, bondability, and host cell associations can significantly improve the material's biocompatibility, bondability, and host cell associations. In this investigation, silver, yttria and silver/yttria thin covers preparation and antimicrobial characteristics on St.St. 316L by pulsed laser deposition (PLD). The mechanical property of the coating has been evaluated by Vickers micro-hardness test, surface characterization studies of the coatings such as LOM and an antibacterial test has been conducted to ensure the coating's antibacterial efficacy.

1.Introduction:

Biomaterials have been utilized successfully in medical applications for decades to improve the quality and length of life of many people. Their diverse spectrum of functions includes anything from drug distribution to removing, augmenting, or restoring tissues, organs, or body functions. However, these capabilities have been only attained after several attempts, research projects, and historical developments [1].

Biomaterials are synthetic materials that are inserted into living organisms to replace body organs and perform traditional functions of the entire body for a prolonged period of time or even for the rest of one's life [2]. Metallic materials, 5ceramics, 5polymers, and 5composites are the 5materials 5utilized to make biomedical devices (orthopaedic, dental, bone cements, and so on) [3]. Despite the potential risks of toxic chromium (Cr) and 5nickel (Ni) ions being released into physiological media, 5austenitic stainless 5steel AISI 316 L is one of the most common and cost-impactive implant materials [4]. Austenitic stainless steel (AISI 316L, ASTM F-55, and F-138) is commonly utilized in medicine and contains 17–20 percent Cr, 12–15 percent nickel, 2–3 percent molybdenum, and small quantities of other components [5]. 316L stainless steel has mechanicals characteristics identical to human bone and is less expensive than titanium implants [6]. Sarcomas, fibrous encapsulations, osteolysis, genotoxicity, carcinogenicity, sand metal sensitivity have all been linked to leached metal ions [4]. A bioactive material coating will be applied to the implant metal surface to solve the issue of surface

energy variation [7]. On the surface of implants, bioactive ceramic coatings with the ability to facilitate favourable physiological interactions are added [8]. Ceramics depending on zirconia (ZrO2), such as Yttria-stabilized zirconia (YSZ), have long been regarded as critical components in medical devices [9]. Yttria-stabilized-zirconia (YSZ) in particular has a high refractive index and low losses, as well as excellent thermal stability and corrosion resistance [10].

2. Experimental Part:

A laser is a versatile instrument that can be utilized in a variety of situations. It's particularly useful in the material processing industry. This instrument is utilized in many scientific research projects and experiments due to its narrow frequency bandwidth, coherence, and high power density. Frequently, the light beam is powerful enough to vaporize even the most heat-resistant materials [18]. A target and substrate are placed inside a vacuum chamber and connected to a series of vacuum pumps in a simple PLD setup. A schematic diagram of a simple PLD system is demonstrate in Figure 1. Other inlet and auxiliary ports may be utilized to bring reactive gas and auxiliary attachments into the system. Outside the chamber, a factualizing lens is utilized to focus the laser source on the surface of the target [19].

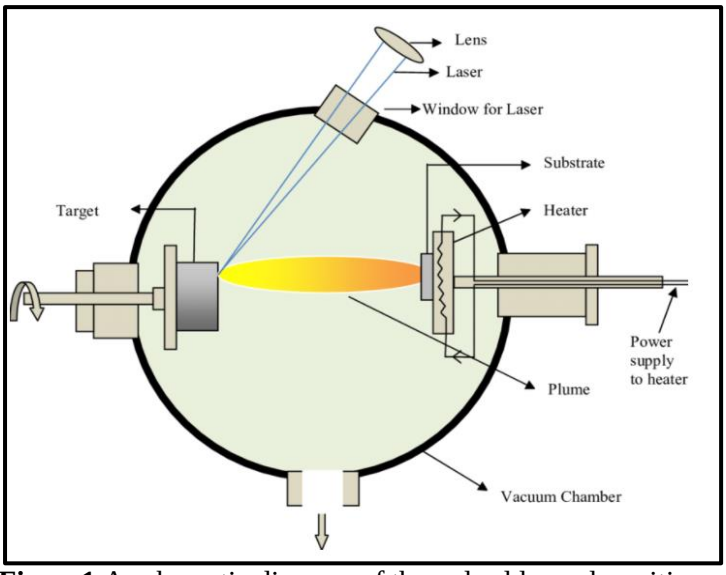

Figure1.A schematic diagram of the pulsed laser deposition setup [20].

2.1. Preparation of St.St.316L Substrates

In this step, a plate of 316L St.St. with thickness of 1mm has been cut in to (2.5cm,4cm). Before coating, the specimens have been ground with successive Al_2O_3 papers (120–1515 grit) and polished. Specimens have been then ultrasonically cleaned in ethanol alcohol for 10 min utilizing ultra sonic cleaning device.

2.2. Targets Impact of yttria and silver

In this step, a 5 g and 13 g from micro powder of both silver and yttria, as a binding material, it has been combined with polyvinyl alcohol (PVA). Then, the mixture powder has been mold in (30 mm) and (20mm) diameters for silver and yttria by compressing moulds, and pressed at pressure (150,75 MPa) respectively utilizing compacted device, to get targets handable. The moulds has been pre-lubricated to reduce friction and to release the compact models easily. Next, the targets has been dried utilizing the dry box at 100°C for 3 hrs for dehumidify and PVA liberating.

2.3. Deposition Procedure and Parameters

To meet the work's goals, the parameters for Ag/YSZ thin cover deposition are described in table (1) and (2).

Table 1. demonstrate Ag/YSZ deposition parameters

Parameters	Selected magnitude of Ag	Selected magnitude of YSZ
Power	100 Mj	180 Mj
Frequency	6 HZ	6 HZ
Pulses	500 P	700 P
Vacuum pressure	10^{-5} mbar	10^{-5} mbar
Temperature	300 °C	300 °C
Distance between the target and specimen	3.5 cm	3.5 cm

Table 2. specimen coding

Specimen code	Thin cove
A	Ag
B	YSZ
C	Ag/YSZ
D	YSZ/Ag
E	Ag/YSZ/Ag
F	YSZ/Ag/YSZ

3. Tests

The following experiments have been conducted in this investigation to assess the efficiency of thin cover (silver/yttria) on specimens.

3.1. Light Optical Microscope (LOM) test

Involved identification and measurement of the phase's shape and grain size are some characteristics of grain boundaries. Each of these has distinct characteristics. The microstructure evaluated with (400-x) magnification utilizing Olympus microscope manufactured by Japan.

3.2. Thickness Test

The thickness measuring device has been utilized to measure the thickness of silver/yttria thin covers on 316l st.st. substrates, in (Babylon/College of Material Engineering/University of Babylon).

3.3. Surface roughness test

The surface roughness of a silver/yttria coated 316l st.st. specimen has been measured by utilizing the (TR-100 surface roughness tester), which is located at the University of Babylon, Faculty of Materials Engineering. The device passes on the specimen surface to measure the surface roughness. The device has a sensor that records the roughness of the specimen surface and takes the reading directly from the device screen. The accuracy of the device (± mμ0.01).

3.4. Hardness test

Vickers Hardness (TH-717 Digital Micro Vickers Hardness Tester) has been measured the hardness of silver/yttria thin covers, at load (300g) and holding time 15 seconds in (college of Material engineering/University of Babylon).

3.5. Antibacterial test

When biomaterials, particularly metallic implants, are utilized inside the human body, they are exposed to germs that cause infections in nearby live cells. Therefore, it must be known if there are bacteria present or not on (silver/yttria) thin cover by the antibacterial test. This test has been performed by has beenhing the specimen in Petri dish with mannitol salt agar and it stayed in dish for 5 minutes. The solution has been then divided into 0.5 ml and put in another petri dish to be incubated at 37 °C for 24 hours.

4. Findings and Discussion

4.1 Light Optical Microscope (LOM)

The microstructure of silver and yttria coating on 316l st.st. plate with number of laser pulses (500 and 700) respectively utilizing the magnification of the microscope (400x) are demonstraten in figure (2).

From figure (2, A) can see the small grains of silver deposited on substrate, in figure (2, B) can see the small grains of yttria deposited on substrate, while in figures (2, (C, D, E, F)) illustrated mix of silver and yttria grains deposited on substrate.

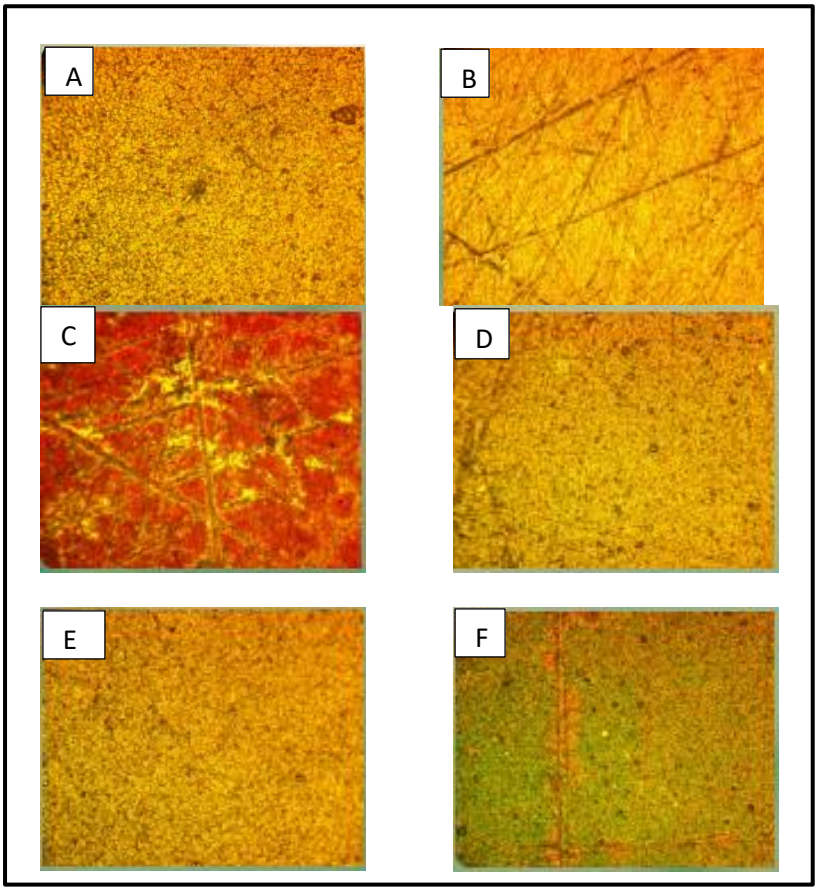

Figure 2. demonstrate the microstructure of specimens (A, B, C, D, E and F)

4.2. Thickness Findings

For A specimen the thickness is 2.76 μm, the thickness increased to 2.92 μm for B specimen because the nature of ceramic materials are easy to deposit compared with metals, and then the thickness reach to 5.45 μm for C specimen, D specimen the thickness is 5.79, while thickness of E and F specimens are 7.63 and 7.97 respectively. Anyhow with increasing number of coated layer, the deposition rate increased and the thickness increased due to increase the deposition layer also as demonstrate in Table (3).

Table 3. The Thickness of coated Layers of silver/yttria

Code of specimens	Thickness (μm)
A	2.76
B	2.92
C	5.45
D	5.79
E	7.63
F	7.97

4.3. Roughness Findings

The roughness of silver thin cover for Aspecimen is 0.432 μm, and it increased until reached 0.485 μm for B specimen for yttria, and then the roughness reach to it is high magnitude 0.571 μm for F specimen. The impact of number of layer coated and kind of coated materials on the surface roughness of coating is demonstrating in table (4).

Table 4. surface roughness findings of silver-yttria thin cover

Specimen code (μm)	roughness
A	0.432
B	0.485
C	0.506
D	0.517
E	0.554
F	0.571

4.4 Hardness finding

Hardness of uncoated specimens are improved after coating it with yttria. Furthermore increasing of number of layer from C to D to E and F specimens could improve the hardness from (264.80 HV) to (289.14 HV) to (237.89 HV) and(302.76 HV) respectively, this findings are agreement with P. Rajesh et.al, [21], Such improvement is due to the improvement in depth morphology, distribution and increasing in thickness which can be clearly observed in LOM findings, as in figure (2). Most likely the pulse increasing could implant more silver and yttria particles on the substrate surfaces.

Table (5) demonstrate the hardness findings and obviously, silver reduce the hardness compared with substrate while yttria increases it compared to the substrate specimen because ceramic materials is more hardness than metals.

Table 5. Hardness findings of 316L and silver-yttria thin cover

Specimen code Hardness	
St.St. 316L	211.20
A	197.68
B	250.41
C	264.80
D	289.14
E	237.89
F	302.76

4.5. Antibacterial findings

After planted of specimens on the media such as Nutrient Agar (figure 3) , Blood agar (figure 4), MacConkey agar, Mannitol salt agar, have been then described in culture by examining the colonies growing on the media mentioned above. After that , microscopically examining has done by staining them with Gram stain. This differential stain is utilized to distinguish between Gram-positive and Gram-negative bacteria. Finally, biochemical tests have been performed to confirm the diagnosis of the bacteria that appeared, such as the Catalase test, which is a test to diagnose the Gram-positive Staphylococcus aureus, while the IMVC test has been utilized to identify Gram-negative bacteria such as (Pseudomonas aueroginosa, Enterobacter sp) [21,22], as demonstrate in table (6).

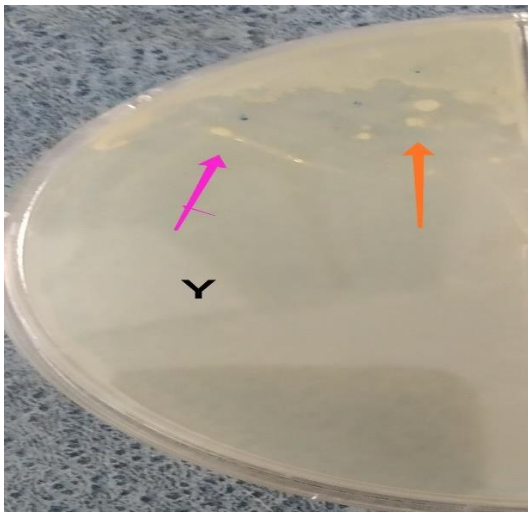

Figure 3. Appears kinds of bacteria on specimen B on general medium (Nutrient Agar).

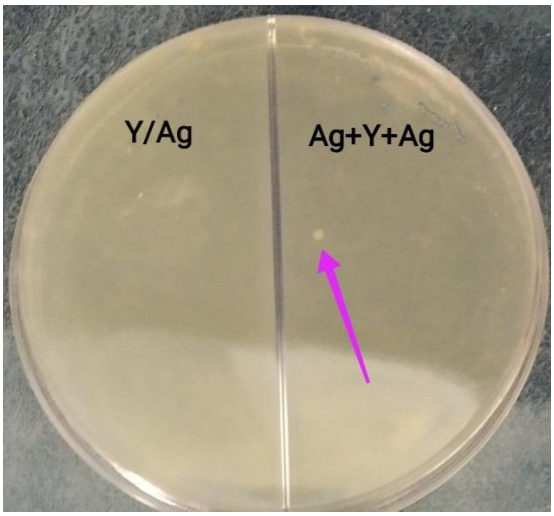

Figure 4. Appears kinds of bacteria on specimen D and E on general medium (Nutrient Agar)

Table 6. Demonstrates kinds of bacteria isolated from the specimens.

specimens	Kind of Bacteria		
	Staphylococcus aureus	Enterobacter sp	Pseudomonas aueroginosa
316l st.st.	+	+	+
A	-	-	-
B	-	+	-
C	-	-	-
D	+	-	-
E	-	-	-
F	-	-	-

Conclusion:

Depending on the gained findings, the following conclusions are made:

1. The thickness of thin cover increased with increasing the number of materials kinds from (2.76 μm) for A specimen to (7.97 μm) for F specimen.
2. The surface roughness of thin cover increased with increasing the number of materials kinds from (0.432 μm) for A specimen to (0.571 μm) for F specimen.
3. The thin covers hardness increased form (211.20 HV) for uncoated specimen to (302.76 HV) for F specimen.

4. No bacteria in (A, C, E and F) specimens.
5. Another interesting increasing in biocompatibility has been gaind throughout the work for the coated specimen compare to the uncoated st.st. specimen.

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