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DEPOSITION OF YTTRIA-STABILIZED ZIRCONIA CERAMIC ON SS316L BY PULSED SOLID-STATE Nd:YAG LASER

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ABSTRACT

DEPOSITION OF YTTRIA-STABILIZED ZIRCONIA CERAMIC ON SS316L BY PULSED SOLID-STATE ND:YAG LASER. Austenitic stainless steel in general has a limited service temperature up to 600°C. Nevertheless, service temperature more than that temperature are one of the key feature of advanced nuclear reactors to gain higher thermal efficiency which is related to economic beneficial, and also to withstand from abnormal condition. However, austenitic stainless steel such as SS316 class is well-known structure material for nuclear power reactors and other power plants. Therefore, one of the key issue is to modify SS316 so that has capability to service higher temperature. One of the technique for that purpose is ceramic-coated SS316L. In this preliminary study, thin films of zirconia-based ceramic i.e. YSZ (Yttria-Stabilized Zirconia) have been deposited on a SS316L using Plasma-Pulsed Laser Deposition (PLD) at Center For Science and Technology of Advanced Materials laboratory – National Nuclear Energy Agency of Indonesia (BATAN). Thin film was deposited with the constant oxygen flow injection of 20 and 40 sccm (Standard Cubic Centimeters per Minute) that produce a chamber pressure of 60 and 200 mTorr for the numbers of laser shots of 7.2×10^4 and 14.4×10^4, respectively. The substrate temperature during deposition was of 850^{o}C. Afterward, the samples were analyzed using Optical Microscope, Scanning Electron Microscope – Energy Dispersive X-ray Spectroscope (SEM-EDS) and Atomic Force Microscope (AFM). The results showed that the YSZ could homogeneously and sticky deposited on the surface of the SS316L surface. The surfaces were very smoothly formed with the surface roughness in the nano-meter scale range of 20-90 nm.

Keywords: Zirconia, YSZ, PLD, Plasma, SS316

ABSTRAK

PELAPISAN *YTTRIA-STABILIZED ZIRCONIA (YSZ)* PADA SS316L DENGAN LASER PULSA *SOLID-STATE ND: YAG*. Baja tahan karat Austenitik pada umumnya memiliki suhu kerja terbatas hingga 600 ° C. Namun demikian, suhu kerja lebih dari suhu itu adalah salah satu fitur utama reaktor nuklir canggih untuk mendapatkan efisiensi termal yang lebih tinggi yang terkait dengan manfaat ekonomi, dan juga untuk bertahan dari kondisi abnormal. Namun, stainless steel austenitik seperti kelas SS316 adalah material struktur yang terkenal untuk reaktor tenaga nuklir dan pembangkit listrik lainnya. Oleh karena itu, salah satu masalah utama adalah memodifikasi SS316 sehingga memiliki kemampuan untuk beroperasi pada suhu yang lebih tinggi. Salah satu teknik untuk tujuan itu adalah dengan melapisi SS316L dengan keramik. Dalam studi pendahuluan ini, film tipis dari keramik berbasis zirkonia yaitu YSZ (*Yttria-Stabilized Zirconia*) telah dilapiskan pada SS316L menggunakan Plasma-*Pulsed Laser Deposition (PLD)* di laboratorium Pusat Sains dan Teknologi Bahan Maju (PSTBM) – Badan Tenaga Nuklir Nasional Indonesia (BATAN). Film tipis YSZ dilapiskan dalam lingkungan gas oksigen dengan injeksi aliran oksigen konstan 20 dan 40 sccm (*Standard Cubic Centimeter per Minute)* yang menghasilkan tekanan ruang 60 dan 200 mTorr untuk jumlah tembakan laser masing-masing $7{,}2 \times 10^4$ dan $14{,}4 \times 10^4$. Suhu substrat selama pelapisan adalah 850^{o}C. Setelah itu, sampel dianalisis menggunakan Mikroskop Optik, Scanning Electron Microscope - Energy Dispersive X-ray Spectroscope (SEM-EDS) dan

Atomic Force Microscope (AFM). Hasil penelitian menunjukkan bahwa YSZ bisa terlapis secara homogen dan melekat dengan baik pada permukaan SS316L. Permukaan lapisan yang terbentuk sangat halus dengan kekasaran permukaan dalam kisaran skala nano-meter 20-90 nm.

Kata kunci: Zirconia, YSZ, PLD, Plasma, SS316

INTRODUCTION

High temperature materials are one of the key issues for the deployment of advanced nuclear reactors [1-4]. There are many types of high temperature materials. Materials of ceramic coating on the structural materials is one of the candidates of high temperature materials for the deployment of advanced nuclear reactors and applications in many fields [5-10]. There are many techniques for the ceramic coating such as plasma spray coating [5], electrophoretic deposition (EPD) [6] and pulsed laser deposition (PLD) [7-10]. One of the issues for the ceramic coating is formation of the roughness and micro porous. It has been reported that the PLD technique has advantage for minimize the micro porous [7-10]. In the utilization of the coating material especially for the deployment of advanced nuclear reactors, it is very important to select the substrate materials for ceramic coating as the base materials. One of the candidate materials for the advanced nuclear materials is SS316 [1-3]. Austenitic stainless steel SS316 class is well-known structure material for nuclear power reactors and other power plants. In this study, YSZ (Ytrria-Stabilized Zirconia, $Y_2O_3 – ZrO_2$) ceramic-coated austenitic steel SS316L with PLD technique is reported. YSZ material has advantages as coating materials to protect the substrate at high temperature [7-10]. The aim of the study is to develop high quality of YSZ ceramic coating layer on the surface of SS316L with smooth roughness of surface and minimizing porosity. It is expected that YSZ-coated SS316L using PLD technique potential to be used for advanced nuclear reactors structure materials.

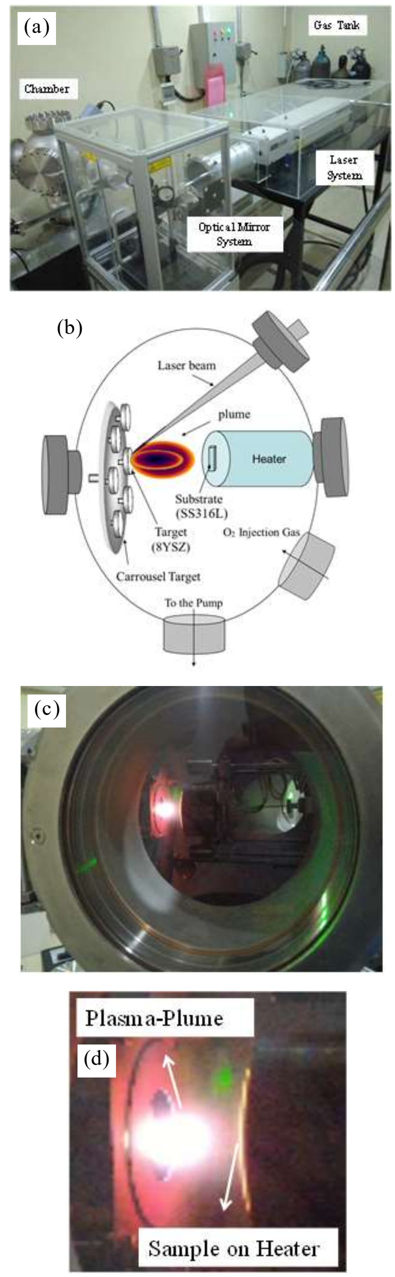

Figure 1. PLD - Pulsed Laser Deposition (a). Device system (b). Schematic (c). Chamber during deposition (d). Plume formation during depostion.

EXPERIMENTAL METHOD

Thin film of YSZ (Ytrria-Stabilized Zirconia) ceramic has been deposited on a SS316L austenitic stainless steel using Pulsed Laser Deposition (PLD) at laboratory facilities of Center for Science and Technology of Advanced Materials –National Nuclear Energy Agency of Indonesia (BATAN). Figure 1 (a) shows the schematic of the PLD and Figure 1 (b) shows the plume formation during deposition. Performance of the plume during deposition is able to be seen and analyzed during deposition through a glass window. The parameter condition of the experiment can be seen in the Table 1. The target was 8YSZ (8%mol Y_2O_3 – 92%mol ZrO_2) and the substrate was SS316L austenitic stainless. Solid state laser of Nd:YAG was used in this PLD with the wavelength of 266 nm and the energy of ~100 mJ. Thin film was deposited with the constant oxygen flow injection of 20 and 40 sccm (Standard Cubic Centimeters per Minute) that produce a chamber pressure of 60 and 200 mTorr for the numbers of laser shots $7,2\times10^4$ and $14,4\times10^4$, respectively. The substrate temperature during deposition was of 850^{o}C.

Table 1. Parameter of experiment

Parameter	Condition	Condition
Target material	8%mol Y_2O_3 – 92%mol ZrO_2	8%mol Y_2O_3 – 92%mol ZrO_2
Substrate material	SS316L	SS316L
Temperature of substrate	850 °C	850 °C
Number of shots	72,000	144,000
Chamber pressure	~200 mTorr	~60 mTorr
Gas backgroud	Oxygen	Oxygen
Laser wavelength	266 nm	266 nm
Laser energy	~100 mJ	~100 mJ
Laser frequency	10 Hz	10 Hz
Laser repetition pulsed	5 ns	5 ns

Afterward, the samples were analyzed using Optical Microscope (OM), Scanning Electron Microscope – Energy Dispersive X-ray Spectroscope (SEM-EDS) and Atomic Force Microscope (AFM).

RESULT AND DISCUSSION

In order to analyze the microstructure of the YSZ ceramic layer on the SS316L substrate optical microscope (OM) and SEM-EDS were employed. Figure 2 shows Optical Microscope image of thin film YSZ with 500 times of magnifications and Figure 3 SEM micrographs (a-c) of the same samples surface to investigate the YSZ film deposition on the SS316L steel.

The results of Optical Microscope and SEM characterization showed that YSZ could homogeneously and sticky deposited on the surface of the SS316L surface. It is found that the film consists of homogeneous particles of YSZ. Furthermore, there was no porous found on the surface of the YSZ film layer.

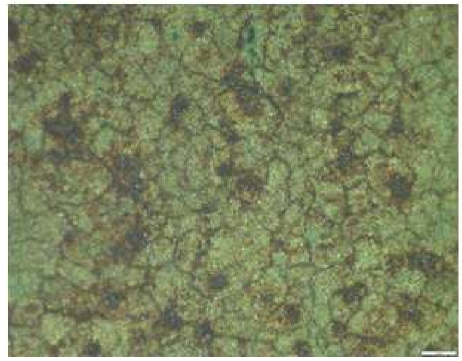

Figure 2. Optical Microscope SYZ thin film on the SS316L.

The EDS analysis of the sample surface was used in order to analyze deeper as shown in Figure 4. The purpose is to investigate chemical analysis of the YSZ film deposition on the SS316L steel i.e. zirconium and yttria elements. Therefore, the analysis of zirconium and yttrium percentages on the SS316L with various positions was analyzed as shown in Table 2.

The results of SEM-EDS characterization showed that the surface of SS316L was YSZ layer with the percentages of zirconium element was higher than yttrium element. Furthermore, the zirconium and yttrium percentage was found in various positions with similar composition ratio. It means that YSZ film-layer was homogeneously deposited on the surface area of SS316L substrate.

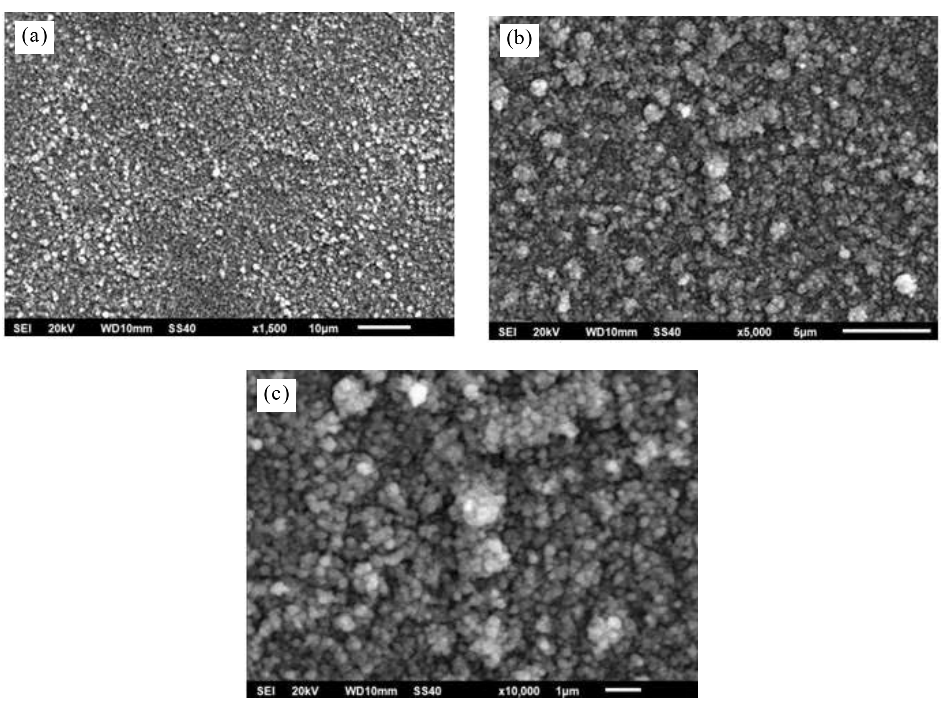

Figure 3. SEM micrographes (a-c) of samples surface with the magnification of (a) 1,500 ; (b) 5,000; (c) 10,000.

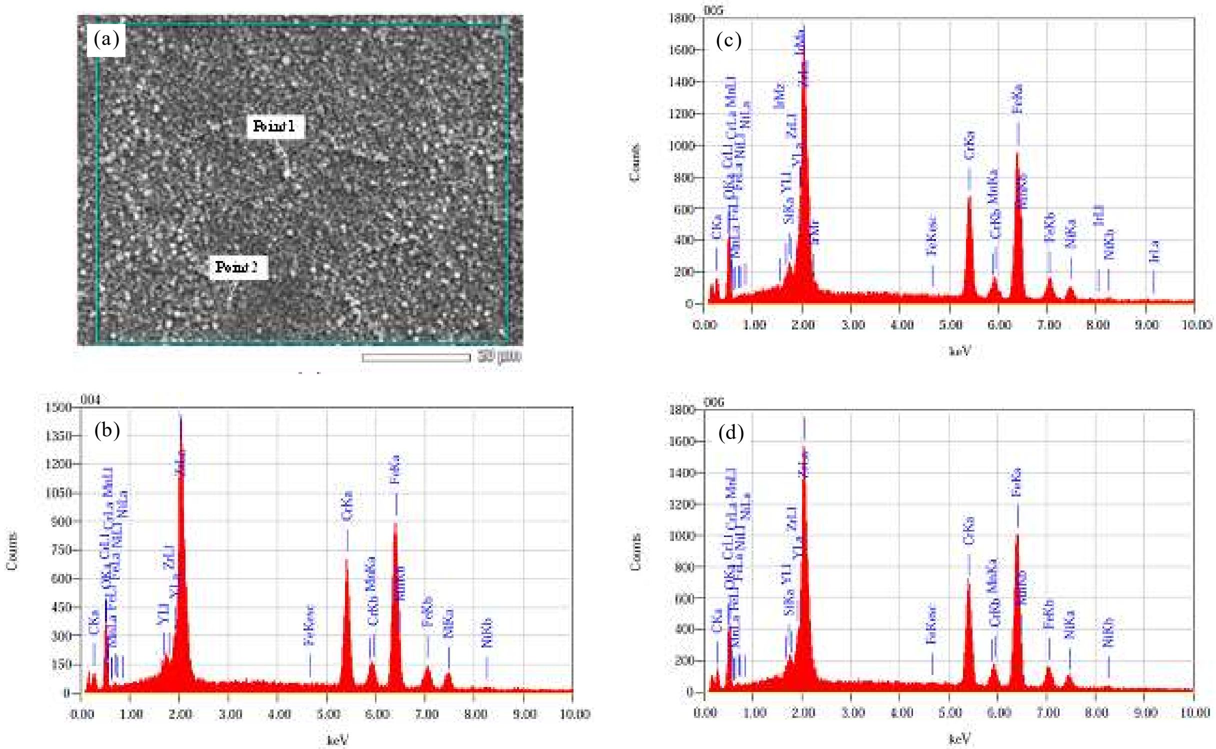

Figure 4. (a). SEM micrographes of samples surface with EDS area analyses to investigate the deposition of YSZ on the steel with various positions (b). point 1 (c). point 2 (d). area.

Table 2. EDS analyses of zirconium and yttrium element percentages on YSZ film-layer on the SS316L with various positions.

Elements/ Paramater	Point 1		Point 2		Area	
	%mass	%atom	%mass	%atom	%mass	%atom
Zr	27.73	12.13	26.11	10.90	26.40	10.97
Y	4.95	2.22	6.80	2.91	6.02	2.57

In order to investigate the roughness of the YSZ thin film layer on the surface of SS316L substrateAFM-Atomic Force Microscope was employed. Figures 5 and 6 show the AFM topography analyses of the samples's surface (2 and 3 dimensions) for $7.2x10^4$ and $14.4x10^4$ number of shots, respectively.

The results showed that the surface roughness of the YSZ ceramic coating layer on the SS316 substrate was in the nano-meter scale i.e. ~20 for 7.2×10^4 numbers of laser shots and ~90 nm for 14.4×10^4 numbers of laser shots. The roughness was increase with the increasing of numbers of laser shots which still in nano-meter scale range. It is found that the PLD technique for YSZ-ceramic coating could develop very smooth layer and minimize the formation of micro-porous. The results showed that the roughness of the layer using PLD technique with able to form nano-meter scale range roughness was much better than using other PVD (Physical Vapor Deposition) techniques which develop micro-meter scale range roughness [16]. It means that YSZ-coated SS316L using PLD technique is potential to be used as high-quality high temperature materials because of very smooth surface of coating.

CONCLUSION

Thin films of YSZ (Ytrria-Stabilized Zirconia) ceramic have been successfully deposited on a SS316L austenitic stainless steel using Pulsed Laser Deposition (PLD) technique with the substrate temperature of 850°C and constant oxygen flow injection of 20 and 40 sccm that produce a chamber pressure of 60 and 200 mTorr for the numbers of laser shots 7.2×10^4 and 14.4×10^4, respectively. The results showed that YSZ could homogeneously and sticky deposited on the surface of the SS316L surface with consist of homogeneous particles of YSZ. Furthermore, the YSZ were very smoothly formed with the surface roughness in nano-meter scale range of 20-90 nm. Therefore, the PLD technique is potential to be used for coating of YSZ on the SS316L for the development of high temperature materials.

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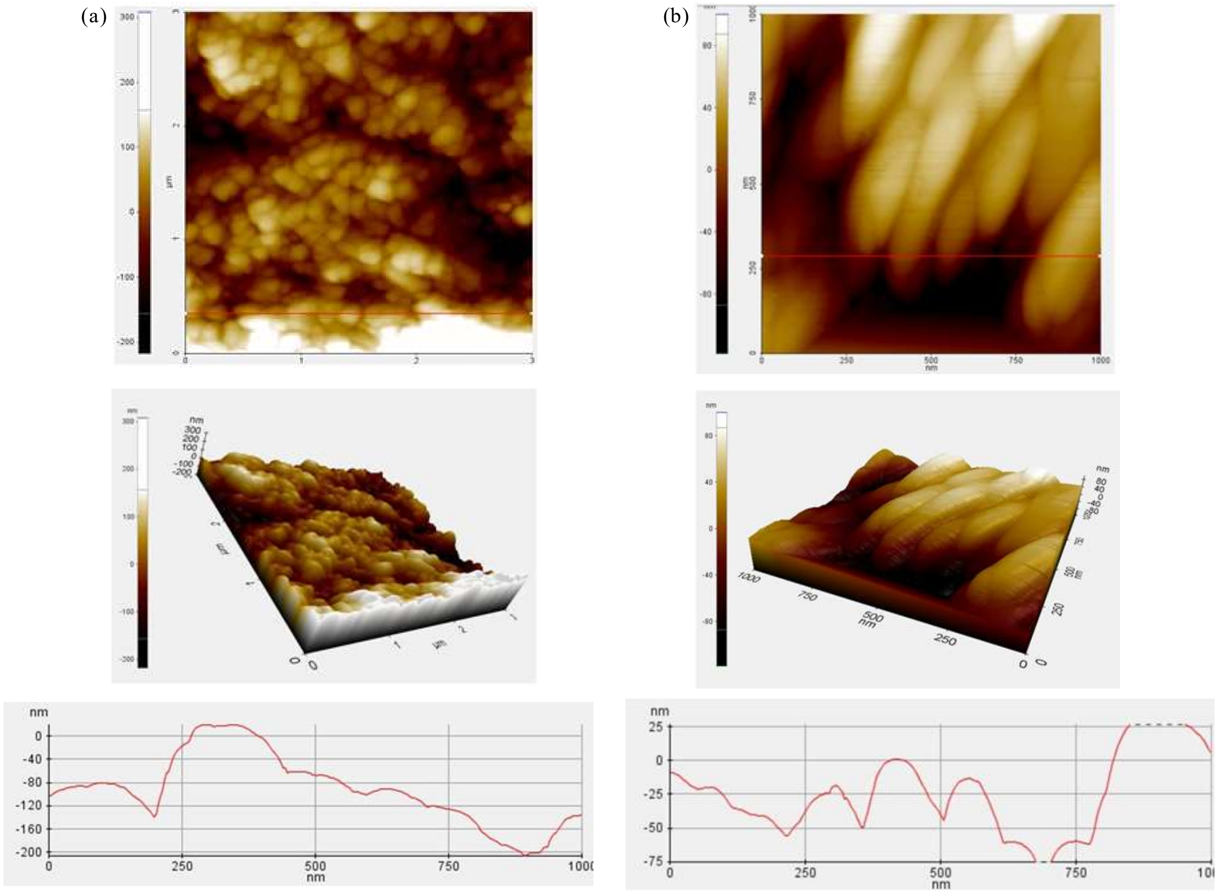
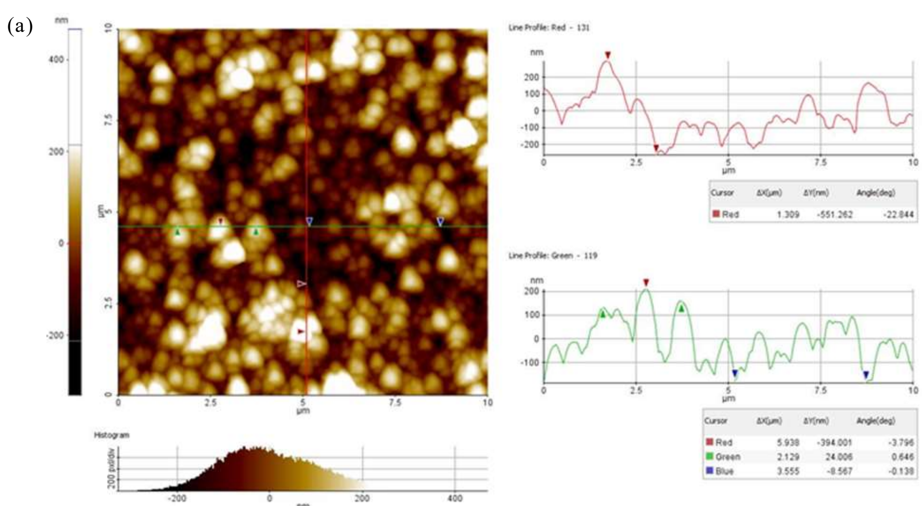


Line	Min(nm)	Max(nm)	Md(Nm)	Mean(nm)	Rpv(nm)	Rq(nm)	Ra(nm)	Rz(nm)	Rsk	Rku
Red	-206.257	19.316	-93.471	-91.958	225.572	60.754	46.491	140.402	-0.152	2.473

Line	Min(nm)	Max(nm)	Mid(Nm)	Mean(nm)	Rpv(nm)	Rq(nm)	Ra(nm)	Rz(nm)	Rsk	Rku
Red	-87.156	38.671	-24.243	-23.941	125.827	30.836	24.467	61.910	-0.387	2.497

Figure 5. AFM - Atomic Force Microscope topography analyses with line scan roughness analysis of the samples's surface with 72,000 of shots (a) 2 dimensions (b) 3 dimensions.



Line	Min(nm)	Max(nm)	Md(Nm)	Mean(nm)	Rpv(nm)	Rq(nm)	Ra(nm)	Rz(nm)	Rsk	Rku
Red	-262.170	293.427	15.629	-23.774	555.598	120.176	96.025	383.393	-0.425	2.930
Green	-187.145	208.164	10.509	-9.001	395.309	90.458	74.132	309.262	-0.282	2.425

Figure 6. (a). 2 dimensions, AFM - Atomic Force Microscope topography analyses of the samples's surface (2 dimensions with line scan roughness analysis and 3 dimensions) with 144,000 of shots.

(b)

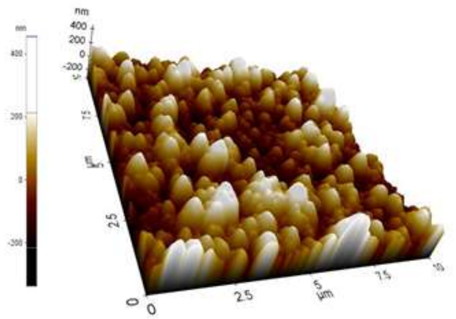


Figure 6. (b). 3 dimensions, AFM - Atomic Force Microscope topography analyses of the samples's surface (2 dimensions with line scan roughness analysis and 3 dimensions) with 144,000 of shots.

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