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Yttria-Stabilized Zirconia Ceramic Deposition on SS430 Ferritic Steel Grown by PLD - Pulsed Laser Deposition Method

Abu Khalid Rivai[1,*], Mardiyanto[1], Agusutrisno[2], Edi Suharyadi[2]

[1]Center For Science and Technology of Advanced Materials – National Nuclear Energy Agency of Indonesia BATAN, Kawasan PUSPIPTEK Gd. 71, Tangerang Selatan, Banten, 15314, Indonesia
[2]Dept. of Physics, Faculty of Mathematics and Natural Science, Gadjah Mada University, Bulaksumur, Daerah Istimewa Yogyakarta 55281, Indonesia

Email: rivai.abukhalid@batan.go.id

Abstract. Development of high temperature materials are one of the key issues for the deployment of advanced nuclear reactors due to higher temperature operation. One of the candidate materials for that purpose is ceramic-coated ferritic steel that one of the functions is to be a thermal barrier coating (TBC). Thin films of YSZ (Ytrria-Stabilized Zirconia) ceramic have been deposited on a SS430 ferritic steel using Pulsed Laser Deposition (PLD) at Center For Science and Technology of Advanced Materials laboratory – National Nuclear Energy Agency of Indonesia (BATAN). The thin film was deposited with the chamber pressure range of 200-225 mTorr, the substrate temperature of 800°C, and the number of laser shots of 3×10^4, 6×10^4 and 9×10^4. Afterward, the samples were analyzed using Scanning Electron Microscope – Energy Dispersive X-ray Spectroscope (SEM-EDS), X-Ray Diffractometer (XRD), Atomic Force Microscope (AFM) and Vickers hardness tester. The results showed that the YSZ could homogeneously and sticky deposited on the surface of the ferritic steel. The surfaces were very smoothly formed with the surface roughness was in the range of 70 nm. Furthermore, thickness, composition of Zr^{4+} dan Y^{3+}, the crystallinity, and hardness property was increased with the increasing the number of the shots.

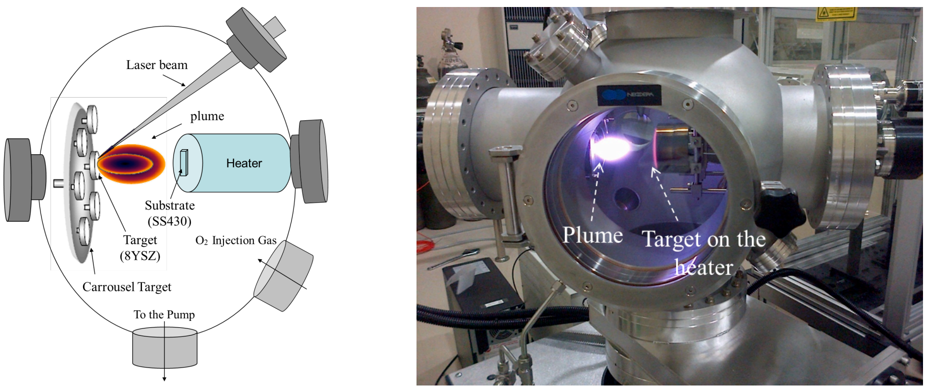
1. Introduction

Development of high temperature materials are one of the key issues for the deployment of systems in many fields such as advanced nuclear reactors due to the need of higher temperature operation. One of the candidate materials for that purpose is ceramic coating on the structural materials [1-5]. The coating is able to protect the substrate materials from corrosion [6]. The techniques of the coating are mostly done by electron beam physical vapor deposition (EB-PVD) [2] and plasma spray coating [4,5,7]. Nevertheless, in order to minimize micro porous the new technique for coating i.e. pulsed laser deposition (PLD) has been reported [1]. It has been reported that the PLD technique is used in many field [8-15] and able to deposit not only ceramic but also polymer material [16].

In this study ceramic-coated ferritic steel that one of the functions is to be a thermal barrier coating (TBC) is reported. YSZ (Ytrria-Stabilized Zirconia, Y_2O_3 – ZrO_2) ceramic was used as the target for coating using PLD technique. YSZ material has advantages as coating materials to protect the substrate at high temperature [17]. The purpose of the study is to develop high quality of coating layer with smooth roughness of surface and minimizing porosity.

2. Experiment

Thin films of YSZ (Ytrria-Stabilized Zirconia) ceramic have been deposited on a SS430 ferritic 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. It can be seen this PLD is able to use multiple target in the carrousel. Furthermore, 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 SS430 ferritic steel with the content of chromium is up to 15%. 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 40 sccm (Standard Cubic Centimeters per Minute) that produce a chamber pressure with the range of 200-225 mTorr. The substrate temperature during deposition was of 800°C, and the numbers of laser shots were 3×10^4, 6×10^4 and 9×10^4.



(a) (b)

Figure 1. PLD – Pulsed Laser Deposition (a) schematic (b) plume formation during depostion.

Table 1. Parameter of experiment

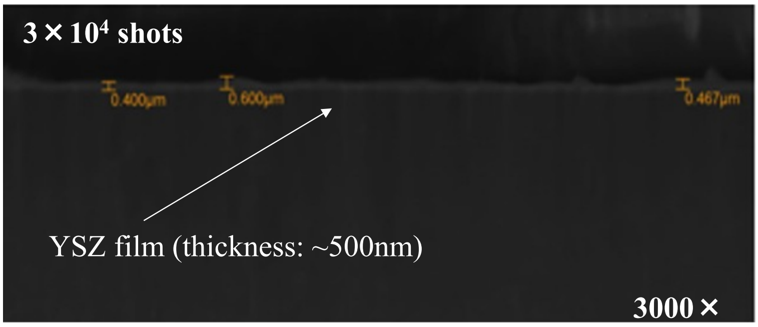
Parameter	Condition
Target material	8YSZ (8%mol Y_2O_3 – 92%mol ZrO_2)
Substrate material	Steel (SS430)
Temperature of substrate	800 °C
Number of shots	$3x10^4$, $6x10^4$ and $9x10^4$
Chamber pressure	200-225 mTorr
Gas background	Oxygen
Laser wavelength	266 nm
Laser energy	~100 mJ
Laser frequency	10 Hz
Laser repetition pulsed	5 ns

After deposition of the steel with YSZ target, the samples were analyzed using Scanning Electron Microscope – Energy Dispersive X-ray Spectroscope (SEM-EDS), X-Ray Diffractometer (XRD), Atomic Force Microscope (AFM) and Vickers hardness tester.

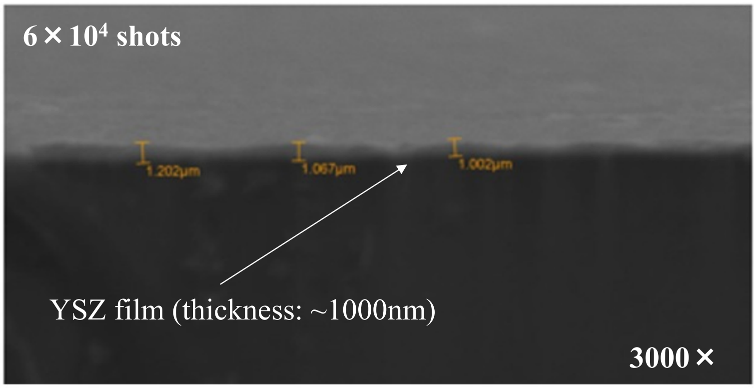
3. Results and Discussion

Figure 2 show SEM micrographs of samples cross section to investigate the film deposition on the steel with various shots i.e. $3x10^4$ shots, $6x10^4$ shots and $9x10^4$ shots. It was found that the thickness of the coating layer were ~0.5, 1, and 1.1 µm for $3x10^4$ shots, $6x10^4$ shots, and $9x10^4$ shots, respectively. The results showed that the thickness of the coating layer was increase with the increasing of the shots.

Figure 3 show SEM micrographs of samples surface with EDS area analyses to investigate the deposition of YSZ on the steel with various shots i.e. $3x10^4$ shots and $9x10^4$ shots. The EDS analyses of zirconium and yttrium percentages on the steel with various shots are shown in Table 2. The results showed that the percentages of zirconium and yttrium were increased with the increasing of the shots. The percentages analyses mean that the thickness of the layer was increased with the increasing of the shots.



(a)



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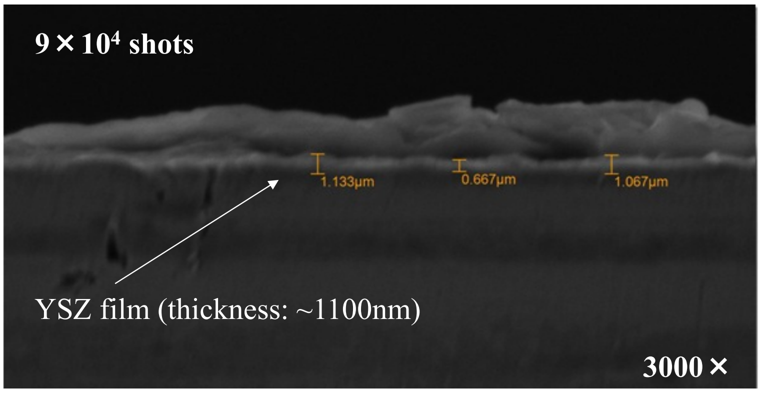


(c)

Figure 2. SEM micrographs of samples cross section to investigate the film deposition on the steel with various shots (a) $3x10^4$ shots (b) $6x10^4$ shots and (c) $9x10^4$ shots

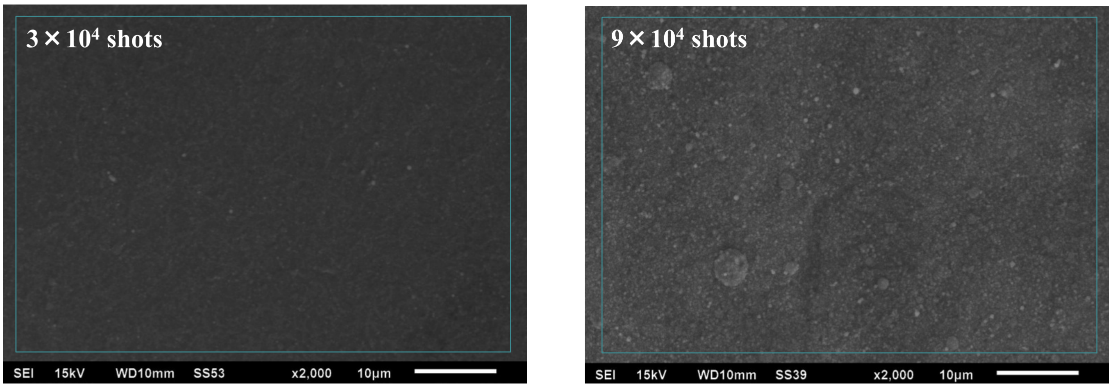
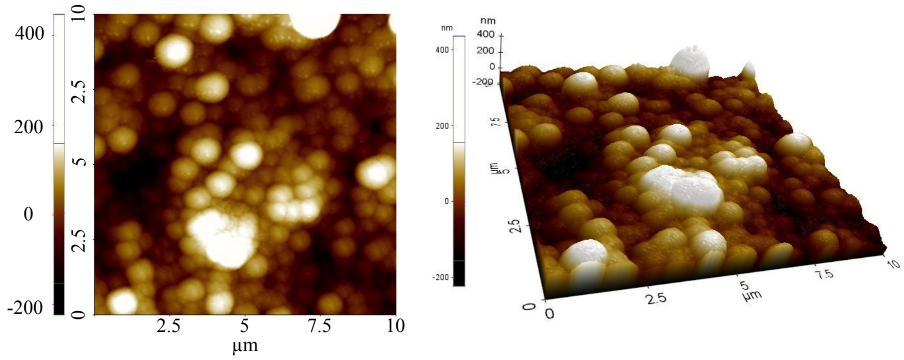


Figure 3. SEM micrographs of samples surface with EDS area analyses to investigate the deposition of YSZ on the steel with various shots (a) $3x10^4$ shots and (b) $9x10^4$ shots

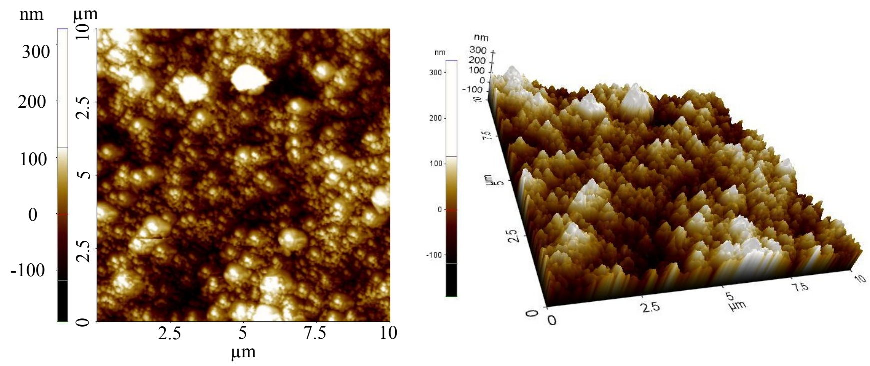
Table 2. EDS analyses of zirconium and yttrium percentages on the steel with various shots

Elements /	$3x10^4$ shots		$9x10^4$ shots	
Parameter	% mass	% atom	% mass	% atom
Zr	8.33	3.72	34.25	13.85
Y	2.88	1.33	6.74	2.87

Figure 4 show the AFM - Atomic Force Microscope topography analyses of the samples's surface (2 and 3 dimensions) for $3x10^4$ number of shots, $6x10^4$ number of shots and $9x10^4$ number of shots. The results showed that the roughness of the coating layer was in the nano-meter scale i.e. ~70 nm for all the cases. It is found that the PLD technique for ceramic coating could develop very smooth layer and minimize the formation of micro porous. As comparison analysis, it has been reported that using plasma sprayed technique the surface roughness was 10 μm and using EB-PVD technique the surface roughness was 1 μm [18]. Therefore, PLD technique has advantage to develop very smooth surface of coating.



(a)



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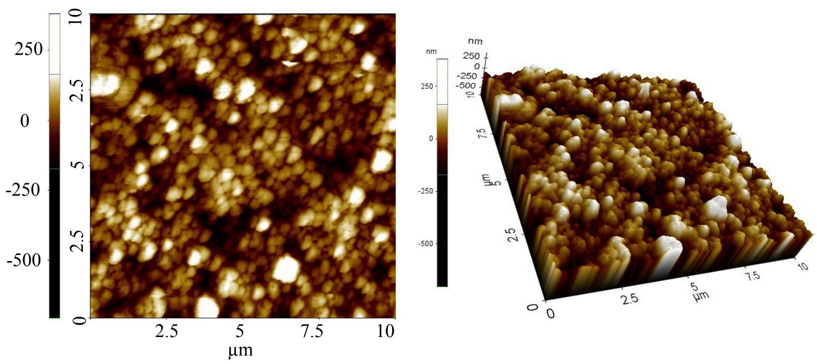
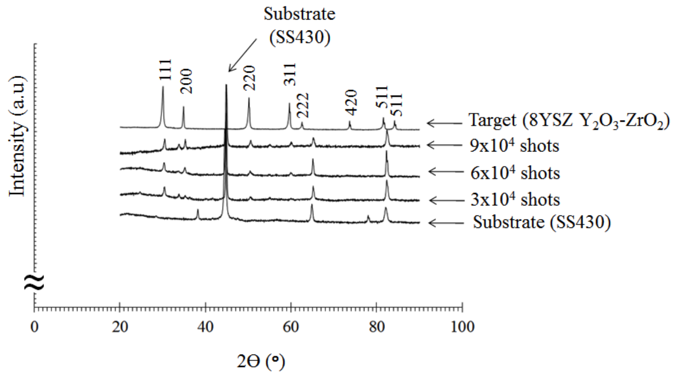


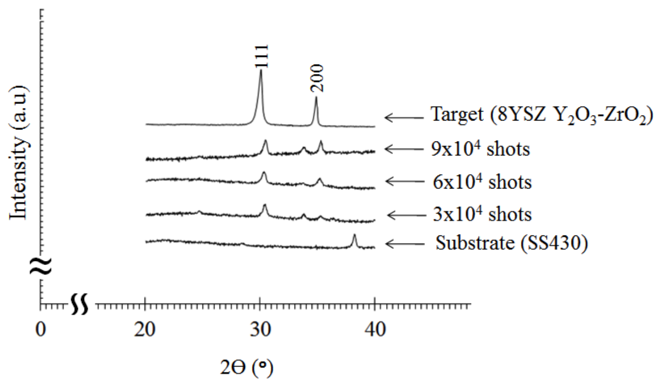
(c)

Figure 4. AFM - Atomic Force Microscope topography analyses of the sample's surface (2 and 3 dimensions) (a) $3x10^4$ number of shots (b) $6x10^4$ number of shots (c) $9x10^4$ number of shots

Figure 5 show XRD analyses of the samples for 2□ between 20-90˚ and 2□ between 20-40˚. The results showed that the ceramic YSZ was deposited on the layer of the substrate with the indication of YSZ peaks. Furthermore, the substrate peaks were decrease with the increasing of the PLD shots and the crystanillity of the YSZ layer was increase with the increasing of the shots. It happen because with the increasing of the shots the thickness of the layer was increase.



(a)



(b)

Figure 5. XRD analyses of the samples: (a) 2□ between 20-90˚, and (b) 2□ between 20-40˚

Table 3. Surface hardness of the samples

Number of shots	Hardness (VHN)
$3x10^4$	167
$6x10^4$	182
$9x10^4$	184

Table 3 show the surface hardness of the samples with the various of the number of shots. The results showed that the hardness property was increased with the increasing the number of the shots. The surface hardness was contributed from the hardness of the thin film and the hardness of the substrate. The increasing of the number of shots increased the thickness of the coating layer therefore it minimized the contribution of the hardness of the substrate. It means that the hardness of YSZ is higher than the steel and finally the hardness is only the property of the YSZ coating layer.

4. Conslusions

Thin films of YSZ (Ytrria-Stabilized Zirconia) ceramic have been deposited on a SS430 ferritic steel using Pulsed Laser Deposition (PLD) technique with the chamber pressure range of 200-225 mTorr, the substrate temperature of 800ºC, and the number of laser shots of 3×10^4, 6×10^4 and 9×10^4. The surfaces were very smoothly formed with the surface roughness was in the range of 70 nm. Furthermore, thickness, composition of Zr^{4+} dan Y^{3+}, the crystallinity, and hardness property was increased with the increasing the number of the shots. The PLD technique is potential to be used for coating of YSZ on the ferritic steel for the development of high temperature materials.

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