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Characterization Study of Thin Film Coatings on Porous Ceramic Material Prepared by Spin Coating Method

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ABSTRACT

Thin films have numerous applications in modern technology, particularly in industry. One common method for producing thin films is spin coating, where the film thickness is influenced by parameters such as spin speed, solution concentration, and coating duration. The present work evaluates the relation between film thickness and coating process, including the porosity, and permeability of porous ceramic materials. The material used is corundum, made from 97% aluminum oxide (Al_2O_3) and 3% Polyethylene Glycol (PEG). The produced porous disk has dimension of 25mm (diamater) and 2mm (thickness). The coating process was carried out using a spin coater with varying spin speeds (1500, 1800, 2100, and 2400rpm) and solution concentrations (10%, 15%, and 20%) for 120 seconds. Testing revealed that higher spin speeds result in thinner films, while solution concentration affects the film thickness of the processed disk. Additionally, the material's porosity and permeability decreased after the coating process. This research highlights the importance of controlling coating parameters to achieve the desired material characteristics.

1. INTRODUCTION

Material processing pushes the boundary of the technical approach, making several innovative solutions are developed in modern industrial process. It comes as reliable solution due to high pressure and concern for the economy, manufacturing and energy sector, making reliable process becomes mandatory [1-3]. Specifically, the attention on manufacturing sector that processed high quality and specific component, typically requires high precision, demands for different technology and specific approach to achieve the intended target. Moreover, the requirement for specific technology, particularly for coating, requires specific approach to achieve a reliable result.

Thin films are types of coatings that have a thickness ranging from the micrometer to the nanometer scale [4]. Thin films have various applications in modern technology [5]. They are used in electronic devices [6], semiconductors, transistors, solar cells, photoconductors, and many others. Thin films are used in most industrial applications. By controlling the thickness and properties of thin films, it is possible to create materials with specific properties useful for various technological applications. The reason of using Titanium dioxide (TiO_2) in thin film material is a low-cost, non-toxic, and excellent optical properties [7-9]. Titanium dioxide (TiO_2) usually was used is in semiconductor and widely used in various industries [10]. TiO_2 thin films refer to thin layers or coatings made of titanium dioxide. TiO_2 is favored in thin film production because of its high stability and resistance to corrosion [11] and appropriate compatible using spin coating.

Spin coating is a method used to deposit thin films within the micrometer to nanometer range by spreading a solution onto the surface of a flat substrate and spinning it at a certain speed, leading to the deposition of the thin film on the surface. This method is commonly used because it can produce thin films with uniform thicknesses ranging from micrometers to nanometers. The spin coating method is employed by dropping material in the form of a solution onto the center-top of a substrate that is placed on a rotating disk, either manually or with the help of a robot. Several parameters affect the spin coating process, including solution viscosity, solution concentration, final layer thickness, coating time, and final rotation speed (ω). When using the spin coating method, centrifugal forces cause the solution to spread toward the edge of the substrate and eventually evaporate, leaving a thin resin film on the surface. The thin film can be improve the porous material.

Porous materials are defined as materials containing holes, cavities, or spaces within their structure that may be interconnected or isolated [12]. A material or solid can be classified as porous if its porosity is between 0.2 and 0.95. Based on pore volume, porous materials can be classified into three types: low porosity, medium porosity, and high porosity. Corundum is a crystalline form of aluminum oxide (Al_2O_3) that typically contains small amounts of iron, titanium,

vanadium, and chromium [13]. Although it is a naturally transparent material, its color may vary due to the presence of transition metal impurities in its crystal structure. One type of ceramic material is corundum, also known as α-alumina (Al_2O_3), which consists of crystalline aluminum oxide minerals that are extremely hard and corrosion-resistant.

Porous materials, such as porous ceramics, activated carbon, and various types of porous polymers, have critical applications in several industries [14]. In the medical field, porous materials are used for medical implants and controlled drug release [15]. In the energy industry, these materials are employed in energy storage and conversion, such as batteries and fuel cells. In environmental sectors, porous materials are useful in filtration and water purification. Furthermore, they are used in catalysis, sensors, and gas filtration, playing crucial roles in technological innovation and industrial sustainability.

Porous materials can be created from various substances, including metals, polymers, and ceramics [16]. Ceramic materials, in particular, can be processed through several stages, such as mixing, compaction, and sintering [17]. The mixing process serves three main purposes: first, to combine different raw materials and ensure uniformity. second, to determine the optimal water content; and third, to form grains or nodules that promote permeability in the sinter bed [18]. The compaction process aims to reduce the material's volume, lower its porosity, and increase its relative density to achieve the desired strength and density. Sintering, a thermally activated process, involves converting solid powder into a solid structure through atomic-scale mass transport events. This process is crucial in the development of porous materials, particularly ceramics, as it strengthens and refines the material's structure, resulting in a porous material with the intended properties [19-21].

Porosity is the ratio of the volume of pores or empty spaces in rock to the total volume of the rock. Porosity indicates the potential volume of water, air, or gas that can be stored within the rock and affects the rock's physical properties such as elastic wave velocity, electrical resistivity, and density [22]. Meanwhile, permeability is defined as the measure of a porous medium's ability to transmit fluids. It is a measure of how well a rock can allow fluid to flow through it. Permeability is one of the important parameters in determining porous materials because it affects the flow of fluids passing through the material [23]. Permeability can be defined as the ability of a material to flow fluids or gases through its pores at a certain speed [24]. Both the fluid's viscosity and the porosity of the substance have an impact on the permeability of the material.

Viscosity is a measure of a fluid's thickness and determines how easily the fluid can flow [25]. In the spin coating process, the viscosity of the fluid affects the flow rate of the fluid as the substrate rotates, as well as the thickness of the layer formed on the substrate. The parameter commonly used in spin coating is dynamic viscosity. Dynamic viscosity measures the internal resistance of a fluid to flow [26] and is crucial in determining how the material solution will spread and evenly coat the substrate during the spin coating process.

Based on the explanations above, this study aims to evaluate the effect of rotational speed and solute concentration on the thickness of the resulting thin film, analyze the effect of the coating process on the porosity of the material after the coating process, and assess the permeability of the material after the thin film deposition process.

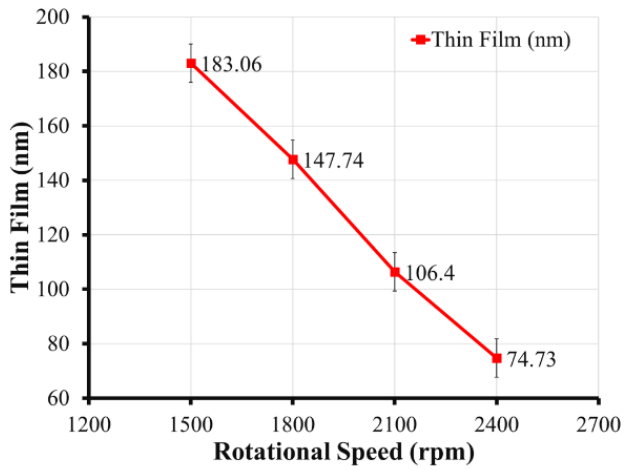
2. MATERIALS AND METHOD

2.1 Materials and tools

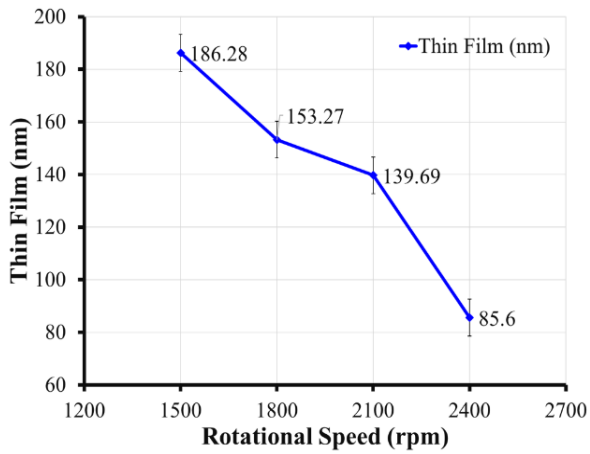
The materials we used in this study are alumina powder, Titanium dioxide (TiO_2) powder, Polyethylene Glycol (PEG), and ethanol 96%. The instruments we used in this study are a furnace chamber, hydraulic press, oven, macro microscope, permeability unit tester, spin coater, hotplate magnetic stirrer, analytical balance, viscometer, and other laboratory equipment for handling chemicals. The porous material used was corundum, which was self-made in the Material Laboratory. The coating material used was a mixture of titanium dioxide solution dissolved in 96% ethanol, purchased from a chemical store.

2.2 Porous ceramic material preparation

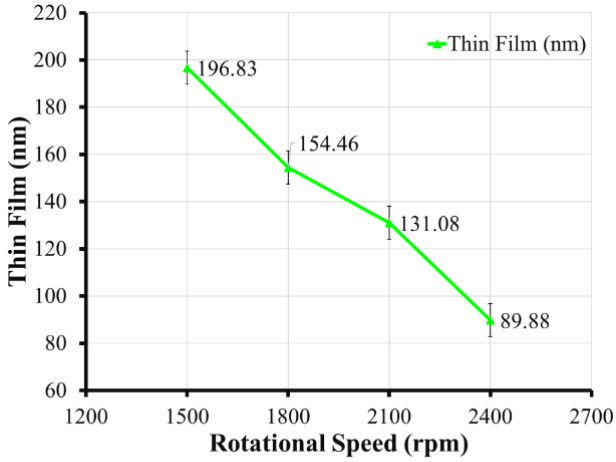
The preparation of porous material specimens was carried out by mixing alumina powder with a binder, then grinding it to homogenize the mixture before compacting it at a pressure of 686MPa. The formed specimens then underwent heat treatment by heating in an oven at 150°C for two hours, followed by sintering at 1200°C, with a heating rate and cooling rate of 10°C/minute and a holding time of 2 hours. The specimen making process can be seen in Figure 1.



(a) Concentration 10%



(b) Concentration 15%



(c) Concentration 20%

Figure 1. Rotational speed to thin film thickness at various concentrations

2.3 Application of a coating solution on porous ceramic materials

2.3.1 Preparation coating solutions

Titanium dioxide was dissolved in ethanol. The solution was stirred at 600rpm for 2 hours. Afterward, the viscosity of the solution was tested using an Ostwald viscometer. Viscosity is a measure of the thickness of a fluid, which indicates the friction within the fluid. The higher the viscosity, the greater the resistance, and the harder it is for objects to move through the fluid.

2.3.2 Coating process on porous ceramic materials

The deposition of the solution was carried out using the spin coating method, with a spin coater as the tool. After coating, the specimens were sintered at 1100℃ for five hours, with a heating rate of 3℃/minute and no cooling rate.

2.4 Identification of coated porous ceramic materials

The thickness of the thin film formed was measured using an analytical method by comparing the thickness of the specimen before and after coating using a macro microscope. The thickness of the thin film was the difference between the thickness after coating and the initial thickness of the coated substrate.

Porosity was measured using the mass method due to its speed and efficiency. Permeability testing was conducted using a permeability unit tester. Macroscopic testing was used to observe the features of the porous material before and after coating using the spin coating method.

3. RESULTS AND DISCUSSION

3.1 Thin film thickness activity on porous ceramic materials

The viscosity and thickness of the solution used in the coating step are examined. Three concentration variations are employed to create the coating solution: 10%, 15%, and 20%. The results of the dynamic viscosity testing of titanium dioxide solutions with concentrations of 10%, 15%, and 20% showed viscosities of 825mPa.s, 1485mPa.s, and 2048mPa.s, respectively.

This phenomenon showed that the higher the concentration used, the higher the viscosity of the solution. When the concentration is higher (20%), the solution will become more viscous. Increasing the viscosity of the solution increases its adhesive force on the specimen. The solution with a higher viscosity has a bigger drag force, therefore even though the centrifugal force increases, numerous particles are still present in the specimen. Consequently, a thicker layer is produced.

Following the viscosity test, the porous ceramic material is coated with the solution to create a thin film surrounding it. Figure 1 shows the results of thin film thickness testing during the coating process with a coating time of 120 seconds and various rotational speed. Figure 1 indicates that at a rotating speed of 1500rpm, film thickness for 10% (Figure 1(a)), 15% (Figure 1(b)), and 20% (Figure 1(c)) concentration is 183.06nm, 186,28nm, and 196.83nm respectively. In contrast, the film thickness for 10%, 15%, and 20% concentrations displays values of 74.73nm, 85.6nm, and 89.88nm at a speed of 2400rpm.

The thickness of the thin layer of the substrate is influenced by the spin coating machine's rotational speed. The tangential speed rises in response to the increased rotational speed (rpm), increasing the centrifugal force acting on the disk. This condition will accelerate the spread of the solution to all of the plates, and some will be carried out. As a result, the solution and particles remaining become thinner. According to research conducted by Ansari and Sartale [27], the results show that the thin film thickness is inversely proportional to the rotational speed during the coating process. In other words, the faster the rotational speed applied, the thinner the resulting film.

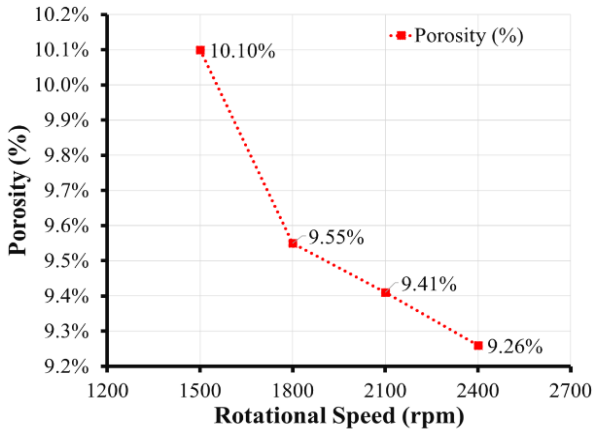
The thin-film thickness against spin speed comparison graph for concentrations of 10%, 15%, and 20% demonstrates that the thinner the final thin-film, the lower the concentration. The solution's concentration and viscosity are intimately correlated. One of the key parameters discussed is the effect of solution viscosity on the thickness of the thin film.

3.2 Porosity activity on coated porous ceramic material

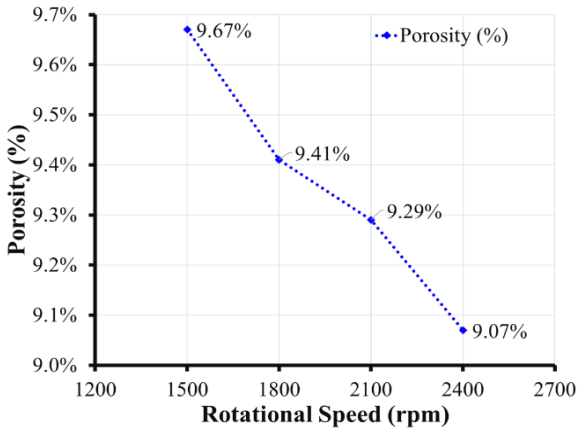
The porosity testing results of the corundum porous material made before coating showed an average porosity value of 10.58%. The porosity value of the porous material after coating can be seen in Figure 2.

Figure 2 illustrates how variations in concentration and rotation speed effect the porosity value. At 1500rpm, the porosity values at 10% (Figure 2(a)), 15% (Figure 2(b)), and 20% (Figure 2(c)) concentrations are 10.1%, 9.7%, and 9.46%, respectively. At 2400rpm, the porosity values at 10%, 15%, and 20% concentrations are 9.25%, 9.18%, and 8.85%, respectively.

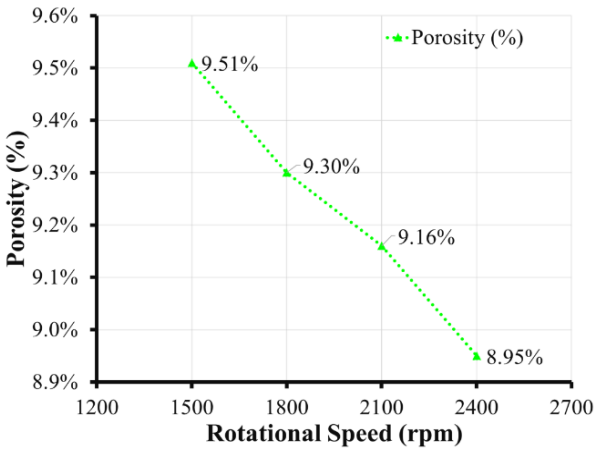
The thickness of the thin film that forms has an impact on the material's change in porosity after coating. The more pores that are sealed the thicker the coating. It is clear from the graph that the coating process parameters have a big impact on how the material's porosity changes after coating. This has to do with how the coating parameters impact the spin coating process's final result. We can conclude that the change in porosity increases with the thickness of the resulting thin sheet. This happens because more of the material's pores are typically covered by thicker thin coatings. Therefore, the porosity of porous materials is directly affected by the coating procedure.



(a) Concentration 10%



(b) Concentration 15%



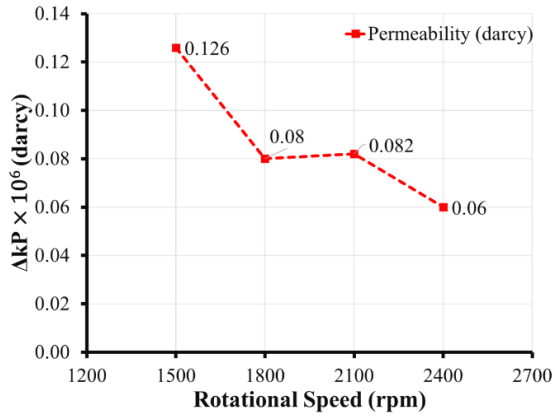
(c) Concentration 20%

Figure 2. Porosity on thin film coated porous ceramic material at various concentrations

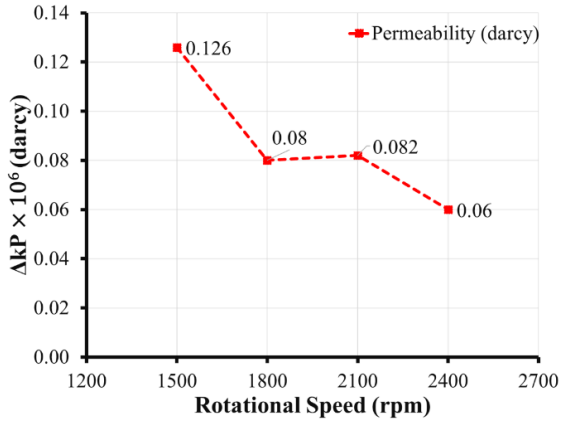
3.3 Permeability activity on coated porous ceramic materials

Permeability tests are conducted to determine the porosity level of coated porous ceramic material. Figure 3 illustrates how variations in concentration and rotation speed effect the porosity value. At 1500rpm, the porosity values at 10% (Figure 3(a)), 15% (Figure 3(b)), and 20% (Figure 3(c)) concentrations are $0.13x10^6$, $0.127x10^6$, and $0.125x10^6$, respectively. At 2400 rpm, the porosity values at 10%, 15%, and 20% concentrations are $0.06x10^6$, $0.043x10^6$, and $0.018x10^6$, respectively.

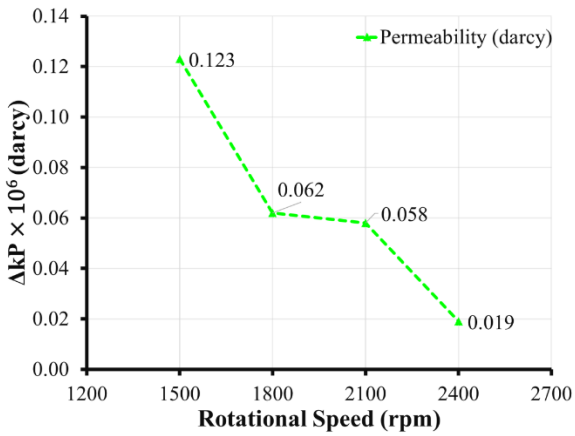
The permeability of the material after coating is greatly impacted by the parameters utilized during the coating process, as can clearly be seen from Figure 3. This relates to how the coating parameters affect the spin coating process itself, where the final coating results are determined by these parameters. According to the results, more material pores are covered as the thin layer gets thicker. Therefore, it may be said that the change in permeability increases with the thickness of the resulting thin layer.



(a) Concentration 10%



(b) Concentration 15%



(c) Concentration 20%

Figure 3. Permeability on thin film coated porous ceramic material at various concentrations

In this study, the sintering temperature used was limited to 1100℃, which is generally sufficient to reduce the material's permeability but inadequate to achieve full densification. At 1100℃, the sintering process can reduce pore size and increase material density to a certain extent; however, it is insufficient for optimal densification. At this temperature, permeability decreases as some of the pores in the material begin to close or diminish, but the material grains have not undergone significant growth or the consolidation necessary for comprehensive densification.

For effective densification under standard sintering settings, higher temperatures are frequently needed. At temperatures of approximately 1400℃, titania grains fuse together, causing substantial grain growth that decreases the material's pore size and quantity. This process, which includes sintering and diffusion, enables the material to achieve greater densification levels with more efficient pore reduction.

The enhanced mechanical characteristics and structural stability of sintered titania are a result of the material's increased density at high temperatures. Grain growth and pore reduction cause titania to densify during the sintering process. Increased sintering temperatures cause titania grains to expand and agglomerate, which reduces the size and quantity of intergranular pores. The density of the substance increases overall as a result of this procedure.

3.4 SEM analysis on coated porous ceramic materials

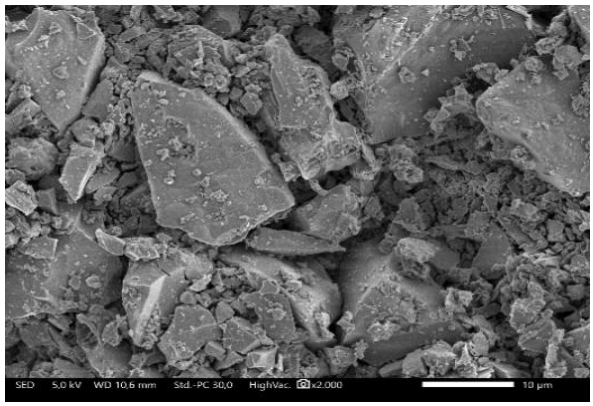
The success of the spin coating procedure and the effect of solvent concentration on the coverage of porous material are shown by the examination of the SEM pictures.

A highly porous structure with big, irregular gaps between particles is visible in Figure 4(a), which shows the substrate prior to coating. To create a smoother, more protective surface layer, the large gaps in this porosity-which is the baseline condition-must be repaired.

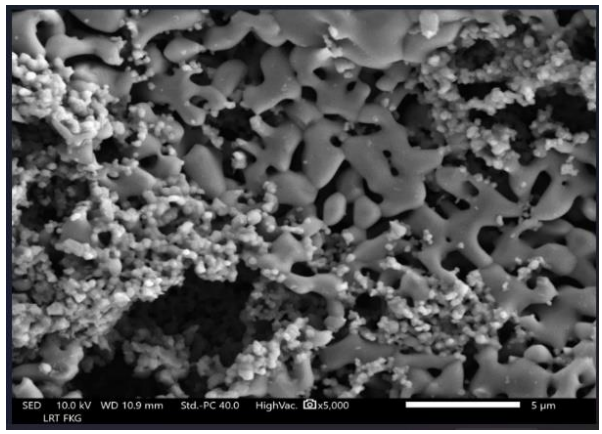
Figure 4(b) shows partial coverage of the porous surface after the initial coating at a moderate solvent concentration. The covering appears rather thin and irregular, with bigger gaps still visible even though some of the smaller pores have been closed. According to this finding, some regions of the porous network remained exposed because the moderate concentration did not produce enough viscosity or material density to completely cover it.

Following a coating application with a greater solvent content, Figure 4(c) demonstrates a noticeable improvement in coverage and homogeneity. In this case, the coating layer is noticeably thicker, has a more continuous surface, and has fewer exposed pores and denser. A higher viscosity solution from the higher solvent content produced a denser, more effective barrier layer by spreading less over the surface. The porous structure is better sealed by this thicker layer, indicating increased surface protection and improved impermeability.

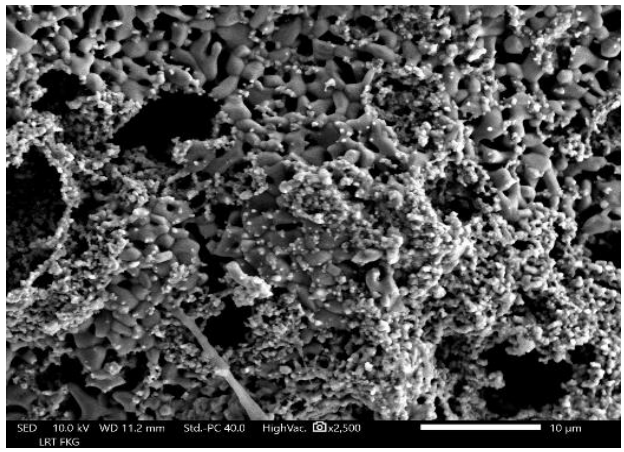
When taken as a whole, these Figure 4 pictures demonstrate how important solvent concentration is to getting the appropriate coating thickness and porosity reduction in spin-coated materials. For applications needing strong surface protection and limited permeability, higher concentrations produce thicker, more consistent coatings. The significance of solvent concentration as a crucial factor in customizing the spin coating procedure for porous materials is shown by this investigation.



(a) Corundum surface before coating



(b) After coating at 20% Concentration with 1800 rpm SpinSpeed



(c) After coating at 20% Concentration with 2400rpm SpinSpeed

Figure 4. SEM test result images of the specimen

4. CONCLUSION

Based on the research results above, it can be concluded that the coating process using the spin coating method with a titanium dioxide solution in ethanol solvent produces thin films on the corundum (alumina) ceramic substrate that become thinner as the rotational speed of the spin coater increases. The thickness of the layer is also affected by the solution concentration, where higher concentrations increase the viscosity of the solution and result in thicker films, even though the coating time used is the same.

After the coating process, there was a significant decrease in the material's porosity, especially in thicker layers, due to the closure of larger pores and the irregular distribution of pores. This decrease in porosity was followed by a reduction in the material's permeability, indicating that the thicker the layer, the more difficult it is for fluids to penetrate the material.

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