Evaluation on the Temperature and Calcination Time During Sol-gel Coating of TiO_2 on Iron Foam substrate

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Abstract. Iron foam is iron based material which is widely applied due to its unique properties. However since corrosion is also a problem for this material, coating with innert material is required in enhancing its applications. In the present research, TiO_2 coating is performed on iron foam suface by sol-gel dipping method. Focus is given on the study of the effect of calcinations temperature and time on the coating characteristics. $TiCl_4$ is used as the precursor with concentration of 0.3 M, added with 1M HCl solution and chitosan soluation with concentration of 1%. Calcination is performed at temperature of 400, 500 dan 600^{o}C and calcinations time of 1 and 3 hours inside controlled gas furnace using nitrogen atmosphere. Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) characterization data shows that TiO_2 particles form on the iron foam substrate. SEM characterization on the sample heated at 400^{o}C and heating time of 1 hour shows the formation of nano particle titania (0.06 μm) which is homogeneously distributed with less agglomeration than others and considered as the best sample in the present research. As the temperature and time of calcinations increase, more inhomogeneous particle distribution and bigger particles form.

Keywords: *iron foam, titania, self-assembled monolayer, chitosan.*

1 Introduction

Iron foam is one type of solid metal with very low density since it contain high volume fraction of pores with 75-95% of its volume is empty. Due to its very high porosity, this material has high compression strength and high energy absorber characteristic as well [1-4]. Based on these facts iron foam has been widely applied for various application such as for applications, which require load bearing characteristic, vibration and sound damper, filter, high temperature catalyst, implant material [5] and for the inner part of vehicle frame to reduce the weight of vehicle.

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As in the case of other metals, corrosion is one of the problems of iron foam. Since corrosion is degradation of material properties due to interaction with environment, therefore if it can be avoided interaction between metal substrate and environment, corrosion can be avoided. This principle is the idea of metal coating, that metal substrate is coated with passive materials (materials that has excellent corrosion resistance) in order to avoid interaction between metal surface and environment, therefore corrosion can be avoided. This metal coating can also be applied for iron foam in order to avoid corrosion and in the present research titania coating on iron foam substrate has been conducted.

Titania or titanium oxide (TiO_2) is ceramic materials which chemically stable and non toxic. Generally Titania has white color with high refractive index [6-8], therefore it is commonly used as dyes of paint paper, implant, tooth paste and coating for the materials susceptible to corrosion. Titania can be found in three different crystal structure, which is rutile, anatase and brookite, and this differences cause differences in their characteristics. In the present research, sol-gel method has been applied as the coating method to coat titania on iron foam substrate. This method is synthesis method to form anorganic compound by chemical reaction at low temperature processes [9-10]. During the process, coloid suspenses (sol phase) precursor transform into gel phase, and if this sysntesis process applied on certain substrate, the product of synthesis can be used as coating on the substrate. The advantages of using this method is good thermal and mechanical stability, with varous optional in surface modification. Rare references can be found on the coating of titania on iron foam, while this works shows potential advanteges for various application. Therefore the present works develop coating of titania on iron foam substrate by sol-gel method. In order to obtain optimum results, evaluation has been made on the variation of temperature and calcination time during the coating processes.

2 Research Methodology

In the present research iron foam substrate is used with the pore size of 450 um, density at 470 g/m2 and thickness of 1.6 mm. Titania was synthesized by TiCl4 precursor using sol-gel water based method using HCl acid catalyst. In addition, chitosan solution was also syntesized, and will be used as support in enhancing the adhesion between titania and iron foam substrate. Coating processes were performed by sol-gel dip coating method and before the process, substrate samples were weighed in order to determine the mass addition of the sample after coating. The coating was performed by dipping the sample into titania precursor for 5 minutes and followed with drying and calcination process. For calcination, variation of calcination temperature was performed at 400^{o}C, 500^{o}C and 600^{o}C, whereas calcination time was varied at 1 and 3 hours.

In order to evaluate the morphology of the titania coated on iron foam sample, scanning electron microscope (SEM) characterization was performed at varioius magnification. In addition, energy dispersive spectroscopy (EDS) and X-Ray Diffraction (XRD) characterization were performed in order to identify the composition of the resulting coating sample.

3 Data and Analysis

Table 1 shows the addition mass of each samples after coating process. It can be seen that there is differences of additional mass for each sample. For samples treated for one hour of calcination, it can be seen that sample treated at 400^{o}C shows highest additional mass, decrease at 500^{o}C and increase at 600^{o}C. On the other hand, for sample treated for 3 hour, there is a fixed trend of data that as temperature increase there is a decreasing of additional mass. These facts are predicted to be related with several mechanisms occur during calcination. At temperature between 100^{o}C and 300^{o}C releasing of H_2O occur, whereas releasing of gases such as CO_2 and salt mineral occur at 500^{o}C. In addition at 500^{o}C, chitosan degradasion occur with amount at around 50% of its total mass. However at high temperature, thereis also possibility for the excessive oxidation of iron foam, especially for its surface which is not covered by titania. Therefore beside there is a tendency for the decreasing of additional mass as temperature increases, thereis also mechanism that migh contributes to the additional mass of the sample during calcination.

Table 1 Summary of sample mass before and after coating.

Samples	Initial mass (g)	Final mass (g)	Δ mass (g)
1 hour 400^{o}C	0.0435	0.0546	0.0111
3 hours 400^{o}C	0.0407	0.0510	0.0103
1 hour 500^{o}C	0.0425	0.0497	0.0072
3 hours 500^{o}C	0.0392	0.0488	0.0096
1 hour 600^{o}C	0.0448	0.0571	0.0083
3 hours 600^{o}C	0.0386	0.0465	0.0079

Figure 1 (a) and (b) shows sample of SEM data of coated iron foam after sol gel dip coating process. It can be seen that TiO_2 layer cover the iron foam surface homogeneously. Excessive layers are also observed covering the pores of the substrate. Type layer growth for this coating is predicted to be layer by layer growth, which the first layer is between TiO_2 layer and substrate, and the subsequent layer is between TiO_2 layers. The first layer is predicted to be stronger the subsequent layers, since the former involving adhesion bonding between titania particles and substrate that is supported by chitosan particle on the substrate surface. On the otherhand subsequent layers on top of the first layer only involving weak cohesion bonding between titania particles.

Figure 2 shows EDS results of the corresponding sample in the Figure 1. It can be seen the mass fraction of O and Ti at 44.09% and 55.91%. This condition indicate that TiO_2 was formed on the iron foam substrate. In addition, by empirically calculating the mass percentage of O and Ti from EDS data, divided with atomic number of the elements, it can be obtained the empiric comparison between Ti and O is 1.16 : 2.81. The higher value of O is predicted due to oxidation of coated sample during calcination.

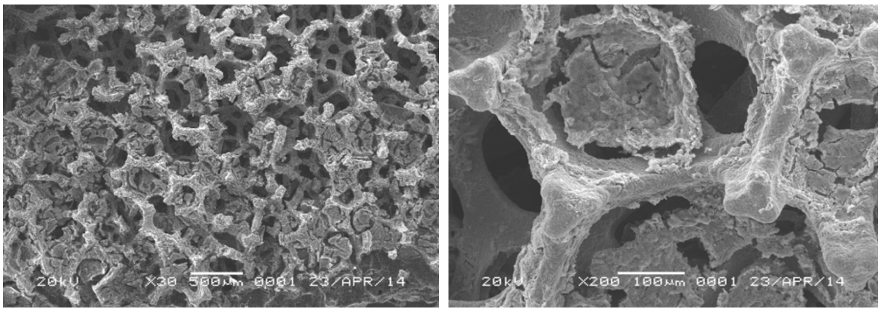
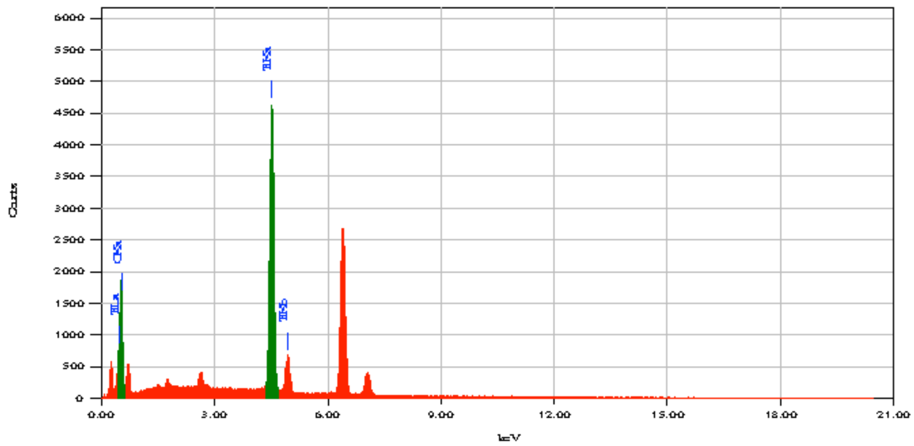


Figure 1 Sample of SEM figures at two different magnification for samples treated at calcination time 1 hour and temperature of 400^oC.



Element	(keV)	mass%	Error%	At%	Compound	mass%	Cation	K
O K	0.525	44.09	2.67	70.25				18.0168
Ti K	4.508	55.91	0.96	29.75				81.9832
Total		100.00		100.00				

Figure 2 EDS of sample treated at calcination time 1 hour and temperature of 400^oC.

On the other hand Figure 3, shows SEM data of sample treated at 400^{o}C calcination for 1 and 3 hours at higher magnification figures. It can be seen in both figures, agglomeration of titania particles with higher agglomeration observed for sample treated at 3 hours of calcination time. This condition is predicted due to higher releasing of chitosan (which has a function as dispersing agent) as the time of calcination increases.

Figure 4 shows titania particles on sample that is resulted from calcination at temperature of 500^{o}C for 1 and 3 hours. It can be seen agglomeration of titania particles which form bigger crystal at calcination time of 3 hours. These crystals were not observed for the sample treated at 400^{o}C, which indicate that higher temperature contribute to crystallization of titania particles. And this condition is also supported by data in the Figure 5, showing that bigger and more compact crystal observed for the sample treated at calcination time of 3 hours and calcination temperature of 600^{o}C. Based on the data resulted in the present research, since in most condition and application, smaller particle with less agglomeration is preferred, sample treated at calcination temperature of 400^{o}C and 1 hour calcination time shows the best condition in the present research. As compared with other parameter, sample treated at this parameter shows more homogeneous coating, less agglomeration and formation of bigger crystal was not observed.

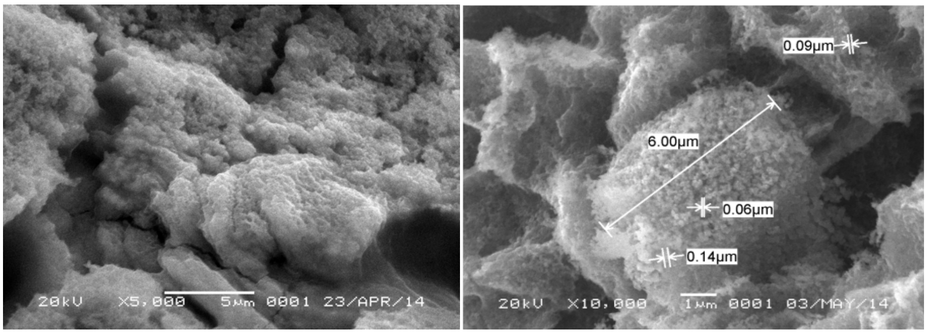


Figure 3 SEM data of samples treated at 1 hour (left figure) and 3 hours at calcination temperature of 400^{o}C.

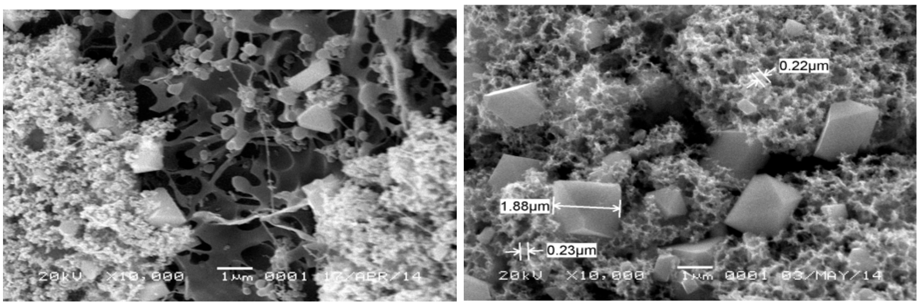


Figure 4 SEM data of samples treated at 1 hour (left figure) and 3 hours at calcination temperature of 500^{o}C.

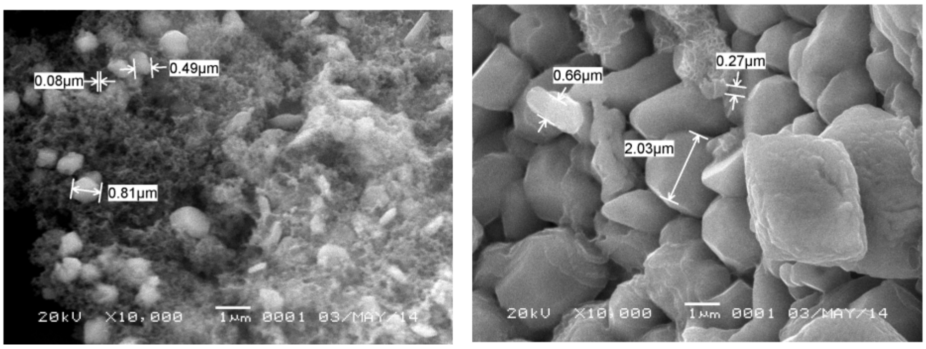


Figure 5 SEM data of samples treated at 1 hour (left figure) and 3 hours at calcination temperature of 600^{o}C.

4 Conclusion

Titania coating on iron foam substrate has been performed by sol gel dipping coating method. It was observed that increasing temperature and time of calcination tend to form agglomeration of titania particles. Agglomeration of titania particles results in the formation of bigger crystal of titania. In the present research, sample treated at calcination temperature of 400^{o}C and 1 hour calcination time shows the best condition in the present research.

5 References

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