Rangkuman Korespondensi:

Judul: "Particle and Crystallite Size Characterisation of Lead Titanate Derived from Solid-state Reaction Method"

Journal : Journal of Physical Science, Vol. 29(Supp. 2), 27–38, 2018

Tanggal	Tema/ kegiatan
20170916	Pemberitahuan pengiriman artikel ke EIC
20170918	Diskusi internal peneliti
20180208	Komunikasi untuk pengiriman artikel dari EIC ke JPS
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20180328	Artikel siap di publikasikan

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Dony Hidayat <aljanan@mail.unnes.ac.id>

EIC 2017: Article Acceptance

4 messages

EIC FT <eic.ft@mail.unnes.ac.id> Sat, Sep 16, 2017 at 7:12 AM
To: Dony Hidayat <aljanan@mail.unnes.ac.id>

Dear author,

On behalf of the organizing committee of the 6th Engineering International Conference on Education, Concept, and Application of Green Technology, held on 11 October 2017, in Semarang, Indonesia, we are very pleased to inform that your article has been accepted. Please find the detail information on the attachment.

We are going to update you with the detailed schedule of the conference soon.

Thank you and we look forward to your presentation at the event.

Regards,
Adhi Kusumastuti
Chairman of EIC 2017

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==========
Engineering International Committee
Faculty of Engineering
Universitas Negeri Semarang
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306K

Dony Hidayat <aljanan@mail.unnes.ac.id> Mon, Sep 18, 2017 at 10:41 AM
To: EIC FT <eic.ft@mail.unnes.ac.id>
Cc: dhoni.hartanto@mail.unnes.ac.id, Dhoni Hartanto <dhoni.hartanto@gmail.com>

Dear Committee,

We are very appreciate for the result.
Here I send to you the revision of my paper based on the reviewer comments.
The revision version with track change on position, then hopefully it can help the reviewer checking more easily.

Please change my email address,
From: aljananku@yahoo.com
To: aljanan@mail.unnes.ac.id

Please confirm while this email is already receipted.

Thank you very much.

Best Regards,

Dony Hidayat Al-Janan, Ph.D
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EIC FT <eic.ft@mail.unnes.ac.id> Sun, Sep 24, 2017 at 6:47 PM
To: Dony Hidayat <aljanan@mail.unnes.ac.id>

Dear author,

Your email is well received, thank you for the revision version.

Thanks
[Quoted text hidden]

Dony Hidayat <aljanan@mail.unnes.ac.id> Mon, Sep 25, 2017 at 8:40 AM
To: rahmat doni <rahmat_doni@yahoo.com>

alhamdulillah diterima pak...

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Dony Hidayat <aljanan@mail.unnes.ac.id>

Artikel EIC 2017 final

2 messages

rahmat doni <rahmat_doni@yahoo.com> Sun, Sep 17, 2017 at 6:58 AM
Reply-To: rahmat doni <rahmat_doni@yahoo.com>
To: Dony Hidayat <aljananku@yahoo.com>
Cc: Dony Hidayat <aljanan@mail.unnes.ac.id>, Aljanan Ku <aljananku@gmail.com>

Assmlkm pak
ini sy kirimkan file artikel utk EIC 2017 yg sdh sy edit kesimpulan dan daftar referensinya semoga sdh tidak ada lagi yang salah, tp supaya lebih yakin lagi sy mengharap sekali lagi jenengan baca bok menowo ada yg keliru, baik bahasa maupun tata tulisnya.
Demikian pak terimakasih banyak atas perhatian dan bantuannya.
Wassallam
Rahmat

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Dony Hidayat <aljanan@mail.unnes.ac.id> Mon, Sep 18, 2017 at 9:08 AM
To: rahmat doni <rahmat_doni@yahoo.com>

wa'alaikumussalam

siap pak

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EIC FT <eic.ft@mail.unnes.ac.id>
to Zulkifli, Akhiar, Fazlina, Abdul

Feb 20, 2018, 8:47 AM

Dear Zul,

Thank you very much for your feedback and information. We are going to share it to the authors.

Moreover, would you please provide official acceptance and declined letter from JPS for the authors? So we can notify them.

•••

Zulkifli Musa <zulkiflimusa@gmail.com>
to me, Akhiar, Fazlina, Abdul

Feb 21, 2018, 3:59 AM

Dear Adhi,

In special issues, extending acceptance and rejection is within the guest editor's jurisdiction.

You can write a recommendation of acceptance (subject to satisfactory revision, etc.) to identified authors/papers. I can provide a template of acceptance if you need. Later.

It should be noted that acceptance letters are too early to write now, as many of the papers require severe revision and are not in publication-ready mode (you can proceed with rejection though). You should wait until the changes are done.

Additionally, we will require Editorial Notes, written by guest editor(s), briefing/introducing about the special issue and the conference theme. Half a page will do. Let me know if you need further guidelines on this.

Thanks.

Rgds
Zul

•••

EIC FT <eic.ft@mail.unnes.ac.id>
to Zulkifli, Akhiar, Fazlina, Abdul

Mar 11, 2018, 8:17 PM

Dear Zul,

Hope this email finds you well.
All authors have sent the revised manuscripts and hopefully, they meet the requirements. Please find the revised manuscripts here: https://drive.google.com/drive/folders/1h4urKCSYq8zxrnFpubsQNbmFAZZVe1xE?usp=sharing

Moreover, attach is the editorial notes that you required.

Thank you and looking forward to hearing from you.

Regards,
Adhi K

•••

Zulkifli Musa <zulkiflimusa@gmail.com> Mar 16, 2018, 6:05 AM

to me, Akhiar, Fazlina, Abdul

Dear Dr. Adhi,

Thank you for all the cooperation in getting the articles revised accordingly, and delivering timely revision.

As per plan, the publication is now set for June 2018 (tentative 25 June).

During the next few weeks, please be alert on the editorial queries that may arise on these articles, which require author's (or guest editor)'s response.

We will also be issuing an invoice at the onset of production (for 22 articles, in accordance with the agreed fee structure).

For this invoicing purpose, please provide us the details of: 1. Contact person, and 2. Full corresponding address. Note that where applicable, the organizer is to bear the bank charges.

We are glad to see things taking shape now. Thank you and we look forward to a fruitful collaboration.

Rgds
Zul

•••

Zulkifli Musa <zulkiflimusa@gmail.com> Mar 28, 2018, 6:15 AM

to me, Akhiar, Fazlina, Abdul

Dear Dr. Adhi,

Two articles require language revision.

[Note that the article numbering has been revised (as a few articles were taken out before), which means they no longer follow previous numbering].

13. Estimation of Battery Capacity Using Voltammetry Method of Lead Acid and Nickel Cadmium Battery Based LMNN at Jember Electric Substation

16. Design and Construction of Chem-E-Car SMARTTRONS Project Utilizing Heat Gradient of Thermoelectric Generator

Apart from language, some of the references still did not fully comply to the requirements (incomplete references being among the issues).

Please remind the authors their responsibility to ensure comformance to expectation and standard. As the journal remains steadfast to safeguard the quality of its publications, we ask the authors to commit to the same, or else the papers will not get past production pipeline.

We recommend a period of 2-3 weeks. Once received, we shall update you with the revised expected publishing date (EPD).

Thanks, and hope to receive new revisions soon.

In the mean time, you should start getting the other article's editorial queries within the coming week.

All the best.

REVIEW FORM

Paper Review

Paper ID	EIC17A007
Paper Title	Particle and Crystallite Size Characterization of Lead Titanate Derived from Solid-State Reaction Method

PART A

	Poor	Average	Good	Very Good
Originality			**X**	
Innovation		**X**		
Applicability			**X**	
Presentation and English		**X**		
Match to Conference Topic			**X**	

*** Please kindly mark with "X"**

PART B

General comment	From my point of view, this paper gives some interesting and original results on the preparation and characterization of Lead Titanate for piezoelectric material. The paper is worthy to publish although there are a few sentences and grammar that need to be corrected. There are several grammatical errors so it needs to undergo a careful edition. This article is still hard to read. Several sentences must be reconstructed to make it easier to understand.

Title	Good
Abstract	- Some words should be corrected. For examples: ..... particle and crystallte size..... ; ..... using particle size analyze (PSA)..... ; ..... to analyze qualitative, quantitative phase..... ; ..... were successfully achieved..... ; - Keyword: lead titatane; - The two last sentences should be revised.
Introduction	- Some words should be corrected. For examples: ..... lead titatane... - Some abbreviations with superscript or subscript should be corrected. For examples: The PbTiO3 property....; should be $PbTiO_3$; ……so-gel.....; should be sol-gel ; etc...
Methodology	The methodology need to re-organize. List all the materials and reagents used in detail in separate section. Then the experimental procedures should be organized properly for better explanation.
Results and Discussion	- All figures and tables must be mentioned first in the text, accompanied by a textual presentation and followed by figures or tables. - Some sentences should be corrected for better understanding. Example of the sentence: "Based on the Rietveld analysis was performed applying High Score Plus program that the crystal system of TiO_2 and $PbCO_3$ is tetragonal dan orthorhombic respectively". etc.....
Conclusion	The conclusions in points 2 and 3 should be rewritten or

	restructured.
References	Please prepare the reference according to guideline
Additional Comment	- The points given above are not a comprehensive list, but rather, just a sampling of a few things that I thought should be corrected. - Some things that need more attention are highlighted in yellow in the article.

*** Please kindly comment per-section of paper**

PART C

	Reject	Accept with revision as per comments	Accept without revision
Recommendation		**X**	

*** Please kindly mark with "X"**

PART D

Name of Journal	**Recommendation**
Journal of Physical Science (Q3 SJR)	**X**
International Journal of Innovation and Learning – Inderscience Publishers (Q3 SJR)	
Jurnal Pendidikan IPA Indonesia (Scopus – indexed)	
AIP Conference Proceedings (Scopus – indexed)	**X**

*** Please kindly mark with "X"**

Particle and Crystallite Size Characterization of Lead Titanate Derived from Solid-State Reaction Method

Abstract: Lead Titanate ($PbTiO_3$) ceramics was produced by solid state reaction via a vibratory ball milling machine and subsequent heat treatment. The effect of milling time on the particle and crystallte size of $PbTiO_3$ powder was investigated. Powder samples were studied using particle size analyze (PSA). The annealing process was up to 1000°C and the products were examined by X-Ray Diffractometer (XRD) to analyze qualitative, quantitative phase and crystallite size. It was found that the average particle's size of powder initially increased due to laminated layers formation and then decreased to an asymptotic value of ~0,8 μm as the milling time extended even to a relatively longer time. Single-phase $PbTiO_3$ were succesfully achieved at 600°C for 1 h holding time of annealing temperature. The crystallite size of $PbTiO_3$ were annealed at 1000°C for 1 h holding time is 64 nm. The crystallite size of $PbTiO_3$ increases as function of temperature of annealing process.

Keywords: *lead titatane, particle size, crystallite size, solid state reaction, annealing*

1. INTRODUCTION

Ferroeletrics are the materials with reversible spontaneous polarization [1-4]. Lead titatane ($PbTiO_3$) is one of the fundamental ferroelectris materials with an ABO_3 compound structure called perovskite and the highest spontaneous polarization among all the ferroelectric perovskites [5-14]. According to the first principle calculations on ferroelectric perovskites, hybridization between the electronic states of A or B atoms and the oxygen atoms is essential for ferroelectricity. $PbTiO_3$ has highest tetragonal distortion ($c/a \approx 1.063$) among all the members of the perovskite's family. This tetragonal distortion corresponds to the highest spontaneous polarization among all the ferroelectric perovskites. Perovskite-type $PbTiO_3$ has a high spontaneous polarization of 86 $\mu C/cm^2$[5], Curie temperature of 364°C[15] - 490°C[10,16], a relatively low permittivity, a large pyroelectric coefficient (250 $\mu C.cm^{-2} K^{-1}$)[17] and small dielectric constant. The dielectric constant increased in the annealing range of 450-750°C. This trend is due to the increased grain size and higher crystallinity with annealing temperatures [18]. The values of saturation polarization *(P_s)*, remanent polarization *(P_r)*, and coercive field *(E_c)* $PbTiO_3$ are 13.1 $\mu C/cm^2$, 3.2 $\mu C/cm^2$ and 5.1 kV/cm, respectively. Because of its character, ferroelectrics are widely used in many applications: the ferroelectric random access memory field, pyroelectric infrared sensor, electro-optic devices, insulator gates in metal-insulator-semiconductor diodes, capacitors, transistor, piezoelectric actuators, high frequency ultrasonic transducers and so on [19-22].

The PbTiO3 property within its applications depends on several aspects: the purity of materials indicated in stoichiometry number; and microstructure that consists of phase, particle's size and then crystallite's size. To obtain stoichiometry, particle and crystallite size $PbTiO_3$ different preparation methods have been introduced, such as co-precipitation [9], emulsion [10] or hydrothermal treatment [20, 23, 24], so-gel [4, 6, 12, 16, 25], spark plasma sintering [26], sonochemical [27] and besides the conventional solid-state reaction of mixed oxides or mechanical alloying [28-35]. All of the methods will produced vary microstructure, processes and then manufactures temperature. The processing time must be determined for quick process and the better quality of final product,- has the higher purity in single phase, also the particle and crystallite formed in nanometer size. Chattopadhyay et al. [36] conducted a detailed study on the influence of particle size on the ferroelectric properties of lead titanate. Studies revealed that size effects are important only below 100 nm. The usual method for producing the fine nanocrystalline materials is mechanical alloying and milling by a ball-milling technique, which has also being adapted to the preparation of lead titanate. The technique is considered simple and less costly for producing very fine particles. In this study, mechanical alloying and milling method has been developed to produce the fine nanocrystalline materials of PbTiO3 ceramics, finally the quality result was judged on material characterization that consist of particle and crystallite size.

2. EXPERIMENTAL METHOD

$PbTiO_3$ (coded PTO) were obtained from the mixture of lead (II) carbonate ($PbCO_3$) and titanium (IV) oxide (TiO_2) powders, by using high-energy ball milling and heat treatment processes. Stoichiometric quantities of the analytical-graded precursors $PbCO_3$ and TiO_2 with purity better than 98 % were mixed and milled in a vibratory ball mill up to 60 hours. The weight ratio of balls to milled material was 1:10. After milling process the diameter sizes of examined powder particles were determined using Particle Size Analyzer (PSA) Malvern ZS nanoseries. Phase analysis and crystallite size of milled powders were carried out using the X-ray Philips diffractometer equipped with

Cu $K\ \alpha$ radiation. The X-ray diffraction patterns were recorded by "step-scanning" method. The powders were annealed in the electric chamber furnace (Nabertherm N31/H) at 500, 600, 700, 800, 900 and 1000°C in the air under atmosphere pressure up to 1 hours. The Rietveld analysis was performed applying High Score Plus program that is an update version for Rietveld refinement with PC and mainframe computers. The pseudo-Voigt function was used in the describing of diffraction line profiles at Rietveld refinement. The crystallite sizes for $PbCO_3$ and TiO_2 also $PbTiO_3$ phases were estimated using Williamson-Hall method [37]. Intensity data during scanning 2 seconds taken for each step of the diffraction angle 0.005°. Diffraction peak width ***(B)*** is given by equation 1 and the mean crystallite size ***(D)*** obtained from equation 2.

$$B = \frac{0.9\,\lambda}{D\,cos\theta} + \eta\,tan\theta \tag{1}$$

$$B\,cos\theta = \frac{0.9\,\lambda}{D} + \eta\,sin\theta \tag{2}$$

Where, λ is the X-ray wavelength, η is the strain in the materials and θ is the Bragg angle, while the peak width B obtained after the correction due to instrument broadening according to equation 3.

$$B = \sqrt{B_O^2 - B_S^2} \tag{3}$$

Where, B_o is the Full Width at Half Maximum (FWHM) of the test sample. B_s is the FWHM standard samples that used an in silicon (Si).

3. RESULTS AND DISCUSSION

The diffraction patterns of TiO_2 and $PbCO_3$ precursors (Fig.1) which these were matched diffraction patterns of TiO_2 and $PbCO_3$ in *data base Inorganic Crystal Structure Database* (ICSD) number 98-009-6946 and 98-016-6089 respectively. Based on the Rietveld analysis was performed applying High Score Plus program that the crystal system of TiO_2 and $PbCO_3$ is tetragonal dan orthorhombic respectively.

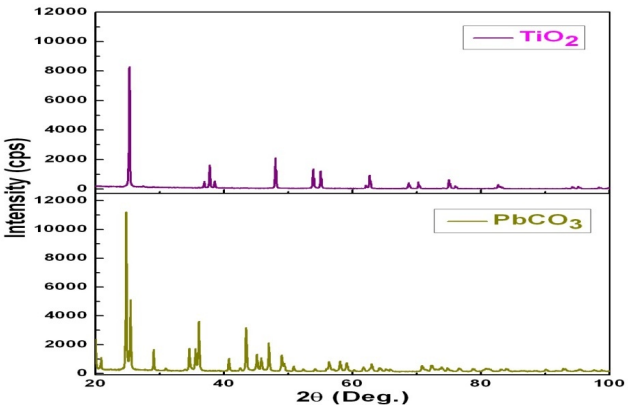

Fig.1. X-ray diffraction patterns of TiO_2 and $PbCO_3$ precursors

Fig.2 are showing results of evaluation for mean particle size of TiO_2 and $PbCO_3$ mixture up to 60 hours of milling. All the sample powders go through the four stages of the mechanical alloying process, namely: (a) initial stage; (b) intermediate stage; (c) final stage; (d) completion stage [38]. It shows that mean particle sizes of mechanically milled for TiO_2 and $PbCO_3$ mixture in initial or early stages of milling are characterized by the increase in the mean particle size due to incorporation of particles of component compounds. The mean particle size of the material increased from 4.2 to 17μm at the 1-10 hour mixing. The core compounds experienced cold weld, namely the integration of the two particles of the basic compounds to form a close bond between the particles as a consequence of the ball mill impact. The process of impact between ball mills continuously occurred. The largest mean particle size were achieved after 15 hours milling times, that the mean particle size of TiO_2 and $PbCO_3$ mixture is ~19 μm. Extention of milling time beyond 15 hours have decreased progressively the mean size towards a settle value. Long terms of mechanical treatment during advanced stages of mechanical alloying have caused particles experiencing embitterment due to accumulation of internal stresses [39]. Continuous plastic deformations to the brittle particles should caused further reduction in particle size towards an average value of ~ 0.8 μm and eventually settle down to that value even if the deformation continues to grow after the duration of 60 hours milling time at completion stage.

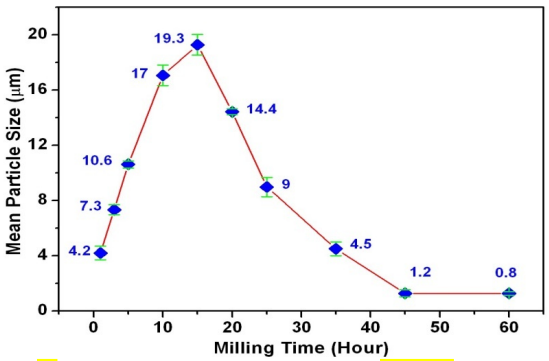

Fig.2. the mean particle size of TiO_2 and $PbCO_3$ mixture as function of milling time

In Fig.3, the comparison of diffraction patterns of TiO_2 and $PbCO_3$ mixture after 1, 5, 10, 25, 40 and 60 hours of milling process is shown. Identification of the diffraction peaks ensured that the all peaks are matched with that of TiO_2 and $PbCO_3$ phase.

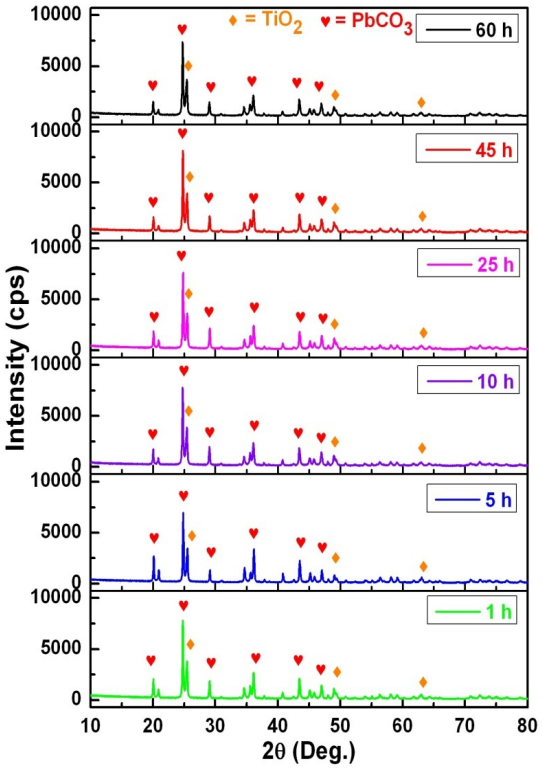

Fig.3. X-ray diffraction patterns of TiO_2 and $PbCO_3$ mixture up to 60 hours of milling

Fig. 4 is results of the evaluation of mean crystallite size in milled particles after milling process based on XRD patterns using Williamson-Hall method. In Fig.4, shows the ball milling process for 60 hours in a mixture of TiO_2 and $PbCO_3$ lead to a decline in the value of the average crystallite size. The average size of the TiO_2 crystallites decreased not so significantly exponentially with time. In contrast to TiO_2, after mechanical alloying for 60 hours, the crystallite size of $PbCO_3$ decreased significantly, which is about 1.5 times smaller. It shows that the process of milling up to 60 hours resulted in a more brittle and easily shattered $PbCO_3$ when compared with TiO_2. The mechanical milling process causes decrease of the crystallite size of tested phases and leads to homogenizing the milled mixture. The mean crystallite size of TiO_2 and $PbCO_3$ phases diminishes to 87 nm and 80 nm, respectively of the milling time up to 60 hours.

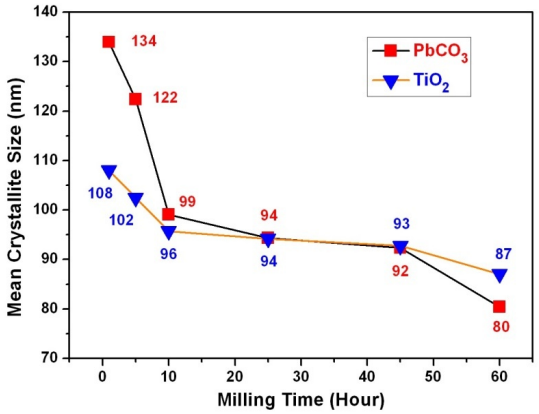

Fig.4. The mean crystallite size of TiO_2 and $PbCO_3$ mixture up to 60 hours of milling

The X-ray diffraction investigations of TiO_2 and $PbCO_3$ powder mixture milled for 60 hours and after different temperature and times of annealing treatment up to 1000°C (Fig.5).

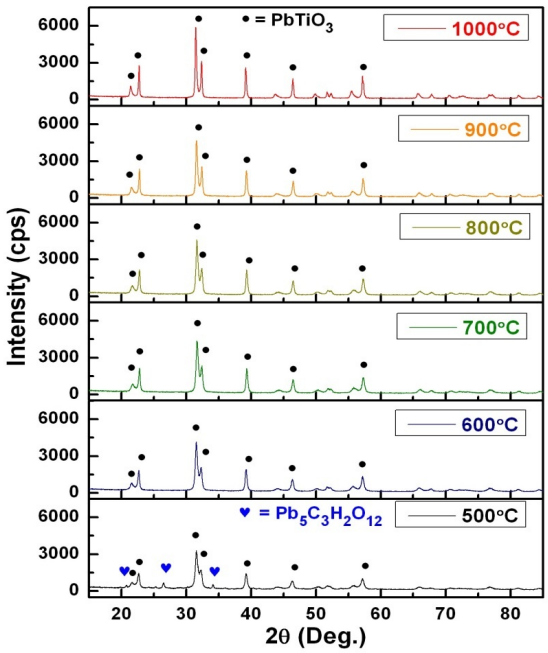

Fig.5. X-ray diffraction profile of TiO_2 and $PbCO_3$ mixture after annealing up to 1000°C.

At 500°C with a holding time 1 hour, the single phase $PbTiO_3$ has not yet been formed where there is still another phase that is present, $Pb_5C_3H_2O_{12}$. Single phase $PbTiO_3$ with tetragonal *perovskite* crystal *structure* was formed after annealing at 600°C up to 1000°C for 1 hour. X-ray diffraction pattern of the sample which has undergone annealing process at 600°C for 1 hour matched with the data based on the diffraction pattern on Inorganic Crystal Structure Database (ICSD) number 98-009-0693. The lattice constant calculated from the XRD data is $a = b = 3.9116$ Å and $c = 4.0943$ Å. The structure matches with the $PbTiO_3$ that can be used in various applications. Based on the x-ray diffraction patterns in Figure 5, the average size of crystallites of each phase can be found, where the result is shown in Figure 6.

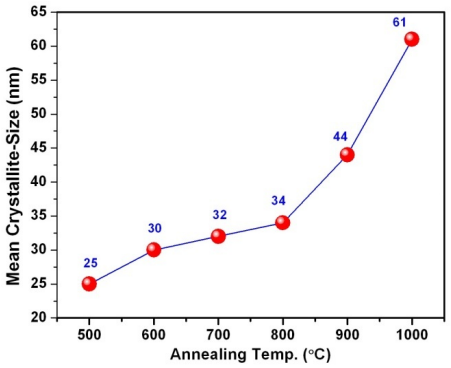

Fig.6. The mean crystallite size of $PbTiO_3$

To know for sure, temperature and time of the most optimum is achieved in the transformation process into a single phase $PbTiO_3$ phase in full, then it should be tested thermal analysis. The intensity and sharpness of the X-ray diffraction (XRD) peaks of $PbTiO_3$ phase were found to increase with annealing temperature at 600°C up to 1000°C. Based on Figure 6, it can be concluded that the average size of crystallites $PbTiO_3$ increased exponentially as the rising of annealing temperature. The increase in intensity and sharpness of the XRD peaks with annealing temperature may be attributed to the increase in the grain or crystallite size thereby increasing packing density of the samples annealed at higher temperature.

4. CONCLUSION

Tests on a mixture of TiO_2 and $PbCO_3$ as a piezoelectric material, $PbTiO_3$, after undergoing a process of milling and sintering resulted in several conclusions, which are:

1. Mechanical alloying process for 60 hours in a mixture of TiO_2 + $PbCO_3$ caused the mixture of the two compounds decreased their average particle size to 0.8 µm and a crystallite size of 87 nm and 80 nm, respectively. The reduction of the size of particle and crystallite was the result of the continuous collision between sample powder and ball mill. As a consequence, the samples underwent embrittlement and deformation.
2. Heating of the particles results in the mechanical integration of the annealing temperature of 600 °C up to 900 °C promote the formation of crystallites with nanometer-scale size and material make the integration of mechanical and annealing the material with a particle containing nanocrystallite.
3. $PbTiO_3$ ceramic has been prepared by conventional solid-state reaction processing technique. The as fired powder was found to be amorphous and crystallized to tetragonal $PbTiO_3$ after annealing at 600 °C up to 900 °C for one hour. Dense ceramic samples were obtained by annealing at different temperatures and holding times.

5. REFERENCES

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