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Denpasar - Bali, 15 - 16 Oktober 2015

PUSAT TEKNOLOGI DAN KESELAMATAN REAKTOR NUKLIR (PTKRN)
PUSAT KAJIAN SISTEM ENERGI NUKLIR (PKSEN)
BADAN TENAGA NUKLIR NASIONAL

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Bali, 15-16 Oktober 2015

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Pusat Teknologi dan Keselamatan Reaktor Nuklir

Pusat Kajian Sistem Energi Nuklir

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KATA PENGANTAR

Puji syukur kami panjatkan kehadirat Tuhan Yang Maha Esa, yang telah melimpahkan rahmat dan hidayah Nya sehingga Prosiding Seminar Nasional Teknologi Energi Nuklir 2015 dapat diselesaikan. Prosiding ini memuat makalah yang dipresentasikan pada Seminar Nasional Teknologi Energi Nuklir, dengan tema Kontribusi Teknologi Energi Nuklir bagi Kemandirian dan Keberlanjutan Pembanguan Nasional, yang diselenggarakan pada hari Kamis – Jumat, 15 – 16 Oktober 2015 di Gedung Pascasarjana Universitas Udayana, Denpasar, Bali. Seminar tersebut terselenggara atas kerjasama Pusat Teknologi dan Keselamatan Reaktor Nuklir (PTKRN-BATAN) dengan Pusat Kajian Sistem Energi Nuklir (PKSEN-BATAN) didukung oleh Fakultas Teknik dan Fakultas Matematika dan Ilmu Pengetahuan Alam, Universitas Udayana.

Penerbitan Prosiding ini dimaksudkan untuk menyebarluaskan hasil penelitian dan pengembangan iptek energi nuklir. Diharapkan dengan terbitnya prosiding ini dapat menggalang kesinambungan komunikasi di antara para peneliti, akademisi, dan pemerhati terkait dengan iptek energi nuklir di Indonesia, dalam rangka mengantisipasi pesatnya perkembangan iptek energi nuklir di dunia.

Panitia menerima sebanyak 83 makalah teknis dari berbagai instansi. Setelah melalui seleksi dan evaluasi oleh Dewan Editor, Panitia memutuskan 77 makalah dapat diterima untuk dipresentasikan dalam Seminar Nasional Teknologi Energi Nuklir 2015. Hasil seleksi ulang dan evaluasi oleh Dewan Editor terhadap makalah yang dipresentasikan, memutuskan sebanyak 74 makalah dapat diterbitkan dalam Prosiding Seminar Nasional Teknologi Energi Nuklir 2015. Ke 74 makalah tersebut terdiri dari : 67 makalah dari BATAN, masing-masing 2 makalah dari BAPETEN dan Universitas Udayana, dan masing-masing 1 makalah dari Universitas Sriwijaya, ATK Kemenperin Yogyakarta, dan STKIP Sumedang.

Kami menyadari bahwa prosiding ini tentu saja tidak luput dari kekurangan, untuk itu segala saran dan kritik kami harapkan demi perbaikan prosiding pada terbitan tahun yang akan datang. Akhirnya kami berharap semoga prosiding ini bermanfaat bagi yang memerlukan.

Jakarta, Maret 2016

Dewan Editor

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KEPUTUSAN

KEPALA BADAN TENAGA NUKLIR NASIONAL

NOMOR: 77/KA/III/2015

TENTANG

PERUBAHAN LAMPIRAN KEPUTUSAN KEPALA BATAN NOMOR 41/KA/II/2015 TENTANG PENYELENGGARAAN SEMINAR NASIONAL TEKNOLOGI ENERGI NUKLIR 2015 DAN PEMBENTUKAN PANITIA

KEPALA BADAN TENAGA NUKLIR NASIONAL,

Menimbang: a. bahwa dengan Keputusan Kepala BATAN Nomor 41/KA/II/2015 telah ditetapkan Penyelenggaraan Seminar Nasional Teknologi Energi Nuklir 2015 dan Pembentukan Panitia;

b. bahwa untuk kepentingan dinas, maka perlu mengubah Lampiran Keputusan sebagaimana dimaksud pada huruf a;

Mengingat : 1. Peraturan Presiden Nomor 46 Tahun 2013 tentang Badan Tenaga Nuklir Nasional;

2. Keputusan Presiden Nomor 72/M Tahun 2012;

3. Peraturan Kepala BATAN Nomor 14 Tahun 2013 tentang Organisasi dan Tata Kerja Badan Tenaga Nuklir Nasional sebagaimana telah diubah dengan Peraturan Kepala BATAN Nomor 16 Tahun 2014;

MEMUTUSKAN:

Menetapkan : KEPUTUSAN KEPALA BADAN TENAGA NUKLIR NASIONAL TENTANG PERUBAHAN LAMPIRAN KEPUTUSAN KEPALA BATAN NOMOR 41/KA/II/2015 TENTANG PENYELENGGARAAN SEMINAR NASIONAL TEKNOLOGI ENERGI NUKLIR 2015 DAN PEMBENTUKAN PANITIA.

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KESATU : Mengubah Lampiran Keputusan Kepala BATAN Nomor 41/KA/II/2015 tentang Penyelenggaraan Seminar Nasional Teknologi Energi Nuklir 2015 dan Pembentukan Panitia menjadi sebagaimana tersebut dalam Lampiran Keputusan ini.

KEDUA : Keputusan ini mulai berlaku pada tanggal ditetapkan.

Ditetapkan di Jakarta
pada tanggal 25 Maret 2015
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$Li_4Ti_5O_{12}$ SYNTHESIS AS A BATTERY ANODE MATERIALS WITH SOLID STATE REACTION METHOD

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ABSTRACT

$Li_4Ti_5O_{12}$ SYNTHESIS AS A BATTERY ANODE MATERIALS WITH SOLID STATE REACTION METHOD. $LI_4TI_5O_{12}$ synthesis of research has been done as a battery anode materials with a solid state reaction method. The synthetic materials were the anatase titanium dioxide and lithium hydroxide from Merck products. Titanium dioxide and lithium hydroxide were mixed with sonochemistry method in deionized water for two hours. The suspension formed is dried at temperature of 150 °C and compacted at 4000 psi and the resulting pellets calcined in a furnace at differ temperatures at 400, 500, 600, 700, and 800 °C respectively for 2 hours. Characterization was performed using X-ray difractometer (XRD) to determine the crystal structure, scanning electron microscope (SEM) for observation of microstructure. The conductivity was measured using LCR meter. From XRD data characterization were compared with the reference data of JCPDS 49-0207, Showed that the calcination result at temperature of 800 °C is assuring the formation of succesfully. $Li_4Ti_5O_{12}$. $Li_4Ti_5O_{12}$ has solid particles surface of DC conductivity value $1,5066x10^{-6}$ $S.cm^{-1}$ at frequency range 3K-30KHz.

Keywords: $Li_4Ti_5O_{12}$, battery anode, solid state method

ABSTRAK

SINTESIS $Li_4Ti_5O_{12}$ SEBAGAI BAHAN ANODA BATERAI DENGAN METODE REAKSI PADATAN. *Telah dilakukan penelitian sintesis $Li_4Ti_5O_{12}$ (LTO) sebagai bahan anoda baterai dengan metode reaksi padatan. Bahan sintesis berupa titanium dioksida anatase dan litium hidroksida dari produk Merck. Titanium dioksida dan litium hidroksida dicampur dalam media deionized water selama dua jam. Suspensi dikeringkan pada suhu 150 °C. dikompaksi pada 4000 psi dan pelet yang dihasilkan dikalsinasi dalam furnace dengan suhu bervariasi 400, 500, 600, 700, dan 800 °C masing-masing selama 2 jam. Karakterisasi dilakukan menggunakan X-ray difractometer (XRD) untuk mengetahui struktur kristal, scanning electron microscope (SEM) untuk observasi struktur mikro, dan untuk mengetahui konduktivitasnya digunakan impedance capasitance resistance (LCR) meter. Hasil karakterisasi XRD dibandingkan dengan acuan JCPDS 49-0207, menunjukkan bahwa pada hasil kalsinasi suhu 800 °C terbentuk $Li_4Ti_5O_{12}$. $Li_4Ti_5O_{12}$ yang dihasilkan mempunyai permukaan partikel padat dengan nilai konduktifitas DC $1,5066x10^{-6}$ $S.cm^{-1}$ pada rentang frekuensi 3K Hz-30K Hz.*

Kata kunci: *$Li_4Ti_5O_{12}$, anoda baterai, metoda solid state*

INTRODUCTION

Battery is one of chemically electrc component that has an ability to save and pruduce power as an electrical energy. Battery divided into two types, namely battery primary (non rechargeable battery) and secondary batteries (rechargeable battery). Both the primary battery and secondary battery convert chemical energy into electrical energy. Primary batteries can only be used once, because its chemical reactions are irreversible (irreversible reaction). While the secondary battery can be recharged because the chemical reaction is reversible (reversible reaction). A battery usually consists of three major components, namely: the anode (negative pole of the battery), the cathode (positive pole of the battery), and electrolyte (conductor). Anode used in lithium batteries (Lithium Ion Battery

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LIB =) generally are composed of graphite. Excellence graphite has high capacity and also has limitations like the inability in conditions of high discharge rates[1].

Lithium titanate ($Li_4Ti_5O_{12}$) as an alternative anode materials for lithium ion batteries offer higher safety and cycle life more than the anode material use commercial carbon (graphite) that are widely used today pad[2-5]. $Li_4Ti_5O_{12}$ has a spinel structure (space group Fd 3m, ≈ 8:35 Å) consisting of an oxygen atom resembles a cubic closed with lithium atoms occupy tetrahedral (8a) and oktahedral (16c, 16d), while Ti partly replaced by Li, placed in octahedral 16d. Overall Li insertion capacity is limited by the number of octahedral sites, but $Li_4Ti_5O_{12}$ can accommodate $3Li^+$ ions per unit cell with no changes in unit cell parameters[6].

Intercalation process is based on the phase transition of spinel Li4Ti5O12 like NaCl structured in a way that enables the reduction of Ti4 + ions 3 of 5 ions, which correspond to the theoretical capacity of 175 Ah kg-1.3 Detailed studies of the chemical and electrochemical insertion of Li into $Li_4Ti_5O_{12}$ have been done by Aldo and Ariyoshi[7-8].

Phase transition occurs without or with a very small displacement of oxygen atoms from its initial position in the spinel structure. Structural changes due to insertion of lithium ignored during manufacture $Li_4Ti_5O_{12}$ anode material for battery applications with long life cycle. Compared with natural graphite, $Li_4Ti_5O_{12}$ shows better thermal stability and sharp voltage waveforms that can serve as an indicator of the overcharge[9]. Various methods of synthesis anode has been done with the sol-gel technique[10-11], mixing wet[12], hydrothermal[13], and a thin layer[14], has been studied extensively. This study aims to synthesis $Li_4Ti_5O_{12}$ with solid state reaction method and determine the growth phase due to the heat treatment temperature calcination at 400, 500, 600, 700, and 800 ° C. Microstructure analysis and determination of the conductivity of materials $Li_4Ti_5O_{12}$ conducted to determine the character of the sample.

METHODOLOGY

Equipment and materials used in this study were glassware, magnetic stirrer hotplate, furnace, X-ray diffraction (XRD), scanning electron microscope (SEM). While the materials used are deionized water, titanium dioxide, and lithium hydroxide. Anatase titanium dioxide and lithium hydroxide from Merck products.

Titanium dioxide was mixed with lithium hydroxide in deionized water under ultrasonic treatment for two hours until formation a suspension.The suspension then drying using hot plate magnetic stirrer at temperature 150 °C. The drying powder obtained then compacted at 4000 psi using hydrolic press and the resulting pellets calcined in a furnace at differ temperatures varying 400, 500, 600, 700, and 800 ° C respectively along 2 hours. These results are then characterized using XRD. Morphological observation using SEM, and measuring the impedance and conductance using LCR meter to the sample of 800 °C calcination treatment.

RESULT AND DISCUSSION

The powder produced from various calcination temperatures of 400-800 ° C in the pellets formula were white and no color change during the calcination. . Identification the phase formation $Li_4Ti_5O_{12}$ were done using XRD analysis of each sample calcination results, as shown below in Fig 1.

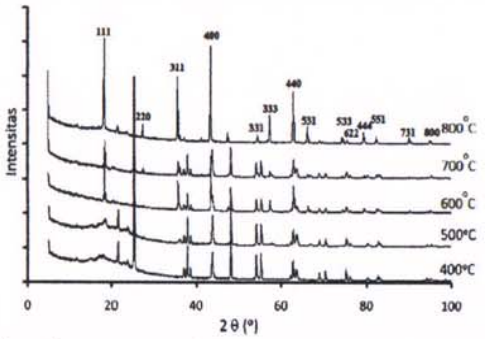

Fig. 1. X-ray diffraction pattern of the sample $Li_4Ti_5O_{12}$ calcination results 400, 500, 600, 700, and 800 ° C respectively for 2 hours.

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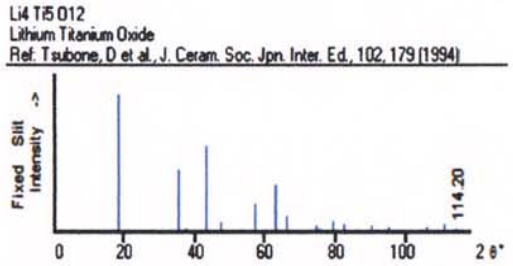

Fig. 2. The diffraction pattern standard $Li_4Ti_5O_{12}$ JCPDS 49-0207.

The diffraction pattern was compared to a standard reference JCPDS 49-0207, observed at point corner of 5-115° , as shown in Fig. 2. The results showed that the sample of 400 °C were ;0: 25. 209diffraction peaks are not $Li_4Ti_5O_{12}$, namely TiO_2 anatase (2θ: 54.862^0; 53.532^0; 47.873^0, and 37.526^0) TiO_2 peaks are still visible on the sample of 400 °C calcination showed that not all react to form TiO_2. These peaks decreased at the temperature rises to 700 °C but however at the temperature of 500-700 °C to form the peaks rutile TiO_2. 2θ= 18,38° looked sharper at While the main peak $Li_4Ti_5O_{12}$ on calcination temperatures rising to 800 °C showed higher crystallinity. The observation of micro structure made using a scanning electron microscope (SEM) with a magnification of 750 times, on samples of calcination are shown in Figure 3. It is seen that the sample results 400 to 700 °C calcination there are irregularities while the particles in the sample of 800 °C calcination seem more homogeneous. This homogeneity in accordance with the observations using X-ray diffraction showed that the results of the 800 °C calcination formed $Li_4Ti_5O_{12}$.

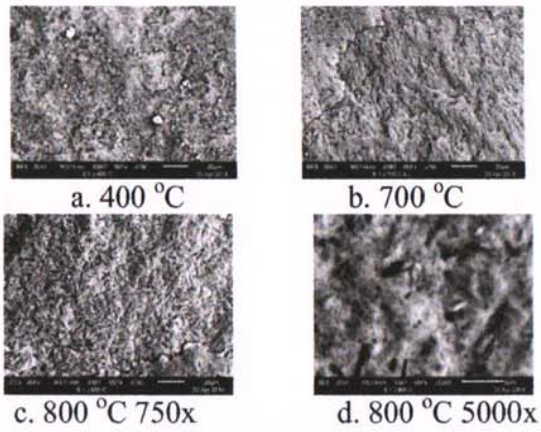

Fig. 3. The structure of the sample micro $Li_4Ti_5O_{12}$ calcination results 400, 700, and 800 ° C.

Impedance measurements performed using electrochemical impedance spectroscopy (EIS). Measurements carried out voltage of 1 V at 42 Hz frequency rentangg - 1M Hz. The measurement results show a pattern semicircle arc (semicircle), as shown in Fig. 4.

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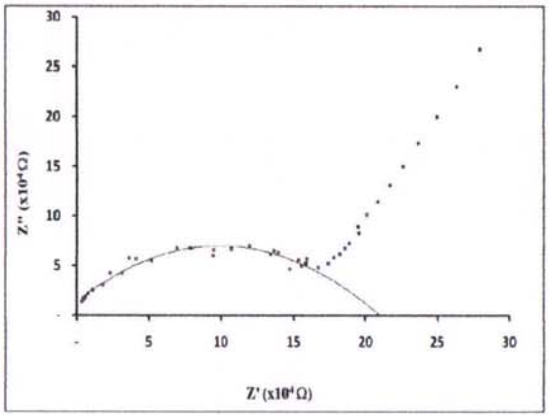

Fig. 4. The impedance spectra of the sample $Li_4Ti_5O_{12}$ 800 °C calcination results measured 2 hours at room temperature.

Electrode materials for Li ion batteries requires electronic conductivity and ionic conductivity of Li^+ are good. Conductivity value were calculated based on the interpretation of the size of the semicircle arc formed on the impedance spectrum as indicated by the value of the impedance R_b (bulk resistance) and R_{gb} (grain boundary resistance). R_b value shows the characteristics of the bulk material that is ohmic, while R_{gb} shows the qualitative characteristics of grain boundary which is capacitive. R_b apparent on higher frequency data, while R_{gb} at low frequencies[1].

The semicircle shown in Fig. 4 is known that the bulk resistance 224,810 ohms and 12,900 ohms resistance grain boundary. The value of bulk resistance indicates the characteristics of the bulk material that is ohmic, while the grain boundary resistance shows the qualitative characteristics of grain boundary which is capacitive.

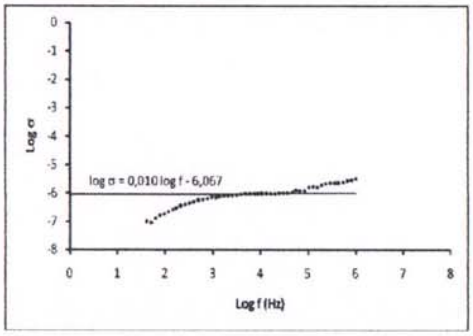

Fig. 5. Conductivity DC $Li_4Ti_5O_{12}$ results calcined sample of 800 °C measured 4 hours at room temperature.

The $Li_4Ti_5O_{12}$ in Figure 5 looks relations frequency and conductivity (sample of 800 °C calcination) measured 2 hours at room temperature. In the frequency region 3K to 30K Hz Hz looks relatively stable the conductivity value, that's why the value of the conductivity of the material expressed $1,5066x10^{-6}$ $S.cm^{-1}.cm^{-1}$ at the frequency range Hz 3K to 30K Hz.

CONCLUSION

$Li_4Ti_5O_{12}$ as the anode material of lithium batteries can be synthesized succesfully with solid state reaction method using anatase titanium dioxide and lithium hydroxide at 800°C calcination temperature for 2 hours. This is shown by X-ray diffraction patterns that were compared with JCPDS 49-0207. Observation of a scanning electron microscope (SEM) showed that the morphology of the material is more homogeneous results 800 °C calcination. DC conductivity value of the material is $1,5066x10^{-6}$ $S.cm^{-1}$ in the frequency range 3K Hz to 30K Hz.

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