NANOGRAIN STUDY OF POLYCRYSTALLINE CERAMICS BASED ON CERATE-ZIRCONATE VIA TWO-STEP SINTERING

RESEARCH MANAGEMENT INSTITUTE (RMI)
UNIVERSITI TEKNOLOGI MARA
40450 SHAH ALAM, SELANGOR
MALAYSIA

BY:

AZLIANA RAMLI
NAFISAH OSMAN
SUHAIDA DILA SAFIAN
IBRAHIM ABU TALIB

DECEMBER 2015
Contents
1. Letter of Report Submission ................................................................. iii
2. Letter of Offer (Research Grant) ............................................................. iv
3. Acknowledgements ........................................................................... v
4. Enhanced Research Title and Objectives ............................................ vi
5. Report ............................................................................................... 1
   5.1 Proposed Executive Summary ......................................................... 1
   5.2 Enhanced Executive Summary ........................................................ 2
   5.3 Introduction .................................................................................. 3
   5.4 Brief Literature Review ................................................................. 4
   5.5 Methodology ............................................................................... 5
   5.6 Results and Discussion ................................................................ 7
   5.7 Conclusion and Recommendation ............................................... 11
   5.8 References/Bibliography ............................................................... 12
6. Research Outcomes ......................................................................... 14
7. Appendix ........................................................................................ 15
1. Letter of Report Submission

Puan Azlana Binti Ramli
Ketua Projek
Fakulti Sains Gunan
Universiti Teknologi MARA (Perlis)
02690 Arau
Perlis

Puan

PENERIMAAN BORANG TAMAT PROJEK PENYELIDIKAN (RAGS)
TAJUK PROJEK: Nanogram Effects And Its Correlation On The Electrical Conductivity Of Polycrystalline Ceramics Based On Create-Zirconate

Dengan segala hormatnya perkara di atas adalah dirujuk.

2. Adalah dimaklumkan, Pusat Pengurusan Penyelidikan (RMC) telah menerima satu (1) salinan asal borang tamat projek bertajuk seperti di atas daripada pihak puam. Pihak RMC mengucapkan setinggi-tinggi tahniah. Sehubungan itu, projek penyelidikan pihak puam telah didaftarkan sebagai TAMAT.

3. Pihak puam perlu menghantar laporan akhir penyelidikan dalam bentuk satu (1) salinan cakera padat (CD) kepada RMC dalam tempoh satu (1) bulan dari dari tarikh surat ini.

4. Pihak RMC akan memutamadkan dan menutup akaun penyelidikan puam dengan kadar segera. Sebarang pertanyaan puam boleh menghubungi Unit Pemantauan Penyelidikan RMC di talian 03-5543 7874 / 03-5544 2753.

Selaras dengan dasar penyelidikan UiTM disamping meningkatkan kesarjanaan akademik, pihak puam adalah diharapkan untuk terus aktif memohon geran dan menjalankan penyelidikan berterusan.

Sekian, terima kasih.

Yang benar,

PROFESOR DR. HADARIAH BINTI BAHRON
Penolong Naib Canselor (Penyelidikan & Inovasi)
5.2 Enhanced Executive Summary

(Abstract of the research) – 1 page only

The properties of ceramics material are strongly depending on their preparation method during synthesizing process and heat treatment. In this study, ceramic perovskite-type oxide based on Ba(Ce,Zr)O₃ was prepared by a sol-gel route and heat treated using a two-step sintering (TSS) method. The first temperature profile was set at T₁=1400°C and the second temperature were varied at T₂= 1150°C, 1200°C, 1250°C, 1300°C and 1350°C, respectively. The sintered pellets were labeled as S1, S2, S3, S4 and S5, accordingly. XRD results showed that all the samples exhibit single-phase of cerate-zirconate ceramics except for S4. The crystalline peaks for single-phase samples are matched to the standard Ba(Ce,Zr)O₃ with JPCDs card no. 01-089-2485. On the other hand, the presence of secondary phases of CeO₂, (Ce,Zr)O₂ and BaCO₃ along with main phase of Ba(Ce,Zr)O₃ were detected in S4. SEM analysis revealed that the samples formed clear and compact grains with submicron sizes. As the second sintering temperature increased, the size of grain decreased from 336.40 nm to 162.00 nm. Therefore, the used of different sintering profile in TSS method was found to give significant effect on the phase and morphology of Ba(Ce,Zr)O₃ solid solution.
5.3 Introduction

Increasing in the energy demand lead the world to focus on an alternative energy conversion technologies. Solid oxide fuel cells (SOFCs) offer more efficiency in conversion of chemical energy in fuels into electricity. There is worldwide interest in reducing the operating temperature of SOFCs to 500-700°C for long-term stability and lower cost. Proton conducting solid oxide fuel cells (PCFCs) are under intense study due to their lower operating temperature < 800°C compared to the conventional oxygen ion conducting solid oxide fuel cells [1]. Cerate-zirconate ceramics with perovskite structure of ABO₃ is one of the promising candidates as solid electrolyte for PCFCs applications due to its low activation energy for proton conduction. A better understanding of proton conduction in this material requires a systematic study on the role of synthesizing process and heat treatment in controlling the material’s microstructure.

Researchers have found one of the cost-effective sintering methods which can be used to improve the ionic conductivity of the materials is a two-step sintering technique (TSS). This technique was developed by Chen and Wang [2] for sample Y₂O₃ to obtain full dense and nano-grain ceramics. The main goal of this novel techniques is to control the microstructural features of ceramics like grain size, density and shorten the heating time to hinder the grain growth [3]. However, to obtain high density pellet at relatively low temperature is very challenging process particularly for the electrolyte ceramics based on perovskite type-oxide. TSS method is successfully done for the sample of Y₂O₃, BaTiO₃, ZnO, Y-TZP, MgSi₂ and Al₂O₃ [2,3,4,5]. To the best of our knowledge, the use of TSS method in Ba(Ce,Zr)O₃ ceramic pellet are rarely documented. Therefore, this work aims to study the effect of sintering profile on the phase formation, density and grain size of Ba(Ce,Zr)O₃ using TSS technique.