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on Materials Science and Technology

ICMST 2010

Materials Science & Technology

**Research and Development of Advanced Materials
for a Better Future**

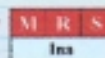
Editors

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Center for Technology of Nuclear Industry Materials
Indonesian National Nuclear Energy Agency

Materials Research Society
INDONESIA



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Serpong, Indonesia

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PREFACE

Materials Science is an interdisciplinary branch of Science and Technology. It is a very wide spectrum of science covering subjects such as soft matters, eg. polymers, gels, bio materials, as well as hard matters, eg. semiconductors, superconductor, metallic material etc.. The applications of materials science range from energy, environment, health, transportation, military, as well as the multitude of manufacturing industries. This means that our daily life is not separable from the use of advanced materials coming out from research in this field.

The International Conference on Materials Science and Technology (ICMST) 2010 was held in Serpong, Indonesia, on 20-22th of October 2010. The event has provided not only regional but also international forum to discuss the latest development, and to share knowledge in interdisciplinary materials research along with its applications. Furthermore, the ICMST 2010, has also provided a platform for international collaborations among national and international participants from more than 10 countries. It was a good opportunity for the Indonesian scientists to promote their research in the international forum

The editors received about 101 articles that were distributed into several publications, namely 34 articles in *Indonesian Journal of Materials Science*, 8 articles in *Atom Indonesia Journal*, and 49 articles in *this proceeding book*. We are pleased to inform all authors that the editors decided to publish these articles into a book style, in the hope that it can optimize its uses for learning the latest development in materials sciences and technologies. The book is divided into four sections i.e. (i) invited papers, (ii) hard matter, (iii) soft matter, and (iv) material energy & devices. There were five invited papers mainly on the research and application of solid state ionics. All these articles have been presented by the invited lecturers in the 'Asian Workshop on Solid State Ionics' that was held one day prior to the ICMST2010. The hard matter part was dedicated for contributed papers that cover magnetic and metallic materials, composite, ceramic, catalyst converter and the application of various neutron scattering methods. The soft matter section covers various topics from cellulosic material, natural rubber, nano-sphere, and nano-emulsification for drug delivery system. The 4th part contains materials useful for energy and devices, starting from different storage materials for fuel-cell, rechargeable batteries, sensors, ion transport modeling, and soft phonon in copper selenide.

We are grateful to all the plenary and invited speakers and also to all the authors of papers for participating in this conference. Our gratitude also goes to all the funding agencies and sponsors for their generous support. Finally, we are also thankful to those who served in International & Local Advisory Committee and Technical Committee for their efforts in making the event a successful one.

24 February 2011

Editors

Message from President of Asian Society of Solid State Ionics

It is my pleasure and honor to greet you on behalf of the Materials Research Society of Singapore (MRS-S), Asian Society for Solid State Ionics (ASSSI) and the International Union of Materials Research Societies (IUMRS). I am delighted that Dr. Evvy Kartini took lead in organizing the Workshop on Solid State Ionics and The International Conference on Materials Science and Technology (ICMST 2010). I am particularly pleased to learn that a large number of overseas delegates are participating in this Workshop and upcoming Conference.

Both Materials Science and Solid State Ionics are multidisciplinary in nature drawing researchers from Chemistry, Physics, Chemical-, Electrical- and Mechanical- Engineering disciplines. I hope all participants take advantage of this unique situation and interact among themselves to contribute to the advances in this new field of research.

It is often difficult for members from Asia, particularly students, to travel to overseas for participating in conferences of interest. In this context, efforts put in by ICMST 2010 organizers will go a long way in promoting scientific research within the country and also establishing international networks. I congratulate the organizers for bringing such a high quality speakers to the door steps of Indonesia and enabling large number of students and non-specialists to participate and get benefited. I am particularly happy that part of MRS-S sponsorship has gone towards supporting students' participation. I sincerely hope that the efforts put in by the organizers and the contacts developed through this will lead to international collaborations and the organization of much larger conferences.

I wish you all a fruitful Workshop and look forward for the pleasure of meeting you at ICMST 2010.

Professor B.V.R. Chowdari

*National University of Singapore (NUS), Singapore
President of Asian Society of Solid State Ionics (ASSSI)
President of Materials Research Society, Singapore*

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$Fe_{1-x}Ni_xO_{3±5}$ CATALYST FOR CONVERTING CO_2/H_2 TO ALCOHOL COMPOUNDS

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ABSTRACT

$Fe_{1-x}Ni_xO_{3±5}$ catalyst was prepared using citric acid method from its precursor of nitrates. The preparation of $Fe_{1-x}Ni_xO_{3±5}$ is carried out for a various ratios of Ni/Fe (b/b). To examine its activity, the catalyst was used in a CO_2/H_2 conversion experiment at different temperatures (100 to 400 °C). The experimental results indicated the dependency of CO_2/H_2 conversion on temperature and the highest value for alcohol formation (291.24 ppm) was achieved at 400 °C under $Ar/CO_2/H_2$ (34/4/12, 50 mL min⁻¹) as a feed. X-ray diffraction examination proved that the crystalline phase is $NiFe_2O_4$ spinel structure, which is also the major phase and appear stronger as the Ni content in the structure increases. FTIR analysis indicated that both the Lewis acid sites which is located at wave numbers of ± 1490 and ± 1450 cm⁻¹ respectively and Bronsted-Lowry acid sites which is located at wave numbers of ± 1645 and ± 1622 cm⁻¹ respectively, were involved in the catalytic conversion of CO_2/H_2 . As Ni content increased, both acid sites' adsorption shifted to a relative higher wavelength. Furthermore, SEM analysis implied that particles distribution of $NiFe_2O_4$ spinel structure relatively spreads out as Ni content increased. The particle's size of the catalyst is close to ≤ 50 μm.

Keywords: CO_2/H_2 Conversion, alcohol formation, Lewis and Bronsted-Lowry acid

INTRODUCTION

Presently, air emission regulation has been largely enforced in most countries throughout the world, especially with regards to activities which tend to emit pollutant gases such as NO_x , SO_x , and CO_x . Moreover, these pollutants are strongly believed to be one of the source which causes global warming. Therefore, handling out these pollutants as a potential energy resource and simultaneously protecting the environment has genuinely become an interesting and challenging task for scientists (Bell et al., 2004, AtlanticBiomass@aol.com, www.acrion.com, and www.methanol.org).

It has been common knowledge that CO_2 emission is a major contributor to global warming effect, therefore since CO_2 gas is used as a raw material in the chemicals manufacture industry, it is now becoming very urgent to find means to solve this problem. Many efforts have been attempted to mitigate CO_2 into light olefin or liquid hydrocarbon (Indala, 2004 ; and Jun et al., 2006). This mitigation is also an alternative way to overcome the fossil fuel dependency to satisfy the need for energy (Williams et al., 2006). The CO_2 conversion method which is widely developed is the catalytic hydrogenation method because, in this method, CO_2 gas can be converted to methanol (Cabrera et al., 1998; and Joo, 1999), Liquid Petroleum Gas, ethylene and propylene (Fujiwara et al., 1995). In addition, this

method can also produce CO gas which is used in Fischer-Tropsch process (Srinivas *et al.*, 2009).

In general, results of CO₂ catalytic hydrogenation reaction are predominantly affected by the type of catalyst used (Lapidus *et al.*, 2006; and Srinivas *et al.*, 2009). Catalyst that can be used in this reaction must possess both the activity and the stability against the *Reverse Water-Gas Shift*, RWGS and Fischer-Tropsch reaction (Riedel *et al.*, 1999; Joo and Jung, 2003; Luo *et al.*, 2007; and Joo, 1999). Furthermore, reaction conditions such as reaction temperature and pressure will also affect a product's diversity. For example, if a reaction proceeds at low temperatures (220 – 250°C and 25 bar), then the finished product tends to be both wax and diesel fuel. Otherwise, the product will most likely be olefin (Dry, 2002).

Based on these informations as mentioned above, this research is focused on the preparation of Fe_{1-x}Ni_xO_{3±δ} Catalyst and the study of its catalytic activity performance to convert CO₂/H₂ in the temperature range of (100 – 400 °C).

EXPERIMENTAL METHOD

Catalysts Characterization.

X-rays power diffraction pattern of Fe_{1-x}Ni_xO_{3±δ} was recorded from 2θ = 10 to 90° on a Philips diffractometer Model PW 1710 using Cu K_α radiation in step of 0.02° per second. Electron micrographs were obtained with Scanning Electron Microscopy in order to investigate its morphology. To determine the Lewis and Brønsted – Lowry sites, catalysts were previously contacted over night to pyridin saturated vapor at ambient temperature, then analyzed by using a FT-IR spectrophotometer. Surface area and total pore volume is measured by S_{BET} quantachrome instrument.

Catalysts Preparation.

Solid Fe_{1-x}Ni_xO_{3±δ} was prepared by dissolving Fe(NO₃)₃ · 9 H₂O, and Ni(NO₃)₂ · 6 H₂O in citric acid solution. Then, these solutions (by Fe/Ni ratio variation, x = 0.1 to 0.5) was mixed, heated and evaporated at 80 °C until a sol – gel solution formed. Furthermore, this precursor was calcinated at 600 °C for six hours (with an increment rate of 2 °C min⁻¹). The solid sample thus formed was crushed into fine grains before performing the characterization.

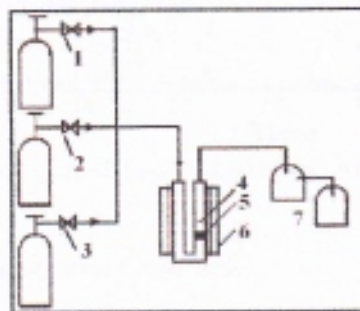


Figure 1: A schematic diagram of the experimental set up: Carbon dioxides (1), hydrogen (2), and Argon (3) were supplied from the gas cylinders equipped by micrometric valves. The mixing reagents were passed by valve to the quartz reactor (4) and catalyst is located on (5), furnace with temperature program (6). The gas products was collected on trapper containing aquadest (7)

Catalytic Measurements.

The CO_2/H_2 reaction was carried out in a vertical down flow U – tube natural quartz reactor (with inner diameter 12 mm) at atmospheric pressure. In a typical experiment, ca. 50 mg of sieved catalyst was placed in a U-tube squeezed between two quartz wool. The reactor was placed vertically inside a programmable cylindrical furnace, which was heated electrically. Before the reaction, the catalyst was pretreated in flowing process gas (Ar/H_2 , $2 L h^{-1}$) at $500\text{ }^\circ C$ for 30 minutes. The experiment was performed at $100 - 400\text{ }^\circ C$ using a designated amount of argon, carbon dioxides and hydrogen. The product stream coming from the reactor was passed through collector tube with water as a trapper and immersed into ice cubes. Then, the product was analyzed with liquid chromatography in order to separate the product. A schematic diagram of the experiment is shown in Fig. 1 below.

RESULTS AND DISCUSSIONS

Catalyst Characterization.

In Figure 2, it is shown that $Fe_{0.9}Ni_{0.1}O_{3\pm\delta}$ catalyst consists primarily of $NiFe_2O_4$ crystalline phase which has representative peaks appearing at 2θ : $35,46^\circ$ (100%), $62,76^\circ$ (34,72%), $30,16^\circ$ (29,17%), $57,4^\circ$ (25,69%). These peaks were matched to JCPDS file of 23-1119. However, other catalysts of $Fe_{1-x}Ni_xO_{3\pm\delta}$ (where $x=0,2 - 0,5$), prepared by the same method, have crystalline phases which corresponds more to the JCPDS file number 10-0325. Furthermore, formation of $NiFe_2O_4$ (JCPDS 10-0325) crystalline phase is much more pronounced as Ni content increased in the $Fe_{1-x}Ni_xO_{3\pm\delta}$ (where $x=0,1 - 0,5$) structure. It has been shown that peaks at $2\theta = 18,6 ; 37,8 ; 43,3 ; 57,54 ; 63,1 ;$ and $75,8^\circ$ emerged stronger by increasing the Ni content in the $Fe_{1-x}Ni_xO_{3\pm\delta}$ structure.

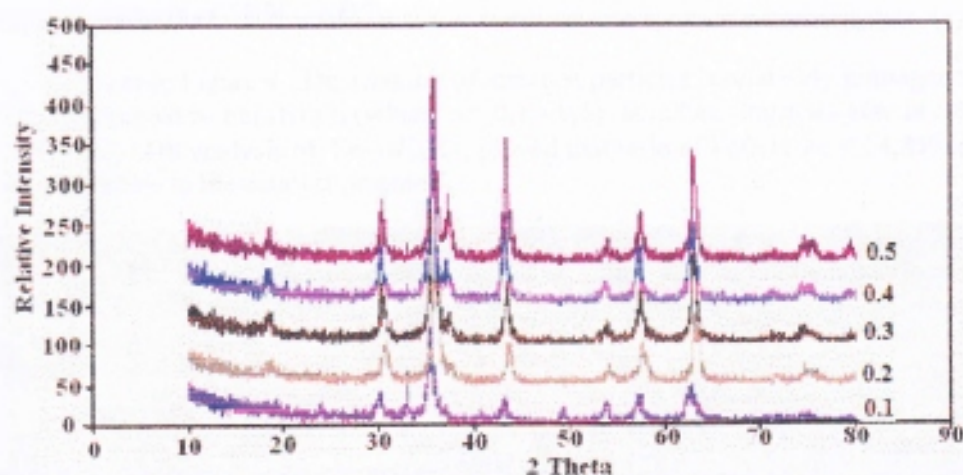


Figure 2: Diffractogram of $Fe_{1-x}Ni_xO_{3\pm\delta}$ (where $x=0,1 - 0,5$) catalysts

FTIR Analysis of Bronsted-Lowry and Lewis acid sites.

Both Brønsted – Lowry and Lewis acid sites of $Fe_{1-x}Ni_xO_{3\pm\delta}$ catalysts (where $x = 0,1 - 0,5$) were investigated by FTIR instrument. Infra red spectrum showed that $Fe_{1-x}Ni_xO_{3\pm\delta}$ catalysts, in general, has Brønsted – Lowry acid sites at wavenumber of ± 1645 and $\pm 1622\text{ }cm^{-1}$. Then, Lewis acid sites appeared at wavenumber of ± 1490 and $\pm 1450\text{ }cm^{-1}$. However increasing Ni content in the $Ni_xFe_{1-x}O_{3\pm\delta}$ structure, would in fact cause a shift to a greater wavenumber as shown in Figure 3.

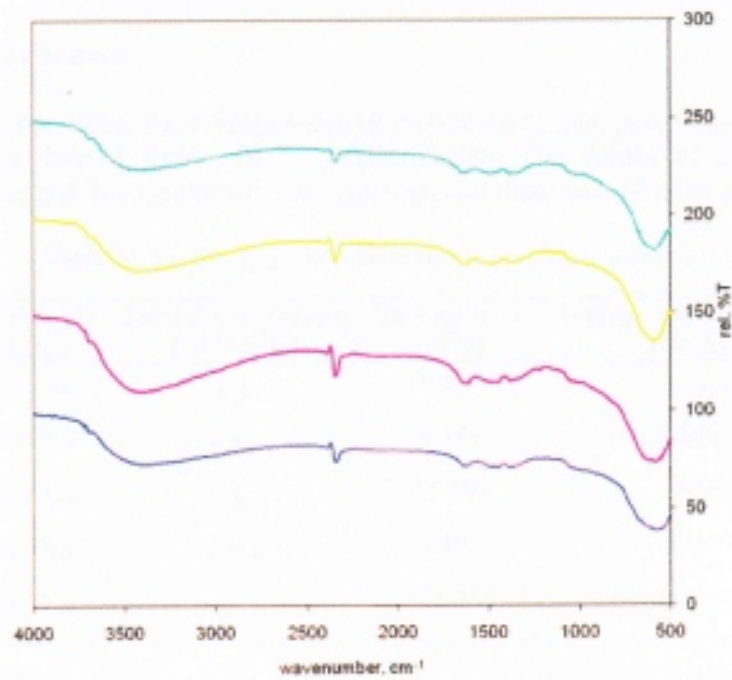


Figure 3: Infra red spectra of $Fe_{1-x}Ni_xO_{3.5}$ catalyst which had been treated by Pyridine (where $x = 0.2$ to 0.5).

Micrograph analysis of SEM – EDS .

As shown in Figure 4, Distribution of catalyst particles is relatively homogeneous as Ni content increased in $Fe_{1-x}Ni_xO_4$ (where $x = 0,1 - 0,5$) structure. Particles size is closed to $\leq 50 \mu m$. Then, EDS analysis of $Fe_{0,5}Ni_{0,5}O_4$ proved that ratio of Fe/Ni (Fe = 14,85% and Ni 14,42%) is suitable to the catalyst prepared .

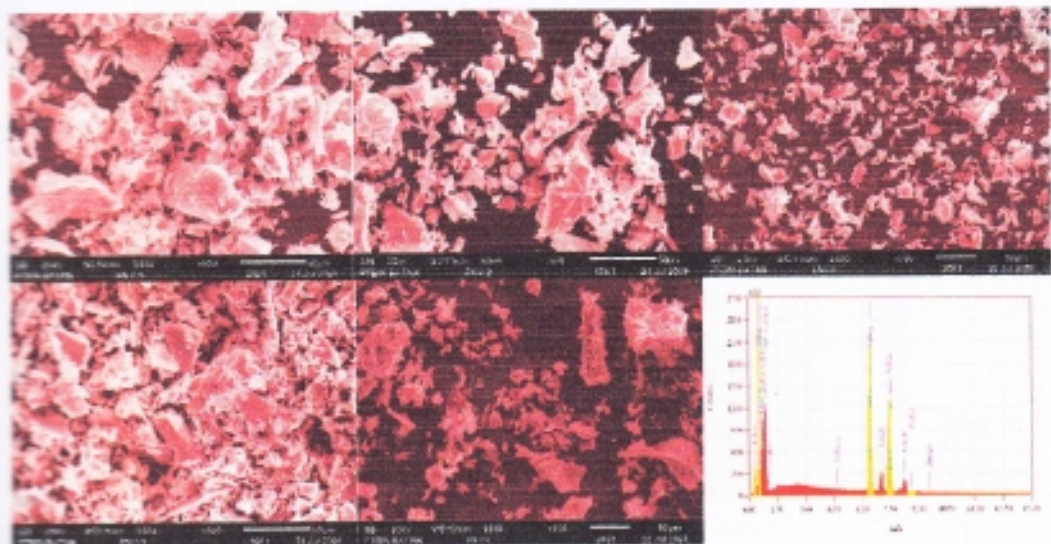


Figure 4: Micrograph of various $Fe_{1-x}Ni_xO_4$ catalyst (where $x = 0,1 - 0,5$; from left to right).

S_{BET} Analysis of Catalysts.

Table 1 represents the average value of surface area , total pore volume and average diameter pores derived from BET measurements. The values of surface area for $Fe_{0.5}Ni_{0.5}O_{3+\delta}$ catalyst is considerably very much greater than those of other catalysts.

Table 1: Results of S_{BET} Determinations of $Fe_{1-x}Ni_xO_4$ catalysts

Type of Sample $Fe_{1-x}Ni_xO_{3+\delta}$	Total Pores Volume ($10^{-3} \text{ cm}^3/\text{g}$)	Surface Area (m^2/g)	Average Pore Diameter (10^2 \AA)
$Fe_{0.9}Ni_{0.1}O_{3+\delta}$	4.1	0.507	3.235
$Fe_{0.8}Ni_{0.2}O_{3+\delta}$	2.81	0.144	7.806
$Fe_{0.7}Ni_{0.3}O_{3+\delta}$	115.3	16.259	2.837
$Fe_{0.6}Ni_{0.4}O_{3+\delta}$	4.519	1.195	0.944
$Fe_{0.5}Ni_{0.5}O_{3+\delta}$	4.1	126.365	0.669

Values of total pores volume and average diameter of pores of a $Fe_{1-x}Ni_xO_4$ catalyst apparently do not follow a regular pattern, nevertheless the Ni content in $Fe_{1-x}Ni_xO_4$ catalysts is still very much influential over the surface area, pores volume and diameter of pores, respectively. The higher effect appears at higher values of the nominal nickel content.

Catalyst activity.

Figure 5(a) shows that catalysts such as $Fe_{0.8}Ni_{0.2}O_4$, $Fe_{0.7}Ni_{0.3}O_4$, and $Fe_{0.6}Ni_{0.4}O_4$ are actively engaged in a CO_2/H_2 reaction to produce alcohol compounds. The most active catalyst is actually $Fe_{0.8}Ni_{0.2}O_4$, especially at 400°C . For other catalysts which are not active even though they have a greater surface area, it could be deduced that CO_2/H_2 reaction apparently did not require much time to react CO_2 with H_2 while catalyst was adsorbing these gases. Another possibility is attributed to the competitive reaction between CO_2 and H_2 to produce alcohol, water and others. Unfortunately, other products such as acid, ketones, aldehyde and carbon monoxide were not analyzed.

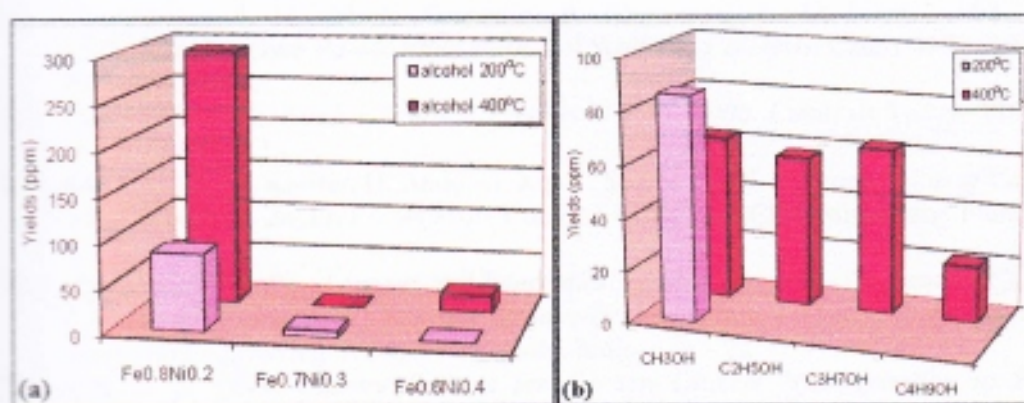


Figure 5: (a). Alcohol formation (yields in ppm) by some catalysts in different reaction temperature. (b). Alcohol products' distribution by catalytic reaction of CO_2/H_2 on $Fe_{0.8}Ni_{0.2}O_{3+\delta}$ catalyst.

As shown in Figure 5(b), $\text{Fe}_{0.8}\text{Ni}_{0.2}\text{O}_4$ catalyst is the most active at 200 °C and 400 °C compared to another catalyst. Product diversification occurs at 400°C in which methanol, ethanol, propanol, and butanol were turned out. It can be implied that catalyst was delaying or impeding the intermediate products, so any other reactions could possibly have taken place. It was also possible to form C_2^+ , C_3^+ and C_4^+ cations in order to produce those alcohol compounds.

The general finding of this research is that among the various $\text{Fe}_{1-x}\text{Ni}_x\text{O}_4$ catalysts investigated in this study, the $\text{Fe}_{0.8}\text{Ni}_{0.2}\text{O}_4$ catalyst system has the most promising potential to induce the CO_2/H_2 reaction under mild temperature conditions.

CONCLUSION

Based on this experiment, it could be concluded that the temperature reaction affect the formation of alcohol compound from CO_2/H_2 reaction. In general, three composition catalysts such as $\text{Fe}_{0.8}\text{Ni}_{0.2}\text{O}_4$, $\text{Fe}_{0.7}\text{Ni}_{0.3}\text{O}_4$, and $\text{Fe}_{0.6}\text{Ni}_{0.4}\text{O}_4$ are actively involved in this CO_2/H_2 reaction. In fact, $\text{Fe}_{0.8}\text{Ni}_{0.2}\text{O}_4$ catalyst is a promising system for performing the CO_2/H_2 reaction under mild temperature conditions. Furthermore, Oxygene bulk plays a role in the CO_2/H_2 reaction.

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