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Oxygen Reduction and Hydrogen Production at the Liquid/Liquid interfaces

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Abstract-Molecular electrocatalysis for oxygen reduction at a polarized water/1,2-dichloroethane (DCE) interface was studied, involving aqueous protons, ferrocene (Fc) in DCE and cobalt porphyrin catalysts like cobalt porphine (CoP) cobalt 2,8,13,17-tetraethyl-3,7,12,18-tetramethyl-5-*p*-aminophenylporphyrin (CoAP) at the interface. The reaction is electrocatalytic as its rate depends on the applied Galvani potential difference between the two phases. We also report herein hydrogen evolution by direct proton reduction with DMFc (Decamethylferrocene) at a soft interface between water and DCE.

The soft interface between two immiscible electrolyte solutions (ITIES) is formed between two liquid solvents of a low mutual miscibility, such as water and 1,2-dichloroethane (DCE), each containing an electrolyte. Electrochemical polarization of ITIES allows studies of electron transfer and ion transfer reactions, as well as the adsorption phenomena. With this advantage, an ITIES has been considered to be a suitable model for investigation of heterogeneous reactions occurring in biological systems, which are in most cases ion-coupled electron transfer reactions such as the proton-coupled oxygen (O_2) reduction. Recently, we have studied Proton-Coupled Oxygen Reduction at liquid-liquid interfaces catalyzed by cobalt porphine [1] and cobalt 2,8,13,17-tetraethyl-3,7,12,18-tetramethyl-5-*p*-aminophenylporphyrin (CoAP). The reaction proceeds as a proton coupled electron transfer process (PCET), with protons supplied by the aqueous phase and electrons provided by Fc (ferrocene) in DCE as shown in Figure 1.

We also present a heterogeneous hydrogen evolution reaction at a soft interface, formed between an aqueous acidic solution and an immiscible organic solvent, 1,2-dichloroethane (DCE), containing DMFc as an electron donor [2]. The reaction proceeds by assisted proton transfer by DMFc across the water–DCE interface with subsequent proton reduction in DCE. The interface

essentially acts a proton pump, allowing hydrogen evolution by directly using the aqueous proton.

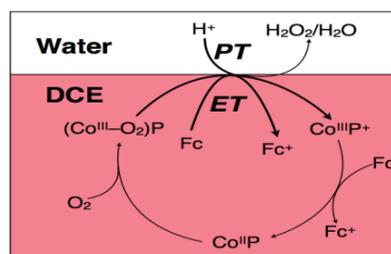


Figure 1: Interfacial PCET mechanism

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Hydrothermal preparation and electrochemical properties of Sm^{3+} and Gd^{3+} , codoped ceria-based electrolytes for intermediate temperature-solid oxide fuel cells

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Abstract- The structure, ionic and electronic conductivities of $\text{Ce}_{0.8}\text{Sm}_{0.2-x}\text{M}_x\text{O}_{2-\delta}$ (for M: Gd, and La, $x = 0-0.1$) solid solutions, prepared for the first time hydrothermally, are investigated. The uniformly small particle size (23-64 nm) of the materials allows sintering of the samples into highly dense ceramic pellets at 1300-1400°C, significantly lower temperature, compared to that at 1600-1650°C required for ceria solid electrolytes prepared by solid state techniques. The maximum conductivity, $\sigma_{700^\circ\text{C}} \sim 6.50 \times 10^{-2} \text{ Scm}^{-1}$, $E_a = 0.59 \text{ eV}$, is found at $x = 0.1$ for Gd-codoping. The electrolytic domain boundary (EDB) of $\text{Ce}_{0.8}\text{Sm}_{0.1}\text{La}_{0.1}\text{O}_{2-\delta}$ has been found to be lower than that of singly doped samples.

These results suggest that co-doping can further improve the electrical performance of ceria-based electrolytes.

Fuel cells are electrochemical devices that directly convert the chemical energy of a fuel into electrical energy in a highly clean, cheap and efficient way [1]. Electrolytes used for fuel cells are usually the main components determining the performance of the cell. A typical solid oxide fuel cell electrolyte, 8mol% yttria-stabilized zirconia (YSZ), having thermal and mechanical strength both toward anode reduction and cathode oxidation requires to operate at high temperatures (800–1000 °C) to provide high level of ionic conductivity. This limits the range of materials used for interconnection, electrodes and sealing due to the corrosion of metallic components [2]. Some singly doped-electrolytes, such as $\text{Ce}_{1-x}\text{Gd}_x\text{O}_{2-\delta}$ (GDC), $\text{Ce}_{1-x}\text{Sm}_x\text{O}_{2-\delta}$ (SDC), $\text{Ce}_{1-x}\text{Y}_x\text{O}_{2-\delta}$ (YDC), etc., show high oxide ion conductivity at intermediate temperatures (500–700°C) [3–5]. Substitution of the Ce^{4+} cations by a lower valent metal ion (e.g., M^{3+}) in the lattice results in the oxygen vacancy formation and increases the ionic conductivity.

In this research, with the aim to develop new ceria-based electrolyte materials with improved electrochemical properties, Sm^{3+} and Gd^{3+} co-doped ceria materials were prepared for the first time hydrothermally. Similar to the previously reported systems [6–7], the electrical conductivity of $\text{Ce}_{0.8}\text{Sm}_{0.2-x}\text{Gd}_x\text{O}_{2-\delta}$ increases systematically with increasing gadolinium substitution and reaches a maximum for the composition $\text{Ce}_{0.8}\text{Sm}_{0.1}\text{Gd}_{0.1}\text{O}_{2-\delta}$, ($\sigma_{700^\circ\text{C}} \sim 6.50 \times 10^{-2} \text{ Scm}^{-1}$) Fig. 1)

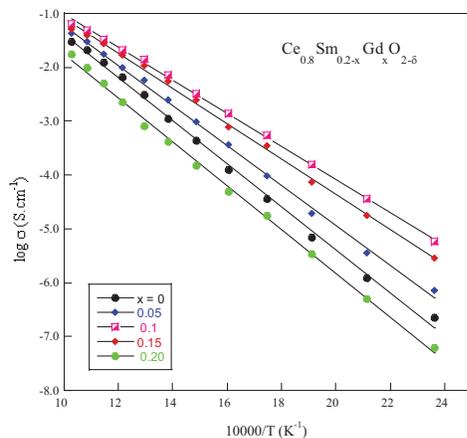


Fig.1 Arrhenius plots of the ionic conductivity of $\text{Ce}_{0.8}\text{Gd}_{0.2-x}\text{Sm}_x\text{O}_{2-\delta}$ solid solutions

The ceria-based electrolytes easily develop n-type electronic conduction when exposed to the reducing atmosphere of the fuel cell anode which decreases the fuel cell efficiency. It is therefore important to make efforts towards the reduction of electronic conductivity. The dependence of total conductivities of $\text{Ce}_{0.8}\text{Sm}_{0.2-x}\text{Gd}_x\text{O}_{2-\delta}$ as a function of oxygen partial pressure has been shown in Fig. 2. As can be seen, the total electrical conductivity (σ_t) is predominantly ionic and remains constant at moderate P_{O_2} , whereas at low P_{O_2} , the total electrical conductivity increases as P_{O_2} decreases and is predominantly electronic..

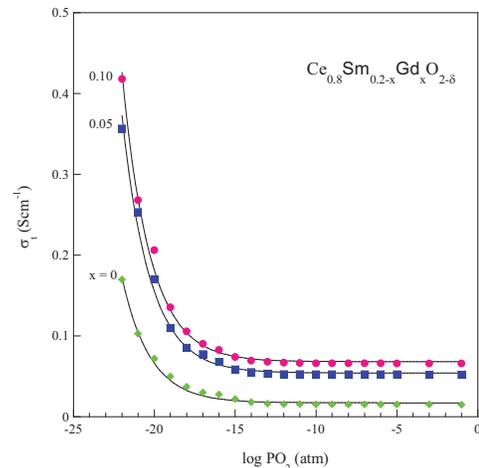


Fig.2 Oxygen partial pressure dependence of the total conductivity of $\text{Ce}_{0.8}\text{Gd}_{0.2-x}\text{Sm}_x\text{O}_{2-\delta}$ solid solutions at 973 K. The data are fitted with $\sigma_t = \sigma_i + kP_{\text{O}_2}^{-1/4}$

From these results we can conclude that co-doping with Sm^{3+} and Gd^{3+} can lead to an improvement of the stability of ceria-based electrolytes at intermediate temperatures.

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Molecular Hydrogen Storage Systems: A First-Principles Study

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Abstract—Here, we present systems that are able to store hydrogen in molecular form. We show that polyolithiated carbon and oxygen molecules store huge (~40 wt % hydrogen) and significant amounts (~8 wt %), in the gas and condensed phases, respectively. The hydrogen binding energies are between 0.1 and 0.2 eV/H₂. As an alternative storage system, we consider the boron sheets that have recently been proposed as novel structures. Dispersing alkali metal (AM = Li, Na, and K) atoms onto the boron sheet results in good hydrogen binding energies (0.2 eV/H₂) and improved storage capacities (10 wt % hydrogen).

A sustainable provision of energy is one of the greatest challenges to mankind. Energy generated from sustainable sources has to be transported and stored in an efficient and ecologically friendly way. Hydrogen is an important energy carrier in current sustainable energy scenarios. Such scenarios are only realistic, however, if hydrogen can be stored in a light, compact, fast and reliable way, and under moderate conditions. Here, hydrogen storage properties of a number of promising molecular systems and nanomaterials are studied from first-principles [1].

In the first part, polyolithiated carbon and oxygen molecular structures are considered for hydrogen storage applications. Using first-principles calculations we predict the interaction of hydrogen molecules with such materials. Within these compounds it is found that the Li atoms connected to a central C or O atom bear partial positive charges. Hydrogen molecules are then clustered around these Li atoms via electrostatic interactions (Figure 1). According to our calculations such molecules can attach hydrogen up to ~40 wt % with average hydrogen binding energies between 0.1 and 0.2 eV/H₂. To prevent clustering of polyolithiated molecules, we attach them to (doped)graphitic templates. The immobilized molecules have a similar interaction with hydrogen molecules as free molecules. Naturally, hydrogen weight percentages are reduced to 5-8 wt % due to the additional weight of the graphitic templates [2].

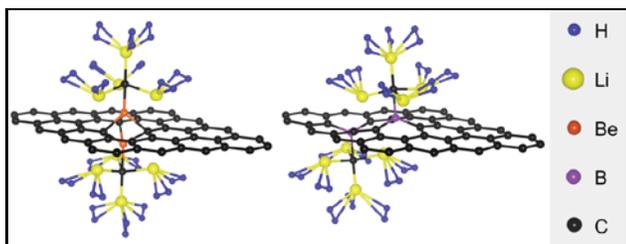


Figure 1. Perspective views of polyolithiated carbon (CLi₄) molecules immobilized on the Be(B)-doped graphene. Hydrogen molecules, shown as blue spheres, gather around the Li atoms.

In the second part, we discuss the hydrogen storage properties of novel boron sheets [3]. The binding of molecular hydrogen to the naked systems is weak. We find that dispersion of alkali metal (AM = Li, Na, and K) atoms onto the boron sheet markedly increases hydrogen binding energies and storage capacities. The unique structure of the boron sheet presents a template for creating a stable lattice

of strongly bonded metal atoms with a large nearest neighbor distance. The strong interaction between the boron sheet and the AM atoms results in a partial transfer of the AM valence electrons to the boron sheet. In particular, Li is found to be a very promising doping element for hydrogen storage purposes (Figure 2). Electrostatic interactions between the well exposed Li atoms and the hydrogen molecules leads to an average binding energy of 0.2 eV/H₂, and up to a maximum of 10 wt % hydrogen [3].

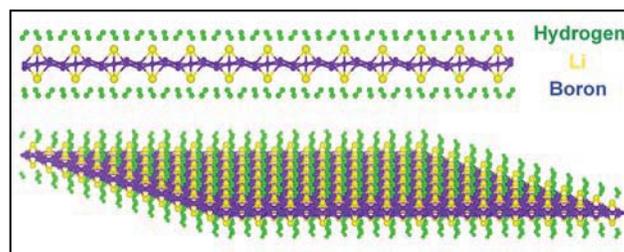


Figure 2. Side and perspective views of the Boron-Li system in its fully hydrogenated state. Up to three hydrogen molecules surround each Li metal on the boron surface.

In conclusion, we show that polyolithiated molecules [2] or alkali metal decorated boron sheets [3] can be used as versatile building blocks for hydrogen storage materials.

This work is part of the research programs of "Advanced Chemical Technologies for Sustainability (ACTS)" and the "Stichting voor Fundamenteel Onderzoek der Materie (FOM)". The use of supercomputer facilities was sponsored by the "Stichting Nationale Computerfaciliteiten (NCF)". These institutions are financially supported by "Nederlandse Organisatie voor Wetenschappelijk Onderzoek (NWO)".

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Engineered Nanolaminates for Energy Release by Oxygen Exchange

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Abstract— The oxygen exchange mechanism was investigated in CuO-metallic multilayer systems with the intent of engineering reaction initiation mechanisms. Thin film nanolaminates were produced from the CuO-Cr, CuO-Zr, CuO-Al, CuO-Ti and CuO-Al_xTi_y (x = 25, 50, 55, 60, 65 and 75) systems. All multilayers were deposited via magnetron sputtering on glass, silicon and sapphire substrates. In most cases, films were 19 layers in total with 100 nm thick metallic layers, however some laminates were fabricated a lower interface density to demonstrate the effect on reactions. The activation energy required for the ignition was provided by fast heating and applied voltage under various atmospheres, *i.e.*, vacuum, air, and Ar. The study revealed that CuO-Cr and CuO-Al multilayer samples have high stability, whereas CuO-Zr and CuO-Ti samples are highly reactive and explosive even inside the sputtering chamber. To obtain control on the reaction, samples based on Al-Ti binary phase diagram were produced. It was shown that multilayer films with 60:40 at.% Al:Ti metallic layers can be activated under controlled conditions and geometry. Ultimately, we use the combination of results to demonstrate the potential for new energetic materials whose reactivity can be predicted and rationally engineered by understanding basic thermodynamics, crystal chemistry, and transport.

The description of thermite refers to an exothermic reaction between an oxidizer and a fuel, which finally, forms oxide of the fuel and reduced state of the oxidizer [1]. Reduction of the oxidizer yields a large heat release, which is generally sufficient to melt the products during the reaction. Thus, thermites have been utilized in joining applications for nearly 100 years, especially for railroad line joining [2]. Today, thermite reactions include a wide range of material couples such as reactions between metal/alloy and metal/non-metal oxide. Once the reaction is ignited, it proceeds as an oxidation-reduction reaction between the reactants by oxygen exchange mechanism and releases a large quantity of heat to the surrounding, and may be expressed as follows;



where M is a metal or alloy and A is either a metal or a non-metal, MO and AO are their corresponding oxides, and ΔH is the heat generated by the reaction [3].

Today nano-engineered thermites are in a class of their own, due to the ability to tune their performance and sensitivity, which separates them from conventional propellants and explosives [4,5]. These structures are of interest for commercial applications including materials joining and smart fusing, and military applications requiring concentrated chemical energy storage. Simply the reaction control makes those nanoscale thermite materials good candidates for micro-devices applications[3] such as detonators, actuators and power generators.

Studies on the utilization of thin film synthesis techniques with flexible sample design showed that a variety of materials and sample geometries are possible with thin film approach instead of using conventional powder systems [6-8]. CuO-Al system is one of the most attractive nano-energetic material couple among the others as multilayers[6] and core-shell structures [7,8].

This study aims to understand the fundamentals of reactive oxygen exchange mechanism between metal and metal oxide layers in the form of thin film multilayers. By the investigation of the thermodynamics and kinetics of the reaction, the study expects to reveal a correlation between the

size, microstructure, and controlled energy release for specific material-systems. For this purpose, multilayers of metal-metal oxide thin films were produced. Two different methods were employed to ignite the exchange mechanism between the adjacent layers *i.e.* thermal annealing and voltage applying.

We used CuO as an oxidizer for all samples. The study focused on two different kinds of metal groups as a fuel. In the first group we produced multilayers of CuO-Cr and CuO-Al, and called them as stable samples. Metals in this group are known to form a very strong native oxide. For the second group we chose Zr and Ti as a fuel, where both have very high oxygen self diffusivity coefficient. All films were deposited in a dual magnetron sputtering system with nm precision and accuracy on the substrates.

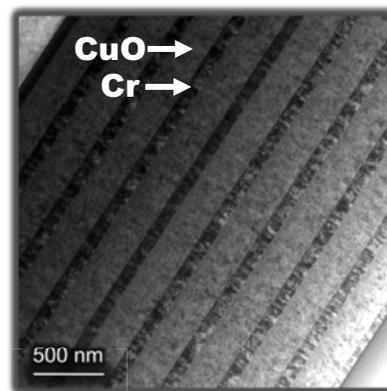


Figure: TEM image of CuO-Cr multilayer thin film in as deposited state

Microscopy and XRD analysis showed that multilayers of CuO-Cr and CuO-Al samples could be synthesized by sputtering and all films are stable under ambient conditions. After thermal annealing and electrical ignition experiments, Cr samples complete the oxygen exchange reaction successfully, however, they suffer from sluggish reaction kinetics. Samples with Al showed similar reaction kinetics to Cr, further, reaction is observed to be incomplete under the experimental conditions applied.

Catalyst Development for Durability and Performance Improvement in PEM Fuel Cells

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Abstract- In this study, supporting material, Vulcan XC-72, was modified with nanosized SiO₂ to improve the durability and performance of PEM fuel cell.

Silica is an insulator material and it has a negative effect on active surface area of the catalysts. In contrast to this, it has a positive effect on performance and durability when it forms a thin layer on carbon surface [1]. It was shown that it enlarges the catalysts surface area and compensate for the loss of ASA due to being insulator and its hydrophilic properties should enhance catalysts-layer wetting what reduces the losses results from proton deficiency in three phase zone [2]. Furthermore, silica hinders the sintering effect that lead to performance drops during service.

Firstly, modified C-SiO₂ supporting materials that include various SiO₂ loadings were prepared. At the next step, Pt/C-SiO₂ catalysts were synthesized with different Pt loadings. Sol-gel technique was used in order to obtain modified C-SiO₂ support materials and low temperature chemical precipitation technique was selected during catalysts synthesizing. NaBH₄ was used as reducing agent. The amount of %SiO₂ content was determined by thermo gravimetric analysis method. MEAs with an active surface area 5cm² were prepared by hot pressing the anode, Nafion 212 membrane and cathode together. Anode and cathode contain 0, 3 mgPt/cm². Single cell PEM Fuel Cell test system was used during performance experiments. Figure 1 shows the performance of MEAs prepared with various loadings of modified C-SiO₂.

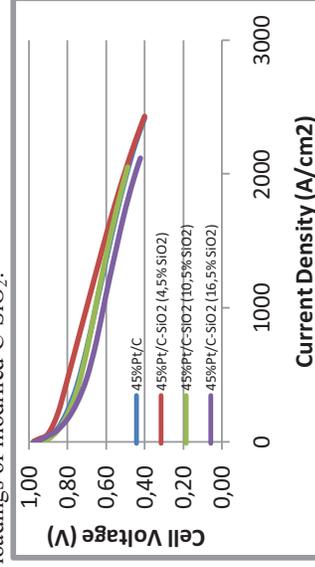


Fig 1. Polarization curves obtained at 80 °C with the back pressure of 30psi. H₂/O₂ gases with %100 RH.

As shown in Figure 1, maximum performance was obtained from the catalyst consist of 4,5 % SiO₂. It was seen that both greater and lower amount of SiO₂ than 4,5 % has a reducing effect on the performance. Being insulator, further silica makes the performance worse.

10% to 45% Pt/C-SiO₂ catalysts were prepared and performance changes based on Pt loadings were investigated. It was found that, as expected, while the amount of Pt loading scales up, the performance becomes better.

In the second stage of this study, the durability tests will be performed. Ex-situ techniques, cyclic voltammetry, etc, will be used during durability tests. The effects of Pt loading and % Si contents on durability will be explored.

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GROWTH OF ZnO NANOSTRUCTURES FOR DYE SENSITIZED SOLAR CELL

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Abstract: The growth of ZnO nanostructures such as nanowire, nanoflower and nanorods was carried out on FTO, ITO and zinc foil substrate by hydrothermal method. With different morphology ZnO nano-architectures were constructed with average diameters of 20-150 nm and lengths in the range of 1-10 μ m. The room temperature PL measurement shows that ZnO nanowires do not have any deep level emission as different nanorods and nanoflowers structures. The UV-Visible absorbance spectrum of the ZnO nanostructures shows a strong absorption between 300 nm and 360 nm (\approx 4.14 eV and 3.45eV). Raman scattering spectra of the ZnO nanostructures showed that the peaks at around 331, 380 and 438 cm^{-1} correspond to the Raman active modes E_2 (M), A_1 and E_2 of the perfect wurtzite ZnO crystal, respectively and extra Raman band was indicated at around 582 cm^{-1} which is known to be related to the E_1 mode of the ZnO nanostructures. After growth of ZnO nanostructures were used as the wide band gap semiconducting photoelectrode in dye sensitized solar cell (DSSCs). Solar conversion efficiencies and incident photon-to-current conversion efficiencies (IPCE) were investigated as depend on surface morphology of ZnO nanostructures. The highest solar-to-electric energy conversion efficiency of 1.3 % obtained by using the ZnO nanoflowers/N719 dye/I⁻/I₃⁻ electrolyte. In addition to, Zn nanowires shows solar-to-electric energy conversion efficiency of 0.6 % while ZnO nanorods show solar-to-electric energy conversion efficiency of 0.27 %.

1. Introduction

The increasing demand for fossil fuels and the environmental impact of their use are continuing to exert pressure on an already stretched world energy infrastructure. Significant progress has been made in the development of renewable-energy technologies, such as solar cells, fuel cells, and biofuels [1]. However, although these alternative energy sources have been marginalized in the past, it is expected that new technology could make them more practical and price competitive with fossil fuels, thus enabling an eventual transition away from fossil fuels as our primary energy sources [2]. Solar energy is considered to be the ultimate solution to the energy and environmental challenge as a carbon-neutral energy source. Although those photovoltaic devices built on silicon or compound semiconductors have been achieving high efficiency for practical use, they still require major breakthroughs to meet the long-term goal of very-low cost (US\$0.40 kWh⁻¹) [3]. To aim at further lowering the production costs, dye-sensitized solar cells (DSCs) based on oxide semiconductors and organic dyes or metallorganic-complex dyes have recently emerged as promising approach to efficient solar-energy conversion [4]. The DSCs are a photoelectrochemical system, which incorporate a porous-structured oxide film with

Catalyst Development for CO_x - free Hydrogen and Nanocarbon Production via Direct Decomposition of Methane

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Abstract- Heterogeneous catalyst was developed for conversion of methane to CO_x - free pure hydrogen and nanocarbon. Catalysts have been prepared by using various supports such as different oxides and zeolites and mainly VI and/or VIII group metals. Catalysts containing VIII group metals have showed more activity in direct decomposition of methane. Optimum conditions of the process have been found. Hydrogen efficiency mainly has been observed that it depends on the reaction temperature. Nanocarbon formed in the reaction contains like nanotube and nanofiber structures.

Nowadays, mainly hydrogen in industrial scale has been fabricated from gas and the other hydrocarbon resources by steam reforming[1] and partial oxidation[2] methods. Recently, the production of hydrogen from direct decomposition of methane has been more attracting. This method has important advantages compared with industrial methods: process is more simple, to obtain pure hydrogen without any extra purification and nanocarbon production in addition to hydrogen[3-5]. The main goal of this study is to develop effective catalyst to produce CO_x - free hydrogen and nanocarbon.

Direct decomposition of methane has been performed in special internal design stainless steel laboratory reactor with fixed bed. Catalysts have been prepared by different impregnation methods. Catalysts and nanocarbon formed in the process have been investigated by XRD, SEM, TEM, BET, DTA and etc.

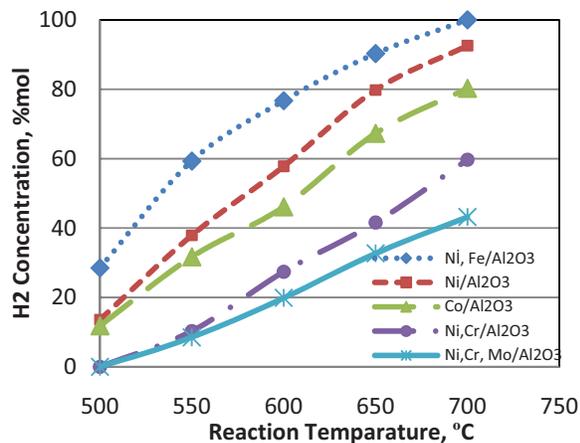


Fig.1. Influence of catalyst type and reaction temperature of the H₂ formation by direct decomposition of pure methane.

Catalysts have been prepared by using various supports such as different oxides and zeolites and mainly VI and/or VIII group metals. Methane decomposition main parameters (temperature, reaction time and partial pressure of methane) effect on catalysts' activities and nanocarbon properties formed in the reaction have been studied. It has been found that hydrogen efficiency mainly depends on reaction temperature.

Activity of some catalysts prepared on γ -Al₂O₃ at different reaction temperatures are shown in Fig.1.. As shown, in methane conversion, the catalysts having VIII group metals have shown more activity. Methane has been converted 85-90

% to H₂ and nanocarbon on Ni,Fe/ Al₂O₃ catalyst at optimum process parameters.

Properties of nanocarbon obtained in methane decomposition reaction were studied. SEM image of nanocarbon grown on Ni,Fe/ Al₂O₃ catalyst is demonstrated in Fig.2.

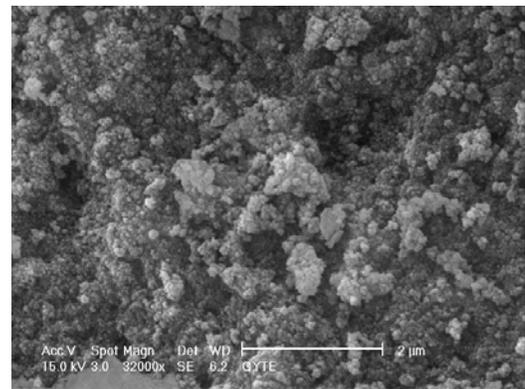


Figure.2. SEM image of nanocarbon grown by direct decomposition of methane (Ni,Fe/Al₂O₃, T=650°C)

Finally: Catalysts containing VIII group metals have showed more activity than VI and VI+VIII group metals. Optimum process conditions have been found. Hydrogen efficiency has stated that it mainly depends on the reaction temperature. Nanocarbon formed in the reaction contains like nanotube and nanofiber structures.

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TMMOB KİMYA MÜHENDİSLERİ ODASI

**6269 Sayılı Kimyagerlik ve Kimya Mühendisliği Hakkındaki Kanun kapsamında kamu kurumu statüsünde faaliyet gösteren Odamızın amacı: Ülkenin ve kamu-
nun çıkarlarının gözetilmesinde, yurdun yeraltı ve yerüstü doğal kaynaklarının
bulunması, korunması ve işletilmesinde, tarımsal ve sınai üretimin
arttırılmasında, ülkenin sosyal, ekonomik, kültürel ve teknolojik kalkınmasında,
çevrenin ve tüketicinin korunmasında, kimya mühendisliği mesleğinin
gelişmesinde, mesleğin ülke, toplum ve üyelerinin yararları doğrultusunda
uygulanması ve geliştirilmesinde, üyelerinin mesleki çıkar, hak ve yetki, onur ve
ortak çıkarlarının korunmasında ve emeklerinin değerlendirilmesinde, üyeleri
arasında dayanışmanın sağlanmasında, diğer meslek odaları üyeleri ve halkla
ilişkilerinde dürüstlüğün ve ahlakın korunmasında, haksız rekabetin önlen-
mesinde, uzmanlık alanında ülkenin ve
üyenin çıkarlarına uygun politikalar üretilmesinde, oda etkinliklerini ilgilendiren
yasa, tüzük ve yönetmeliklerin hazırlanması, geliştirilmesi, eğlendirilmesinde,
gerekli önlemleri almak, yurt içinde ve yurt dışında üyelerini temsil etmek ve
başlıca aşağıdaki amaçların gerçekleştirilmesini sağlamaktır.**

**Meslektaşlarımızı, örgütlülüğümüze güç katmaya, birlikte çalışma, birlikte karar
alma, birlikte paylaşım kültürümüze ortak olmaya davet ediyoruz.**

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Structural characterization of single-phase AgInSe_2 photoabsorbing thin films grown by the selenization of magnetron sputtered Ag/InSe precursor layers

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Abstract—The growth of single-phase polycrystalline AgInSe_2 thin films as a photoabsorbing layer for thin film solar cell applications was successfully realized by means of the two-stage process for the first time. Ag and InSe precursor layers were deposited by DC and RF magnetron sputtering technique in a single deposition cycle, respectively. The prepared ten successive Ag/InSe layers were selenized through thermal evaporation of elemental Se source at different substrate temperatures between 200 and 450 °C. The effect of selenization temperature on the structural properties of the grown films was studied by means of XRD, SEM/EDS and SPM. The single chalcopyrite AgInSe_2 crystalline phase with (112) preferred orientation was observed in thin films after selenization at 350 °C and further increase in substrate temperature caused a spectacular increase in grain size. As a result of this study, a concise production procedure has been developed for the growth of high quality single-phase polycrystalline AgInSe_2 thin films for photovoltaic applications.

Thin film chalcopyrite I-III-VI₂ (I=Ag,Cu; III=In,Ga,Al; VI=S,Se,Te) semiconducting materials with direct band gap energies have proven to attract technological interest owing to their optical and electrical properties suitable to be used as photoabsorbing materials for thin film solar cell structures. For photovoltaic applications, AgInSe_2 thin films were deposited by flash evaporation, laser pulsed deposition, thermal evaporation, rf magnetron deposition and electrodeposition techniques, previously [1-4]. However, low Ag incorporation in the films is a commonly encountered problem for the vacuum evaporation of AgInSe_2 thin films that prevents producing high quality p-type thin films.

In this study, high quality p-type AgInSe_2 thin films with high absorption coefficient within the wavelength region that matches solar spectrum were successfully deposited by selenization of DC and RF magnetron sputtered Ag and InSe thin film precursor layers, respectively for the first time that is so called two-stage process. Furthermore, optimum value for selenization temperature that is an important parameter affecting the structural, optical and electrical properties of deposited films was determined.

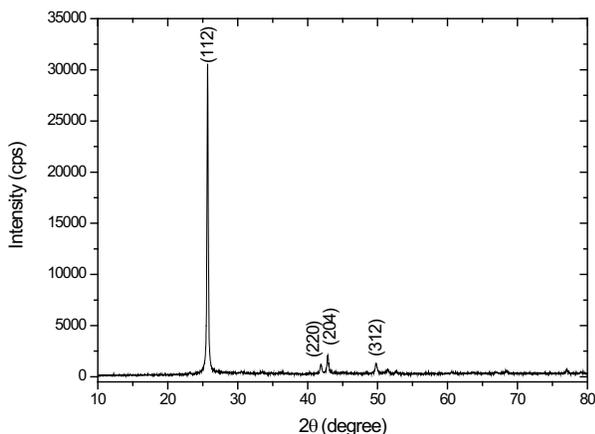


Figure 1. The XRD pattern of AgInSe_2 thin films selenized at 400°C under nitrogen atmosphere.

The evolution of the structural properties of AgInSe_2 thin films as a function of selenization temperature was investigated by means of XRD, SEM/EDS and SPM. As a result of XRD analysis, it is required that the Ag/InSe precursor layers should be selenized at above 350°C in order to prepare single-phase AgInSe_2 thin films with (112) preferred orientation which is the prominent peak for chalcopyrite phase. Moreover, intensity of main orientation peak (112) increases while FWHM values decreases as selenization temperature increases up to 450°C which is an indication for grain size enlargement. Along with this fact, the increase in grain size was confirmed by SEM analysis.

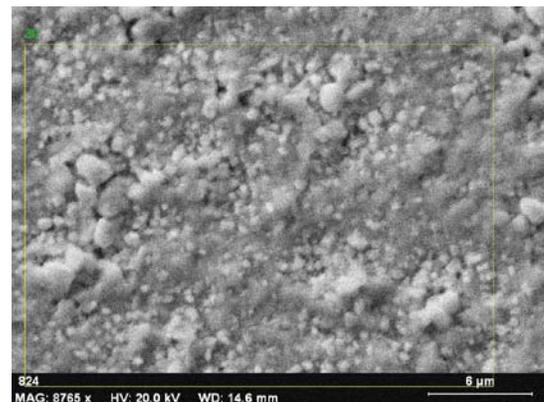


Figure 2: The SEM image of AgInSe_2 thin film selenized at 450°C under high vacuum.

The compositional analysis were performed by EDS and the selenized films at 450°C were found to be composed of 28% Ag, 25%In and 47% Se that proved the stoichiometry of the films. As a result of this study high quality AgInSe_2 thin films were successfully deposited by two-stage process.

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