

Electrical power generation with PEM FC using hydrogen supply through Mg-Ni reaction with water

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Hydrogen is considered an energy vector of the future because of its potential use for clean energy generation. Portable electronic devices can be powered when hydrogen is supplied to fuel cells. In order to avoid massive equipment for hydrogen storage, direct hydrogen production can be achieved on-site during the reaction between metals/metal alloys/metal hydrides and water. Magnesium hydride offers great perspective for widespread applications as its weight yield of hydrogen reaches 6.4% according to the reaction with water and it can even increase to 15.2% if water produced in the fuel cell is used in the reaction again.

In the present work, Mg powder with the content of Ni was synthesized under low temperature hydrogen plasma conditions changing the DC magnetron current from 0.5 to 1 A. As pure Mg powder was immersed into hydrogen plasma, the simultaneous hydrogenation process was ensured. Nickel was chosen as a catalyst capable to influence the growth of hydride. The process of electric power generation was investigated when reaction between modified Mg powder and water was applied to laboratory-built equipment consisting of a reactor for hydrogen production, gas dryer before H₂ introduction to the fuel cell, fuel cell, load and energy meter. Solutions of acetic acid and sodium chloride were used as promoters during powder-water reactions. The characterisation of predicted magnesium hydride powder was done using scanning electron microscopy, electron dispersive spectroscopy and X-ray diffraction. XRD analysis showed only Mg, MgO and Ni peaks indicating that hydrogen generation during powder-water reaction was evoked because of microgalvanic corrosion at Mg-Ni intersections.

Key words: hydrogen generation, Mg-Ni powder, water, electrical power generation

INTRODUCTION

Constantly growing demand for portable multi-functional electronic devices leads to increased power requirements. Despite market needs for

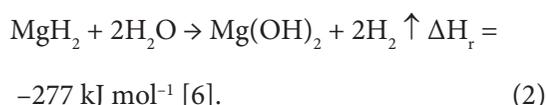
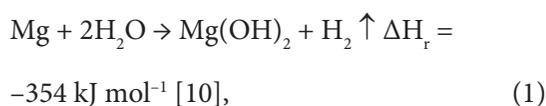
power sources in reasonable size and longevity, an autonomous electricity generation must be with high power density as well [1, 2].

High gravimetric energy density of hydrogen (142 MJ/kg) makes it an energy vector of the future.

Because of its promising utilization for environmentally friendly energy generation without CO₂ emissions, the development of an appropriate carrier where hydrogen would be safely stored is essential [2–4]. Hydrogen can be stored either in pressurized, cryogenic tanks or solids capable to ensure reversible absorption/desorption reaction of hydrogen with low energy exchange [1, 5, 6]. However, storage in high pressure or cryogenic tanks is not suitable for many hand-held applications as it requires massive equipment with additional safety fittings [6].

On the contrary, gas storage can be excluded if direct hydrogen production is achieved on-site during the reaction between metals, metal alloys, hydrides and water [3, 4, 6–9]. For example, aluminum, magnesium, lithium, zink, boron, beryllium or silicon are successfully applied for *in-situ* hydrogen generation using the previously mentioned water oxidation of light metals [4, 8].

Particular attention is paid to magnesium (Mg) due to its low density, high capacity, reactivity and abundance in the earth [10, 11]. Magnesium hydride (MgH₂) offers a great potential for widespread use as its weight yield of hydrogen reaches 6.4% and it can be increased up to 15.2% if water produced in a fuel cell (FC) is used in the reaction again [6]. Therefore, Mg and MgH₂ are used for hydrogen production by the following reactions:



However, Mg does not show good reaction with water either at normal or elevated temperatures because of its native thin oxide layer that forms hydroxide in the presence of moisture preventing water to penetrate into the core and cause the start of hydrogen production. The same tendency is observed in case of MgH₂ when powder-water reaction is hindered by the formation of a magnesium hydroxide layer reducing the rate of hydrogen production.

The kinetics of Mg and MgH₂ hydrolysis can be enhanced when the formation of Mg(OH)₂ is

affected by decreasing pH value of the solution used during the reaction. Therefore, this process can be essentially improved using low concentration of acid or aqueous solution of sodium or potassium chloride causing increased rate of Mg corrosion [8–10, 12, 13]. Moreover, the corrosion rate of Mg to Mg²⁺ is enhanced when alloys with Ni, Fe, Cu or Co are used [8, 11]. This is due to the fact that local galvanic acceleration of corrosion is achieved and corrosion resistance of Mg is reduced [11, 14, 15]. For instance, Mg with increased content of Ni (Mg-2.7 Ni) showed significantly faster hydrogen production compared with pure Mg. The difference in the rate of H₂ production was 1300 times [11].

In the present work, Mg powder with the content of Ni was synthesized under low temperature hydrogen plasma conditions aiming to understand if hydride can be created. It is known that the dissociation barrier of Mg can be reduced by the catalyst of Ni, and the formation of Mg-Ni contact may influence the growth of hydride nucleation centres [16–19]. According to the reactions (1) and (2), magnesium hydride would generate more hydrogen than pure Mg during its on-site reaction with water. The modified Mg powder was characterized using scanning electron microscopy (SEM), electron dispersive spectroscopy (EDS) and X-ray diffraction (XRD). The process of electric power generation was investigated using laboratory-built equipment consisting of a reactor for hydrogen production, gas dryer before H₂ introduction into the fuel cell, fuel cell, and load and energy meter.

EXPERIMENTAL TECHNIQUES

Preparation of Mg-Ni powder

Ni films were deposited on Mg powder (315–630 μm, 99%) using a magnetron sputtering system equipped with 99.99% Ni target. The distance between the sample and the target was 7 cm. The procedure was done at room temperature. Ni films were deposited changing the DC magnetron current (0.5 A, 0.75 A, 1 A) under hydrogen (AGA Gas AB, 99.999%) atmosphere at 7 Pa. Deposition was performed for 60 min for each sample.

A simultaneous hydrogenation process was applied to pure Mg powder when it was immersed into hydrogen plasma consisting of

hydrogen ions, neutral atoms and molecules. In consequence, energetic ions were capable to get into the bulk of material and make bonds with Mg creating MgH_2 .

Figure 1 shows an experimental scheme of hydrogenation process used in this work. Experimental parameters are indicated in Table 1.

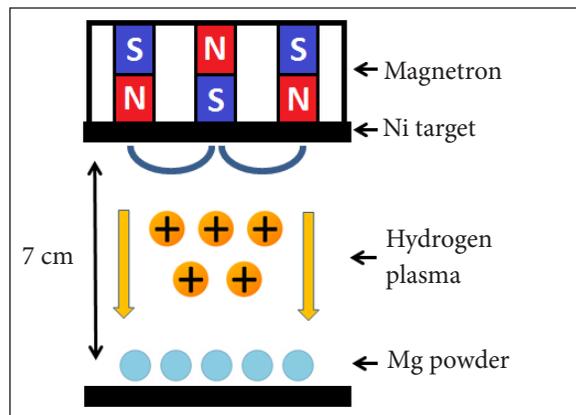


Fig. 1. Experimental scheme of hydrogenation process

Table 1. Experimental parameters

Sample	Mg-Ni-0.5A	Mg-Ni-0.75A	Mg-Ni-1A
Mg powder	315–630 μm , 99%, ChemPUR		
Ni target	4.00" Diameter \times 0.125" Thick, 99.99%, Kurt J. Lesker		
Current	0.5 A	0.75 A	1 A
Pressure	7 Pa		
Time of treatment	60 min		

Characterization

The surface morphology of Mg powder covered with Ni was characterized using a scanning electron microscope (Hitachi S-3400N). The elemental composition of the samples was identified by energy dispersive X-ray spectroscopy (EDX,

Bruker Quad 5040). Microstructural characterisation was carried out using the Bruker diffractometer D8. The diffractometer was used for $20^\circ < 2\theta < 70^\circ$ with Cu K α radiation in 0.01° steps. The EVA Search Match software was used for identification of peaks.

Hydrogen generation

Hydrogen generation based on the reaction between Mg powder and water was measured using an inverted burette (250 mL, tolerance ± 2 mL) filled with water. An amount of produced H_2 gas was indicated by the change of water level in the burette, and then the total volume of generated H_2 was defined by integrating gas flow with time. In order to enhance hydrogen production, low concentration of acetic acid (3% CH_3COOH) and aqueous solution of sodium chloride (5% NaCl) were used. In order to ensure an objective comparison of the results, an equal amount of Mg powder (0.05 g) was used in a volume of 30 mL of prepared solution during every experiment.

Power generation

The exploration of hydrogen production and its application for on-site electricity generation was carried out according to the scheme shown in Fig. 2. The equipment used in these experiments consisted of a reaction tank where Mg reaction with water took place, filtration unit, pressure valve ensuring constant H_2 pressure supplied to the fuel cell, 1.5 W PEM fuel cell for energy generation, energy monitor from Horizon and resistance as a load of the system. Voltage alteration $U(t)$ and generated energy in mWh were measured during these experiments. In order to ensure an objective comparison of the results, an equal proportion of Mg powder and solution was used during experiments. The reaction took place when 0.1 g of Mg was introduced into 30 mL of prepared solution.

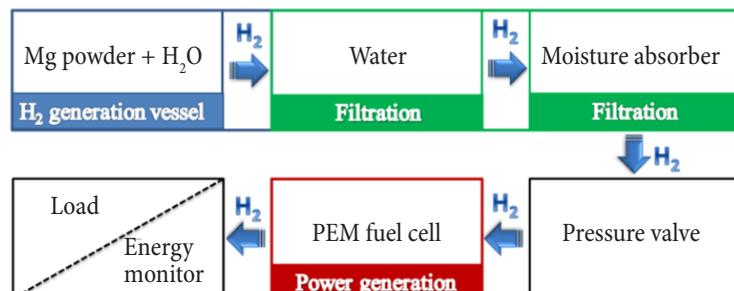


Fig. 2. Experimental scheme for energy generation based on H_2 production during powder reaction with water

RESULTS AND DISCUSSION

Characterisation of Mg-Ni powder

Figure 3 shows the surface morphology of pure Mg powder and its irregularities after hydrogen plasma treatment using a magnetron with Ni target as a source of plasma during the sputtering process. Ni films were deposited changing the DC magnetron current (0.5 A, 0.75 A, 1 A). The SEM micrograph (Fig. 1a) of pure Mg demonstrates a relatively smooth surface without any precipitates. On the contrary, the images of Mg-Ni powders (Fig. 1b–d) reveal non-uniform surface structure. However, no significant difference in morphological appearance is noticed among these Mg-Ni samples. The reason of this resemblance could be explained by the duration of a relatively long sputtering procedure (60 minutes) and properties of Ni itself demonstrating a high rate of sputtering in general [20].

The elemental mapping analysis revealed uneven distribution of Ni (Fig. 4). The size of primary Mg powder (315–630 μm) and spherical shape could be the reason for such inhomogenous distribution of a catalytic element, i. e. partial coverage of Ni is reached because of relatively large particles exposing only a part of their surface in plasma. This tendency is observed for all samples despite deposition conditions.

The XRD analysis of Mg-Ni samples showed only Mg, MgO and Ni peaks. The formation of expected hydride was not observed when the sputtering of Ni was applied under low temperature hydrogen plasma. It is assumed that relatively higher temperature on the surface of powder can bring reversible chemical reactions when hydrogenation and decomposition take place simultaneously [21]. Therefore, at the end of sputtering nothing, except increase of Ni peak, was noticed. Despite the failure to form magnesium hydride, results showed successful deposition of Ni and its

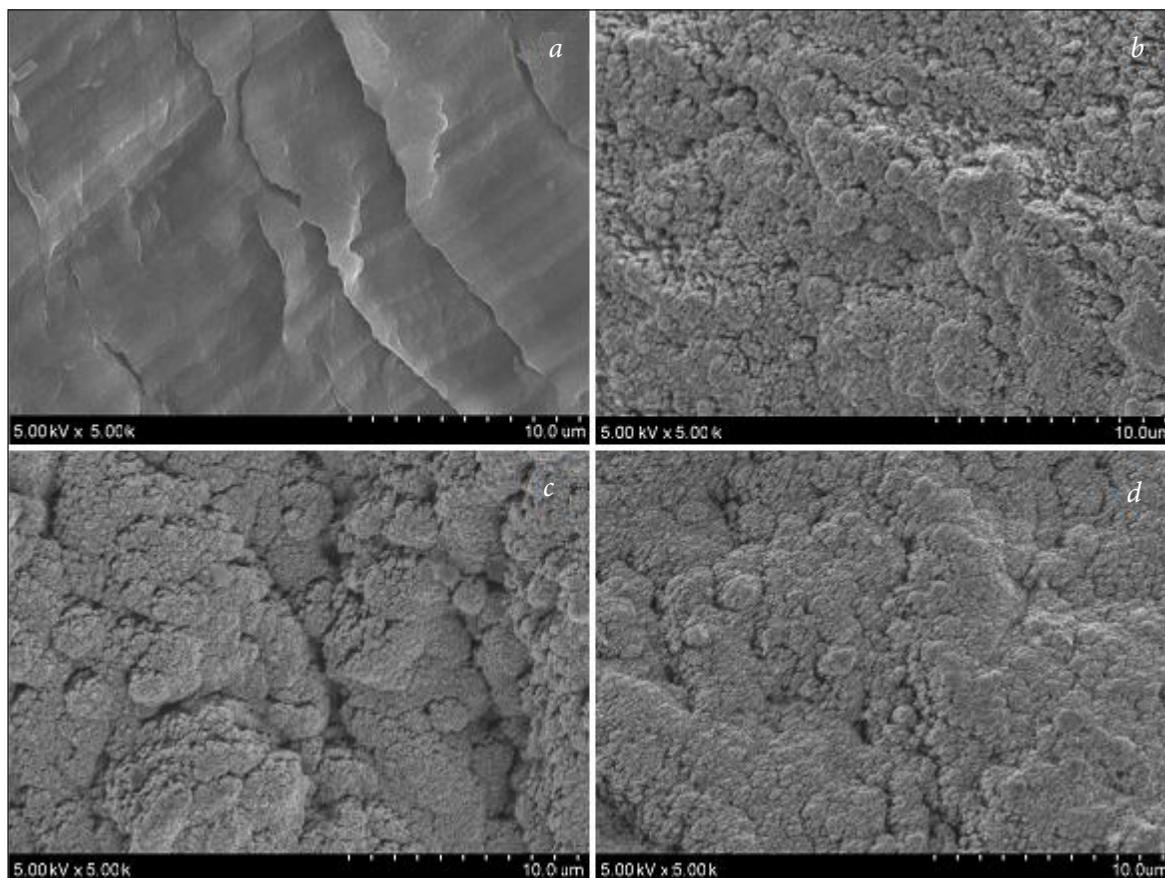


Fig. 3. SEM images of pure Mg (a) and its modifications after sputtering of Ni by changing the DC magnetron current: 0.5 A (b), 0,75 A (c) and 1 A (d)

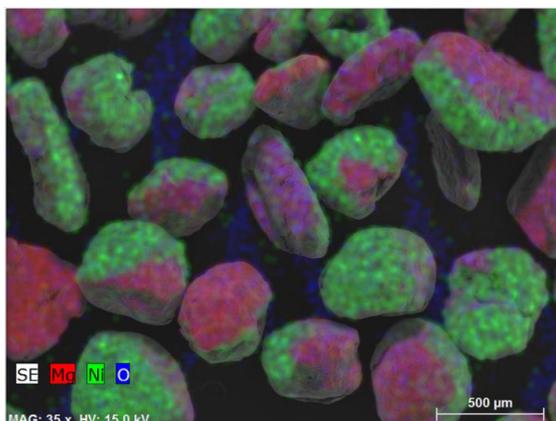


Fig. 4. Element mapping image of Mg-Ni-1A sample

growth by increasing the DC magnetron current from 0.5 A to 1 A (Fig. 5). It is clear that Ni deposition rate is increased by increasing the current. Therefore, XRD registered the growth of the cubic Ni phase which is marked as black triangles in Fig. 5.

Nevertheless, Mg powder covered with Ni can be implemented in the reaction with water for H_2 generation. The micrographs of Mg-Ni powder (Fig. 3 b–d) indicate a positive change in the specific surface area with increase of roughness compared to pure Mg. This result takes an important role during the reaction with water. Moreover, unevenly distributed Ni makes intersections with Mg creating centres where local galvanic corrosion can arise leading to the improvement of hydrogen generation. In general, microgalvanic corrosion is initiated because of the difference of Mg and Ni electrochemical potential, where these parameters are -2.37 V and -0.25 , respectively [22].

Power generation using hydrogen generation during Mg-Ni powder reaction with water

The second part of this study targets on exploration of hydrogen production and its application for electricity generation.

The H_2 generation based on the reaction between Mg-Ni powder and water was measured using an inverted burette filled with water. As pure water did not show any effect on hydrogen generation, reactions were done in two different solutions (promoters) in order to understand if it had any significant influence on the kinetics of H_2 production. All reactions took place when 0.05 g of Mg was introduced into 30 mL of prepared solution.

First of all, H_2 production was measured in the solution with low concentration of acetic acid (3% CH_3COOH). Very similar hydrogen generation kinetics was observed among all Mg-Ni samples regardless of different sputtering conditions (Fig. 6 a). However, the efficiency of these reactions was higher compared with the process when pure Mg was introduced into water, i. e. such reactions demonstrated faster kinetics, and the total capacity of H_2 was proportionately higher.

On the contrary, hydrogen production in aqueous solution of sodium chloride (5% NaCl) varied more evidently depending on sputtering conditions (Fig. 6 b). Only pure Mg did not react with this salty liquid assuming that the key reason was the absence of Mg-Ni intersections inducing microgalvanic corrosion.

Summing up, current variation during DC magnetron sputtering had influence on hydrogen production rate. Moreover, it is important

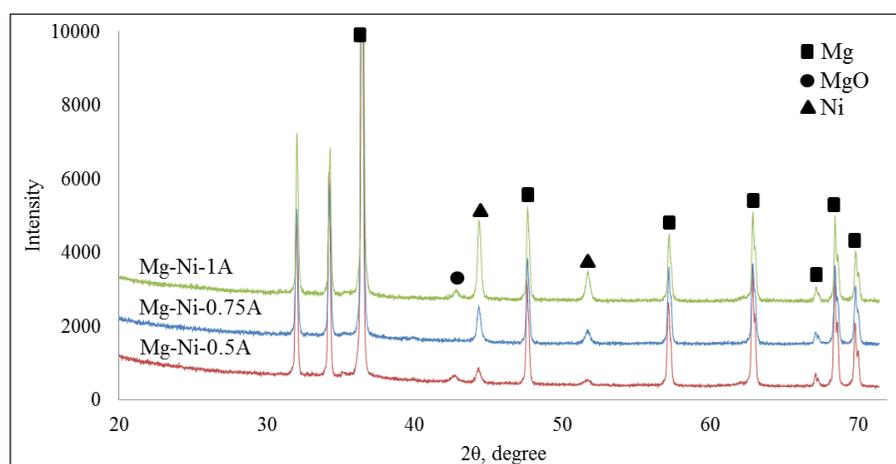


Fig. 5. XRD diffraction patterns of Mg-Ni samples

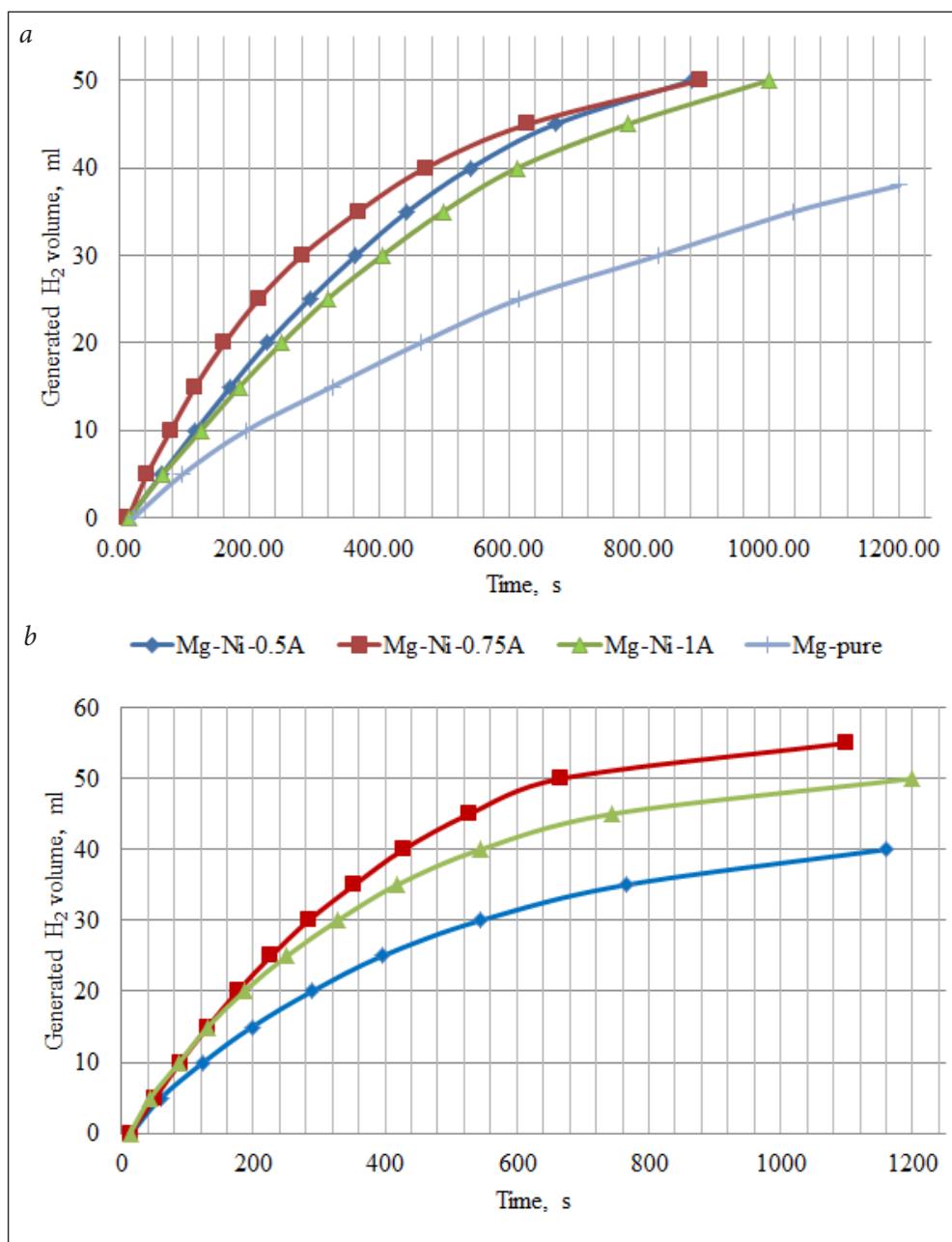


Fig. 6. Hydrogen generation during the reaction between Mg-Ni powder and water using different promoters (a – 3% of CH₃COOH; b – 5% of NaCl). Samples Mg-Ni-0.5A, Mg-Ni-0.75A, Mg-Ni-1A are prepared under different DC magnetron current during deposition of nickel – 0.5 A, 0.75 A, 1 A, respectively

to notice that Mg-Ni-0.75A showed superior kinetics compared with other samples during all experiments. In this particular case, MgO peak was not observed in XRD analysis (Fig. 5). Therefore, the limiting barrier for the start of hydrogen generation was avoided.

Finally, the energy generation system was operated to confirm a possibility to implement H₂ produced from the Mg-Ni powder reaction

with water. The voltage alteration in time $U(t)$ was measured during experiments. In this case, acetic acid was selected as a promoter. As mentioned previously, the reaction took place when 0.1 g of Mg was placed into 30 mL of prepared 3% CH₃COOH solution.

According to the graph given in Fig. 7, the start of energy generation is very similar for all Mg-Ni samples. Only pure Mg exhibits the delay of

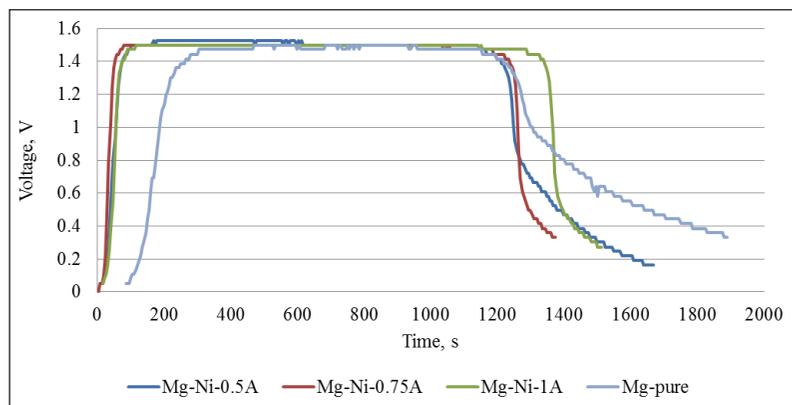


Fig. 7. Voltage alteration during energy generation. Samples Mg-Ni-0.5A, Mg-Ni-0.75A, Mg-Ni-1A are prepared under different DC magnetron current during deposition of nickel – 0.5 A, 0.75 A, 1 A, respectively

the reaction. Based on SEM and EDS results presented in Figs. 3 and 4, it is evident that hydrogen production highly depends on morphological surface variation and appearance of Ni caused after treatment in plasma compared with pure Mg in general. Hence, a relatively smooth surface of pure Mg restricts the reaction with water.

Finally, the total amount of energy generated during Mg-Ni powder reaction with water was identified (Fig. 8). As it was expected, the least amount of energy was generated during the reaction with pure Mg (17.22 mWh). A very similar amount of generated energy was observed for Mg-Ni-0.5A (18.61 mWh) and Mg-Ni-0.75A (18.89 mWh). Energy generation for all these samples took place for about 20 minutes. However, energy generation caused by Mg-Ni-1A reaction with water was measured for about 24 minutes and the total amount of produced energy reached 20.00 mWh.

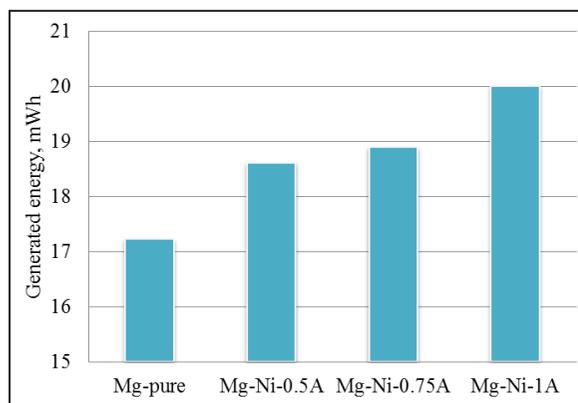


Fig. 8. Energy generation during Mg-Ni powder reaction with water. Samples Mg-Ni-0.5A, Mg-Ni-0.75A, Mg-Ni-1A are prepared under different DC magnetron current during deposition of nickel – 0.5 A, 0.75 A, 1 A, respectively

The results presented in Figs. 7 and 8 confirm the reaction's feasibility for on-site electricity generation. However, additional attention must be paid to the determination of the most appropriate ratio of powder to water used in the reaction. This assumption was raised because of disagreement in experimental results. In case of hydrogen production, Mg-Ni-0.75A demonstrated the highest rate of H_2 generation. Therefore, it was expected that the total amount of produced energy would be observed for the same sample. However, Mg-Ni-1A was superior in this experiment. It is important to notice that 0.05 g of Mg powder were used during measurements of hydrogen production, and 0.1 g of Mg was introduced into water during investigation of energy generation when the volume and concentration of the solutions were kept as a constant.

CONCLUSIONS

Ni coatings were deposited onto Mg powder using magnetron sputtering technique under low temperature hydrogen plasma. It was expected to form hydride that could be implemented for hydrogen generation during reaction with water. However, nucleation centres of hydrides did not appear at Mg-Ni intersections. An assumption for such result could be related to a relatively higher temperature on the surface of powder during sputtering. In consequence, reversible chemical reactions of hydrogenation and decomposition could take place simultaneously.

Nevertheless, the synthesized Mg-Ni powder was investigated for *in-situ* hydrogen production and its integration for energy generation. This

composition was confirmed as feasible for energy production on-demand, because hydrogen can be produced during microgalvanic corrosion taking place at Mg-Ni intersections. The optimal energy generation can be achieved if an appropriate ratio of powder to water is found. Therefore, consistent experimentation is mandatory.

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ELEKTROS ENERGIJOS GENERAVIMAS PANAUDOJANT PEM KURO ELEMENTUS, KAI TIEKIAMAS VANDENILIS YRA GAUNAMAS Mg-Ni REAKCIJOS SU VANDENIU METU

Santrauka

Dėl potencialaus vandenilio panaudojimo aplinkai draugiškos energijos generavimo procese šis elementas yra laikomas vienu iš ateities energijos vektorių. Nešiojamiems prietaisams elektros energija gali būti užtikrinama tiekiant vandenilį į kuro elementus. Siekiant išvengti masyvios įrangos, skirtos vandeniliui saugoti, siūlomas vandenilio dujų generavimas pagal poreikį, panaudojant metalų, jų lydinių, metalo hidridų reakcijas su vandeniu. Magnio hidridas pasižymi plačiomis taikymo galimybėmis dėl santykinai didelės vandenilio išėigos pagal masę. Remiantis magnio hidrido reakcija su vandeniu, išėiga gali siekti 6,4 %, o dar kartą panaudojus iš kuro elemento gautą vandenį – 15,2 %.

Šiame darbe Mg milteliai su Ni priemaisomis buvo gauti panaudojus žematemperatūrinį magnetroninį

nusodinimą vandenilio dujų plazmos aplinkoje, keičiant prie magnetrono prijungto nuolatinės srovės šaltinio srovę nuo 0,5 iki 1 A. Mg milteliams sąveikaujant su vandenilio dujų plazma buvo užtikrinamas vienalaikis magnetroninis Ni nusodinimas ir miltelių hidrinimas. Visų eksperimentų metu išlaikytas nekinantis darbinis slėgis (7 Pa) ir poveikio plazmoje laikas (60 min.).

Nikelis pasirinktas kaip katalizatorius, galintis pagerinti hidrido susidarymą, sukuriant nukleacijos centrus. Elektros energijos generavimas buvo tiriamas laboratorijoje sukonstruotu stendu, kuriame vyko plazmoje modifikuotų Mg miltelių reakcija su vandeniu. Siekiant paspartinti šią sąveiką kaip aktyvatoriai buvo naudojami acto rūgšties ir natrio chlorido tirpalai. Vandenilio gamybos bei elektros energijos generavimo eksperimentai patvirtino, kad MgO nebuvimas, morfologiniai paviršiaus pakitimai, Ni priemaišos bei tinkamas miltelių ir vandens santykis lemia vykstančių reakcijų efektyvumą.

Plazmoje modifikuotų magnio miltelių charakterizavimas buvo atliekamas panaudojant skenuojantį elektronų mikroskopą, rentgeno spindulių energijos dispersijos spektroskopą ir rentgeno spindulių difraktometrą. Skenuojančiu elektroniniu mikroskopu buvo stebimi morfologiniai Mg miltelių paviršiaus pakitimai lyginant su plazmoje nepaveiktais milteliais. Naudojant rentgeno spindulių energijos dispersijos spektroskopą nustatytas nehomogeniškas Ni pasiskirstymas, priklausantis nuo pradinių miltelių dydžio ir formos. Atliekant rentgeno struktūrinę bandinių analizę identifikuotos tik Mg, MgO ir Ni smailės, indikuojančios, kad vandenilio generavimas, panaudojant miltelių reakciją su vandeniu, yra sukeltas mikrogalvaninės korozijos Mg-Ni sandūros taškuose.

Raktažodžiai: vandenilio gamyba, Mg-Ni milteliai, vanduo, elektros energijos generavimas