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## SEPARATION OF WASTE INDUSTRIAL GASES WITH MAGNESIUM

BY

IOAN BARABULICĂ\* and IOAN MĂMĂLIĞĂ

“Gheorghe Asachi” Technical University of Iași, Romania,  
“Cristofor Simionescu” Faculty of Chemical Engineering and Environmental Protection

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**Abstract.** The metallic magnesium is a material with an interesting mix of properties which makes it attractive for research area and is often found in nature, but in a more scattered way. The disadvantages of using magnesium came from its costs and its danger presented by the high reactivity with gases closer to the melting point. The expensiveness of magnesium came from the high energy intake to extract the metal from magnesium raw materials existed in nature. Over time and related to new technological development magnesium has become a very useful material either in an elementary state or in bind state, the fact that put pressure on optimizing production technologies.

This study uses the property to react with gases of magnesium for some industrial applications and came with a few ideas of research directions that can lead to energy savings, optimizing of some existent technologies and brings its contribution in some environmental issues.

**Keywords:** magnesium; nitrogen; carbon dioxide; magnesium nitride; reactions.

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\*Corresponding author; *e-mail*: ioan.barabulica@tuiasi.ro

## 1. Introduction

The gaseous industrial waste products represent at the present time a serious problem from the environment point of view. Industrial development tends to affect the basic elements that sustain life. One of these elements is the air with oxygen content, temperature, humidity as parameters that influence life development on our planet, represented by terrestrial species. Huge amounts of gas mixtures are released into the atmosphere from industrial activities, the most common being burned gases. The major component of burned gases is the  $\text{CO}_2$  which in large quantities influences the parameters of air that sustain life. A material that can be used to diminish this effect is the metal magnesium. These particular element benefits of a large occurrence in nature being the third element in seas water and the fourth compositional element of the earth, Mg covering 13% by mass of the composition of our planet. It is the fifth most abundant element in the hydrosphere (the topmost 3.8 km of oceans) and the only extractable structural metal from hydrosphere (Pekguleryuz *et al.*, 2013, p.1). In the earth's crust is considered to be the eighth-most abundant element and the ninth most abundant element in the universe by mass. (Mathandhu *et al.*, 2014, p.1). Magnesium was first isolated by Sir Humphry Davy in 1808 by electrolysis of a mixture of  $\text{MgO}$  and  $\text{HgO}$ . Antoine Bussy prepared the metal in bigger quantity by heating magnesium chloride and potassium in a glass tube in the year 1831. Magnesium in the separation of gas mixtures was used for the first time by William Ramsay and Lord Rayleigh when argon was isolated by removing nitrogen from the air with magnesium in 1894-1895.

## 2. Production of Magnesium Metal

Natural occurrence of magnesium is in mineral form and in solved ionic form in seawater. The most used minerals to obtain magnesium are: magnesite, dolomite, bischofite, carnallite, serpentine and seawater (although the seawater it's not a mineral it represents a reliable source of magnesium compared with the mineral sources). Obviously, the minerals presented above require purifying steps in order to be used in processes regarding metal magnesium production.

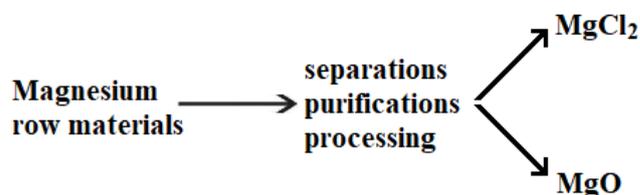


Fig. 1 – Production of magnesium precursors from raw materials.

A simplified scheme of producing magnesium precursors that are used in the extraction of metallic magnesium is presented in Fig. 1. The production technologies can be grouped into two big branches by the type of energy used to extract the metal: the electrochemical and thermochemical methods. The electrochemical methods involve the electrolysis process applied to a mix of melt salts. The mixture contains the precursor (anhydrous  $\text{MgCl}_2$ ) and other salts added to optimize the process (lowering the melting point, reducing the undesired surface electrode processes that can raise the energy consumption). The overall process can be described by the equation:



The most used salt for lowering the fusion point is KCl. In mineral carnallite, KCl is naturally mixed with  $\text{MgCl}_2$  that makes it very suitable for the electrolysis process. The temperature for running the electrolysis is around  $700^\circ\text{C}$ , and the specific consumption of power is around 13 kWh for one kg of magnesium obtained. This value can slightly vary with the type of electrolysis cell, operating procedures, electrolytes used.

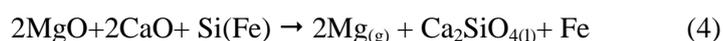
The thermochemical methods imply, as the name suggests, high temperature for the separation process of magnesium metal. The form of energy that runs the reaction is heat. At high temperatures magnesium is obtained in gaseous form, the boiling point being  $1090^\circ\text{C}$ . Besides thermal energy needs, these processes require additional materials that can reduce the magnesium to metal state. The materials used for magnesium production are generally giving the name of the method. The most used material is the alloy Si-Fe (ferrosilicon alloy) and the name of method is the silicothermic process also named the Pidgeon method or the Pidgeon process. The reactions that describe processes in silicothermic methods (Friedrich and Mordike, 2006, p.52-53) are:



Working conditions:  $P = 1 \text{ at}$ ,  $T = 2300^\circ\text{C}$  or  $P = 1 \text{ mmHg}$ ,  $T = 1500^\circ\text{C}$



Working conditions:  $P = 10 \text{ mmHg}$ ,  $T = 1220^\circ\text{C}$



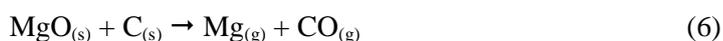
Working conditions:  $P = 1 \text{ at}$ ,  $T = 1700^\circ\text{C}$  or  $P = 1 \text{ mmHg}$ ,  $T = 1150\div 1200^\circ\text{C}$

As can be seen, this kind of processes requires high temperature and very low pressure, so a high amount of energy intake is needed for the development of the processes.

Other reducing agents are aluminum for aluminothermic methods and carbon for the carbothermic processes, the reactions involved (Friedrich and Mordike, 2006, p.57) being the following:



Working conditions: P = 1 at, T = 1300°C or P = 10 mmHg, T = 900°C



Working conditions: P = 1 at, T = 1854°C

For the aluminothermic process, the temperature requirements are lower, but the cost of aluminum rise the price for magnesium metal obtained in this way. The carbothermic process requires the highest amount of energy and the product is a gaseous mix from which is difficult to separate Mg, because it can react with CO as the reversal of reaction (6). Another disadvantage is related to the toxicity of CO. The main advantage is that coal can be used directly in the process, avoiding the use of other, more expensive, reducing agents. But the mentioned disadvantages make this method very less used.

### 3. Uses of Magnesium

The production ways presented above are the main methods for obtaining magnesium metal at an industrial scale. A common disadvantage of the methods described above is the high amount of energy needed for producing this element and rise significantly the price of the metal which, in turn, lead to a decrease in interest of using magnesium at industrial scale.

Although the price of magnesium is approximately double for that of aluminum, the element presents some properties that make it suitable for research, as proved by the multiple uses of this metal. Since 1808, the discovery year of magnesium, only over around 100 years a real demand for magnesium can be seen (Friedrich and Mordike, 2006, p.63) mainly for alloying element with aluminum, deoxidation of steel and other chemical development procedures. The most attractive property of this metal is the low density, in value 1.74 g/cm<sup>3</sup> and this aspect makes the aviation industry to be one of the big consumers of magnesium. Today the main uses of magnesium global production are (Mathandhu *et al.*, 2014, p.1): 41% as an alloying element (most of it for aluminum alloy), 32% a structural metal for casting and wrought products, 13% in the processes of iron and steel production (mainly for desulfurization steps) and the rest of 14% of magnesium is used in other branches of activity. There are numerous technical activity areas that use magnesium in the form of compounds. The most used compound is MgO (Shand, 2006) especially in the refractory industry because of the high melting

point, but it has other applications in agricultural, constructions, chemical, environmental and other industrial related domains (Shand, 2006, preface). Although the other domains that use magnesium require smaller quantities, this element is of great importance. For example, in the biological area of activities magnesium is a key element in life development. It is so important that is necessary to be administrated artificially, producing biological compatible compounds (Nelson, WO2018) that can be easy assimilated by living organisms. In organic synthesis magnesium and its compounds are very useful reagents. Other new domains that begin to use magnesium are the ceramic industry, where the category of chemical-bonded phosphate ceramics (Wagh, 2016) has the magnesium phosphate ceramics as the most developed and studied chemically-bonded phosphate ceramics (Wagh, 2016). In the last decades, a new domain appears in the technical research area, that is solid-state hydrogen storage materials, where magnesium is an interesting reagent by compound  $MgH_2$  (Walker, 2008, p.357-358), that releases  $H_2$  in contact with water even in vapor state as humidity or by thermal decomposition.

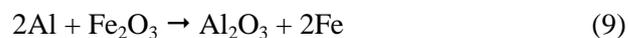
#### 4. Magnesium Reaction with Gases

Another property of magnesium less studied over the years is the high reactivity with a wide range of gases, this aspect is often considered as a disadvantage in using magnesium. Its property to react with many gaseous compounds is the main argument for selecting magnesium for the study of the possibility to separate a particular gas from gaseous mixtures.

Magnesium can easily and highly exothermally react with gases like chlorine and oxygen:



The reaction described by (8) is so exothermic that can increase temperature closer to  $3000^\circ C$ , this property is used to trigger other reactions like the one described by (9)



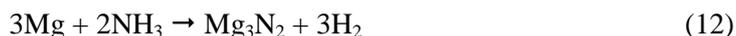
Other diatomic gases that react with magnesium are  $H_2$  and  $N_2$ :



The reaction (10) take place at 200 bar pressure and a temperature of  $577^\circ C$  (Walker, 2008, p.358); recent inventions make the process more

accessible (Jianxing and Wenjiang, WO2019). The reaction (11) is very important in two ways. One way concerning magnesium fires safety issues and the other for the importance of  $Mg_3N_2$  that will be discussed further in this paper. The safe matter of magnesium fires resides from the possibility that magnesium continues to burn in nitrogen too, so nitrogen cannot be used to extinguish magnesium fires. This aspect was most studied in regard of magnesium-nitrogen reaction and most of the articles published are related with magnesium firefighting area of research, but the ways of reactions specified in those articles can be used to characterize the magnesium-nitrogen system. Another area of interest that can be useful in revealing magnesium-nitrogen system are those that specify methods for obtaining  $Mg_3N_2$  as an important reagent, being a very good ammonia source.

Magnesium can react with various gaseous compounds such as ammonia, carbon dioxide, sulphur dioxide:

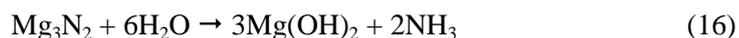


Most exothermic is the reaction (13), the reaction (12) can be used to prepare  $Mg_3N_2$  of greater quality (Kim *et al.*, 2011) and the reaction (14) occurs at temperatures over  $600^\circ C$  (Aleksandrova and Roshchina, 1977). Returning to reaction (13) it can be seen that  $CO_2$ , a gas that is well known for the lack of reactivity being used in fire extinguisher production, its decomposed by magnesium, releasing a considerable amount of energy as heat. This aspect makes  $CO_2$  useless against magnesium firefighting and together with the reaction (11) characteristics make magnesium metal a material that presents important hazard issues at high temperatures. In this article we tend to add another point of view over these reactions, almost absent over the literature existed about magnesium.

## 5. The Reaction of Magnesium with Nitrogen and Carbon Dioxide

In this study, the nitrogen and carbon dioxide were taken into consideration as gaseous reactants for the reason that these are the most encountered in waste industrial gases, especially carbon dioxide. Reaction with nitrogen is very important because it is a way to bring nitrogen into compounds. Nitrogen it's almost chemically inert and it's hard to chemically bind it. The largest method used for this purpose is ammonia synthesis by Haber-Bosh process which implies tough conditions for the development of the reaction. Magnesium nitride obtained as described by (11) or (12) has the property of

releasing ammonia in contact with water even as humidity from the air, reactions that take place are:



Magnesium and nitrogen were treated as a system in the article "Heat Contents and Heat of Formation of Magnesium nitride" (Mitchell, 1949) for the purpose of studying heat implications in binding nitrogen by magnesium. Over time other studies have come to public attention, mostly regarding the flammability of magnesium, because of increase in the use of it in the aircraft industry and the exothermic reactions of magnesium with gases are of concern, especially those reactions that do not imply oxygen. Some alloying elements are lowering the ignition point of magnesium (Fassell *et al.*, 1951), but the  $\text{SO}_2$  presence has the effect of inhibiting magnesium ignition. Almost every study of magnesium combustion mentions the existence of a coating layer of MgO at the surface of the metal that prevents and delays the ignition (Fig. 2).

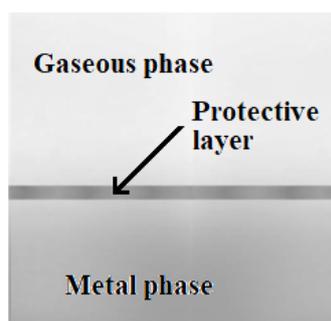


Fig. 2 – Coating layer at the surface of metal.

This MgO film at the surface of magnesium is produced by the action of oxygen from air on magnesium at environmental conditions and is of great importance in using magnesium as a metal because of the protection offered against other reactions that could take place otherwise at temperatures much lower than the melting point of magnesium, which is very close to the ignition point. Magnesium does not ignite so easily (Boris, 1964), but once ignited it continues to burn even if the oxygen from the air is consumed. The heat of combustion in nitrogen is 25% of that in air and the  $\text{CO}_2$  combustion of magnesium releases 60% of the heat of the air one (Boris, 1964). This fact means that even magnesium will not burn with flames in the absence of oxygen; the temperature remains high in the presence of nitrogen or carbon dioxide.

The nitridation mechanisms and kinetics were shown for the first time in the article "The Kinetics of Magnesium Nitriding by Pure Nitrogen" (Dupre and

Streiff, 1970) were two stages temperature-related was indicated. The temperature of reference is 500°C and the stages of reaction (11) that takes place is below 500°C were the mechanism that runs the reaction is at interface metal-gas, implying the diffusion of gaseous phase thru protective layer of MgO and the stage that occurs at temperatures higher than 500°C were the sublimation of magnesium begin to be important, implying the reaction to take place in gaseous phase to. Great contributions to mechanisms involved in the reaction (11) come with the article: "Thermal analysis of magnesium reactions with nitrogen/oxygen gas mixtures" (Chunmiao *et al.*, 2013) were can see that diffusion of gaseous phase or the magnesium vapor thru the surface layer are the processes that influence the reaction development (Fig. 3).

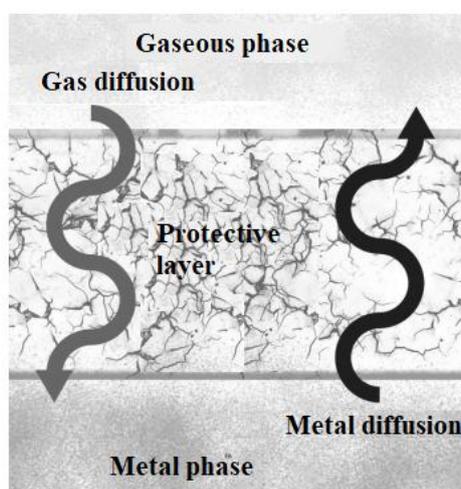


Fig. 3 – Diffusion processes involved at heating the metal.

Over time the interest for magnesium nitride, the only compound of nitrogen-magnesium system (Boris, 1964), was rising and new studies about obtaining magnesium nitride appear, for example the 1995 article "Preparation of Magnesium Nitride Powder by Low-Pressure Chemical Vapor Deposition" (Murata *et al.*, 1993) presented the influence of NH<sub>3</sub> in gaseous mix, and the reaction (12) was highlighted showing the manner of developing along with the high affinity of magnesium nitride to react with water according (16). The fact that diffusion thru the protective layer at the surface of magnesium it's the key process in the development of any reactions of magnesium with gases was leading in attempts to eliminate this effect. One way of working without protective layer is to use pre-activated magnesium, that can be obtained by decomposition of MgH<sub>2</sub> (Shen *et al.*, 1999) and together with the doping of pre-activated magnesium with NiCl<sub>2</sub> or La lead to lowering the starting temperature for reaction in the range 300÷450°C with very good conversion rates compared to the commercial Mg powder. These kinds of studies appear as a result of a

growing demand for magnesium nitride. The property of magnesium to react easily with nitrogen together with the property of solving other transitional metals and the relatively lower melting point make this metal suitable for preparing nitrides of transitional metals (Reckeweg *et al.*, 2001). The use of magnesium to prepare other kinds of nitrides was increasingly developed along with the beginning of using magnesium nitride to prepare special materials like thermal conductivity ceramics and in hydrogen storage materials industry. These aspects are leading to new methods of producing magnesium nitride by using argon dilution (Mei and Li, 2008) of the nitrogen, preventing in this manner the agglomeration of magnesium particles due to nitridation heat reaction. The formation of  $Mg_3N_2$  is desirable at temperatures in a range of 650 to 800°C (Zong *et al.*, 2010) where vaporization of magnesium occur at a faster rate that leads to faster conversion rates. Preparing of magnesium nitride from bulk magnesium was investigated (Kim *et al.*, 2011) by heating magnesium with thermal plasma and the vapor resulted to react with ammonia according (12). In this manner, the reaction was conducted in the gas phase, vaporization of magnesium being faster than the reaction and in no gaseous reactant was obtained magnesium powder, which demonstrates the process of sublimation of magnesium.

Another low-reacting gas that is decomposed by magnesium is carbon dioxide, process described by the Eq. (13). This reaction was observed primarily in combustion of magnesium studies (Boris, 1964) were the heat of reaction between Mg and  $CO_2$  are 60% of the air combustion of Mg. In later studies of this reaction was propose the formation of an oxygen atom in the process (Campbel *et al.*, 1986) and the development of combustion in two stages (Shafirovich and Gol'dshleger, 1990). Then a kinetics analysis of the process (Valov *et al.*, 1992) was adding new information about the influence of diluted  $CO_2$  with Ar which is lowering the activation energy compared to pure  $CO_2$ . According to (Valov *et al.*, 1994) the reaction of magnesium with  $CO_2$  takes place in two parallel reactions:



Overall, the process that implies Mg and  $CO_2$  reaction was demonstrated to be diffusion conducted (Legrand *et al.*, 1998; Gol'dshleger and Shafirovich, 1999) and two reaction zones were proposed (King, 2002), one in the gas phase that surrounding the magnesium particle characterized by the reaction (17) in witch magnesium is in the vapor state that diffuse thru the MgO film and the second zone of reaction characterized by diffusion of CO formed in reaction (17) and the reaction with magnesium at the interface metal-gas described by (18). The decomposition of  $CO_2$  by magnesium is important due to its heat effect. The possibility of using this reaction in some rocket engine is currently under study (Li *et al.*, 2018).

## 6. Discussions and Conclusions

The literature analysis presented before highlighted the metallic magnesium property to react with nitrogen and carbon dioxide. The reactions are exothermic and are influenced by the composition of gaseous phase, particle dimensions of magnesium powder, the distribution of powder particle in the gas phase and the presence of other compounds that can influence the processes. Although some gaseous mixtures reacting with magnesium was analyzed over time (Aleksandrova and Roshchina, 1977; Chunmiao *et al.*, 2013), the possibility of using magnesium to separate gases was not studied. The reaction presented of magnesium with nitrogen and carbon dioxide can be used, theoretically, to separate some mixes of gases such as the burned gases released from burning carbon containing fuels or the purge gas from ammonia synthesis where the recovery of hydrogen is an important issue.

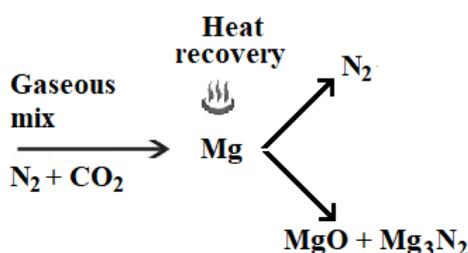


Fig. 4 – Using of magnesium to separate a gaseous mix of nitrogen and carbon dioxide.

In Fig. 4 is presented a sketch of a possible application of magnesium and carbon dioxide reaction. The gaseous mix chosen is encountered as a waste industrial gas in the form of burned gases from carbon related fuels which contain nitrogen and carbon dioxide in the biggest quantity. The sketch represents a research idea that was not treated before and the benefits of reactions development would be a heat source (magnesium and  $\text{CO}_2$  react exothermally) and a source of nitrogen without oxygen traces that is highly important due to the cost of pure nitrogen especially oxygen free.

Magnesium-nitrogen system evolutions can be used as shown in Fig. 5 to separate purge gases from ammonia synthesis. The composition of purge gases is approximately 55%  $\text{H}_2$ , 32%  $\text{N}_2$ , 10%  $\text{CH}_4$  and 3%  $\text{NH}_3$ . From this mixture of gases nitrogen and ammonia react easily with magnesium, the other components of the mix can be recycled in the ammonia gas preparation section after the magnesium reacts with nitrogen and ammonia. The nitrogen extracted with magnesium are not lost, the magnesium nitride obtained release very easily ammonia and is often use as an ammonia source in some organic synthesis (Bridgwood *et al.*, 2008; Veitch *et al.*, 2008a; Veitch *et al.*, 2008b). As the purge gas came from ammonia synthesis, it can be use water in form of steam (in ammonia plants steam is in large quantities) to recover the ammonia from

magnesium nitride. The solid compound that can be considered as a residual product is magnesium oxide, but according to reactions (2), (3), (4), (5), (6) it can be used to recover the magnesium. However, this step of magnesium recovery is highly energy consuming.

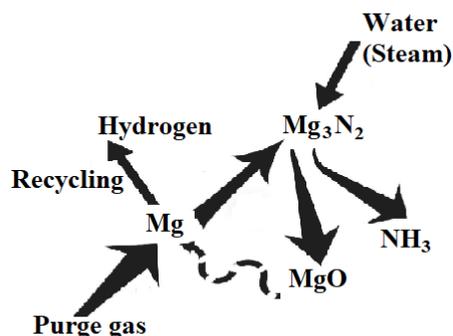


Fig. 5 – Using magnesium in separation of purge gas from ammonia synthesis.

The hypothesis of magnesium uses in reacting with purge gas is an interesting idea of research.

The ideas presented above are examples of magnesium uses in the separation of waste industrial gases. From the reaction with nitrogen, the valuable product is magnesium nitride. From the reaction with carbon dioxide, heat recovery and the elimination of carbon dioxide from gaseous mixes are the main points of interest.

In Fig. 6 is presented the sketch of obtaining ammonia by using magnesium. The key aspect is the magnesium oxide from the ammonia production point of view is a residual matter, but it can be considered as a primary compound like interest in magnesium production. The problem consists of a high amount of energy required for magnesium extraction from MgO as the thermochemical class of magnesium production processes can express.

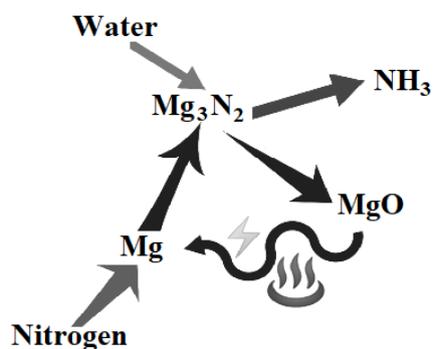


Fig. 6 – Alternative way of producing ammonia.

Another way would be to transform MgO in  $\text{MgCl}_2$ , the electrochemical methods being less energy consuming for obtaining Mg, but this aspect implies new substances in the process and not easy to handle ones like chlorine gas or hydrochloric acid resulting in more steps in order to enrich magnesium. The signs along with the arrow that make connection between MgO and Mg in Fig. 6 represented the two technological branches of producing magnesium thermochemical (below the arrow) and electrochemically (the  $\swarrow$  sign). Although the processes of recover magnesium metal from magnesium oxide are energetically expensive, recycling magnesium oxide from magnesium nitride use to produce ammonia can lead to lower energy consumption to reach from magnesium raw materials to magnesium oxide.

As the final remark the authors suggest that magnesium represents an attractive material that possessed many interesting properties as presented. The reaction of magnesium with gases, for many of its uses represents a hazard issue. Exploiting the reactions of magnesium with hardly reacting gases can be used in optimizing production technologies and in environmental protection, if the  $\text{CO}_2$  from burned gases resulted from combustion of carbon containing fuels is decomposed by magnesium along with energy saved from recovery of magnesium oxide to enclose the cycle. There are more steps to be done in order to set the processes described in Figs. 4, 5, 6 but the key one remain to lower the energy consumption of magnesium production as a future trend.

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## SEPARAREA GAZELOR REZIDUALE INDUSTRIALE CU MAGNEZIU

(Rezumat)

Având în vedere abundența naturală a magneziului și reactivitatea deosebită a acestuia cu gazele se poate lua în considerare utilizarea acestui element în separarea amestecurilor gazoase rezultate în urma diverselor procese chimice industriale. Dintre acestea cele care conțin azot sau dioxid de carbon reprezintă categoria cea mai potrivită pentru studiul separării cu ajutorul magneziului. Deși este un element întâlnit frecvent în natură, extragerea magneziului din compuși necesită cantități importante de energie și condiții de lucru dificile ceea ce reduce utilizarea mai largă a acestui metal. Pe de altă parte azotul și dioxidul de carbon sunt gaze puțin reactive și deseori se întâlnesc în compoziția amestecurilor gazoase rezultate din instalațiile industriale, iar faptul că magneziul reacționează exoterm cu acestea reprezintă un motiv în plus pentru studiul separării amestecurilor gazoase ce conțin azot și/sau dioxid de carbon cu magneziu. Reacția magneziului cu aceste gaze a fost puțin studiată în decursul timpului, iar studiile existente au adus contribuții importante în clarificarea mecanismelor implicate în aceste

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processe din punct de vedere termodinamic și cinetic, iar în cazul reacției cu azotul s-au studiat și elemente de optimizare a reacției în vederea obținerii produsului de reacție superior calitativ. Literatura actuală ce tratează reacția magneziului cu gazele amintite atestă existența reacțiilor și efectul termic al acestora. Posibilitatea aplicării industriale a acestor reacții nu a fost evaluată satisfăcător, astfel că reprezintă un domeniu important pentru cercetările viitoare. Ca exemple practice se pot considera gazele arse pentru eliminarea dioxidului de carbon sau gazele de purjă de la sinteza amoniacului pentru eliminarea azotului. Reacțiile menționate mai au câte un factor teoretic favorabil, pentru reacția cu dioxidul de carbon fiind căldura de reacție și pentru azot importanța azotului de magneziu ca furnizor de amoniac.