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A STUDY OF THE FLAMMABILITY OF MAGNESIUM IN ABSENCE OF OXYGEN

BY

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Abstract. This study was performed to provide information on the flammability of magnesium in absence of oxygen. An investigation of the ignition and burning characteristics was conducted on prepared specimens of magnesium ribbon from magnesium ribbon coil, by cutting samples 7 to 10 mm in length and about 11 to 13 milligrams in weigh. Source of ignition was represented by a kanthal wire that is passed through magnesium samples and positioned on the device for connecting to electricity. The electrically connectable device is added in to a pressure resistant chamber in volume of 350 cm^3 connected with gas sources, vacuum pump and provided with an evacuation outlet for depressurisation. Gases (CO_2 and N_2) was added in to the chamber after using vacuum pump to extract the air. The major results of the prepared specimen tests are presented as a family of curves which indicate the heating effect of kanthal wire, the thermal effect of magnesium ignition and the time and temperature were ignition take place. In general, tests indicated that magnesium can ignite in gaseous mixtures of CO_2 and N_2. The manner of developing combustion can be considered an indicator of gaseous mixtures composition.

Keywords: carbon dioxide; combustion; heat; magnesium; nitrogen.

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1. Introduction

Magnesium is a metal with a good abundance in nature (Pekguleryuz *et al*., 2013; Mathandhu *et al*., 2014), but in a much-scattered manner, as makes it difficult to extract the metal with acceptable costs and considering the high energy demands (Friedrich and Mordike, 2006; Barabulică and Mămăligă, 2019) for extracting the metal as reasons for not using magnesium at large industrial scale. However, the special properties magnesium possesses become attractive for research and studies, a fact illustrated by the multitude of domains that use magnesium in elementary state and its compounds. The most used compound of magnesium is magnesium oxide (Shand, 2006) and in recent decades other domains begin to use the compounds of this metal. From these domains, it can be an example the ceramics technologies with magnesium phosphate ceramics (Wagh 2016) in particular. The solid-state hydrogen storage materials include magnesium as a part of this category of research with the compound MgH_2 (Walker, 2008). Another large domain that uses magnesium compounds is the biological one, were magnesium organic compounds are key ingredients for life development in plant and animal organisms and the research area seek new bio-compatible compounds of magnesium (Nelson, 2018). For magnesium metal, the most use is in the industry of alloy making, especially the lighter ones and as structural metal (Mathandhu *et al*., 2014).

A problem in choosing magnesium metal, besides the high price, is that at relatively high temperatures it reacts with gases that are, apparently, chemically inert ones, like nitrogen or carbon dioxide. This problem is a potential danger in the case of fire hazards events that affect magnesium-containing parts and is the reason for research magnesium reaction with these gases for improving care measures and safety issues in the use of magnesium metal.

In a previous theoretical study (Barabulică and Mămăligă, 2019) it was showed that magnesium reactions with carbon dioxide were analysed for safety matter and for clarifying the mechanisms involved in processes (Campbel *et al*., 1986; Shafirovich and Gol'dshleger, 1990; Valov *et al*., 1995) that take place between magnesium and carbon dioxide. The reaction of magnesium with gaseous mixtures was studied before (Aleksandrova and Roshchina, 1977), highlighting the processes related to the carbon dioxide content.

This study is dedicated to the reaction between magnesium and different mixtures of nitrogen and carbon dioxide in absence of oxygen. In the course of tests development, the evaluation of carbon content influence over the combustion parameters was considered. For this purpose, a reaction chamber was made from stainless steel with a volume of 350 cm^3 and in this chamber, all tests of magnesium combustion were made. Magnesium probes consisted of pieces of magnesium ribbon 4mm wide. The pieces are cut at 10 mm in length. Magnesium heating for combustion initiation was made with a kanthal wire connected at a power source of continuum current. The test chamber was connected to a vacuum pump for the air removal step and to a storage tank of the gas mixture that play the role of preparation tank as well.

2. Testing Equipment

For analysis of magnesium combustion in binary mixes of carbon dioxide and nitrogen, a lab designated circuit that includes three main groups of equipment:

– The A group – includes measurement instruments and electrical devices;
– The B group – includes a testing system;
– The C group – includes auxiliary equipment.

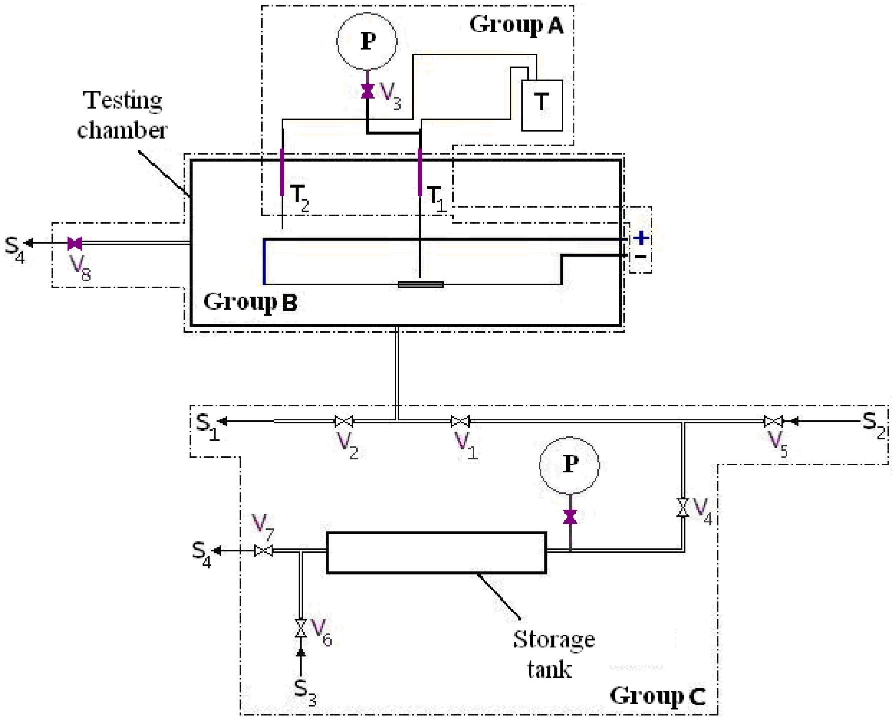

Fig. 1 – Testing circuit.

In Fig. 1 it can be seen a general view of the designated circuit and configuration of groups entered before that will be described forward:

Group A – the measurement instruments and electrical devices. In this category, they are the thermocouples T1 and T2 connected to an electronic device with digital display (T), the pressure gauge manometers (P) and AC/CC transducer for low voltage with amperes measurement which forms the electrical power source.

Group B – the testing system. Here is the testing chamber as the primary constituent made from a piece of stainless-steel pipe that has at one end a welded cover from the same material and provided with a nozzle for depressurizing the chamber (V8), and to the other end a removable cover made from a stainless-steel disk which contains the electrical connections. The cylindrical part of the testing chamber contains the nozzles for measurements, gas inlet and for watching.

Group C – the auxiliary equipment. These are the devices or parts that help the circuit function properly, like vacuum pump (S1), carbon dioxide source (S2), nitrogen source (S3), storage tank, hoses, pipes and valves. With S4 was noted the exterior atmosphere.

2.1. Preparation of Testing Probes

The magnesium used in the experimental series was extracted from a coil of magnesium ribbon by cutting. The magnesium ribbon has the following characteristics: 4.5 mm wide and 0.25 mm thickness. The ribbon was cut at around 10 mm in length. The mass of magnesium probes was around 10 mg (±1.5)

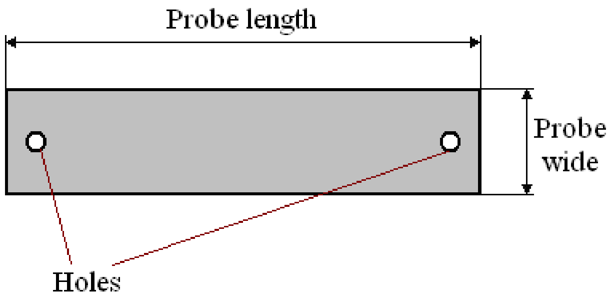

Fig. 2 – The magnesium probe.

In Fig. 2 it is drawing a sketch of a magnesium probe with essential parts illustrated. The holes that appear in Fig. 2 are made after the probe was cut from bulk material for passing the kanthal wire through them and then connected with electrical source from removable cover (Fig. 1 group B).

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3. Testing Procedures

The procedures for test development include several steps which are followed for every test made and these are:

– The step of preparing the magnesium probe and kanthal wire;
– The step of introducing the magnesium probe in the test chamber;
– The step of preparing the gas mix for testing;
– The step of preparing the chamber for testing;
– The step of testing;
– The step of finalizing the test.

The steps listed above will be described as follow:

3.1. The Probe Preparing Step

This step supposes the following actions: measurement from bulk magnesium ribbon of a 10 mm piece and cutting it, then holes making (Fig. 2). Separately it will measure 90 mm kanthal wire Ø = 0.7 mm, then is measure the weight of the wire. The wire is passed through probe holes so that the probe is positioned in the middle of the wire, then is weighted the wire with the probe. The difference between the two weights is the mass of the magnesium probe.

3.2. The Step of Introducing the Magnesium Probe in Test Chamber

In this step, the kanthal wire with magnesium probe is linked at connection made in removable cover for coupling electricity (Fig. 1 +/–), then the cover is fixed at the end of test chamber and sealed with O-ring gasket. After these actions, a check in work must be done for electrical devices for properly made connections.

3.3. The Preparing Gas Mix Step

This step is illustrated in Fig. 1, group C. The essential part is the storage tank that is been used as a preparation tank. This tank is a piece of stainless-steel pipe with two welded covers at the ends of cylindrical shape provided with nozzles for gas connections. Before primary use of this tank as a storage tank for gas mix, it was vacuumed by realizing the next circuit of valves: V8 closed; V1, V2 open; V5 closed; V4 open; V6, V7 closed. After vacuuming it was pressurized with CO2 to 4 bars, then exhaust by open the V7 valve for lowering the air traces from the tank. For adding gases for mixing the following position of valves must be realized: for CO2 V4 and V5 are open, V1, V6, V7 closed until pressure read at the gauge is at wanted value, after that V4 is closed. And for adding nitrogen V6 is open, and V4, V7 closed until the pressure rich the wanted value. V7 valve can be used to release some of the gas content from the tank to rich at the desired composition. The sources of gases are the commercial pressurized gas cylinders.

3.4. The Preparing Test Chamber Step

In the time of preparing gas mixes, the test chamber is isolated by preparing circuits by keeping closed V1. The test chamber has the inner side of the magnesium probe on kanthal wire connected to removable cover connections. The removable cover is closed and sealed with O-ring gaskets so the first stage of preparing the test chamber was done at the second step of procedures. The second stage of preparing the test chamber is related to the atmosphere in the chamber and adapting it for testing. For this purpose, first, the air from the chamber must be extracted with a vacuum pump (S1) provided with self gauge. The pressure in the chamber was lowered to around 50 mbar. Positions of valves related to the chamber are: V1 closed; V2 open; V3 closed (for protection of gauge to vacuum). After vacuuming V2 will be closed, V1 is open and V3 open after that V4 will be open slightly (with V5 closed) so the pressure in the chamber to rise slowly until is riches the wanted value. For a better concordance of the theoretical value of gas mix composition with the practical one, before setting the pressure for test a depressurization of the chamber was done after the first pressure. The depressurization was done by opening the valve V8, this valve can be open for lowering the pressure in the chamber if it rises too much.

3.5. The Testing Step

This step needs that previous steps must be done, which means the test chamber is pressurized with a gas mix with known composition and the kanthal wire with the magnesium probe is properly connected and positioned. Before closing the electrical circuit a video recording device is positioned in front of the watching nozzle to record the events that happened in the chamber. Aside from watching tube is placed the electronic device that shows the values of temperature measurements made with T1 and T2 thermocouples so that these displayed values are caught on video material. The testing step begins with start the video recording followed by closing the electrical circuit that includes the kanthal wire.

3.6. The Finalizing Test Step

After an experiment of magnesium combustion takes place, the video recordings continue until the temperature drops to an equilibrium with the temperature generated by kanthal wire and in some cases, temperature begin to rise again, this time electrical generated. When the conditions mentioned are reached, the electrical circuit must be open and continues to register the temperature drop by changing heat with the environment and after that, the video recording is closed and the test is considered finalized. Forward is needed that the chamber to be depressurized, the removable cover to be removed from the chamber and watching how the probe appears. Sometimes the interior of the steel cylinder must be checked for cleaning issues in order to set the chamber for another test.

4. Results and Discussions

The experiments made according to with the procedures described above contain the results in form of video material. The video presentations from all test made were used for extracting the experimental data values. These experimental data values are composed of the two temperatures measured T1 and T2, and the time from starting the test to the end of it. In this manner, to every test, it corresponds two series of temperature variation over time which is represented in a diagram.

The experiments were organized so that the composition of gas to be easier to control and to vary. The test was developed starting with lower content of nitrogen and rise progressively to higher content, then lowering back the nitrogen content to lower values.

After gathering the tests diagrams and studying them it can be observed 3 types of curves for 3 different intervals of composition: with low content of nitrogen (higher of CO_2), with almost equal content of the gases and with a high content of nitrogen. Low content means that concentration is lower than 40% and high content means that concentration is higher than 60%, between 40% and 60% are the middle compositions (with almost equal content).

Further 3 tests are exemplified and their diagrams are shown in the next Figs. 3 to 5:

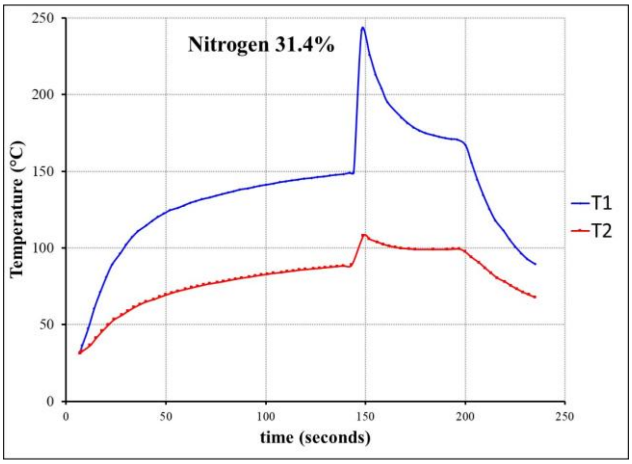

Fig. 3 – Temperature variations diagram for 31.4% nitrogen test.

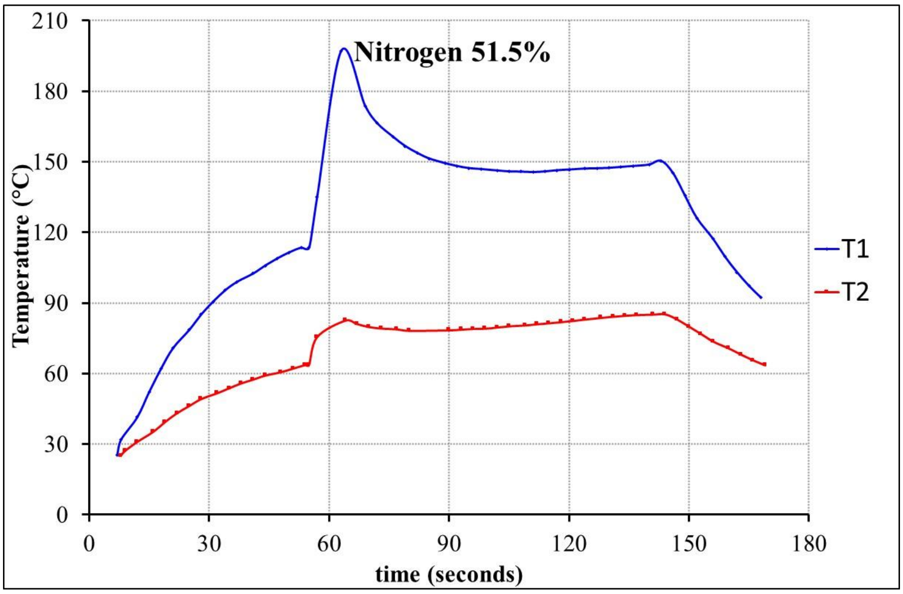

Fig. 4 – Temperature variations diagram for 51.5% nitrogen test.

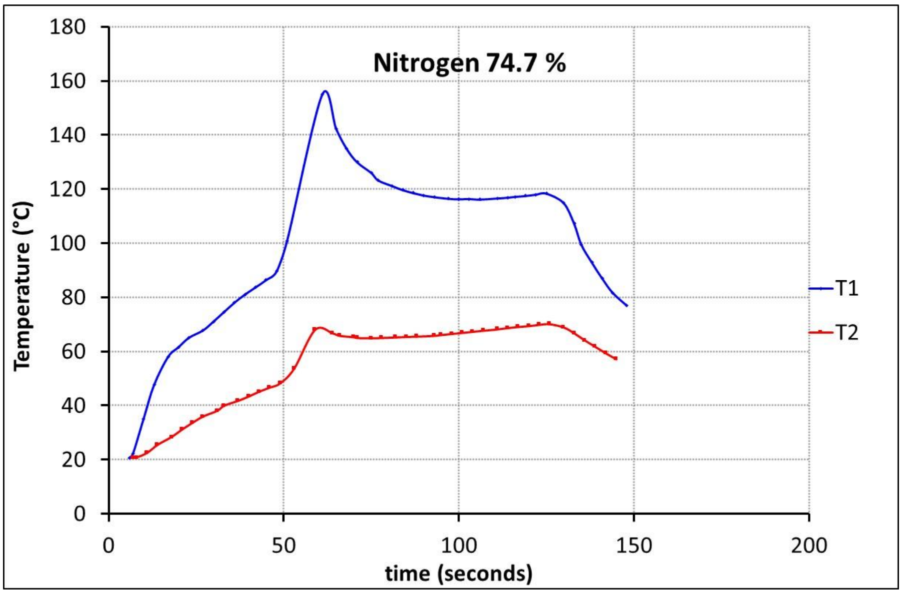

Fig. 5 – Temperature variations diagram for 74.7% nitrogen test.

The temperature measurements are in the following places: T1 at 5 mm distance to probe (the wire passes through the centre of cylindrical chamber) and T2 it measures temperature in the gas phase (atmosphere in the chamber) closer to the inner wall of chamber as it's represented in Fig. 1.

As it can be observed from the above diagrams, the temperature varies profile describes the combustion process and the curve can be analyzed to extract combustion parameters in terms of ignition time and combustion time. For this reason, the curve obtained from experimental data can be sub-grouped into the following zones:

– The heating zone;
– The combustion zone;
– The first cooling zone;
– The second cooling zone.

This parts of a temperature variation from experimental data diagrams are represented in Fig. 6 for T1 curve series and will be described forward:

The heating zone – includes the part of temperature rise from the heating effect of kanthal wire thermo-electric properties. This zone is the first part of the test, it begins from the moment of turning the electric switch as it was described at the testing step procedure and can be assimilated with ignition time because it ends at the ignition point.

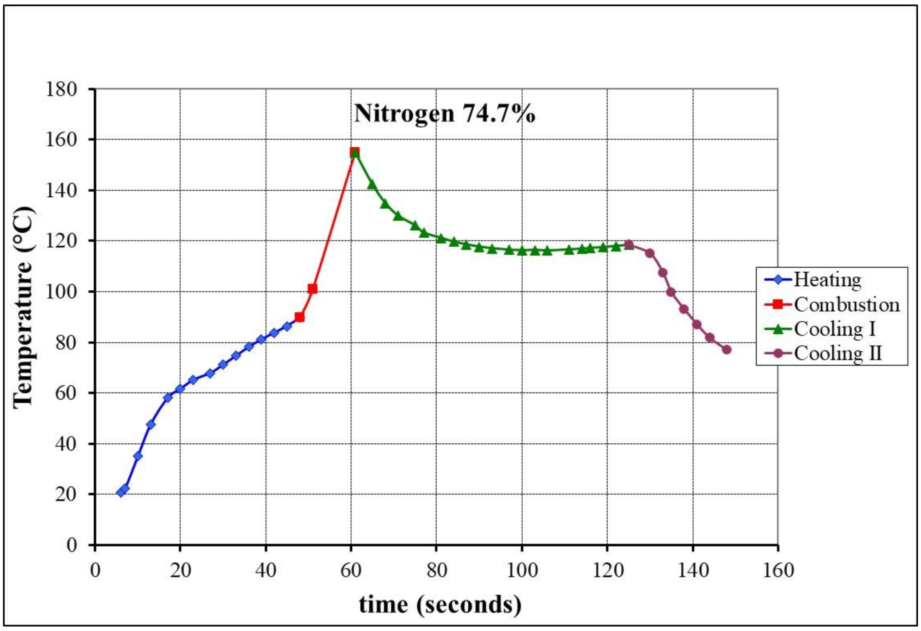

Fig. 6 – Diagram parts details.

The combustion zone – it is the part where temperature records a big rise in a short time (combustion time). This zone corresponds to a bright flash recorded in video material. The ending point of this part is the highest temperature obtained in the test developed and the corresponding time is the combustion time as a combustion parameter.

The first cooling zone – this zone shows the cooling process that takes place after the combustion process and in this part, the electrical switch is not turned off so the cooling takes place until the temperature T1 reaches a relatively constant value. When the temperature starts to slowly rise is the moment of turning off the electrical switch because this temperature rise is from the kanthal wire. Opening the electrical circuit is the ending of the first cooling zone.

The second cooling zone – starts from the moment of turning off the electrical switch and the atmosphere within the test chamber begins the process of natural cooling. The ending moment of this part was arbitrary choose after the temperature drops below 100^{o}C and it represents the ending moment of the test.

From description of the temperature variation curve zones the parameters of combustion can be extracted and from the diagram the values of them.

The ignition time it's the interval initiated with turning on the electrical switch and ends with ignition moment (combustion start). The ending moment of this interval can be extracted easily from video material corresponds the test being the moment when the flash starts.

The combustion time its duration of the flash from video material or the highest value of temperature from diagram. This is the time interval of magnesium reaction with carbon dioxide from gas mix.

From the 3 tests exemplified in this paper it can be extracted the parameters of combustion defined above and make next table:

Table 1
Registered Combustion Parameters

Concentration (N2)	**Combustion time** (s)	**Ignition time** (s)
0.314	2	142
0.515	5	54
0.747	7	50

5. Conclusions

From experimental data of the test developed and from closely analyzing diagrams made. It can be seen some proportionality issues between concentration value of gas mix and combustion characteristics:

– Magnesium reacts in a highly exothermic process with gaseous mixes of carbon dioxide and nitrogen namely a combustion process;

– The ignition times vary direct proportional with nitrogen concentration;

– The combustion times vary inverse proportional with nitrogen concentration;

– The temperature peak is inverse proportional with nitrogen concentration;

– The magnesium combustion process in gaseous mixes of carbon dioxide and nitrogen with high nitrogen content takes place readily, the combustion process takes longer and with smaller a temperature peak.

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UN STUDIU AL FLAMABILITĂŢII MAGNEZIULUI ÎN ABSENŢA OXIGENULUI

(Rezumat)

Acest studiu a fost derulat pentru a furniza informații despre flamabilitatea magneziului în absența oxigenului. O investigare a caracteristicilor de aprindere și ardere s-a realizat utilizând probe de bandă de magneziu provenită prin secționarea unui fragment cu lungimea de 7-10 mm şi masa de 11-13 mg dintr-o bobină de bandă de magneziu. Sursa de aprindere a fost reprezentată de un fir de kantal trecut prin proba de magneziu şi montat într-un dispozitiv pentru conexiunea electrică. Dispozitivul cu conexiune electrică este montat într-un recipient rezistent la presiune de 350 cm^3 volum, conectat cu sursele de gaze, pompa de vacuum și prevăzut cu ștuț de evacuare pentru depresurizare. Gazele (CO_2 și N_2) au fost adăugate în recipient după extragerea aerului cu o pompă de vid. Rezultatele testelor sunt prezentate ca o familie de curbe care indică efectul termic al firului de kantal, căldura reacției magneziului, timpul și temperatura aprinderii magneziului. În general testele indică faptul că magneziul poate fi aprins într-un amestec gazos de CO_2 şi N_2. Modul desfăşurării combustiei poate fi considerat un indicator al compoziției amestecului de gaze.