



# Oxygen and Hydrogen Reaction “*In situ*” with Magnesium and Tantalum in a Mechanical Milling System When a Solvent is Used

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## Authors' contributions

This work was carried out in collaboration between both authors. Author JLIG designed the study, performed the statistical analysis, wrote the protocol, wrote the first draft and revised the final manuscript. Authors BELM and JLIG managed the analyses of the study. Both authors read and approved the final manuscript.

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## ABSTRACT

The purpose of this paper is study the preparation “*in situ*” of magnesium–tantalum phases when reacted with oxygen and hydrogen during a mechanical milling process. Different phases were formed as function of the milling time. High energy ball milling was utilized to prepare hydrogenated compounds by controlling the milling between 5 and 20 hours with a ball powder weight ratio of 10 and using methanol as the control agent. Scanning electron microscopy (SEM) and X-ray diffraction (XRD) were used to characterize the compounds. The XRD results at 20 h of milling revealed that only hydrogenated species ( $Ta_2H$  and  $TaH_{0.5}$ ) and  $Mg(OH)_2$  were present.

**Keywords:** Tantalum hydrides; high energy ball milling; solvent dissociation; XRD; SEM.

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## 1. INTRODUCTION

Different techniques, such as the wet chemistry method conventional technique have been used to prepare metal alloys. Other various techniques use physical reactions to prepare alloys, such as electrical arc, induced thermal fusion or high energy mechanical alloying process, and the latter was used in this study, the use of these techniques allow the preparation of nanocrystalline, amorphous, nanocomposites, and quasicrystalline materials including intermediate phases, etc. [1-5]. Currently, the mechanical alloying process has been used extensively to principally obtain metal alloys, but its mechanism is not completely understood and depends on each milling system. Mechanical alloying using high energy has also been utilized in the synthesis and modification of metal hydrides and other inorganic solids, while organic compounds such as transition metal complexes have been obtained in the presence or absence of a suitable solvent [6]. Many materials used in hydrogen storage, including mainly metallic combinations and nanoporous compounds, have been synthesized and studied to contribute to the development of hydrogen technology [7]. To meet the requirements of many laboratories at the international level, studies are performed to obtain new materials that can be used for practical purposes particularly in fuel cells. The aim of this technological challenge is to store the maximum amount of hydrogen in the minimum volume or mass of storage system safety (gravimetric and volumetric hydrogen density as gas and liquid systems) [8-10]. Various compounds among the Mg-base alloys are considered for the reversible absorption/desorption hydrogen process [11-17]. On the other hand, the preparation of hydrogenated compounds such as hydrides or complex materials that are based on the tantalum element, is very limited and mainly utilizes mechanical alloying techniques used in the present investigation. Certain studies related to the preparation of tantalum hydrides consider the monohydrides as complex compounds that are the basis of this metal; these studies include crystallographic properties and thermodynamic aspects, high temperature reactions in the Ta-H system and the liberation of hydrogen achieved by a hot vacuum treatment at 1000°C and pressure of  $10^{-5}$  mm Hg. Other physicochemical parameters, such as hardness, mechanical properties, electrical resistance, density, and superconductivity have been reported [18-22]. In this work chemical reactions between oxygen

and hydrogen with Mg and Ta metals were carried out "*in situ*" during a mechanical milling procedure using methanol as the process control agent, which resulted in different phases depending on the milling time. One of the goals of reacting Mg and Ta with hydrogen within a mechanical milling system was to determine if magnesium hydride could be formed together with tantalum hydride *in situ* like previous works in which hydride tantalum was obtained by dissociating the control agent into its elementary components, including hydrogen [23]. Based on our experience, when a certain type of solvent is used during mechanical milling, specific experimental conditions are created inside the container that separate the solvent compounds in their elementary components.

## 2. MATERIALS AND METHODS

Magnesium and tantalum metal particles with a purity of 99.8% were mixed at desired quantities to reach a nominal composition of 25wt% Mg and 75wt% Ta, which corresponds to the stoichiometric mole ratio of 1:3. The chemical reactions were carried out by a mechanical alloying technique with a spex-type high energy ball mill; this system was fabricated at our institute. Then, Mg and Ta powders, three stainless steel balls 12.7 mm in diameter and methanol as the process control agent were put into a stainless steel vial for milling. The ball to particles weight ratio was 10. In a little lucite glove box under argon gas, all metal samples were handled to prevent exposure to air. The milling time is an important parameter for material preparation; the milling time period was defined to be between 5 to 20 h at room temperature. To monitor the possible formation of a phase, a small amount of ball milled material was removed from the stainless steel vial at time intervals of 5, 10 and 20 h. From a series of experiments, it was found that these periods were the most appropriate times for checking the possible changes in the structural materials, particularly the formation of hydrides. During milling, the temperature increases and warms the vial; to keep the temperature as low as possible, a ventilator was placed on the container to avoid overheating of the whole milling system. The structural evolution during milling was determined by X-ray diffraction analysis using a Siemens D5000 diffractometer to explain the possible chemical processes in the formation of metal hydrides and identify their corresponding phases. The measurements were taken at a power between 20 – 25 kV and 25 mA. A

diffracting beam monochromator and Cu K $\alpha$  radiation were used. The material was placed in a portable sampler with a scanning angular interval from 15 – 20 to 120° for 2 theta. A JEOL 5900 LV Scanning Electron Microscope equipped with an energy dispersive microanalysis using the X-ray (EDAX) system was also utilized before and after milling to determine the morphology and elemental composition of the powders.

### 3. RESULTS AND DISCUSSION

Fig. 1 shows a micrograph of the Ta and Mg metals utilized in this investigation. The image in Fig. 1A corresponds to the Ta particles. Tantalum powder with a particle size of 50 microns according to the provider (sigma-aldrich) has the appearance of a sintered material with a heterogenic morphology possibly because the preparation method affected the structure of this powder, which appears in the image of these particles as agglomerates that have empty spaces type pores. The Mg particles are shown in the micrograph in Fig. 1B. The Mg particles were treated with an abrasive file from an ingot, which was prepared in the materials department of our institution (ININ) before the mechanical milling process. These particles have elongated shapes and are toothed at the sides due to the way the abrasive file roughened the surface, which is that of a relatively soft metal. According to the scale reported on the image, the particle size was on average greater than 1 mm. The images were obtained at 5,000 and 100 magnifications for tantalum and magnesium respectively. According to the results by the EDAX for the elemental composition, only Ta and Au elements were present in Fig. 1A, which shows that tantalum is a pure metal without any impurity. Gold appears because the sample surface was subjected to a coating with this element to prevent an electric over charging when the microscopic analysis was performed. On the other hand, the same results of the EDAX corresponding to the magnesium sample (Fig. 1B) showed that only Mg and Au were identified; the presence of gold was for coating sample before microscopic analysis, as previously mentioned. Generally, the material must be reduced in size to nanometer scale to increase the probability of reacting with hydrogen.

Fig. 2 shows Ta and Mg powders after 20 h of milling. According to the results of the SEM, the particle size after 20 h of milling had morphologically changed. A great difference

exists between the images in regards to the particle sizes. Some particles smaller than a micron has been obtained and agglomerate accumulations are observed, which are almost uniformly distributed. Nanometric sizes were obtained when the powder material was diminished after this milling time. The results registered by the SEM show cumulus, which is composed of very small particles. Image A represents the material whose morphology, in general is not defined, while image B is more detailed because the visualizations were obtained at 5,000 and 25,000 magnifications respectively. According to the results of diffraction, the images correspond to the phases of mainly tantalum hydride. Both physical changes such as colour from silvery to dark and particle size from large to small, and chemical changes, such as the metal changing to its corresponding oxide, were observed of this material. The material after 20 h of milling was analyzed by the EDAX and the Mg, Ta, C, O and Au elements were identified. The presence of C is mainly because of the tape where the Mg – Ta powders were placed for analysis; the tape is made of carbon and additional carbon is possible from methanol used as a control agent process. The presence of oxygen may be due to the methanol because the conditions created in the container during milling and solvent dissociation resulted in the components of oxygen, carbon and hydrogen, as demonstrated elsewhere [23]. The oxygen may be reacted during milling with the magnesium in a small concentration that was enough for the results of the XRD to detect the corresponding hydroxide. Most of the hydrogen was reacted with Ta and after finishing the mechanical milling process, only their hydrogenated species were identified. The carbon probably was mixed with the Mg – Ta phases without combining chemically with these compounds and was not detected because to the small concentration. Au is used to cover the sample surface to avoid an electrical overcharge during the SEM analysis.

Fig. 3 shows three diffraction patterns that represent the Mg, Ta elements and the phases after 5 h of milling process. Analyses were performed at the intervals in the figure to document the evolution of the formation of different phases for different milling times. The first spectrum at the bottom of the figure corresponds to the Mg sample before milling, and its particles size image is shown in Fig. 1B. Only the Mg phase was identified according to the JCPDS (00-035-0821) card; no other phase

appeared as an impurity because the magnesium particles are of high purity. More intense deflections appear at relatively small  $2\theta$  angles between 30 – 45 degrees, and a majority of the peaks are perfectly separated. The spectrum in the middle was obtained for the tantalum powder with its micrograph as indicated

in Fig. 1A. Similarly, with the 00-004-0788 JCPDS card, the material was only identified as the tantalum element. The most intense line was registered as 38.463 for the  $2\theta$  angle; the intensity and position of both spectra are not interposed between them. The rest of the peaks are perfectly separated at high angle values.

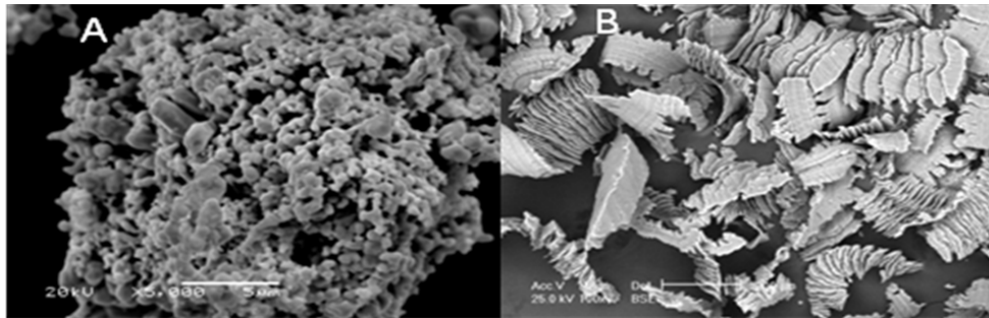


Fig. 1. Micrographs of tantalum (A) and magnesium (B) before the mechanical milling process

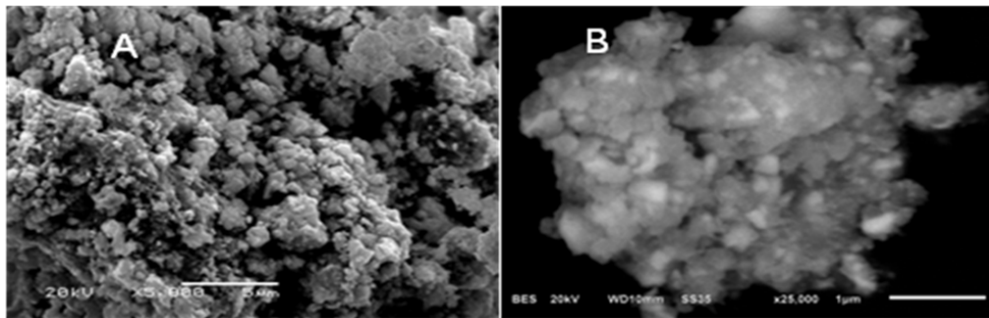


Fig. 2. Micrographs of the Mg-Ta powders after 20 hours of milling

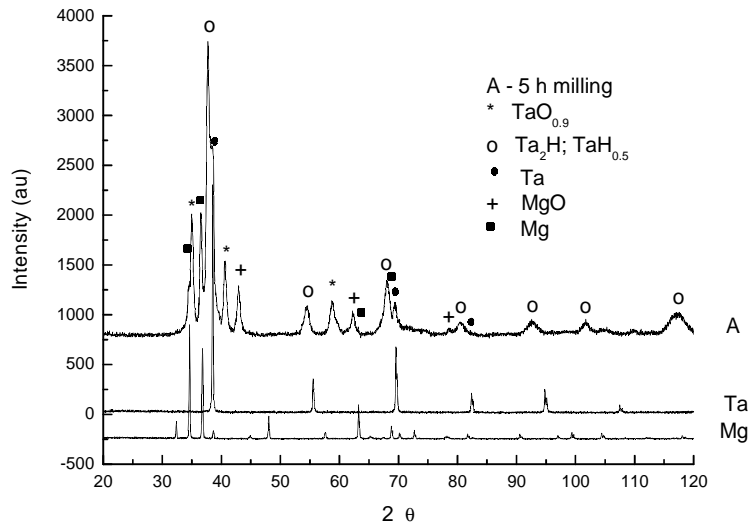


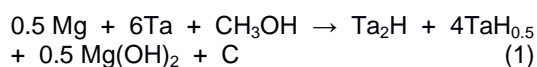
Fig. 3. X ray diffraction patterns of Mg, Ta and different phases formed (A) after 5 h of milling

Spectrum A shows that the material milled for 5 h had some peaks that indicate partial formation of tantalum hydrides. At this time, tantalum hydrides ( $Ta_2H$  and  $TaH_{0.5}$ ) and magnesium and tantalum oxides ( $MgO$ ,  $TaO_{0.9}$ ) phases were formed and identified with JCPDS cards (01-089-4074, 10-089-7746 and 03-065-7449) respectively. The tantalum element appears equally because it has not reacted completely at this milling time. It is assumed that the hydrides formation and oxides takes place simultaneously, because when the appropriate experimental conditions are created inside the vial, the methanol can be dissociated at the same time as the particles size is being reduced; therefore, the corresponding reactions are realized partially or completely depending on the elements and milling time. Based on the diffraction results, it is important to note that magnesium first partially reacted with the oxygen causing the formation of magnesium oxide ( $MgO$ ). Similarly, the elemental magnesium was identified with its respective card and was unreacted after 5 h of milling. The formation of hydrides "in situ" may be due to the high impact energy generated between the stainless steel balls, the sample ( $Mg$  and  $Ta$ ) and the methanol. In these circumstances, the milling reaches high temperatures at the atomic level which creates, the experimental conditions that cause the solvent to dissociate into its elementary components. At the same time, the pressure generated in the solid which is trapped between two colliding balls, can rise to an extremely high level of several Gpa; this facilitates genuine ultra-high pressure activated processes that are reported in the literature [6,24]. It is interesting that the sequence formation of the tantalum hydrides phases during the milling process initiates at short milling time (5 h) and that the magnesium and tantalum oxides were identified at the same time that the magnesium and tantalum metals remained unreacted, possibly because the temperature inside the system at short milling time was not high enough to dissociate the methanol completely. In these milling conditions, magnesium first reacts with oxygen before reacting with tantalum to form the  $MgTa$  alloy in such a way that the diffraction analysis did detect the corresponding oxide ( $MgO$ ).

Fig. 4 shows the diffraction pattern after 10 h of milling. At this milling time different phases, hydrides and oxides (spectrum A) were formed. During this time, the magnesium and tantalum elements were not detected and the hydride phases ( $Ta_2H$ ,  $TaH_{0.5}$ ) appeared with greater

intensity in the diffractogram. The tantalum oxide ( $Ta_2O_5$ ) was identified using the JCPDS cards 00-005-01315 which contrasts with the ( $TaO_{0.9}$ ) obtained at 5 h. Magnesium oxide ( $MgO$ ) and mixed magnesium-tantalum oxide ( $MgTa_2O_6$ ) were also registered and identified with the JCPDS cards 01-084-1679. The main deflections of the tantalum and magnesium hydrides phases are at low values of  $2\theta$  angles, and all intensities of the different compounds were observed without any interference. It is assumed that after 10 h of milling, magnesium and tantalum have completely reacted to form their corresponding phases. According to the diffractogram some exchange reactions occurred by increasing the milling time for example, when the  $TaO_{0.9}$  to  $Ta_2O_5$  phases and the combination of  $Mg$ ,  $Ta$  and oxygen to form the corresponding mixed oxides.

Fig. 5 shows the diffraction pattern of the hydrogenated phases after 20 h of the milling process, in which only the tantalum hydride and magnesium hydroxide phases were identified this last one with small intensity lines (spectrum A). All deflections in the spectrum correspond to the  $Ta_2H$ ,  $TaH_{0.5}$  and  $Mg(OH)_2$  phases; during this stage no  $Mg$  and  $Ta$  oxide compounds were detected. XRD results indicate that tantalum and magnesium were completely reacted within the container and the final products were hydride and hydroxide phases, according to Equation 1:



It shows clearly that the main tantalum hydrides intensities of the  $2\theta$  angle do not overlap with the magnesium or tantalum elements lines whose values are 37.762, 36.624 and 38.463 respectively. Results of the XRD at a milling time of 30 h were analysed. The importance of these results is that the magnesium hydroxide and only the tantalum hydrides conserved the same phase after this period of milling (30 h), spectrum no reported. Furthermore, at 20 h of milling or less, only tantalum hydrides and magnesium hydroxide phases were obtained. On the other hand, in this research, due to differences in the physicochemical properties between these two elements, especially their melting points (3017 and 923°C for  $Ta$  and  $Mg$  respectively), and in these conditions, it was difficult to obtain the  $MgTa$  alloy using mechanical alloying. During the mechanical milling process, high energy is generated by large impacts between the container walls, milling media and material. This

energy is probably large at the atomic level which creates the conditions necessary to break the chemical bonds of the methanol that is used as a control agent and dissociate into hydrogen, carbon and oxygen. Each binding energy between the various atoms of methanol has a

different value. Of the four hydrogen atoms in the methanol, three are bonded to a carbon with a bonding energy of 415 kJ/mol. Another bond in the solvent is a carbon – oxygen bond that has a value of 352 kJ/mol. The fourth hydrogen atom is bonded to oxygen and has a binding energy

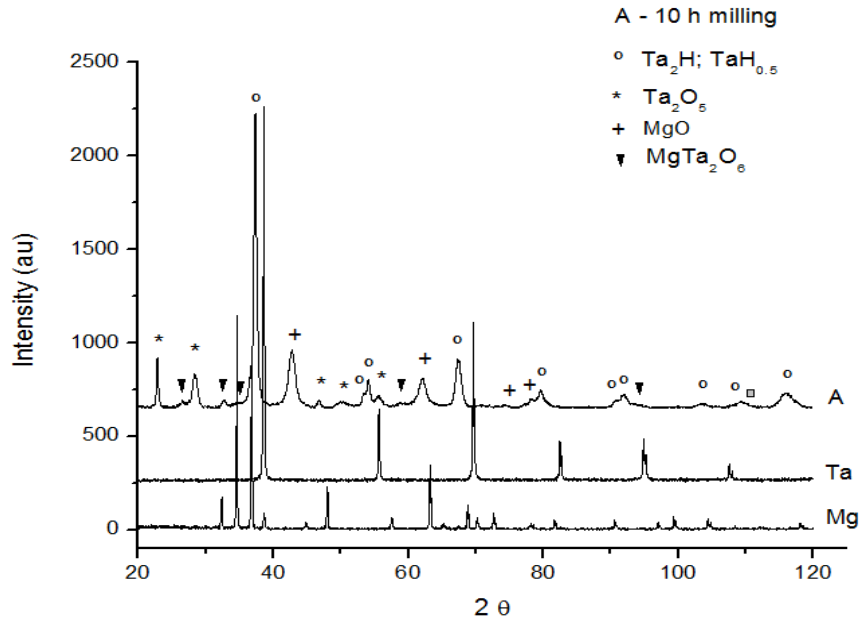


Fig. 4. XRD patterns of Mg, Ta and intermediate phases (A) formed after 10 hours of milling

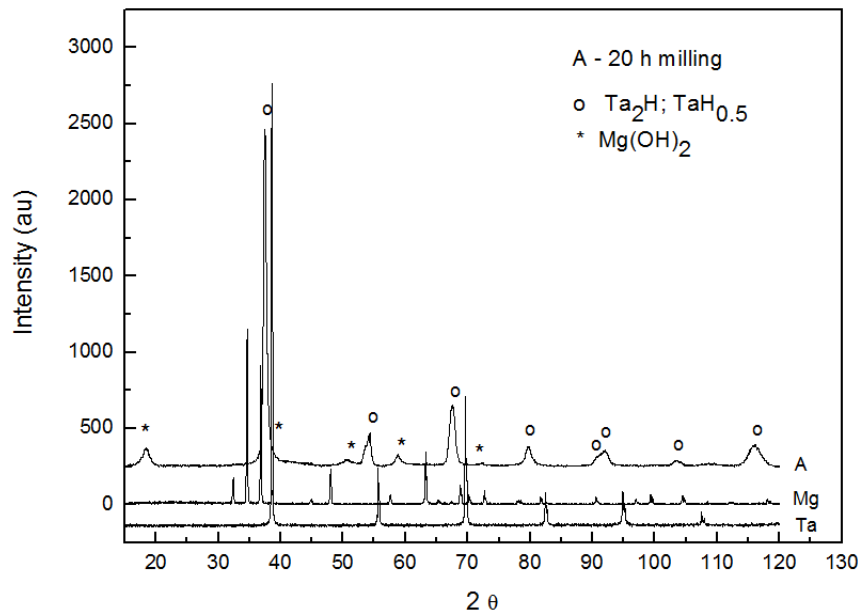


Fig. 5. XRD patterns of the Mg and Ta elements and different phases formed after 20 h of milling and spectrum (A), in which only the tantalum hydrides and magnesium hydroxide phases were observed

of 460 kJ/mol. Possibly during high-energy mechanical milling, the necessary physicochemical conditions are created for different types of events to occur within the container of the milling system that allow the solvent to dissociate into its elemental components. It should be mentioned that these values are average energies because the energy of a given bond slightly depends on the other atoms bonds in a given compound. We suppose that the preparation of Mg and Ta compounds using mechanical milling is influenced by general chemical properties for example, the melting points, electronegativities, atomic radius, etc. The Ta and Mg metal atoms have few electrons in their valence shells, usually 5 and 2 respectively. In these circumstances it is likely that the atoms easily lose these electrons (valence electrons) during the milling process to become positive ions. When the solvent dissociates into its elements oxygen, hydrogen and carbon ions are formed, then the oxygen and hydrogen ions probably remain as negative ions and they are arranged in the space and form compounds upon impact the hydrogenated phases. The carbon rests in its elemental form and mixes with the other compounds without reacting with Ta, Mg or its oxides, but the concentration is so low that the analytical methods did not detect it. In these experimental conditions, the hydrides formation may have occurred because some of the elements, i.e., Mg, Ta and subsequently their intermediate compounds formed during milling acted as catalysts that improved the reaction conditions that caused the dissociation of the control agent into its components, H, O and C. Another possible cause of the formation of different phases of magnesium and tantalum is the particle size reduction as a function of milling time. A smaller particle size facilitates the formation of tantalum hydrides as shown in the diffractograms. It could be a combination of different events occurring within the milling process that encourage the methanol dissociation. It is interesting to note that the tantalum hydrides phases were synthesized "in situ" during the short milling time. By varying the milling time, different phases of tantalum (oxides and hydrides) were formed, the oxides changed to hydrides, and the magnesium hydroxide phase was identified during less than 20 h of milling. These results suggest that another alternative method is available for preparing some "in situ" metal hydrides at low temperature and practically normal pressure, while simultaneously having a source of hydrogen from

the conversion of methanol or any other solvent during the mechanical milling process. In these experimental conditions with the different milling times used in this work, magnesium hydride was not synthesized; it appears that the mechanical milling was selective and only obtained tantalum hydride. On the other hand, it is very difficult to measure the local microscopic temperature during milling because of the dynamic nature of the milling process. In our system, an external temperature measurement was performed on the top cap of the vial using a digital thermocouple; the results of monitoring this parameter during the different milling times show a gradual increase in temperature. In this working condition, the initial temperature was 18°C (room temperature), and after one hour of milling, it increased to 31°C. As the milling time increased to 5 h, this one was of 45°C. Another series of experiments was performed by controlling the temperature by blowing air over the external surface of vial during milling to maintain the temperature as low as possible. The recorded temperature throughout during the milling time was 27°C from the initial time until 5 h of continuous milling. The increase in temperature is not generated throughout the powder, it is only at the site where the impact between the milling media and the material occurred; therefore, we suppose that in this particular case, the local temperature does not influence the compounds preparation process because the same results were obtained for all the milling times tested, and the dissociation phenomena of the control agent into its elemental components and the chemical reactions that occur with the Ta and Mg metals that results in their corresponding phases remained unchanged. Although the exact mechanism of the phenomena that occur within the container should be considered case by case, it seems that the mechanical milling processes in solid materials are mainly the results of structural changes. In effect, a ball mill with a high energy produces a wide variety of defects due to the collision of the balls, which results in continuous fragmentation, coalescence, vacancies, dislocations, and others events at the collision sites; high pressure is also generated in the compound during the milling process. It has been reported that prolonged milling can also destroy the crystallinity of the material, which leads to partial or complete amorphization [24]. On the other hand, when the milling includes a solvent, dissociation may occur in each of its components and then react with the mixture of metals inside the sealed vial, which causes the formation of chemical compounds including

hydrides that depend on the type of solvent and ball mill used. During the last two decades, a number of promising new materials have been developed and studied. However, still no materials satisfying each of the main targets of storage capacity, thermodynamics and kinetics aspects have been found, so further research in this field is required. Developing hydrogen storage in solid materials having high volumetric and gravimetric densities is the long-term goal for a hydrogen economy. These efforts will involve combinations of theory and experiments, development and improvement of novel methods for synthesis, and “in situ” methods and developments of strategies and combinations of different techniques for hydrides preparation. Many research studies are currently under way to develop new technologies to prepare new materials for use in hydrogen storage. Metal hydrides are attractive candidates for a safe way of storing hydrogen for a broad range of practical purposes such as portable, mobile or static applications. However, more research is needed to develop metal hydrides preparations using alternative methods that meet all the technological requirements.

#### 4. CONCLUSION

The mechanical alloying technique can be used to obtain a few metal hydrides “in situ”. Because of the high impact energy between the components in the container, the necessary conditions to dissociate methanol which was used as process control agent, into its elementary components were created. The oxygen and hydrogen gases were reacted with the Ta and Mg metals “in situ” inside the container. Intermediate phases composed of mainly oxides of the two elements (Mg and Ta) were formed and subsequently transformed during the mechanical milling process. Short milling times were utilized to obtain the hydrogenated phases. Hydroxide and hydride phases were the final products after 20 hours of milling for Mg and Ta respectively. The tantalum hydride was readily formed. Mechanical ball milling can be used as an alternative method to prepare some hydrides; with an appropriate catalyst, the hydride preparations using a variety of metals can be improved.

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#### COMPETING INTERESTS

Authors have declared that no competing interests exist.

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