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Sintesis de polvo de Titanio por reducción metalotermica

Synthesis of Titanium powder by metalothermic reduction

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Resumen: Se obtuvo polvo metálico de titanio (2,98% en peso O) de tamaños de partículas irregulares y semiesféricas entre 0,5 y 3,5 µm mediante reducción magnesiotérmica de TiO_2 y un proceso de purificación por lixiviación. Se evaluó la influencia de la temperatura, tiempo, tamaño de partícula de TiO_2, forma de magnesio, relación molar de Mg / TiO2. Se diseñaron y usaron tres reactores diferentes que debieron soportar alta temperatura y promover las reacciones sólido-líquido y sólido-gas. La mejor configuración resultó cuando se promovió la reacción del modelo de gas sólido.

Palabras clave: Producción de titanio, Metalotermia, Dioxido de titanio.

Abstract: Titanium metallic powder (2.98 wt% O) of irregular and semispherical particles sizes between 0.5 to 3.5 µm was obtained by magnesiothermic reduction of TiO_2 and a leaching purification process. Experiments were carried out to evaluate the influence of temperature, time, size of TiO_2, shape of magnesium and molar ratio of Mg/TiO2. Three different reactors were design and use at high temperature and to promote solid-liquid and solid-gas reaction. The best configuration resulted when solid-gas model reaction was promoted.

Keywords: Titianium production, Metallothermy, Titanium dioxide.

1. INTRODUCCIÓN

Titanium powders can be used as reactive agents in the production of alloys in ferrous and non-ferrous metallurgy; also, in alloying, refining and modifying components to produce titanium carbide and nitride as well as in welding and smelting electrodes. Titanium P/M components can be welded by argon-arc, contact and diffusion welding and processed using metal-cutting machine tools. Titanium powders are readily formed and sintered into components that possess exceptional corrosion resistance, biological inertness, high strength at room and high temperatures, and relative lightness. They are used to produce refractory com-pounds, particulate-reinforced titanium matrix composites, metal–polymer anticorrosive compositions, mechanical components, ceramic-metal porous filters, gas absorbers of electronic vacuum devices, surgical tools, prosthesis, frames for lenses, weapon details, and different components in the automobile, aerospace and chemical industries (Prasad, Ehrensberger, Gibson, Kim, & Monaco, 2015)(Workshop & Pm, 2007)(Yang, Zhang, Dai, & Luo, 2017) (Hurless & Froes, 2002) (Gopienko & Neikov, 2009).In spite of this great properties, titanium is not used commonly in engineering and general applications due to his prices. The high price of titanium is caused by the high technical multi-step production process. Norgate (Norgate & Wellwood, 2006) has estimated that if a low cast production process is developed, reducing price should to be about 50% and the penetration of titanium into new markets could increase to about 220%. The production technology includes different process technology and produced components with a huge range of properties and prices. This variation can be associated with the volume production capacity, cleanliness, handling and kind of process. Generally, prealloy powder (PA) produces high-performance fully dense components but it needs an expensive investment for the special melt stock, ultra-clean handling and compaction tools which increase its price twice as much in comparison with the BE components. Also, for the component produced by each technology the market will be quite different and much more specific. Table 1 estimates the penetration of titanium-powder-made components by the year 2020, when the demand is projected to come close to 18944 yearly metric tons (mt), additive manufacturing (AM) exhibiting the largest projected consumption counts (German, 2009)(Dutta & Froes, 2015)(Froes, 2012)(Bolzoni, Ruiz-Navas, Neubauer, & Gordo, 2012)(Cui, Hu, Zhao, & Liu, 2011)(Vlad, 2008)(Capus, 2017),(Whittaker & Froes, 2015)

Table 1. Projected market penetrations of various PM titanium product types within a 5-year time frame (2020) (Whittaker & Froes, 2015).

Product/technology type	**Sector**	**Tonnes p.a.**
Blended Elemental (BE)	Automotive	6800
Additive manufacturing (AM)	Aerospace, motorsport, medical, others	10000
Metal Injection molding (MIM)	Aerospace, medical, other	45
		2000
Prealloy Process (PREP)	Aeroengineer parts	
Hydride-dehydrided (HDH) material	Automotive, engineering	90
Spray formed products	Surface engineering	9

The recent development of powder metallurgy has proved to be a rapid and economic manufacturing technology which can produce component savings up to about 80% of the total raw material plus energy expenditure. It consumes only half resources in comparison to wrought processes. A synergy between the desired low-cost titanium powder process and the energy and the material savings when powder metallurgy is used could be the way of reducing the final titanium component price leading to a high growth on new application fields. Currently powder production is carried out by high technological processes that use bars of metallic titanium as raw material. In the last two decades, the research race began again to obtain low cost titanium powder directly from its titanium dioxide (TiO_2) or tetrachloride ($TiCl_4$). Electrochemical processes to obtain titanium powder from TiO_2 by reduction through melting calcium or its salts had been researched at the same time by Susuky(Suzuki, Ono, & Teranuma, 2003)(Suzuki & Inoue, 2003) and Fray (FFC process)(Fray, 2001)(Mohandas & Fray, 2004). The Kroll and Hunter processes have improved to produce continuous titanium powder by the Australians (TiRo™ process) (Hongan, L.; McGinn, E.; Kendall, 2008) and the American (Armstrong ITP Process)(Borys, S; Anderson, R. P.; Benish, A.; Jacobsen, L.; Ernst, W.; Kogut, D.; Lyssenko, 2005) respectively. Okabe(Okabe, Oda, & Mitsuda, 2004) researched the direct production of titanium through calciothermic reduction of TiO_2. Unfortunately, nowadays, there is no industrial scale up for these processes to assure a massive production of titanium powder at low cost. This aspect makes this technology more expensive than the production of titanium metal. However, if the powder could be produced directly from titanium dioxide, replacing the expensive steps of titanium metal production by melting and re-melting, its purpose is worthy of investigation. The basis of the idea developed at IME “Process Metallurgy and Metal Recycling, Institute and chair of RWTH Aachen University”, is the direct production of titanium powder by metallothermic reduction from titanium dioxide as can be visualized in Figure 1. The proposed four step process consists in a first reduction of titanium dioxide by magnesium to a low titanium oxygen content, about 2.9 wt%, following an acid leaching step to recover the obtained powder which is used as raw material in subsequent calcium deoxidation process. Finally, another acid leaching step would obtain the low oxygen content titanium powder. The requirement of the oxygen content depends on the application were the component will be used. Possible new daily applications could use titanium with higher oxygen content than that commonly used in high technical application which gives high range on the process control. The magnesiothermic reduction of titanium dioxide and the subsequent acid leaching are the first and second step of the proposed process leading to a high content of oxygen in the titanium powder. This is the focus of this research (Bolívar & Friedrich, 2009)(C. Oosterhof, Reitz. J, Bolivar. R, 2010).

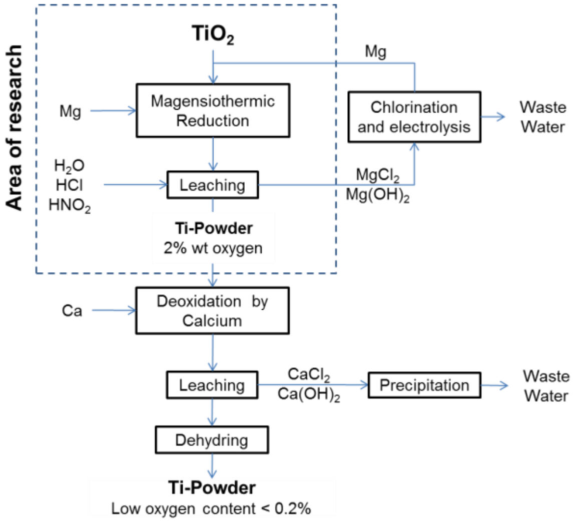

Fig. 1. Proposed process to produce titanium powder by magnesiothermic reduction from Rutile

2. MATERIALS AND METHODS

2.1. Raw materials

The experiments were carried out using pure magnesium of ≥ 99.9 wt% and titanium dioxide powder “Kronos 3000” with the following properties: TiO_2 purity ≥ 99 wt%, size distribution 86 wt% of particles > 100 µm and 0.2 wt% of particles < 800 µm, X_{50}-Value (media size particle) of 200 µm. The studied variables were 1) temperature, 2) molar composition ratio φ, 3) sizes of TiO_2, 4) shape of Mg, 5) reaction time and 6) kind of reactor. Three different reactors were used in order to provide the possibility to investigate the mechanism reaction.

2.1. Magnesiothermic reduction

2.2.1 Reactor I

The first reactor (RI) was made of normal carbon steel DIN 1.0402 (Figure 2) with a capacity of 500 ml and some gadgets on top for temperature measurement and protective gas injection. Argon (2 l/min) was injected into both the reactor and the furnace during the experiments. Magnesium (95 g) in different shapes (in cubes, plates or turnings) and titanium dioxide powder (the amount depends on the molar ratio, labelled “φ”). These were always placed in contact with each other within the reactor. Magnesium was generally placed at the bottom and then TiO_2 was poured on top of it.

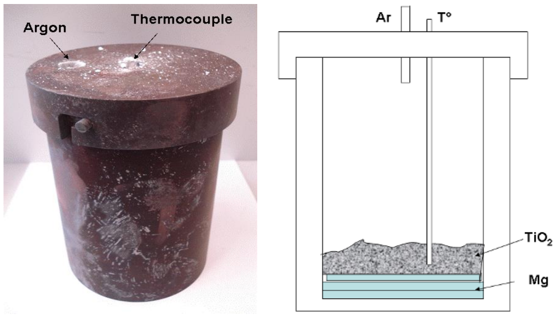

Fig. 2. Reactor I (RI) with facilities to inject protective gas and to measure temperature.

In reactor I three different experimental series were conducted use factorial experimental design statistical methodology. The assessment criterion was the grade of the reduction which was valued by the amount of the strongly reduced product obtained.The following parameters and characteristics were investigated in this reactor:

Starting temperature of the reaction, product distribution in the reactor, influence of the set temperature (710°C or 850°C), influence of the size and shape of magnesium (turnings, plates and cubes in 20 mm wide) or cubes (20*20 mm), influence of the size of titanium dioxide particles (710°C or 850°C), influence of the molar ration φ (2.5 or 4), pathway of the reaction with mixed reagents (liquid-solid mechanism). One replica of each trial was always carried out in order to obtain a minimum statistical concordance. For each experimental series carried out on the different reactors, an analysis of variance was calculated in order to identify the influence of the dependents variables on the degree of reduction.

2.2.2 Reactor II

The second reactor (RII, Figure 3) was built with high elevated-temperature strength stainless steel DIN 1.4841. The 500 ml capacity reactor could support a pressure of 9.8 bars at 1000°C. The reactor was sealed by a blind flange, screws and a gasket seal. Sponge titanium lumps were placed as getter into the reactor, in order to remove the enclosed air by forming titanium oxides and nitrides.

Fig. 3.Sealing reactor II of special steel to support 9.8 bars at 1000°C.

Two different experimental series were implemented the using factorial experimental design. In this reactor the following influences were studied. Reactant agents in separated form (Gas-liquid mechanism on pathway reaction), degree of reduction, influence of the set temperature (850°C or 1000°C), particle size of TiO_2 powder (as received, +355 μm, pellets 1cm), molar ratio φ (2.5 or 4), holding reaction time (90 or 240 min)

2.2.3 Reactor III

The last (RII, Figure 4) design was a tube reactor formed by two different compartments joined by a joint flange in-between (Figure VIII). Each compartment had a capacity of 350 ml. It was made from high elevated-temperature strength stainless steel DIN 1.4841. The reactor was sealed on its ends with blind flanges and screws made out of the same material. A gasket of “Sigraflex® Economy” was used as well. The reactor was designed to support pressure of 9.8 bars at 1000°C. It was heated in a rotary furnace (2 rpm) with facilities to inject argon (2 l/min) as protecting gas. The trials were carried out on an atmosphere controlling rotating furnace. Sponge titanium was placed into each compartment to remove the enclosed air by reaction at low temperature. Three different experimental series were implemented the using factorial experimental design. In this reactor the following influences were studied: reactant agents with each other or in separated form (Gas-liquid and solid-liquid reaction), temperature of starting of reaction at 1030°C and partial pressure vapor Mg of 0.58 bar by special blind plug of nickel vapor gas of magnesium, degree of reduction, influence of the set temperature (850°C or 1100°C), molar ratio φ (2.5 or 4), holding reaction time (90 or 240 min)

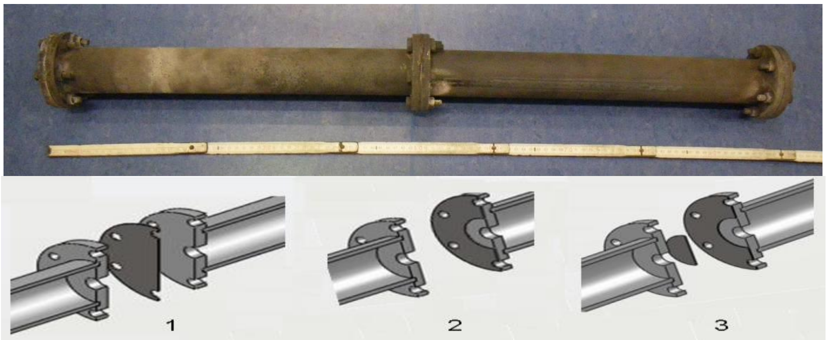

Fig. 4. Up. Reactor III with two compartments. Down. Different configuration for Reactor III.

2.3. Products characterization

Five kinds of particles grouped of different grades of reduction and commonly grouped in the form of sinter cake or placed on a specific place of the reactor. These were obtained during the reduction from different conditions studied. Grey strong reduced particles, black metallic intermediate reduced, black no metallic weakly reduced, no reduced TiO_2 and another one. The strong and intermediate reduced particles are called SRP (strongly reduced product) and the weakly one is WRP (Weakly reduced product). Those products, also SRP and WRP were obtained alone or as a mixture, depending on each particular configuration in the trial. They can be separated by hand and classified by it colour and texture, and where leaching in a different acid solutions. The intermediate product and final obtained powder was analysed by ICP, XRD, SEM, EDX and optical microscopy. The other products were the sub-products of the magnesiothermic reduction; such as, MgO, $MgHN_3$. Titanium dioxide without any reaction was observed as well. The last one was hardly observed.

3. RESULTS AND DISCUSSION

3.1. Products of the magnesiothermic reduction

3.1.1 Strongly reduced product

The strongly reduced products (SRP) exhibit two different colours (Figure 5). The first one is grey silver particles which form a sinter-cake of metallic appearance and is obtained in trials where the reagents are place in contact with each other or separately. The second one is a black metallic detachment powder produced in trials where the reagents were placed separately and only a reaction gas-solid was allowed to run. XRD pattern of the grey silver particle (Figure 6) shows metallic titanium (Ti), a low proportion of sub-oxide (Ti_2O), magnesium oxide (MgO) and metallic magnesium (Mg).

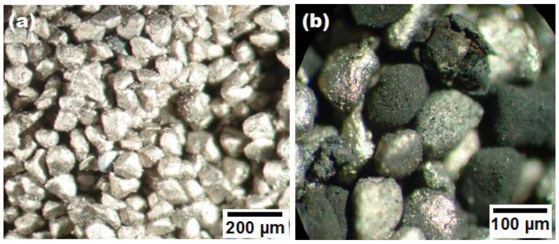

Fig. 5. The strongly reduced product (a) grey metallic appearance (b) black metallic appearance

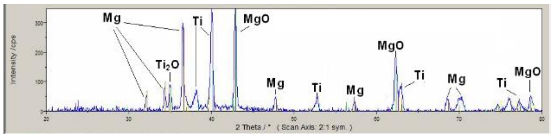

Fig. 6. XRD patterns of the grey silver metallic strongly reduced product.

The XRD pattern of the black metallic shows that titanium metal (Ti) and magnesium oxide (MgO) are its main components, together with low peaks of Quandilite ($Mg_{1.5}Ti_{1.5}O_4$), titanium oxide (Ti_2O_3) and suboxide (Ti_2O) (Figure 7). Although the peaks of Ti and MgO phases show higher intensity in comparison to the $Mg_{1.5}Ti_{1.5}O$, Ti_2O and Ti_2O_3 ones, the presence of those products suggests a partial reduction in some places.

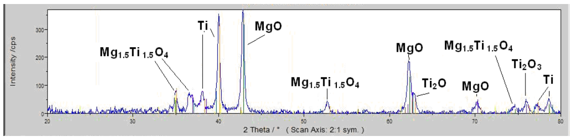

Fig. 7. XRD patters of the metallic black strongly reduced product

3.1.2. Weakly reduced product

This kind of product was obtained together with the strongly reduced product in almost all cases when the reactants were placed in contact with each (reactor I and some configuration of reactors II and III) or as a single product when a nickel blind plug was used on the reactor III. The XRD pattern shows that titanium and magnesium oxide are the principal phases and the quandilite ($Mg_{1.5}Ti_{1.5}O_4$) and titanium oxide (TiO_2 and Ti_2O_3) phases are in a lower proportion (Figure 8)

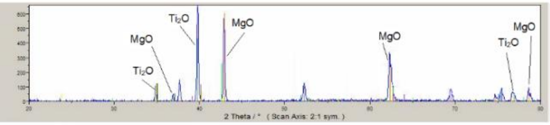

Fig. 8. XRD patters of the weak reduced part of product.

3.2 Impact of parameter variation on magnesiothermic reduction

3.2.1 Influence of the molar composition ratio (φ)

The molar composition ratio was only taken into account when the reactants were in contact with each other (Figure 9). The maximum degree of reduction that theoretically could be reached by the magnesiothermic reduction depends on the molar composition ratio (φ) and reaction temperature lower than 1400°C. If the reactants are in contact with each other, the heat generated during the reduction process is used to melt/evaporate the magnesium bulk and to maintain the self-propagating reaction. The reaction starts invariably at 650°C, the fusion temperature of magnesium. At φ values as high as 4 a big share of the energy generated by the exothermal reaction is used to melt/evaporate most of the magnesium material, while only a minor part is employed to heat the reaction bed. This determines a low reaction temperature, which prevents the re-oxidation of the already reduced titanium. In turn, the highest strongly reduced product (SRP) yield was obtained. Nevertheless, at low φ = 2.5, the effect is opposite; a big share of the generated heat is used to evaporate magnesium and increase the temperature of the TiO_2 bed, even coming close to 1400°C. This leads to a reoxidation of the evolved titanium to its sub-oxides, thus resulting in lower amounts of SRP.

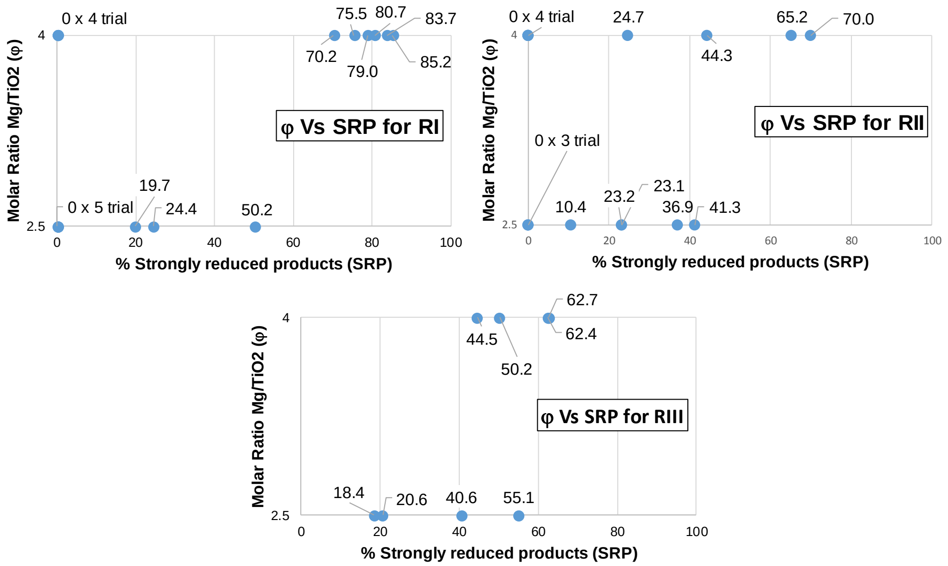

Fig. 9. Influence of the molar ratio Mg/TiO2 (φ) on the strongly reduced product (SRP).

The analysis of variance (Anova) for the RI and RII experimental series shows that the molar composition ratio influences the SRP at fairly low probability rates (44.7% for RI and 51.7% for RII), which implies that the independent effect of this variable on SRP is relatively weak. However, in the case of RIII the probability that φ influences the produced amount of strongly reduced product (SRP) is 99.4%, which implies a strong independent effect on SRP.

3.2.2 *Set temperature and holding reaction time*

Set temperature and time for preheating were investigated for every reactor and can be considered as a “soaking time and temperature”. The set temperature was fixed by the experimental plan and achieved by adjusting the furnace temperature. The holding reaction time started when the furnace achieved the set temperature. Based on the experimental results obtained from RI, RII and RIII and their corresponding statistical calculations, it can be concluded that holding time and set temperature have no influence on the amount of strongly reduced product obtained in those experiments in which the reactants were in contact with each other. The analysis of variance for the experimental series of RI and RII showed that temperature influences SRP at a fairly low probability rate (51.1% for RI, 61.7% for RII and 79.8% for RIII), thus implying a relatively weak independent effect of this variable on SRP. In the case of holding time in RII, this effect was 61.7%.

3.2.3 The shape of magnesium used for reduction

The form of magnesium becomes important for the degree of reduction only when the reagents have been put into contact with each other. Magnesium was used as cubes, plates and turnings in RI. The analysis of the result of first experimental series for RI and the anova calculation for the second one show that the influence of shape of magnesium over of SRP obtained is 100%. Due to this fact, only plates of magnesium were used on RII and RIII. The other shapes as cubes or turnings never obtained the SRP as can be observed on Figure 10.

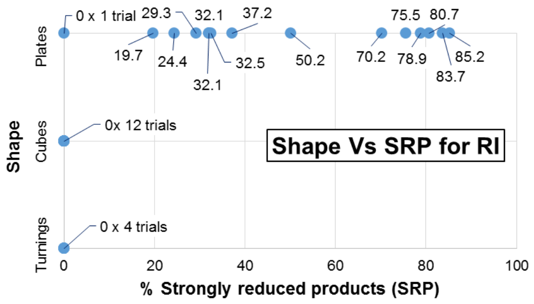

Fig. 10. Influence of the shape of magnesium on the percentage of strongly reduced product.

When plates are used, an initial liquid-solid reaction model occurs only in the small portion of the TiO_2 bed that is in contact with the sides of the plates. The heat thus generated is enough to both melt the quasi-liquid magnesium and evaporate a part of it. The vapor flows through the bed and a bubble fluidized bed structure which produces SRP is evolved, thus conforming to a gas-solid reaction model. The heat generated by the reaction gradually evaporates the remaining liquid magnesium or is lost through the reactor's wall. This prevents both a large temperature increase in the bed and, consequently, the re-oxidation of already reduced titanium. The excess of magnesium vapor is condensed on top of the bed, and a liquid-solid reaction starts again. Also in this case, titanium forms a metallic coating, this time because the heat is not enough to re-oxidize it. Thus, a strongly reduced product is obtained.

3.2.4 *Particle size and form of the titanium oxide*

TiO_2 particle size only had a strong influence (99.9%) on the obtained SRP when the reagents were in contact with each other (Figure 11); there was no movement and plate shaped magnesium particles were used. In these conditions, particles > 355 µm ensured a higher void volume than smaller ones and the process yielded up to 70% SRP. Provided that magnesiothermic reduction in those trials are dominated by the gas-solid reaction model, and that the void volume has a considerable effect on the behavior of the bed with regard to the flow of the reactive gas, it follows that the effect of size can be observed and demonstrated statistically. However, in other experimental configurations where pellets or other particle sizes were used, no influence of size on SRP yield was observed. In RIII (particle size as received), where the trials were carried out in a rotary furnace to guarantee the continuous movement of the TiO_2 powder, a homogeneous reaction was observed through the whole dioxide bed. Some trials rendered 100% SRP, while others rendered only weak ones, demonstrating again no influence of TiO_2 particle size.

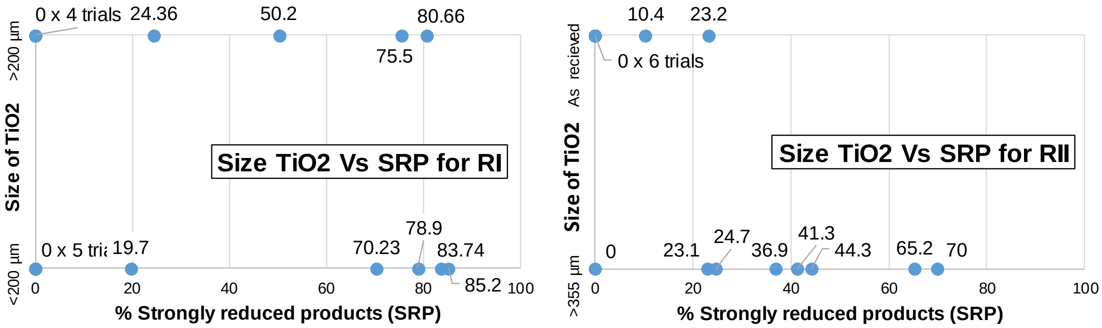

Fig. 11. Influence of TiO_2 size on the strongly reduction products.

3.3 The obtained powder

After a leaching acid treatment leached (8 HCl% + 3 HNO_3%) to purified the titanium powder from magnesium, titanium oxides, magnesium titanates and some elements dissolved from the reactor as Fe, Ni, Cr, the obtained powder was analysed by SEM and EDX. The EDX chemical analyzes show that the oxygen content in the strongly reduced particles exhibit a share from 2.9 wt% to 7.46 wt% with an average of 4.23 wt%. Chloride was not found on the powder. The powders exhibit different morphologies, such as equiaxed as well as spherical particles and their size is on the order of the nanometres. In order to obtain a quantitative value of the degree of reduction from TiO_2 to Ti in the obtained products, the amount of oxygen was fixed as the reference parameter. Provided that TiO_2 contains 40.03 wt% oxygen while grade 1 commercial titanium contains 0.18 wt% oxygen, it was established that a degree of reduction of 0% corresponded to 40.03 wt% oxygen. Correspondingly, a 100% degree of reduction was assigned 0.18 wt% oxygen.

Morphology of powder from RI (Gas Protective Reactor)

At lower temperatures, such as 850°C and short times of 30 minutes titanium metal was formed with a high content of oxygen (about of 12.7 ± 3 wt%; corresponding to a reduction degree of 68.58%). The titanium particles (Figure 12a) exhibit a distribution size from 0.1 μm to 1.4 μm. Their general shape is semi-spherical but some of the bigger ones exhibit an angular morphology. Porosity produced by nucleation and releasing of MgO could only be observed on the bigger particles. The size and quantity of small particles suggest a quick transformation to the TiO_2 to Ti metal, which agrees with the pathway proposed and the chemically driven consideration. The EDX shows that the oxygen content is approximately 12.7 wt%, leading to a degree of reduction of 68.58% (Table IV).

Morphology of powder from RII (Sealed Reactor)

The titanium particles of the SRP (Figure 12b) exhibits a distribution size from 0.28 μm to 0.96 μm. Their shape is similar to RI but the semi-spherical shape is more abundant than the angular ones. The EDX shows the presence of oxygen and magnesium. The oxygen content is approximately 4.02 wt% (90.36% degree of reduction), with magnesium only on point 2 with 1.98 wt%.

Morphology of powder from RIII (Two Compartment Reactor)

Three different morphologies were observed in the SRP obtained using Reactor III, depending on variations in the processes, as described in the following:

Equiaxed particles: The particles from trials in which the reagents were in contact with each other were obtained under the following conditions: set time was for 4 hours, molar ratio φ = 4, and set temperature was 1100°C. These particles exhibited an equiaxed form with holes on their surface (Figure 12c) Particle size ranges from 0.5 μm to 3.5 μm. The SEM micrograph renders a facetted growth of the titanium particle from the prior magnesium titanates particle. The prior ones are decomposed on both MgO and titanium particles. The MgO particles are released leaving behind a high porosity of octahedral holes on the surface, which is then transformed into titanium metal by the removal of magnesium and oxygen. This micrograph shows larger particles than the other sample; it could be that the smaller particles have been adsorbed by the bigger ones in a ripening process promoted by the high temperature and longer time. The titanium particle shapes are semi-spherical. The EDX shows the presence of oxygen and magnesium. The oxygen content is approximately 4.38 wt% (89.46% degree of reduction), and magnesium is only on point 1 with 2.79 wt%.

Spherical and semi-spherical particles: Two different morphologies (Figure 12d) were observed in strongly reduced products obtained under the same conditions: reagents separated from each other, 4 hours set time, molar ratio of 4 (φ = 4) and set temperature of 1100°C.

The first one of these morphologies corresponded to spherical and semi-spherical titanium particles with a 0.53 µm to 1.93 µm size. They were detached from the continuous matrix structure under a "free flow" behavior mode. Besides those two morphologies, an angular monolithic material was observed, containing an elevated amount of nickel (circa 52 wt%) that suggests chemical reactions between some of the reactants and the nickel contained in the high temperature resistant steel alloy of the reactor´s wall. The EDX shows that the oxygen content is approximately 3.25 wt%, leading to a degree of reduction of 92.29%.

The second morphology corresponds to a continuous titanium matrix spotted by traces of small, detached titanium particles (Figure 12e) that had been absorbed by the matrix. Small cavities on its surface, left by leached MgO were observed as well. This confirms the formation of a continuous matrix resulting from facetted growth by a sinter/ripening process. The EDX shows that the oxygen content is approximately 5.22 wt%, leading to a degree of reduction of 87.35%.

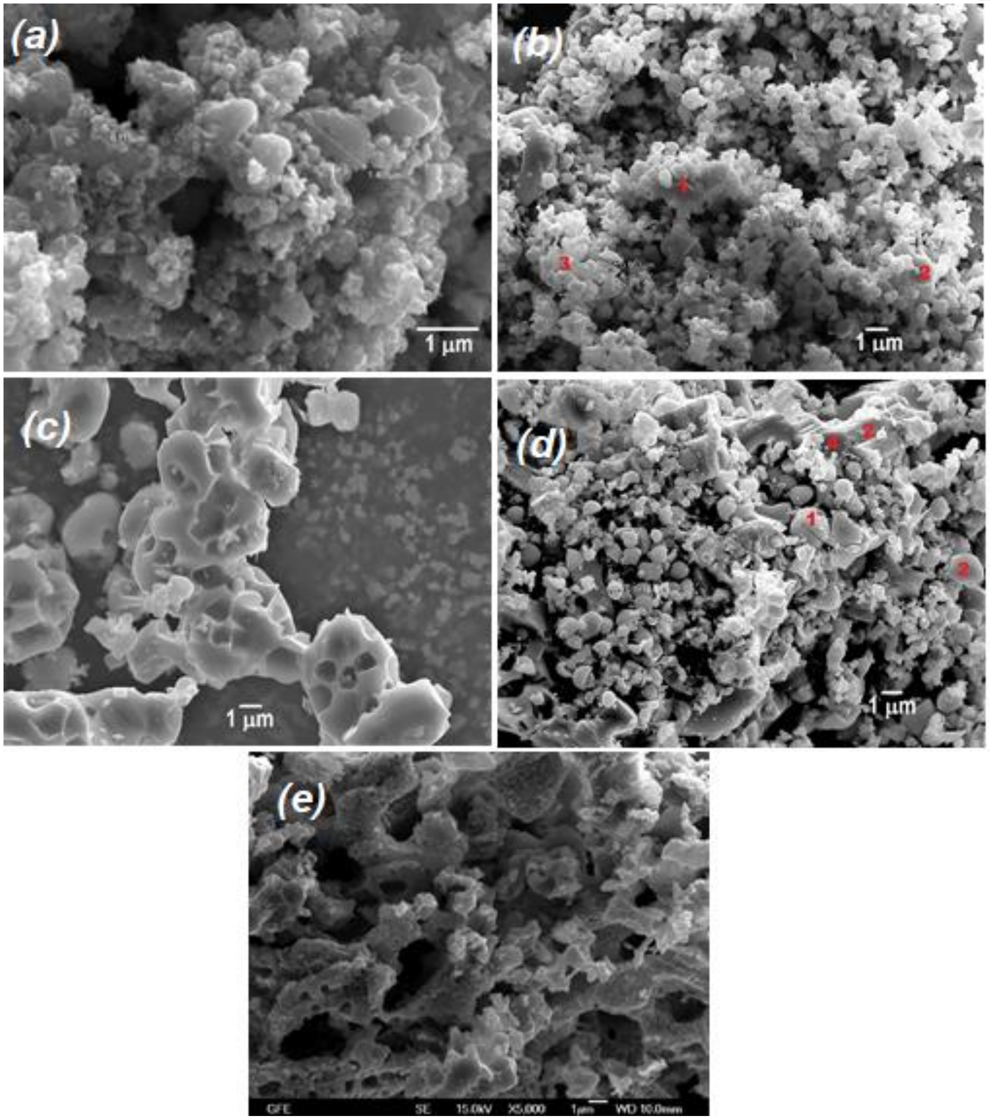

Fig. 12. SEM of the SRP powder obtained from (a) RI; (b) RII, (c) RIII equiaxed, (d) RIII semispherical (e)RIII continuous

4. CONCLUSION

In the second configuration of rotary tube reactor, where the reactants were separated with each other through a rotational movement and the incipient magnesium gas was instantly allowed to react with the titanium oxide, 100% strongly reduced product (SRP) was obtained. Under this configuration, the amount of evolved gas depends only on the increase of the furnace temperature, thus entailing that only a small fraction of the gas is available for the reaction. Furthermore, the movement ensures a homogenous reaction and distribution of the products. On the other hand, when magnesium gas was allowed to react at 1030°C (0.68 bar) and the available gas was enough to complete the reaction, only weakly reduced product was obtained.

Among the variables studied when the reactants were in contact with each other, such as size of the TiO_2 powder, set temperature, holding time, shape of the magnesium, time and molar ratio Mg/TiO_2 only both shape of magnesium and molar ratio Mg/TiO_2 influenced the degree of reduction. The shape of the magnesium is directly related to the reaction area. The use of cubes or turnings result in a high flash reaction under a gas-solid model generating high temperature on the reaction bed which produces only weakly reduced product by re-oxidation of the titanium obtained at the beginning of the reaction. Plate with lower reaction area results in a small share of liquid-solid model and a maximizing the gas-solid one, obtaining more quantity of the strongly reduced product. However, the evaporation of magnesium takes more time and conduces the magnesiothermic reaction slower than the other shapes, sure a high quantity of strongly reduction products.

The semi-spherical titanium particles obtained was approximately of 0.5 μm size with a content of oxygen on an average of 3.25 wt% on average, corresponding to a degree of reduction of 92.3% referring to TiO_2. Lower oxygen concentration was reached in some trials to about of 2.9 wt%, degree of reduction about 93.17%. No chlorine contamination was detected via EDX. The shape of the titanium powder was not homogeneous, and it depended on condition of the trial. Generally, small and detached equiaxed and semi-spherical particles were obtained when reactants were in contact, but when they are placed separately, a continuously sinter structure of big and small particles was obtained. The rough surface structure of the powder morphology results from the formation and releasing of magnesium oxide micro particles on the surface of the raw particles. Very small particles of titanium metal make this sub-micron powder hardly usable for the blended elemental powders (BED) and pre-alloyed powders (PE) processes, where the requirement size is bigger than 25μm, they could be ideal to use on both Powder injection Molding (PIM) or Metal Injection Molding (MIM).

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