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CRITICAL ASPECTS
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OF NEW TECHNOLOGIES
A CASE STUDY

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Critical aspects in the management of new technologies: A case study

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ABSTRACT: The good and effective management of a new technology is fundamental for its success not only among the scientific and technical community, but especially for its entry and expansion in the market.

There are many examples of new product or process technologies that, notwithstanding the technological relevance and many chances to succeed, have found many obstacles to their dimensional development due to a lack of management of technology in their specific sector.

The aim of this paper is to analyse the case of many new process technologies developed along 50 years in the titanium metal sector where some critical aspects and mistakes in the management of these new technologies have limited or stopped their complete development and the following introduction in the sector. Some useful elements will be pointed out in order to reach more general remarks.

KEYWORDS: Technological Barriers, Management of Technology, Electrolytic Process, Titanium.

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INTRODUCTION

There are several cases of new product or process technologies that, notwithstanding the many chances to succeed from a technological point of view, have found many difficulties to be completely developed and applied due to a lack or to some critical aspect in the management of the new technology: the process technologies developed along the last 50 years for obtaining the titanium metal are one of these cases and will be analysed in this paper.

The titanium sector is very limited compared to that of other metals that are equally obtainable from minerals and often give lower performances, for example the quantities of Titanium produced every year are about 1/400 of those of Aluminium.

Titanium is present on the earth's crust in the form of minerals and compounds in the proportion of 0,6% mass, is the fourth most abundant metallic element on the earth's surface among structural metals after aluminium, iron and magnesium and is in ninth place overall; it could be placed, with regards to production, consumption and application, between the stainless steels and aluminium alloys.

In fact, titanium's excellent chemical-physical-mechanical characteristics make its application possible in very different areas, especially when the environmental conditions are particularly difficult and aggressive: aircraft constructions, chemical and electrochemical plants, petrochemical industries, energy and power plants, water desalination equipments, marine installations, food industries, biomedical equipments, civil engineering constructions, archaeology and restorations, sport equipments, mechanical industries in general.

Notwithstanding these wide possibilities, titanium is still considered a "precious metal"

and more than 50% of its little production is used in civilian and military aircraft and spacecraft with the function of structural metal (jet engines, airframes components, space and missile applications).

However, as opposed to stainless steels and aluminium alloys, titanium has a far more recent history, both with regards to its discovery and, above all, to its industrial and commercial development.

In fact, the titanium minerals were discovered only in 1790 and the development of the first production process for obtaining the metal from the titanium dioxide (TiO_2) contained in the mineral dates at 1910, due to its strong tendency to react with the oxygen and nitrogen in the atmosphere. What made it possible to overcome this problem was the introduction of thermo-chemical processes with sodium (Hunter process, 1910) or with magnesium (Kroll process, 1937) which allowed to obtain titanium metal in the form of "sponge".

This kind of processes are still used nowadays and represent the most critical moment of the whole titanium *filiere*, because are expensive to run, erratic and cannot be automated, therefore they cannot be improved technologically anymore.

One solution to this limit of a technological nature must thus be sought in a radical innovation of the metal productive process to obtain titanium automatically with a consequent reduction in costs and therefore in prices, and this could result in an enormous impulse on its application in new outlet markets. However, despite the numerous attempts of new technologies performed until today in the last 50 years none has reached the industrial production.

One of the main reasons has to be researched mainly in a lack of "Management Of Technology (MOT)" in handling these new technologies within the intense cyclical fluctuations of the titanium market.

Furthermore the strategies of the sponge producers, which have always been based on a high added value on low quantities of product, have used the pretext of the technological obstacles to continue their activity with obsolete technologies and do not invest in new ones.

1. WHEN TECHNOLOGY IS THE MAIN LIMIT

The original thermo-chemical processes that are still being used for the production of titanium (basically the Kroll process) produce the metal in the shape of agglomerated grains called «titanium sponge». The sponge must then be separated from the reaction by-products, by means of vacuum smoothing or distillation. At the end of the process, it is melted into ingots that are then transformed into semi-manufactured products.

Still nowadays the production of titanium sponge processes represents the most fundamental and critical moment of the whole titanium production cycle: because they are very expensive to run, erratic and cannot be automated, therefore they cannot be improved technologically. Obtaining the sponge is even now one of the major restraints of the whole filiere that goes from the mineral to the metal, in that the processing at the beginning and at the end of the cycle do not present particular technological limits or limits of other kinds compared to the materials in competition with titanium (mainly steel and aluminium).

In particular, the Kroll Process, still used worldwide for producing industrially the titanium sponge, was invented by Dr. Wilhelm Justin KROLL and patented in the 1940s. It consists to the metallothermic reduction of gaseous titanium tetrachloride with pure magnesium metal.

Even if the Kroll process has been improved since its first industrial introduction it still exhibits several technical problems and many of them imply addictive costs; for example the

magnesium and chlorine must be recovered from reaction products by electrolysis in molten salts accounting for 6% of the final cost of the sponge; it only produces dendritic crystals or powder requiring extensive reprocessing before usable mill products can be obtained and wastage of 50% is common in fabricating titanium parts; furthermore residual oxygen and iron content of the final ingot requires expensive and complex refining steps of the crude titanium sponge in order to remove entrapped inclusions accounting for about 30% of the final cost of the ingot.

One solution to titanium limit of a technological nature must thus be sought in a *radical innovation of the metal productive process*: this can take place using a new process so as to obtain titanium automatically and no longer in an erratic way, with lower costs and higher and constant quality and purity; this way could be pursued following the example of the aluminium, which production in the 30's boomed by transition to the *electro-chemical process*. Thanks to this change, a constant reduction in aluminium production costs and therefore in price was obtained, and this resulted in an enormous impulse on its application in new outlet markets. World-wide production of aluminium, that in fact started at similar rates to the current titanium production rates, is today equal to about 400 times as much in volume.

It is estimated that sponge production using traditional erratic methods that are still used (mainly the Kroll process) affects costs to the extent of around 40% more compared to the constant innovative processes (such as the electrolytic one), which are far closer, both regards to conception and operation, to the process used for aluminium production.

Today potential huge markets such as automotive parts are still looking forward to seeing the cost of the primary titanium metal to decrease by 50-70% to use it extensively. Nevertheless, this cost is mainly maintained high because of the expensive old technology used to produce the metal.

2. A REVIEW OF THE ATTEMPTS TO DEVELOP NEW PROCESS TECHNOLOGIES IN TITANIUM SECTOR

Since the early 1970s, for all the above reasons, there have been many attempts of the titanium industry, in synergy with several academic institutes, to find out new routes for producing high-purity and low-cost titanium metal with a continuous process.

Several alternative methods have been examined beyond a laboratory stage or have been considered for preparing titanium crystals, sponge, powder, and alloys, but until now none have reached industrial production. Most of these methods were considered by the authoritative *National Materials Advisory Board committee* panel (NMAB) in the US to be unlikely to progress to production in the near future except “*electrowinning¹ in molten salts*” which seemed to be the most promising alternative route. Today, among the current industrial electrolytic processes, several utilize an “aqueous electrolyte” instead of “molten salts” to electrodeposit metal (e.g., Cu, Zn, Ni, Pb, Au), but unfortunately, *aqueous electrolytes* are unsuitable for preparing highly electropositive and reactive metals such as titanium. By contrast, *molten salt* based electrolytes were already used industrially since the beginning of the 1900s in the electrolytic preparation of important structural metals (e.g., Al, Mg) because exhibit numerous attractive features over aqueous electrolytes, therefore, it has become clear that the most promising route is to develop a *high temperature electrolytic process conducted in molten salt electrolytes*.

However, despite the numerous attempts performed until today with the aim of

developing a continuous process to replace Kroll's process, there are still no available electrolytic processes in molten salts for producing titanium metal industrially. In order to reach industrial success the new electrochemical route must still solve the major issues of the energy demanding and labour intensive Kroll's process and also overcome the pitfalls that have lead to failures until today.

A brief review of the several attempts made in industry to develop the electrolytic production of titanium metal is following, trying to highlight their main weaknesses, strengths and critical aspects.

Since 1950 early work was done by National Lead Industries, Inc. and in 1956 at the former U.S. Bureau of Mines (USBM) in Boulder City, Nev. A small pilot was built to investigate the electrowinning of titanium. It consisted of a 12-inch cylinder vessel lined with pure iron and containing a molten electrolyte. By conducting experiments USBM claimed that high-purity titanium was electrowon with a current efficiency of 60%. However frequent failures of the diaphragm, which is the cell partition that prevents the reoxidation of titanium, that became periodically plugged or loaded with titanium crystals proved troublesome.

In 1968, the Titanium Metal Corporation (TIMET) disclosed that a new electrowinning cell was patented, designed and operated in Henderson, Nev. This electrolytic cell was a unique pilot based on a non diaphragm basket cathode type.

TIMET claimed that later models of pilot-plants have produced up to 363 to 408 kg of titanium metal in one cathode deposit. This semi-works plant produced about 68 tonnes of electrolytic titanium sponge but discontinued the operation in 1968 owing the overcapacity, for the small titanium market dimension at that time, of world plants for making sponge by Kroll's process, that made them think that was not economic to establish new sponge capacity until the market had not increased.

¹ “Electrowinning” is the extraction and preparation of pure metals using an electrolytic process, based on the electrochemical reduction of metal cations present in a suitable electrolyte by electrons supplied by a negative electrode (i.e., cathode), while at the positive electrode (i.e., anode) an oxidation reaction occurs (e.g., anode dissolution, gas evolution, etc.).

In 1971 Hashimoto et al. have worked extensively in Japan on the electrowinning of titanium metal from its oxides or mixed oxide. Titanium solute was introduced in a molten fluoride bath, as a solid compound. The first electrolysis study was conducted at temperatures above 1600° C. with graphite anode and cathode. Molten titanium was obtained but largely contaminated by carbon and oxygen (2-4 %). In other cases, fine titanium powder was only obtained. After the preliminary results, they focussed on the electrowinning of titanium from pure TiO₂ carried out in molten salt baths at 1300-1420°. The titanium electrodeposited was considerably contaminated by carbon owing to graphite electrodes. Best results, with material yield of titanium superior to 95 %, were obtained using other kind of baths, but the purity of the deposit was not as high as that of the common grade titanium sponge required by the industry and it did not still meet the quality requirements of commercial sponge.

In 1973 D-H Titanium Company was founded by the Dow Chemical Company and the HOWMET Group, for producing continuously high-purity electrolytic titanium at Howmet's plant in Whitehall, Michigan. The cell operated at 520° C under argon atmosphere with a molten salt electrolyte. According to Cobel et al., the direct current required for electrowinning (17.4 kWh/kg) appears to be only about half that required for the Kroll process. Although titanium sponge of apparently satisfactory purity was claimed to be produced in relatively small pilot-plant cells they were not able to overcome a titanium capacity of 86 kilograms per day and the electrowinning of titanium was still far from an industrial scale adequate to a growing market.

At the end of 1982, according to the *American Metal Market*, the expenses for completing the joint program and the economic climate at that time have forced the dissolution of the D-H Titanium Company. The Dow Chemical

has continued some research and development work on the electrolytic process but without success, while Howmet has later focused in the metals fabrication area.

In 1985, the Italian company Elettrochimica Marco Ginatta S.p.A. (EMG), claimed a new upgraded electrowinning process inspired from the previous attempts, using always the dissolution and cathodic reduction of titanium tetrachloride in an electrolyte made of alkali or alkaline-earth metal halides and the electrodeposition of the dissolved titanium cations. The process was supported by the American company RMI Titanium, which built a pilot plant in Ohio (US). Ginatta claimed that the current production capacity of this plant reached 70 tonnes per year in 1985. Unfortunately, in 1990 RMI closed the plant owing to inability of investing other money to complete the resolution of "engineering issues" in concomitance with the coming big world economic crisis which determined also the closure of its traditional Kroll plants.

Between 1997 and 2000, Kawakami et al. have proposed an electroslag remelting process, based on direct electrowinning of liquid titanium metal, in order to avoid common dendritic electrodeposits by producing the electrodeposited titanium metal in its liquid state. It can be seen from the published results that unfortunately most of the deposit was obtained as titanium carbide and not in the liquid state, so that the current efficiency for the reduction was only 1.5%.

In 1999, the process was improved; pure titanium metal pieces were obtained in the solidified salt after this run with the bigger electrode distance. It was concluded that the electrowinning of liquid titanium metal was possible if sufficient heat to form a metal pool could be supplied at the bigger distance between the electrodes. This process (DC-ESR) was patented in 1988, reconducted in 2000 and later presented at ECS (Electrochemical Society) meeting. However,

the formation of an intermediate, oxygen containing species with high melting point, precludes the use of temperatures relatively close to the melting point of titanium, to electrowin molten titanium from titanium dioxide.

More recently (2000-2004) the idea to use a molten pool of titanium was also claimed by the Italian company Ginatta Torino Technology (GTT) who patented a new process for electrowinning titanium based on the recovery of the molten metal using, like for aluminium, a pool of liquid titanium as cathode.

The main idea of GTT was to avoid common dendritic electrodeposits by producing the electrodeposited titanium metal in the liquid state such as for aluminium. The process operates at 1750° C with the dissolution of titanium tetrachloride into a molten salt electrolyte made of calcium fluoride CaF₂ and containing calcium metal. The work is in stand-by phase at present due to a lack of financial support.

In 2000, the Defence Evaluation and Research Agency (DERA) at Farnborough (Hampshire, U.K.), had conducted and patented early trials based on early results obtained by Fray, Farthing, and Chen (FFC's Process) at the Dept. of Materials Science of the Cambridge University. The process claims the electrochemical deoxidation of solid titanium dioxide that was originally applied for refining titanium metal by Okabe et al. in 1993.

The process has been partially demonstrated only in a bench-scale reactor (i.e., 1 kilogram of titanium per day) and there are several important pitfalls to be overcome in scaling-up the process for a future commercial development. Primarily, it is strictly again a non-continuous process, requires expensive preparation and long times, has an extremely low space time yield.

Another attempt in 2000 was carried out by Sharma, who proposed the calciothermic reduction of pure titanium dioxide with a

zinc-calcium alloys performed in a molten salt mixture at 800°C. Titanium powder was later recovered from the alloys formed by vacuum distillation which is anyway highly energy demanding and consequently too expensive.

In 2001, Fortin proposed a process for obtaining titanium metal from ilmenite using a so-called 'shuttle-alloys'. The process which comprises two consecutive steps requires expensive materials and some having environmental issues for an industrial process and is also energy demanding.

Always in 2001, Pal et al. from Boston University suggested and patented a new way for electrowinning reactive metals including titanium using a solid oxide membrane (SOM) process. The method consists to electrolyse a molten salt electrolyte containing the cations of the metal to electrodeposit at the cathode. No further information is available.

From 2001 François Cardarelli of Rio Tinto Iron & Titanium Inc. has developed a method for electrowinning titanium metal from electrically conductive titanium mixed oxide compounds in the liquid state², acting as cathode, covering the cathode material with a layer of molten salt electrolyte (United States Patent n.7504017).

There is no information available on whether Cardarelli's method has been implemented at the pilot plant scale yet. Probably the very high temperatures involved in the process are a challenge to material selection for equipment construction. Also the presence of oxygen containing compounds at the location where titanium metal is formed makes it difficult to produce high purity metal.

² Such as molten titania slag, molten ilmenite, molten leucoxene, molten perowskite, molten titanite, molten natural and synthetic rutile or molten titanium dioxide.

3. MAIN CRITICAL ASPECTS IN THE MANAGEMENT OF THE NEW PROCESS TECHNOLOGIES

A lot of work and many studies have been carried out on the cells for the electrolytic production of titanium in molten salts, but none of the numerous attempts described to develop equipment on a pilot scale has been able to reach the industrial scale. The lack of a proper system specifically designed for this purpose has consequently enabled a full development of this new process technology.

Many reasons can be found, but they can be traced back mainly to:

1. from a scientific point of view: the high electrochemical complexity of the titanium system cannot be fully tackled with the present insufficiently developed fundamental electrochemistry, which persists even today, and does not permit a complete understanding of the electrolytic processes and therefore does not consent the full exploitation of the electrochemical plants;
2. from a managerial point of view: a lack of "Management of technology and innovation" in handling the intense cyclical fluctuations of titanium market with boom periods that are shorter than the time required to complete an industrial R&D program, and bust periods during which no investor wants to finance research, and prototypes and people are dispersed.

These reasons have mainly caused the Titanium industry today to be far inferior to its potential. Analysing the second one, with reference to *a scarcity of Management of technology and innovation*, it is possible to point out the following aspects:

- The technical competence of the financing supporters is almost always a fundamental prerequisite for the success of the R&D

program. For example, Prof. Giulio Natta of Milan Polytechnic could not have created the *polypropylene*, the first man-made material, and won the Nobel Prize in 1963, if his financial supporter, Ing. Giustiniani, President of Montecatini Co., had not persisted in maintaining his support long beyond the other board members and bankers decisions to stop the Natta's research. Without Giustiniani's technical competence and decision, Italy could not have had its only Italian Nobel Prize in Chemistry and the world a very important innovative material.

In titanium sector this kind of MOT approach and determination has not emerged until now in none of the attempts described to develop new technologies.

- From a wider point of view, the need for time continuity of all supports, along the development of the industrial research, the more the work increases in intensity. It is typical that once the corollary problems have been solved, the main ones are to be attacked: however having accumulated much experimental experience on the system object of the research, the researchers are at this time in the best conditions for harnessing and comprehending the more fundamental aspects and for designing experiments really new, which will lead to the discovery of new technologies. In this kind of sectors this conditions occur typically after 4-5 years from the start, generally coinciding with the end of the favourable economical cycle which has promoted the support of the R&D program. At this time, in many of the cases quoted, it occurred the beginning of the about-turn by the boards of directors and/or the annulment of the commitments by the financing supporters, together, as a reaction, with the increase of the intensity of the struggle of the research tasks to try

to advance at maximum speed the R&D work, in order to harvest the most from the performed activities. Finally, in a few months the programs stopped with a grinding halt, with the diaspora of the researchers and the sales of parts of the equipments. The result was very often the impossibility, when the economic conditions had eventually improved again, to restart the R&D programs.

- In some specific research field the level of scientific competence of the researchers, should be elevated not only for the R&D program director responsible for the activities, but most importantly for the co-workers: in many instances this has not been the case of titanium for a variety of reasons, which are all detrimental to the result of the research.
- Since the beginning, the very close tie that has always linked the fate of the Titanium industry to that of the aerospace industry (mainly military) has not push the metal producers to develop an innovative process technology able to reduce the metal price.
The aerospace industry is characterised by relatively low consumption compared to the other manufacturing industries, but it is also characterised by its capacity to bear high purchasing prices for titanium in function of the very high quality required by the aeronautical specifications.

FINAL REMARKS

In conclusion, the policy carried on by sponge producers in the last 50 years has been characterized by high prices, low quantities and the lack or extreme limitation of any technological and marketing effort. With such a policy, that managers and businessmen consider as being a niche and that economists

define as a restriction on quantity, managerial and organisational techniques became stale, along with firm cultures and to face a market different from the original one becomes very difficult. It followed that firm policies hardly considered the development of new application sectors that were much larger and required a less elevated standard of product quality, with the result that the price had to be reduced and new technologies had to be developed in order to favour this price reduction.

Even when it is admitted that oligopolistic firms do not or cannot know the elasticity of the demand, only its increase can bring about an increase in investments, regardless of the shape of the market; however, in the case of a concentrated oligopoly, as with the metal titanium industry, an expansion in demand stimulates production and investments in innovation only if it is sufficiently large as to enable firms to overcome the obstacle of old technologies. It is once more clear therefore that it is extremely important to broaden the market outlets for titanium if new investments in R&D are to be attracted to the sector that contribute to overcoming the technological problems linked to metal production. On the contrary, all the new attempts to develop innovative process technologies risk, as in the past, not to reach the industrial scale, mainly due to both a scarcity of investments in technology and innovation and to the incapability to manage these new investments.

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