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Polycrystalline Powder Synthesis Methods

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Abstract

The synthesis of polycrystalline powder is a key step for materials sciences. In this chapter, we present the well-known methods of preparation of powders such as: solid-state reaction, sol–gel, hydrothermal, combustion, co-precipitation. Moreover, synthesis methods by arc furnace, by heating in a “high frequency” induction furnace and by high energy grinding are presented. The obtained powders could be defined by their purity, grain size, crystallinity, and morphology, which are influenced by the synthesis method. In addition, each method is dependent on some parameters like pH, concentration and temperature.

Keywords: solid-state, sol–gel, hydrothermal, combustion, coprecipitation, intermetallic

1. Introduction

Optimizing the physico-chemical properties of materials is a challenge for many scientific researchers, and it could be altered using different synthesis methods. The synthesis of ceramics is influenced by many parameters; the choice of such a synthesis method is generally conditioned by the morphology as well as by the particle size and the specific surface of the samples, which it is desired to synthesis. Ceramics, when prepared by different routes, exhibit different properties, even with the same starting compositions. There are two ways of synthesizing ceramics:

- Dry method (solid–solid method)
- Wet method (sol–gel, hydrothermal, combustion and coprecipitation methods)

Microstructure plays an important role in the properties of materials. In fact, for ceramic materials, the presence of porosities between the grains has a negative effect on the conductive properties. In addition, ceramic materials made of small grain size are thermally more stable mechanically than samples with larger grain size [1]. For example, barium hexaferrite ($\text{BaFe}_{12}\text{O}_{19}$) could be used either as a permanent magnet or as a recording medium depending on the morphology of the compound, which depends on the route of preparation [2].

A variety of metal oxides, both simple and complex, is prepared by conventional ceramic process. This involves the mixing of metal oxides, carbonates, and their

repeated heating and grinding. This method is used on both a laboratory and industrial scale. Nevertheless, the need for alternative routes to the synthesis of oxides has arisen due to intrinsic problems related to:

- Inhomogeneity of the products obtained by ceramic methods
- Incorporation of chemical impurities during repeated grinding and heating operations. Impurities have an undesirable effect on physicochemical behavior
- Particle size obtained by conventional means is not fine, which makes them unsuitable

To have better control of stoichiometry, structure, and desired phase purity, mild chemical pathways are becoming increasingly important in preparing a variety of oxides, including nanocrystalline oxide materials [3].

This chapter is divided in two parts. The first one is dedicated to powders prepared by chemical methods while the second describes the methods of preparation of intermetallic compounds.

2. Powders prepared by chemical methods

2.1 Solid-state method

It is the simplest and most widely used synthesis method in solid-state chemistry. It consists of making the various solid reagents react directly at high temperature [4]. The reagents used for this method are simple oxides: ZnO, Cr₂O₃, Fe₂O₃, NiO, MgO, fluorides: BaF₂, CaF₂ [5], carbonates: NaCO₃, K₂CO₃ [6], nitrates: CuNO₃ [7] and oxalates: Fe(COO)₂. There are reagents, which are difficult to handle, due to their hygroscopy and high sensitivity to water vapor. For example, lanthanum oxide (La₂O₃), reacts quickly to the air to form carbonates and hydroxides, so it should be calcined first at 900 °C to remove all traces of impurities. The powders are weighed according to the stoichiometric quantities provided by the reaction equation and should be grounded in an agate mortar to obtain the desired phase. It is done in the absence or in the presence of a volatile liquid (acetone) to ensure good dispersion of the grains and good homogeneity of the powder [8]. Then, the materials are subjected to a controlled heat treatment, would to form well-defined crystalline structures. The powder obtained could be annealed as it is or shaped into pellets pressed uniaxially. During calcination, there is release of carbon dioxide or oxygen dioxide and possibly a little water vapor. Successive anneals after intermediate grinding are generally necessary for the reaction to be complete. Synthesis temperatures are generally of the order of 900 to 1000 °C.

Although this method is considered the simplest and most economical compared to other methods, it has drawbacks. In fact, the reagents should be of high purity, because some impurities could have marked effects and do not give the desired phase and therefore, it would influence the properties of materials. In addition, this method leads to the formation of powders with large particles and with low homogeneity. Finally, the synthesis of ceramics by this method requires reactions for hours or even days at relatively high temperatures.

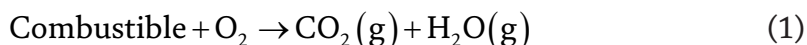
The materials synthesized by this method are numerous, among them, the cuprates A₂BO₄: La₂CuO₄, Pr₂CuO₄, Sr₂CuO₄) [9], the perovskites ABO₃: (CaTiO₃, LaTiO₃, BaTiO₃ [10], the pyrochlore A₂B₂O₇: Dy₂Ti₂O₇, La₂Zr₂O₇, Cd₂Re₂O₇ [11], the brownmillerite A₂B₂O₅: Ca₂(Al,Fe)₂O₅ [12].

2.2 Combustion method

The combustion method is a synthetic route for ceramics that requires less calcination compared to the conventional solid–solid method. It is simple, economical, and fast. It is interesting from research on the mechanism of the physicochemical processes involved, the dynamics of product formation, their limit of stability and process control.

Indeed, the method of combustion or synthesis by fire is known under the name of self-propagating synthesis at high temperature. To generate fire, fuel and temperature are needed. All these three elements form a triangle of fire. Fire could be described as uncontrolled combustion, which produces heat, light, and ash. The process uses highly exothermic redox chemical reactions between an oxidant and a fuel [13].

For the reaction to propagate on its own, the heat released should be greater than the heat required to initiate combustion. A redox reaction involves simultaneous oxidation and reduction processes. The classic definition of oxidation is the addition of oxygen or any other electronegative (non-metallic) element, while reduction is the addition of hydrogen or any other electropositive element (metal). The general formula of the combustion reaction is as follows:



These reactions are highly exothermic or even explosive. This method is used for the synthesis of refractory materials: borides, nitrides, oxides. Reagents are generally used in the form of nitrates because the nitrate group acts as an oxidizing agent and it has high solubility and allows homogenization of greater product yields. They are mixed, then they are “ignited” by laser, electric arc, or heating resistance. Materials based on hydrazine, urea, glycine, or citric acid form the fuel for this reaction [14, 15].

The combustion method was unexpectedly discovered during the reaction between aluminum nitrate and urea [16, 17]. A mixture of $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ and a urea solution, when rapidly heated to about 500 °C in a muffle furnace, to burn with an incandescent flame producing a bulky white product, which has been identified as $\alpha\text{-Al}_2\text{O}_3$.

2.3 Sol–gel method

Sol–gel process is considered as a soft chemistry method used for the synthesis of ceramics [18]. The sol–gel process is a wet-chemical technique that uses either a chemical solution or colloidal particles (sol) to produce an integrated network (gel). This later is a semi-rigid solid where the solvent is held captive in the network of solid material, which could be colloidal (concentrated sol) or polymeric [19].

The elaboration of materials by the sol–gel process, takes place via reactions of inorganic polymerizations in solution from molecular precursors, generally metal alkoxides: $\text{M}(\text{OR})_n$ where M is a metal of oxidation degree n (for example: Si, Ti, Zr, Al, Sn...) and OR an alkoxyl group. After a drying step, a heat treatment leads to the elimination of organic compounds to form the inorganic oxide material. These reactions lead to gelation and the formation of a gel made up of M-OM (or M-OH-M) chains, the viscosity of which increases over time. This gel still contains solvents and precursors, which have not reacted. The “gel” phase in the sol–gel process is defined and characterized by a solid 3D “skeleton” embedded in liquid phase. The solid phase is typically a condensed polymeric sol where the particles have become entangled to form a three-dimensional network. The reactions allowing this material to be obtained are carried out at room temperature (**Figure 1**) [20, 21].

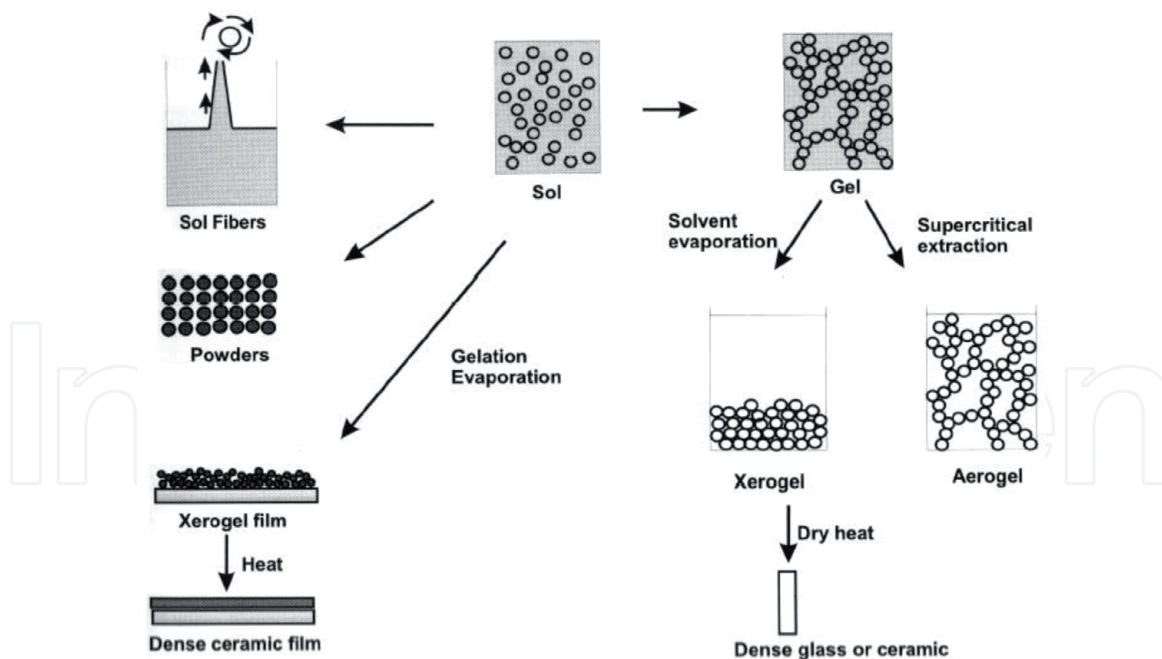


Figure 1.
General scheme of sol-gel process.

The parameters influencing the reactions are the temperature, the pH, the nature of the precursor and the solvent and the concentrations of the reactants. However, the most significant are the pH and the $[H_2O] / [M]$ ratio. The pH influences the morphology of the gel formed. In fact, an acidic pH accelerates hydrolysis and slows down condensation unlike basic pH. A high rate of hydrolysis (acidic pH) therefore promotes network growth and leads to a polymeric solution. Under acid catalysis, which is the fastest synthesis route, the gel formed is called a “polymer gel”: after gelling, an open structure is obtained. However, a low rate of hydrolysis (basic pH) rather favors nucleation and leads to the formation of a colloidal solution. In the case of basic catalysis, the size of the pores is controllable (unlike acid catalysis). The gel formed is called a “colloidal gel” and has a large pore structure (clusters) [22]. Once gelation takes place, the material undergoes drying due to capillary forces in the pores and this drying could result in volume shrinkage. The drying process for obtaining the sol-gel material requires that the alcohol or water could escape as the gel solidifies. The evaporation process occurs through existing holes and channels in the porous sol-gel material. The method of drying dictate whether an aerogel or xerogel is formed. In fact, aerogel is obtained when the liquid phase of a gel is replaced by a gas in such a way that its solid network is retained, with only a slight or no shrinkage in the gel. It was usually achieved under supercritical conditions of the solvent. It is characterized by low density and high porosity. When the drying of gel is occurred by simple evaporation, xerogel is obtained. It may retain its original shape, but often cracks due to the extreme shrinkage that is experienced while being dried. After a drying process, the liquid phase is removed from the gel. Then, a thermal treatment (calcination) may be performed in order to favor further polycondensation and enhance mechanical properties [23, 24].

Although this process requires the use of relatively expensive precursors, it has many advantages:

- The high purity and the homogeneity of the solutions linked to the fact that the different constituents are mixed at the molecular scale in solution

- The controls of the porosity of the materials and the size of the nanoparticles
- The heat treatments required at low temperatures as well as the synthesis of materials inaccessible by other techniques

Several materials were synthesized by the sol-gel process. CeO₂-TiO₂ mixed oxides aerogels with high surface area and stable anatase phase are obtained via sol-gel process [25, 26]. Bismuth barium ferrite nanoparticles doped with sodium and potassium metal ions (Bi_{0.85-x}MXBa_{0.15}FeO₃, x = 0 and 0.1, M = Na and K) were prepared by the sol-gel method by Haruna et al. [27]. Ahmoum et al. [28] synthesized Zn_{1-x}Ni_xO by sol-gel method. La₂Mo₂O₉ nanoparticles were synthesized by Zhang et al. [29] by the sol-gel process using lanthanum nitrate La(NO₃)₃·6H₂O, and ammonium heptamolybdate (NH₄)₆Mo₇O₂₄·4H₂O as precursors.

2.4 Coprecipitation method

Another classic soft chemistry synthesis method commonly developed in the chemical industry is coprecipitation. This method involves bringing into play several metal cations and a precipitating agent, which is most commonly ammonium bicarbonate (NH₄HCO₃) or ammonium hydroxide (NH₄OH). The use of ammonium bicarbonate results in powders, which are less agglomerated than with ammonium hydroxide or urea [30].

The coprecipitation method could be performed by:

- Direct way: addition of a solution of precipitating agent in the solution of metal salts
- Reverse way: adding the metal salt solution to the precipitating agent solution

Synthesis by coprecipitation allows obtaining homogeneous ceramics and a well-controlled particle size. Precipitation is characterized by two main processes: nucleation, the birth of crystals, and crystal growth. These two phenomena determine the size distribution of the crystals. Indeed, nucleation corresponds to the formation of the smallest crystalline entity called “germ” or “nucleus”. Once supersaturation is established (supersaturation is the driving force behind crystallization processes), it takes a certain time, called the induction time, for germs to appear. Crystal growth corresponds to the spontaneous evolution of germs of critical size towards a state of greater stability [31].

The coprecipitation method offers some advantages:

- Fast and easy preparation
- Easy control of particle size and composition
- Various possibilities to modify the surface state of the particles and the overall homogeneity
- Low temperature
- Does not involve the use of organic solvents

However, it has some disadvantages:

- Not applicable to uncharged cash
- Traces of impurities could be precipitated with the product
- This method does not work well if the reagents have different precipitation rates

For example, the synthesis of the $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_2$, cerium and iron nitrates taken in stoichiometric proportions are mixed with stirring. The basic precipitation medium consists of an aqueous solution of ammonia. The precursors are then precipitated dropwise from the reaction medium with vigorous stirring. The solution is left to mature for 2 hours at room temperature before being washed with an aqueous ammonia solution. The powder, strongly hydrated, is then placed in an oven overnight. After a grinding step, a final calcination step at 600 °C for 5 hours, to remove traces of nitrates, would be necessary [32].

2.5 Hydrothermal method

In a sealed vessel (bomb, autoclave), solvents can be brought to temperatures well above their boiling points by the increase in autogenous pressures resulting from heating. Performing a chemical reaction under such conditions is referred to as solvothermal processing or, in the case of water as solvent, hydrothermal processing.

Hydrothermal reactors are mostly metal autoclaves with Teflon or alloy linings or containing an extra beaker or tube made of Teflon, platinum, or silver to protect the autoclave body from the highly corrosive solvent, which is held at high temperature and pressure.

The influence of the conditions of hydrothermal synthesis (pH, temperature, the nature of the precursors or the presence of surfactant) on the morphology and size of the particles has been investigated by several researchers. Indeed, a high pH favors the formation of nanoparticles. On the other hand, a lower pH favors the formation of nanoparticles, which is explained by the evolution of the nature and the proportion of soluble species in solution. The increase in temperature causes an increase in particle size with a change in morphology [33, 34]. The synergistic effect of high temperature and pressure provides a one-step process to produce high crystalline materials without the need of post-annealing treatments. The hydrothermal method becomes useful when it is difficult to dissolve the precursors at low temperatures or room temperature. It proves to be useful to grow nanoparticles if the material has a high vapor pressure near its melting point or crystalline phases are not stable at melting point [35].

In addition, the size of the particles of the precursor is an important parameter in controlling the morphologies obtained. Nanotubes resulting from the hydrothermal synthesis of particles of small grain size (8–10 nm) have been proven to have a larger outer diameter (10–30 nm), thicker layers and an ill-defined tubular shape or incomplete. In contrast, large particles (~ 200 nm) lead to the formation, not of nanotubes, but rather of layers [36].

The main disadvantage of hydrothermal method is the high cost of equipment. However, it has many advantages; it could generate nanomaterials, which are stable at elevated temperatures. In addition, it creates larger sized and high-quality crystals and nanoparticles. In addition, this method could be combined with other process like microwave, electrochemistry, ultrasound, optical radiation and hot

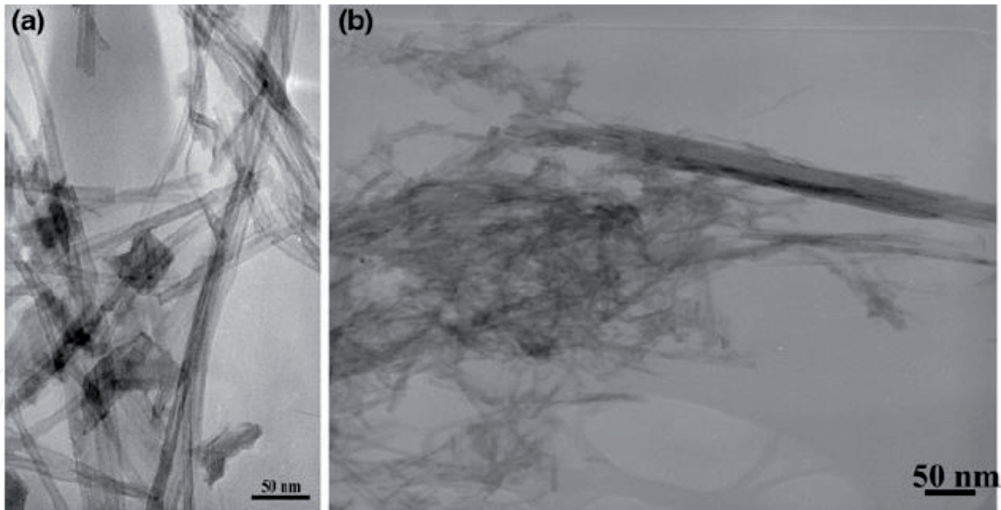


Figure 2.
TEM images of hydrogenotitanate prepared by hydrothermal method (a) nanotubes and (b) nanowires.

pressing. By varying the synthesis conditions (t, T p, etc.) it is possible to vary phase composition and particle size and to change the morphology. Hydrogenotitanate nanotubes and nanowires (**Figure 2**), used as supports for ruthenium catalysts, were obtained by hydrothermal method using highly concentrated NaOH and KOH, respectively [37].

3. Preparation of intermetallic compounds

3.1 Synthesis by arc furnace

The synthesis of the intermetallic compounds was carried out either by induction melting, by electric arc melting or by grinding, from a mixture of pure elements (purity 99.99%). This mixture contains the elements (R: Rare Earth, M: Transition Metal, T: Metalloids) in an amount corresponding to the desired stoichiometry, except in the case of Sm-based compounds for which an excess of Sm (4% - proportion mass) to have a stable phase.

The arc furnace uses the thermal energy of the electric arc established between the tungsten electrode (W) and the metals placed in a copper crucible to obtain a sufficient temperature to melt them (**Figure 3**). The technologies used can be the cold crucible (crucible formed of cooled copper or stainless-steel sectors), the hot

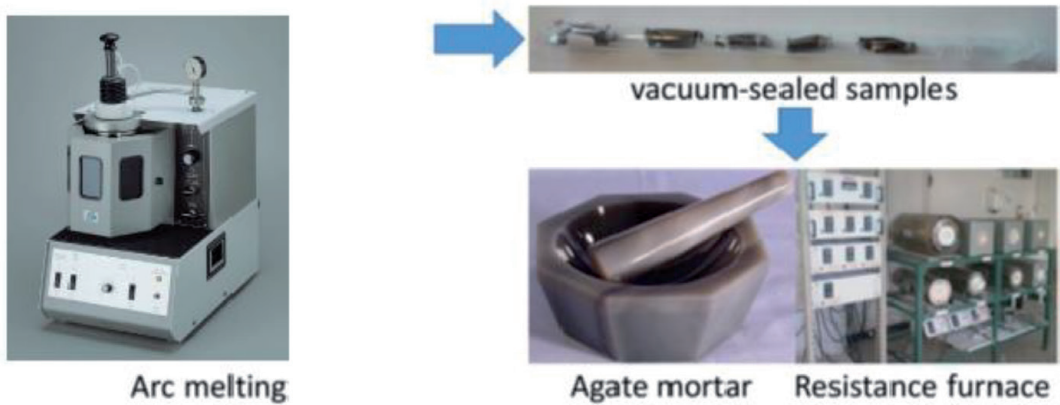


Figure 3.
Synthesis steps using arc furnace method.

crucible (graphite or others) or the use of a susceptor or muffle to radiate on the material to be heated. As a result, cold electrical insulating materials (ceramic, glass, enamels, silicon) can be treated in our induction furnaces. Plasma solutions can be considered as a means of purification of materials. The tungsten electrode and the base copper are connected to a generator that delivers a high intensity current creating an electric arc that could reach temperatures above 3000 °C.

The principle is to create an electric arc between the tungsten tip of a welding machine (Genesis 160, $I_{max} = 160$ A) and the surface of the sample. Care should be taken not to touch the sample to prevent it from sticking to the tip. The electric arc created induces a rise in temperature and the fusion of the various constituents. The crucible is cooled by circulating cold water, under high purity argon gas. Zirconium-titanium alloy was used as an O₂ getter during the melting process, which allows for a sudden quenching when the electric arc is stopped. In order to ensure good homogenization, it is necessary to move the tip to the surface of the sample but also to perform several fusions by inverting the ingot between each heating. Care should be taken to work in a slight argon overpressure and to perform several air-argon rinses in order to purify the atmosphere of the bell. Systematic weighing after reaction makes it possible to assess the loss of mass of the most volatile elements; this is related to the purity of the final products and limits to a certain extent ($\leq 1\%$) the appreciation of the areas of homogeneity.

The arc furnace used could hold approximately 5 g of sample. For larger masses, the use of an induction furnace is necessary. Annealing is a homogenization technique that involves putting the sample at a high temperature for a specified time. Prolonged heating causes an increase in thermal agitation and diffusion coefficients, which allows atoms to organize themselves better, to find an optimal structure corresponding to thermodynamic equilibrium at this temperature.

The furnace used for the annealing is controlled by a Microcor Coreci regulator and the thermocouple is platinum/platinum rhodium (± 5 °C). The bulk was wrapped in a tantalum sheet. Samples were sealed in vacuum quartz tubes and annealed at 800 °C for one week in order to reach a good homogenization and improve the atomic diffusion kinetics. This temperature was chosen as a good compromise between relatively fast diffusion kinetics and absence of reaction with the quartz tube (**Figure 3**).

Intermetallic single crystals of compositions, Er₆Fe_{17.66}Al_{5.34}C_{0.65} [38], GdFe_{0.37}Ge₂, GdFe_{0.27}Ge₂ [39], ErFe_{2.4}Al_{0.6} [40] and polycrystalline SmNi₅ [42], Gd₂Fe_{17-x}Cu_x [43], Nd₂Fe_{17-x}Co_x [44], Er₆Fe_{23-x}Al_x [45], SmNi₂Fe [46], SmNi_{3-x}Fe_x ($x = 0; 0.3$ and 0.8) [47], GdFe_{12-x}Cr_x [48] were prepared by arc furnace. These iron-based ternary diagrams R (Sm, Gd, Er, Nd)–Fe–M (Al, Cr, Cu, Co, Ni) were achieved using arc furnace method [49–56]. The stoichiometric composition of SmNi₅ is confirmed by SEM and XRD analyses (**Figure 4**).

3.2 Synthesis by heating in a “high frequency” induction furnace

The induction furnace or high frequency furnace (**Figure 5**) consists of an external coil inside which a non-inductive copper crucible, divided into sectors and cooled by circulating water, supports the sample.

Prolonged annealing at high temperature under secondary vacuum between 1200 and 1750 °C should be carried out for two reasons:

- To complete the reaction (synthesis)
- To obtain crystals (crystallogenesis)

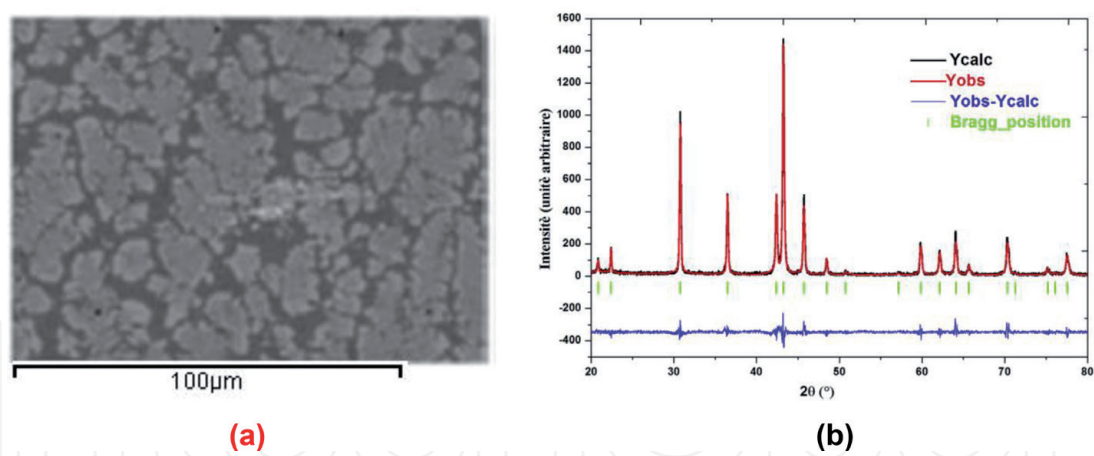


Figure 4.
SmNi₅ characterization: (a) SEM image and (b) XRD pattern and the corresponding Rietveld refinement.

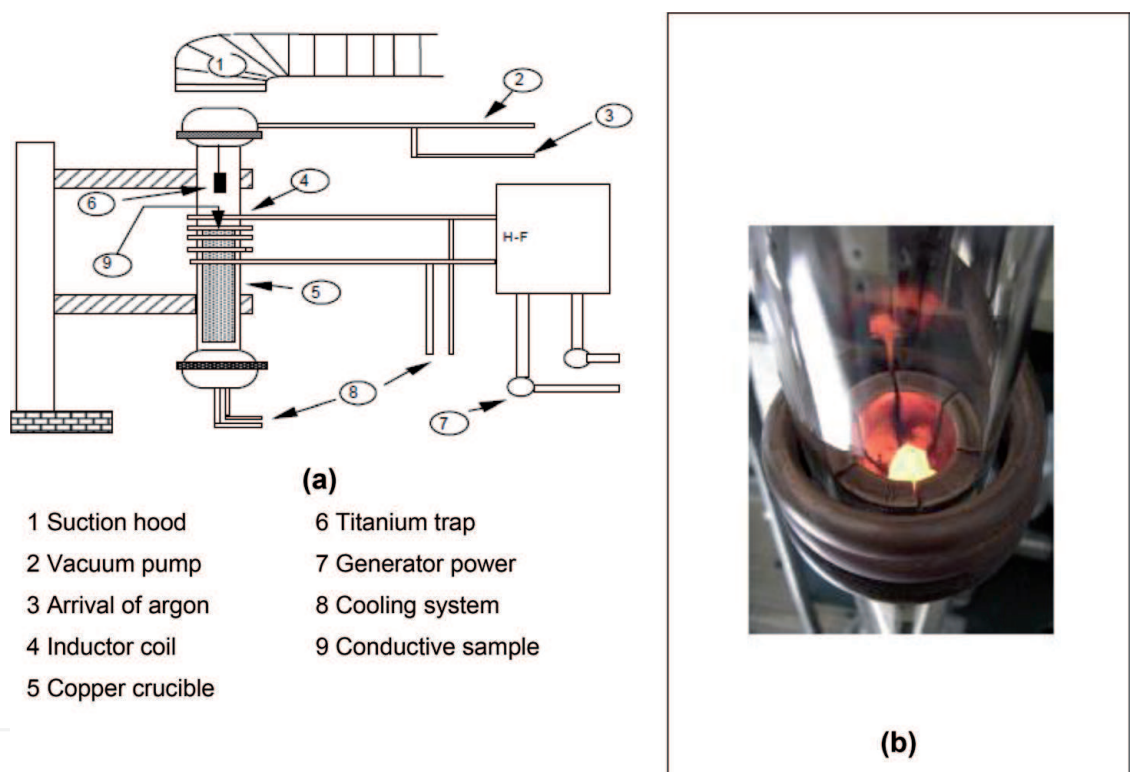


Figure 5.
(a) Diagram of the induction furnace (b) photograph of the induction furnace.

The coil is traversed by a high frequency current (from 10 to 100 kHz), which generates a variation of the magnetic field. Therefore, the metal sample, located in the center of the coil, is subjected to an induced current or eddy current, which causes the metal to heat up by Joule. The sample preparation is identical to that for the arc furnace preparation. The pure metals are placed in the copper crucible.

As the temperature rises, each sector of the latter being cooled with water, any contamination metal through the crucible is avoided. The crucible is protected from the outside environment by a glass tube in which a secondary vacuum is created before melting. This makes it possible to get rid of all gaseous species adsorbed on the internal walls of the tube and on the surface of the sample. The power of the high frequency generator is then gradually increased until all the metals melted to combine with each other. The evaporation of metals is controlled with the manometer indicating the vacuum state. Because of a significant increase in pressure is

observed, argon is introduced into the tube. Five fuses in total are performed with successive turning of the alloy between each of them to ensure the homogeneity of the sample.

The induction furnace could synthesize up to 12 g of massive compounds. Unlike the arc furnace, the melting is less violent because the temperature is controllable. However, the preparation time in the induction furnace is longer because to return the sample between each fusion, it is necessary to disassemble the glass tube and therefore make the secondary vacuum again later to proceed with the next fusion.

The polycrystalline $\text{PrTiFe}_{11-x}\text{Co}_x$ ($x \leq 3$) [57] and $\text{YFe}_{11-x}\text{Co}_x\text{TiC}$ alloys ($x = 0; 0.5; 1; 1.5; 2$) [58]. Ingots were prepared by induction melting under argon atmosphere.

3.3 Synthesis by high energy grinding

To carry out the fragmentation of the particles, it is necessary to set them in motion in suitable equipment. Several studies [59, 60] have been carried out on the grinding of polymers using vibrating fragmentation systems. Even if this technique gives interesting results, this type of device is difficult to extrapolate industrially because of the technological difficulties linked to the vibrating system. Thus, the two crushers used were ball mill and agitated ball mill. These two mills have the same operating principles but the energy transmitted to the powder to effect the fragmentation is different. We would see later that some processes require several tens of hours in the ball mill while they only require a few tens of minutes in the agitated ball mill.

For preparation by mechanosynthesis, the starting materials should be in powder form. This poses a problem due to their sensitivity to oxidation, especially in the case of rare earths, which are very reactive to air. By means of the mechanical composition, it is possible to manufacture amorphous alloys, supersaturated solid solutions of immiscible elements in thermodynamic equilibrium, semi-crystalline compounds, as well as unregulated metal alloys.

The “Pulverisette 4” planetary vario-mill [61] is a mill consisting of an animated plate, with a rotational movement on which two jars are placed; rotating around their axis in both directions relative to that of the movement without rotation of

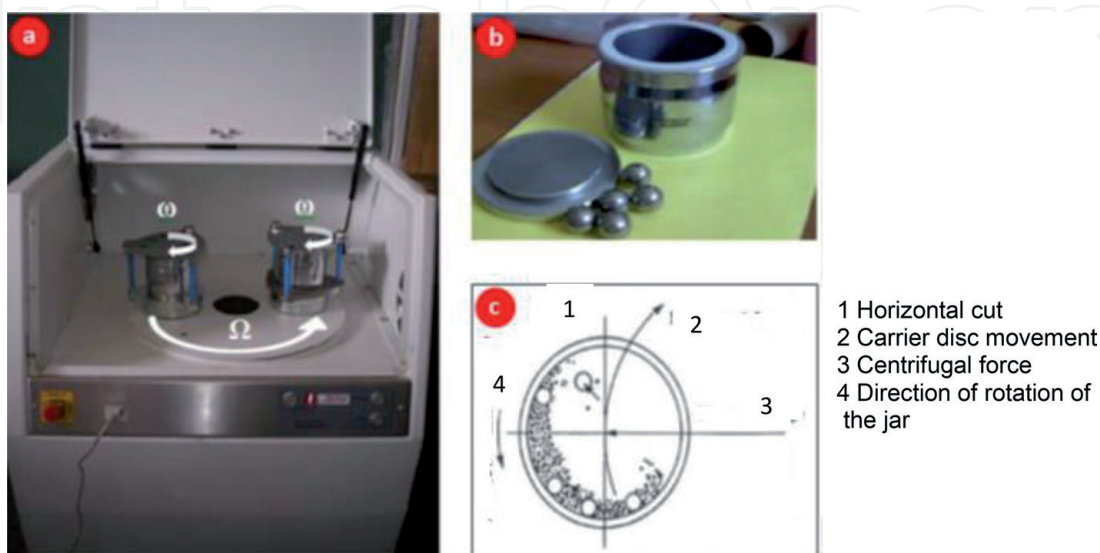


Figure 6. Planetary mill Fritsch (a) planetary mill “Pulverisette 4”; (b) jar and grinding balls; (c) movement of the balls inside the jars.

the plate. The ratio between the speed of the jars ω and the plate speed Ω is called the multiplicity factor, if $\Omega/\omega > 1$ the grinding is in shock mode, and if $\Omega/\omega < 1$ we say that the grinding is in friction. As the directions of rotation of the disc and the jars are opposite, the centrifugal forces resulting from these movements act on the contents of the jars by creating high-energy effects of shocks, friction of the balls on the walls of the jars in all directions and cause the powder to fission. Grinding is carried out discontinuously; it is interrupted every 30 minutes for 15 minutes, to limit the temperature rise inside the jars, as well as to avoid the problems of powder clogging on the walls of the jars, which prevents further crushing. Previous experimental studies have identified the necessary parameters; to avoid increasing the temperature inside the jars, as well as to obtain a satisfactory nanometric powder. The polycrystalline $\text{LaFe}_{13-x}\text{Si}_x$ ($x = 1.4, 1.6, 1.8, 2.0$) compounds were synthesized by high energy ball milling using LaSi as a precursor to prevent oxidation of lanthanum (**Figure 6**) [62].

4. Conclusion

The synthesis of new crystalline materials is a field of intense activity in solid-state chemistry. In this chapter, the most adopted methods of synthesis of the crystalline materials are described. The first part is dedicated to solid-state method, combustion reaction, hydrothermal method, sol-gel process and coprecipitation method. These methods lead to different types of crystalline phase such as organic and hybrids compounds, organometallics, oxides, intermetallic alloys, etc. However, a control of several parameters such as temperature, pressure, etc. is necessary.

The second part is devoted to intermetallic compounds synthesized by arc furnace, induction furnace and high energy grinding methods.

The arc furnace is a system using electrothermal energy provided by an electric arc to heat a load. Electric arc furnaces are used almost exclusively for the manufacture of steel from scrap recovery or arcs develop between each of three electrodes in graphite and metal, this last melt by direct attack bows digging wells in the solid metal charge.

The induction furnace is an electric furnace operating thanks to the phenomenon of metal induction heating. This method presents several advantages; it is clean, energy efficient and allows better control of the metals melting. The capacities of induction furnaces vary from less than one kilogram to one hundred tons; they are used to melt iron and steel, copper, aluminum and precious metals.

Mechanosynthesis is a very high energy milling technique, which allows to obtain metastable crystalline or nanocrystalline phases and to transform crystalline phases into amorphous phases.

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