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Reactive Sintering of Aluminum Titanate

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1. Introduction

The high thermal shock resistance due to the negligible thermal expansion coefficient, additional to its low thermal conductivity and good chemical resistance makes the aluminum titanate (Al_2TiO_5) a suitable material for different technological applications. It is a ceramic material consisting of a mixture of alumina (Al_2O_3) and titania (TiO_2) forming solid solution with stoichiometric proportion of the components: $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$ or Al_2TiO_5 . It is prepared by heating a mixture of alumina and titania at temperature above 1350°C , in air atmosphere. Pure Aluminum Titanate is unstable at temperatures above 750°C , when the solid solution decomposes, following a eutectoid reaction, into two separate phases Al_2O_3 and TiO_2 . For this reason Aluminum Titanate ceramics are doped usually with MgO , SiO_2 and ZrO_2 in order to stabilize the solid solution structure.

Unfortunately, the expansion crystal structure anisotropy that promotes the low thermal expansion coefficients provokes microcracking, as a result of anisotropy of thermal expansion along the three primary axes of the crystal lattice (a single crystal of Aluminum Titanate expands along two axes and contracts on the third one when heated), therefore a low mechanical strength and, on the other hand, the low thermal stability below 1280°C restricts its technical use.

Aluminum Titanate ceramic materials have many technological applications, among others, as thermal insulation liner, soot particulate filter in diesel engines, spacing rings of catalytic converters, in the foundry crucibles, launders, nozzles, riser tubes, pouring spouts and thermocouples for non-ferrous metallurgy and master moulds glass industries.

2. Fundamentals of low thermal expansion coefficient aluminum titanate (Al_2TiO_5) ceramics

There are two important features to achieve a very low thermal expansion coefficient, in crystalline ceramic structures highly anisotropic. The first aspect involves unit cell crystalline chemistry. The coefficients of thermal expansion of the crystal axes are controlled to develop solid solutions, in an attempt to ensure that the sum of the coefficients of the principal axes gives zero. In the case of polycrystalline ceramic materials, the volumetric

thermal expansion coefficient is related to the sum of unit cells coefficients of thermal expansion. In orthorhombic crystal structures as that of the pseudobrookite, material object of this work, the relationship is:

$$\beta_v = \alpha_a + \alpha_b + \alpha_c \quad (1)$$

Where β_v = Volumetric thermal expansion coefficient

α_i = Thermal expansion coefficients of principal crystal axes

As the anisotropic crystalline structures have principal axes with positive and negative expansion coefficients, it is necessary to examine the thermal expansion coefficients of all the members of an isostructural family and chemically design a solid solution whose α_i addition is close to zero. Bayer (1971; 1973), studied the unit cell, of the pseudobrookite structure. Provided that the sum of the thermal expansion coefficients of the principal axes (α_i) add zero, it occurs an inevitable combination of positive and negative values. This condition leads to, very high (at GPa levels), micromechanical stresses at grain boundaries, during cooling from the temperatures of ceramic processing. The development of these internal stresses, promotes the breakdown of the grain boundaries, which causes a decrease in the structural integrity of the polycrystalline ceramic body. However, the existence of this microcracking depends on the microstructural grain size. Kuszyk and Bradt (1973) noted that the rigidity of the ceramic body decreased as increasing grain size, determining a critical grain size. Once determined this size, is simply necessary a process production control to achieve a compromise between the microcracking and the required structural mechanical resistance. Another possibility is to produce a material with large grain size and extensive microcracking with low mechanical resistance but where the main interest is the low thermal expansion (Hasselmann, 1977; Stingl, 1986; Sheppard 1988; Huber, 1988). However, several researchers (Buessem, 1966; Cleveland, 1977; 1978) have suggested that the presence of the extensive internal microcracking, contributes to an increase in the resistance to fracture of these polycrystalline ceramics highly anisotropic, activating mechanisms such as: shielding, branching or cracks deviation. Experimentally, this hypothesis has not been demonstrated, so it is a concept that must be handled carefully.

3. Aluminum titanate (Al_2TiO_5) ceramics

The Al_2TiO_5 is a one mole Al_2O_3 and one mole TiO_2 compound. This material is conventionally prepared by reactive sintering of Al_2O_3 and TiO_2 powders. Its interest as polycrystalline ceramic material arose from the work of Bachmann (1948), who found that the thermal expansion of aluminum titanate, in the studied temperature range, could be lower than that of the vitreous silica. This material has interesting features for applications such as thermal insulator and can also withstand strong thermal gradients. This aluminum titanate emerged as a promising ceramics for technological applications; summarizing its most important physical properties, in Table 1.

The material presents two major problems: the thermodynamics instability of the Al_2TiO_5 below 1280 °C and its poor mechanical resistance related to an extensive microcracking which is, in turn, responsible for the low thermal expansion. Decomposition can be controlled or at least delayed, with oxides additions, such as MgO (Ishitsuka and col., 1987;

Wohlfrom and col., 1990) and Fe_2O_3 (Tilloca, G., 1991, Brown et al., 1994), which forms solid solutions between the Al_2TiO_5 and the isostructurals MgTi_2O_5 and Fe_2TiO_5 . The mechanical strength can be increased with good results preparing composite materials such as: Al_2TiO_5 - Mulita (Morishima and col. 1987), Al_2TiO_5 - Mulita - ZrO_2 (Wohlfrom et al., 1990).

Property	Al_2TiO_5	Reference
Density (g/cm^3)	3.702	Holcombe (1973)
Thermal Expansion Coefficient Average ($\times 10^{-6} \text{ }^\circ\text{C}^{-1}$) $\alpha_{a20-520} - \alpha_{a20-1000}$ $\alpha_{b20-520} - \alpha_{b20-1000}$ $\alpha_{c20-520} - \alpha_{c20-1000}$	-2.9 - -3 10.3 - 11.8 20.1 - 21.8	Wohlfromm (1990)
Thermal Expansion Coefficient Average ($\times 10^{-6} \text{ }^\circ\text{C}^{-1}$) Crystallographic $\alpha_{20-520} - \alpha_{20-1000}$ Macroscopic $\alpha_{20-1000}$ $\alpha_{20-1000}$ Anisotropy $\Delta\alpha_{20-520} - \Delta\alpha_{20-1000}$	9.2 - 10.2 1.0 - 1.5 1.5 - 1.7 23 -24.8	Stingl (1986) Milosevski (1995)
Melting Point ($^\circ\text{C}$)	1860	Lang (1952)
Elastic Modulus E(GPa)	12 - 18 10 - 20 13 - 15	Stingl (1986) Cleveland (1978) Milosevski (1997)
Hardness, Hv (GPa)	5	Wohlfromm (1990)
Bending Strenght, σ (MPa)	4 - 20 25 - 40	Milosevski (1995)
Thermal shock resistance (Wm^{-1})	500	Stingl (1986)
Thermal Conductivity, k(W/mK)	1.5 -2.5	Stingl (1986) Milosevski (1997)

Table 1. Aluminum Titanate Physical Properties.

3.1 Equilibrium diagram

Lang et al. (1952) studied the Al_2O_3 - TiO_2 equilibrium diagram (Fig. 1), finding the existence of two allotropic forms of aluminum titanate: α - Al_2TiO_5 , a high temperature phase, stable between 1820°C and the melting point at $1860\pm 10^\circ\text{C}$ and β - Al_2TiO_5 , a low temperature phase stable from room temperature up to $\approx 750^\circ\text{C}$ and from 1300°C up to inversion temperature 1820°C (at intermediate values, it has instability and decomposes to $\text{Al}_2\text{O}_3 + \text{TiO}_2$). The

transformation between both phases is spontaneous and reversible; it was found that it is almost impossible to obtain α - Al_2TiO_5 at room temperature, being necessary cooling speeds greater than 800 K/h.)

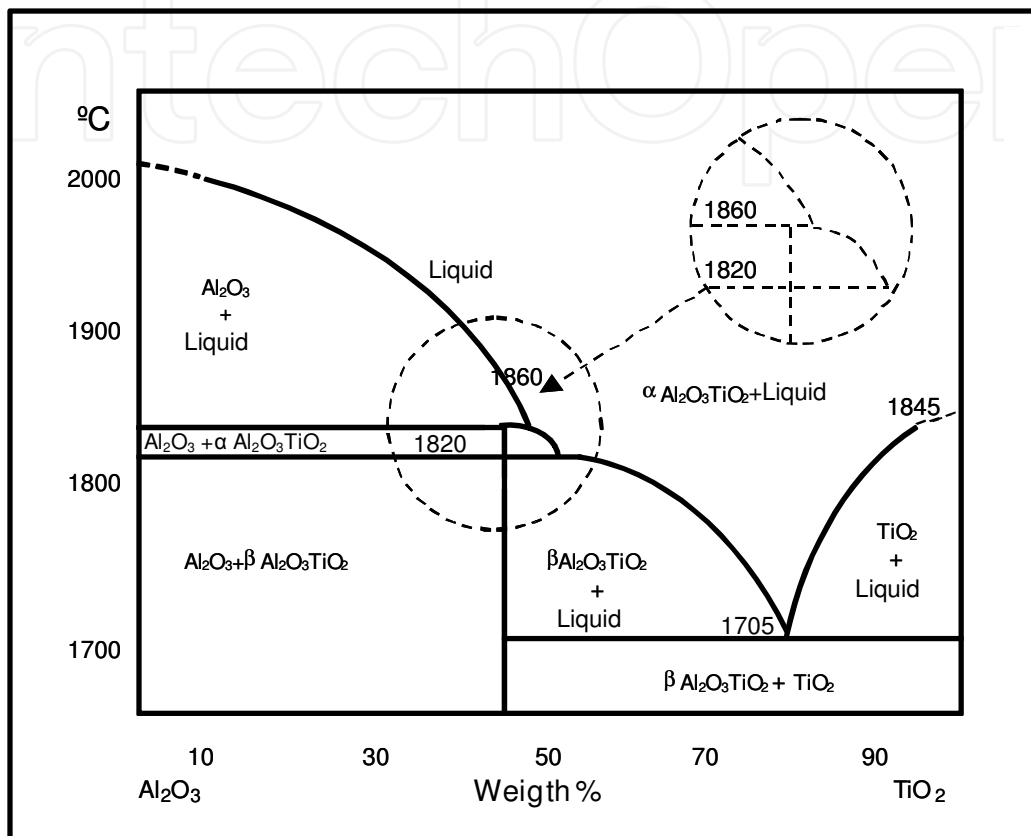


Fig. 1. Al_2O_3 - TiO_2 Equilibrium Diagram by Lang (1952).

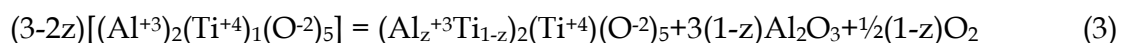
Evidence suggests a congruent transformation of α - Al_2TiO_5 at 1860°C , but the possibility of an incongruent fusion or the existence of a solid solution between Al_2O_3 and Al_2TiO_5 could not be studied, due to the difficulties of obtaining accurate data, because of the high viscosity of liquid formed.

A second point of importance obtained from this research was the suggestion of an instability region for the β - aluminum titanate between 750°C and 130°C . This phenomenon has been confirmed by subsequent research (Fonseca & Baptista 2003). Lang et al., (1952) concluded that opposing formation and decomposition processes occurred in dynamic equilibrium, promoting decomposition at a certain temperature range, i.e. the β - Al_2TiO_5 phase is stable above 1300°C , below this temperature aluminum titanate undergoes an eutectoid transformation according to the reaction:



3.1.1 Effect of oxygen partial pressure in aluminum titanate stability

The composition and mechanical properties of aluminum titanate are strongly influenced by the partial pressure of oxygen from the surrounding atmosphere. It is well known that the valence of the Ti cation in titanium oxides depends on the partial pressure of oxygen (Jürgen et al. 1996). At high oxygen pressures, Ti is tetravalent producing TiO_2 . Due to entropic reasons, at low oxygen pressures (such as for example in air) there is a small fraction of Ti^{+3} , its amount depending on the temperature. By decreasing the partial pressure of oxygen furthermore, the $\text{Ti}^{+3}/\text{Ti}^{+4}$ relationship increases continuously producing $\text{Ti}_n\text{O}_{2n-1}$ Magneli phases and other sub-oxides such as Ti_4O_7 , Ti_3O_5 and Ti_2O_3 . The aluminum titanate phase shows similar behavior (Jürgen et al. 1996). Under high O_2 pressure the aluminum titanate is almost a stoichiometric composition phase Al_2TiO_5 . Decreasing the oxygen partial pressure, due to the increase in the $\text{Ti}^{+3}/\text{Ti}^{+4}$ ratios, it occurs a gradual interchange of Al^{+3} by Ti^{+3} in the aluminum titanate structure. This exchange results in the stoichiometric Al_2TiO_5 decomposition, to the reduced form of aluminum titanate, Al_2O_3 and oxygen according to the reaction:



Decreasing the oxygen potential ($z \rightarrow 0$), the decomposition reaction eventually produces Ti_3O_5 titanium oxide. The degree of decomposition from Al_2TiO_5 to Ti_3O_5 can be related to a continuous change of the aluminum titanate parameter network c , (Asbrink et al., 1967).

Considering the solubility between the Al_2TiO_5 and the Ti_3O_5 under various oxygen pressures, the Al_2TiO_5 was described with a subnet model $(\text{Al}^{+3}, \text{Ti}^{+3})_2(\text{Ti}^{+4})_1(\text{O}^{2-})_5$, this model was derived from the Al_2TiO_5 orthorhombic structure (Epicier et al. 1991), taking into account the mutual exchange of trivalent cations in one subnet, while in the other subnet occupied by Ti^{+4} and O^{2-} species there is no influence.

Subsequently, Freudenberg (1987), brings together all the data obtained and proposed a modified diagram (Fig. 2). Where the only stable compound in the $\text{Al}_2\text{O}_3 - \text{TiO}_2$ system is considered to be, the β - Al_2TiO_5 phase; this compound decomposes above $1280 \pm 1^\circ\text{C}$ (Kato et al. 1980).

The Al_2TiO_5 divides the system in two sub-systems $\text{Al}_2\text{O}_3 - \text{Al}_2\text{TiO}_5$ and $\text{Al}_2\text{TiO}_5 - \text{TiO}_2$ with eutectics at titania 38.5 and 80 weight percent respectively (Fig. 2).

It is important to point out the remarkable solubility difference between corundum and titania, the Al_2O_3 in TiO_2 is \approx one order in magnitude higher than the TiO_2 in Al_2O_3 , and the Al_2O_3 and TiO_2 solubility are practically null in Al_2TiO_5 ; so it is considered aluminum titanate as a stoichiometric compound. However this claim is only correct for oxidizing atmospheres.

The Al_2O_3 has a 2.5% molar maximum solubility in TiO_2 (1.97 ± 0.18 in weight) at 1726°C , (Slepetys, 1969), whereas the solubility of the latter in the alumina is almost non-existent 0.35% molar between 1300 and 1700°C . While both oxides solubility in the Al_2TiO_5 is completely null (Golberg, 1968).

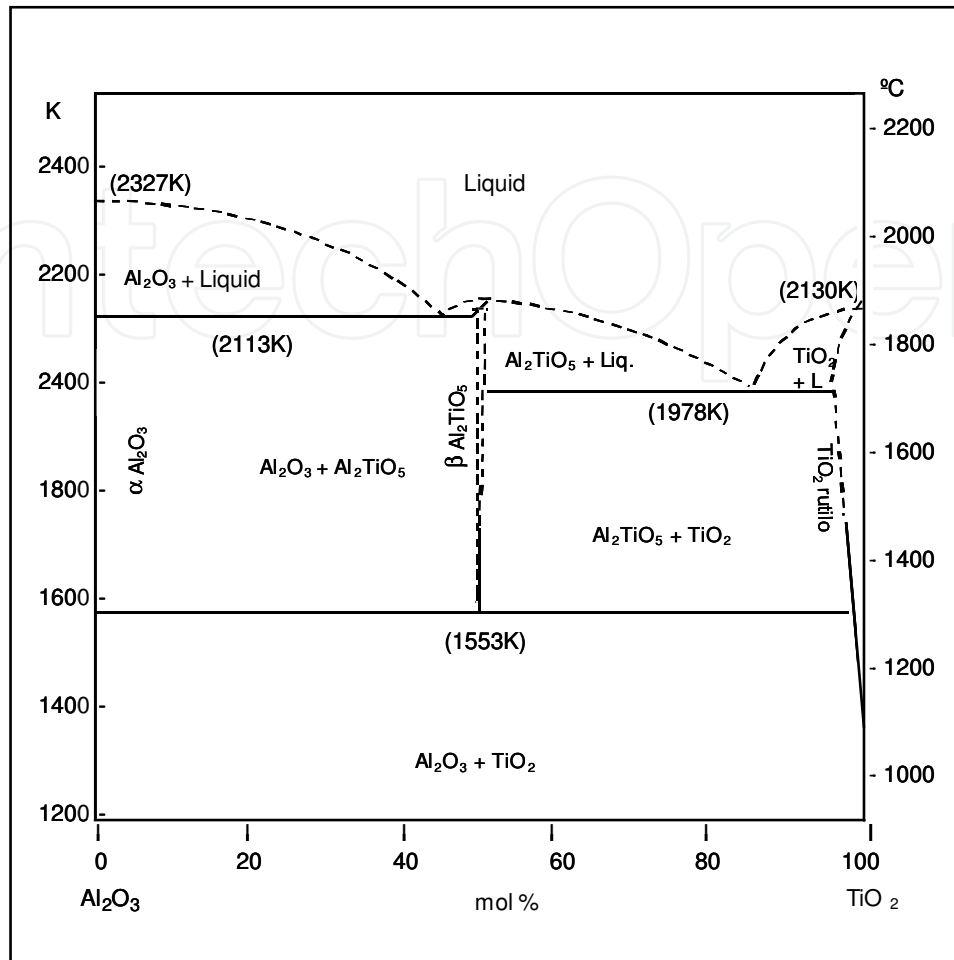


Fig. 2. Al_2O_3 - TiO_2 Equilibrium Diagram calculated in air, from experimental review by Freudenberg (1987).

4. Additives

The thermal instability of aluminum titanate and its low mechanical resistance are the main reasons for the additives use, taking into account these will influence the production process and the final product properties. An important characteristic for all additives is that they do not decrease significantly aluminum titanate thermomechanical properties. Small additions ($\leq 5\%$ by weight) are usually added with the aim of forming aluminum titanate solid solutions.

As was mentioned before, the aluminum titanate is formed above and decomposes below the equilibrium temperature 1280°C (Kato et al., 1980), with a free energy of formation given by:

$$\Delta G^\circ \text{Al}_2\text{TiO}_5 = \Delta H^\circ - \Delta S^\circ T \quad (4)$$

$$\Delta G^\circ \text{Al}_2\text{TiO}_5 = 17000 - 10.95T \quad (5)$$

The endothermic reaction is possible due to the entropy (ΔS°) positive contribution. So as other pseudobrookites, Al_2TiO_5 can be stabilized entropically (Navrotsky 1975), with certain contributions to cation disorder (Morosin et al., 1972). It is conceivable that the positive effect of entropy can be reinforced with additional entropy in terms of mixing by the formation of aluminum titanate solid solutions. It has been determined empirically that solid solutions containing Fe^{+3} and Mg^{+2} , provide a lower decomposition temperature, i.e. increasing stability. On the other hand, solid solutions with Cr^{+3} promote a greater temperature of decomposition, i.e., reducing stability (Woermann 1985).

Jung et al. (1993), studied the replacement of Ti^{+4} by Ge^{+4} and Al^{+3} by Ga^{+3} and Ge solid solutions combined also with additions of MgO and Fe_2O_3 , finding that the stabilizing effect of the additions decreased in the following order: Fe^{+3} , $\text{Mg}^{+2} > \text{Ge}^{+2} > \text{Ga}^{+3}$, corroborating data found in previous research that Fe^{+3} , Mg^{+2} are the best stabilizers so far.

Additions such as Fe_2O_3 , MgO or SiO_2 were studied, the first two promoting structures of the pseudobrookites type Fe_2TiO_5 and MgTi_2O_5 giving complete solid solutions with Al_2TiO_5 (Brown 1994; Buscaglia et al., 1994; 1995; 1997). The SiO_2 has limited solubility (Ishitsuka 1987), however additions up to 3 weight percent produce a slight increase in the mechanical resistance, due to small amounts of liquid phase that densify the material but, larger amounts cause excessive growth of the grain that is detrimental to the mechanical resistance, (Thomas et al., 1989).

Liu et al., (1996), studied the thermal stability of Al_2TiO_5 with Fe_2TiO_5 and MgTi_2O_5 additions finding that material with Fe^{+3} additions did not show any significant mechanical properties decomposition or degradation and the material with Mg^{+2} annealed to 1000 - 1100°C showed an Al_2O_3 and TiO_2 breakdown.

5. Experimental procedure

The raw materials used were reactive grade: Al_2O_3 ($D_{50}=0.60\mu\text{m}$), TiO_2 ($D_{50}=0.88\mu\text{m}$), V_2O_5 ($D_{50}=0.60\mu\text{m}$), MnO ($D_{50}=0.60\mu\text{m}$), ferrosilicon ($D_{50}=0.69\mu\text{m}$), FeTiO_3 ($D_{50}=0.82\mu\text{m}$), and, alumina ball milled 98.5% FeTiO_3 -1.2% SiO_2 purified mineral ($D_{50}=0.88\mu\text{m}$).

Two (2) equimolar mixtures of Al_2O_3 and TiO_2 (56wt% Al_2O_3 - 44wt% TiO_2) were homogeneously mixed with 3, 6 and 9 wt% of each additive using alumina jars and balls, during 6 hours. No binder has been added to the aqueous media powder mixture and it was dried out at 120°C for 24 hours. The material was crushed in an alumina mortar prior to the manufacture of samples by uniaxial die compaction at 300 MPa. Green bodies were reactive sintered at 1450°C, in air for 3 h. Heating was programmed at 5°C/min. whereas cooling at 15°C/min, in order to avoid eutectoid transformation: $\text{Al}_2\text{TiO}_5 \rightarrow \text{Al}_2\text{O}_3 + \text{TiO}_2$ (Kolomietsev et al., 1981).

X-ray diffraction (XRD) analysis has been performed on powders from crushed sintered samples, with grains below 30 μm suitable to obtain rigid specimens. The quantification of Al_2TiO_5 formed was determined by the internal standard method, through direct determination based on the methodology of Klug and Alexander (1954). In this study, the diffraction signals used were: Al_2TiO_5 (023), Al_2O_3 (104) and TiO_2 Rutile (110), which are representative of the three components of interest in the studied samples.

Sintered sample surfaces were carefully ceramographically prepared to minimize damage and, in some cases it was needed to chemically etch in ambient 15%HF solution for 60 s, to reveal grain boundaries. The microstructure characterization was carried out using compositional back scattered electron images (BSEI) from scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS). Evaluation of grain size and phases present has been performed by image analysis.

In order to quantify the stabilization of Al_2TiO_5 , sintered samples previously thermal treated at 1100°C for 100 h. were Si internal standard XRD analyzed, as in the as-sintered condition.

To determine the type of Fe ion in solution, it was used Mössbauer Spectroscopy with the isotope iron ^{57}Fe , in the samples with addition of ilmenite and ferrosilicon. The source used was ^{57}Co , the Mössbauer transition is 14.41 keV, with the excited level of nuclear spin $I = 3/2$ and fundamental level $I = 1/2$. The extent of the isomeric shift provides information on the valence of the atom to which belong the core, as the electronic layers and therefore the density of electrons in the nucleus, are sensitive to chemical bonding.

Thermal expansion analysis in the temperature range of 25 to 1000°C and 1450°C , at $5^\circ\text{C}/\text{min}$ heating and cooling ramps, has been performed on selected samples with good stabilization behavior.

6. Discussions of results

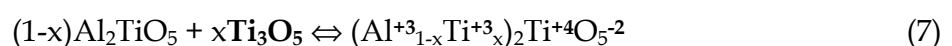
6.1 Additives selection

The additives selection is based on the cation radius, which must be related to the aluminum titanate cations i.e., Al^{+3} and Ti^{+4} , in order to be able to replace them in the solid solution to be formed. The thermal expansion can be related to the degree of crystalline distortion which is known to increase with the difference between the radii of the cations.

Consequently, the octahedral distortion is much greater in Al_2TiO_5 than in Fe_2TiO_5 , due to the small radius of Al^{+3} ions, which facilitate a tendency to the tetrahedral coordination (Bayer, G., 1973). To avoid extreme distortion, the replacing selected cations must have a radii at least close to the $\text{Al}^{+3}=0.54 \text{ \AA}$; i.e., $\text{V}^{+5}=0.59 \text{ \AA}$; $\text{Mn}^{+4}=0.76 \text{ \AA}$; $\text{Si}^{+4}=0.41 \text{ \AA}$; $\text{Fe}^{+3}=0.67$ and $\text{Ti}^{+4}=0.76$. Hence, all additives used in this work fulfill the requirement. On the other hand, the aluminum titanate is formed by an equimolar reaction between Al_2O_3 and TiO_2 . However, due to entropic reasons, in low O_2 pressure conditions, as in air for example, there is a small fraction of Ti^{+3} , its quantity being a function of the temperature. Hence, the reaction of transformation can be expressed in terms of the intermediate reaction of the titanium oxide Ti_3O_5 in the following manner:



The Ti_3O_5 can be seen in terms of $\text{Ti}^{+3} \text{Ti}^{+4} \text{O}_5^{-2}$ for which there is the possibility of forming "limited solid solutions" by cationic substitution as:



Studying the affinity diagram of multivalent oxides of the transition metals with O_2 , it was observed that below Ti, the order of decreasing affinity with O_2 is V, Mn and Fe (Fig.3.).

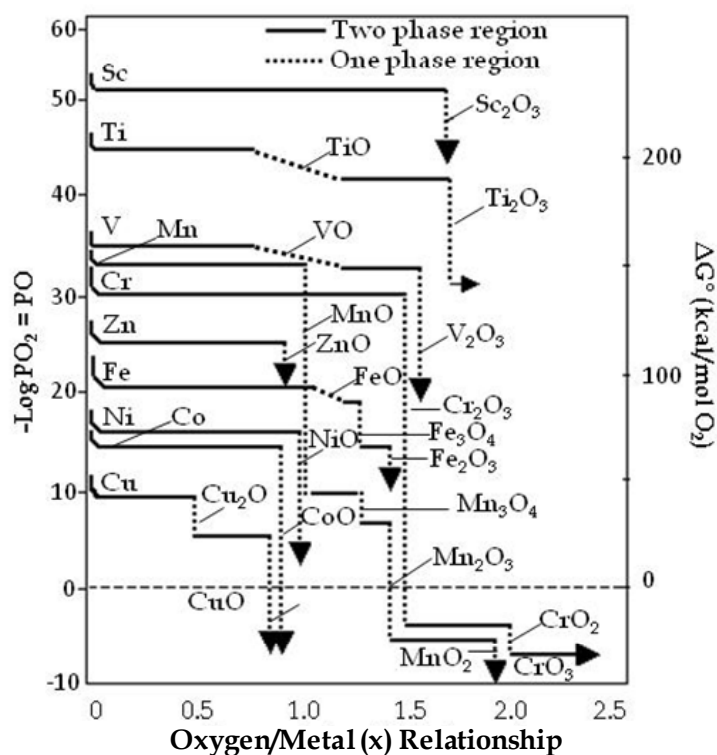


Fig. 3. Oxygen partial pressure (PO_2) vs. Oxygen/Metal (x en MO_x) for the 3d metals.

6.2 Structure analysis

The XRD results showed, for aluminum titanate without additive (Fig. 4.), that the selected temperature and time are sufficient for a near 100% Al_2TiO_5 reaction of formation, as the most important peaks correspond to this compound with a minimum of Al_2O_3 and TiO_2 remnants. It is important to point out that in all XRD, are represented the PDF values for all the constituents expected in each case, although they are not present.

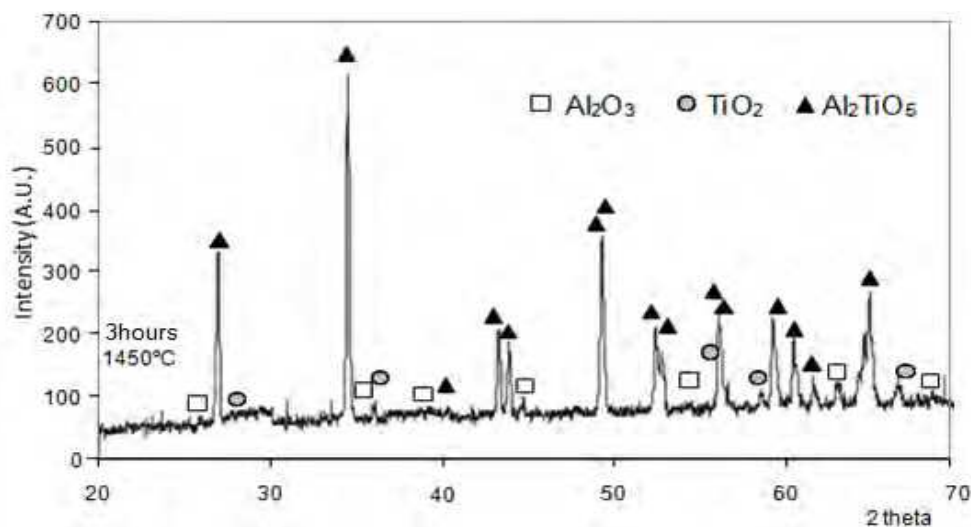


Fig. 4. X Ray Diffraction of equimolar mixture Al_2O_3 and TiO_2 without addition, sintered at $1450^\circ C$ for 3 hours.

In the samples with V_2O_5 , the formation of Al_2TiO_5 decreases as addition contents increase due to an intergranular liquid phase formed, identified by SEM-EDX, that inhibits the reaction between the main constituents (Fig. 5.)

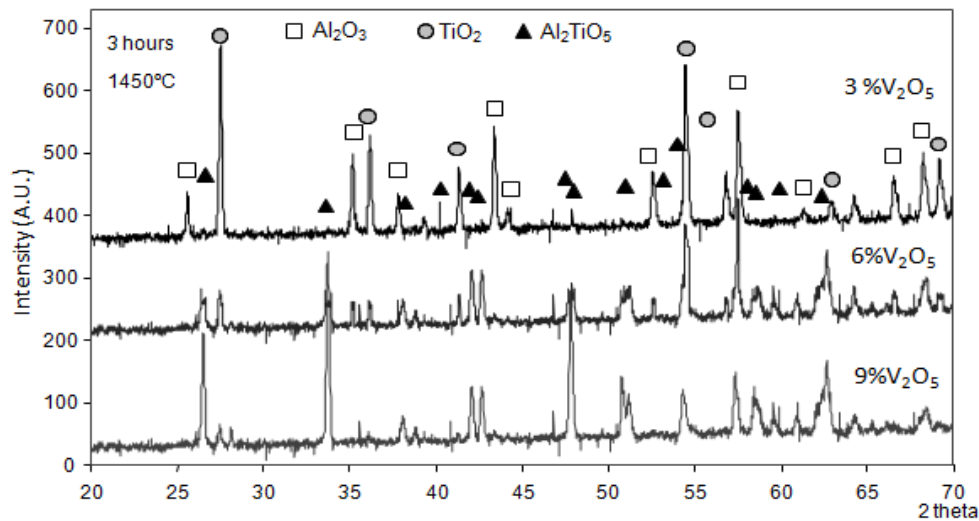


Fig. 5. X Ray Diffraction of equimolar mixture Al_2O_3 and TiO_2 with V_2O_5 : 3, 6 and 9 wt% addition, sintered at $1450^\circ C$ for 3 hours.

Regarding the MnO addition, it promotes Al_2TiO_5 formation with contents, as depicted by the aluminum titanate principal signals which increase in intensity whereas those of Al_2O_3 and TiO_2 are suppressed. $MnTiO_3$ appears as product of TiO_2 and MnO eutectic reaction. (Fig. 6.).

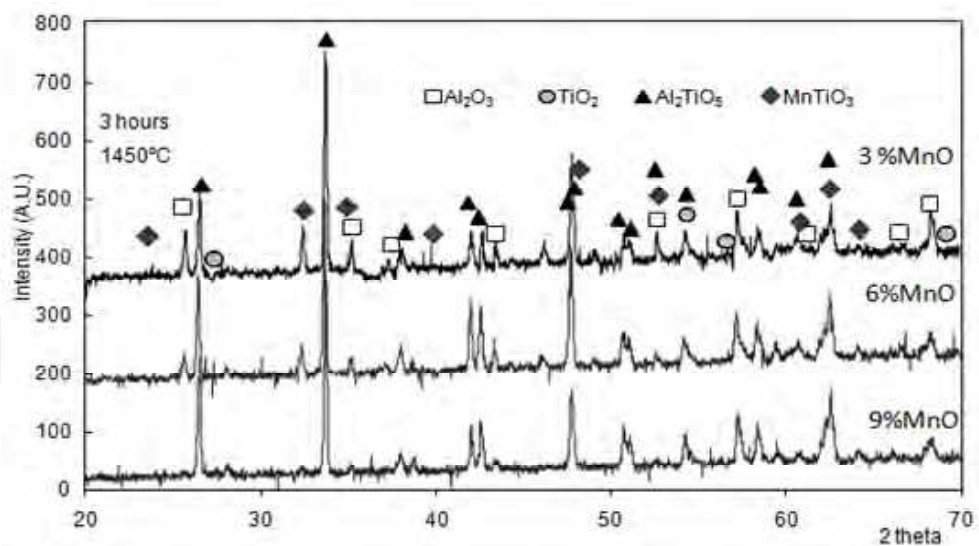


Fig. 6. X Ray Diffraction of equimolar mixture Al_2O_3 and TiO_2 with MnO : 3, 6 y 9 wt% addition, sintered at $1450^\circ C$ for 3 hours.

In the case of ferrosilicon added compositions (Fig. 7), the Al_2TiO_5 formation reaction occurs but not complete and, the main signals of Al_2O_3 and TiO_2 are slightly shifted, corresponding to Al_2SiO_5 and $Al_4Ti_2SiO_{12}$ being the latter, a product of a ternary eutectic transformation.

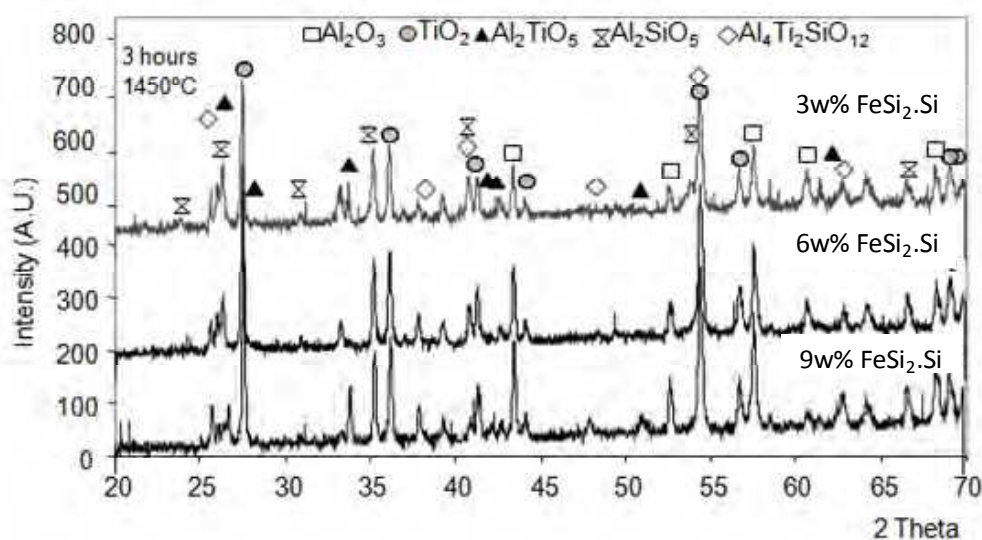


Fig. 7. X Ray Diffraction of equimolar mixture Al_2O_3 and TiO_2 with industrial $\text{FeSi}_2.\text{Si}$: 3, 6 and 9 wt% addition, sintered at 1450°C for 3 hours.

Both pure and concentrated mineral ilmenite (FeTiO_3) (Figs.8 and 9), promoted the formation of Al_2TiO_5 in all compositions studied. Phases such as Fe_2O_3 , TiO_2 , or Fe_2TiO_5 product of decomposition and reaction of the FeTiO_3 , due to the oxidizing atmosphere, were not detected.

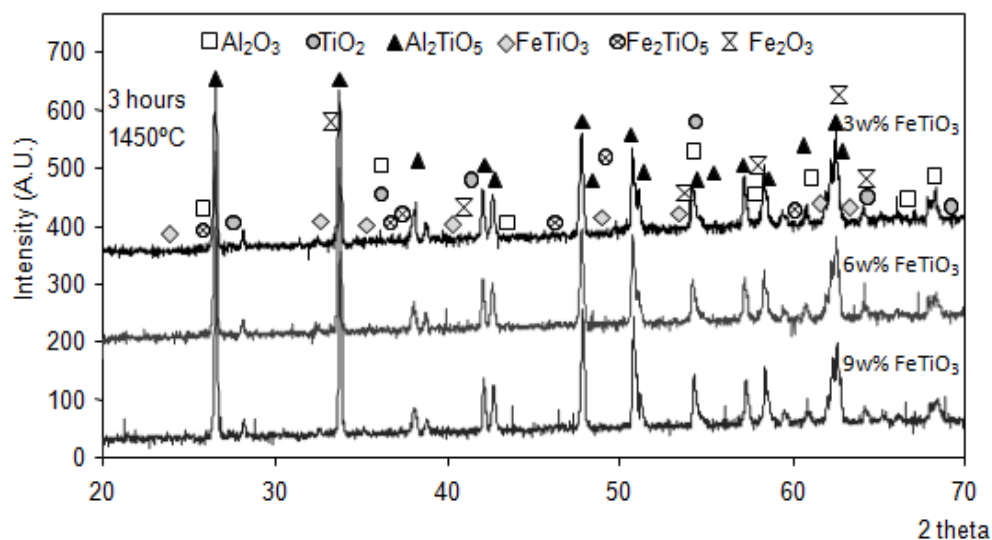


Fig. 8. X Ray Diffraction of equimolar mixture Al_2O_3 and TiO_2 with pure FeTiO_3 : 3, 6 y 9 wt% addition, sintered at 1450°C for 3 hours.

Other remark is that expected phases, product of the reaction of contaminant SiO_2 with the parent Al_2O_3 and TiO_2 , in the samples containing mineral did not show in the XRD spectra. Nevertheless, the most important reflections correspond to the Al_2TiO_5 corroborating the beneficial effect of this additive in its formation (Fig. 9).

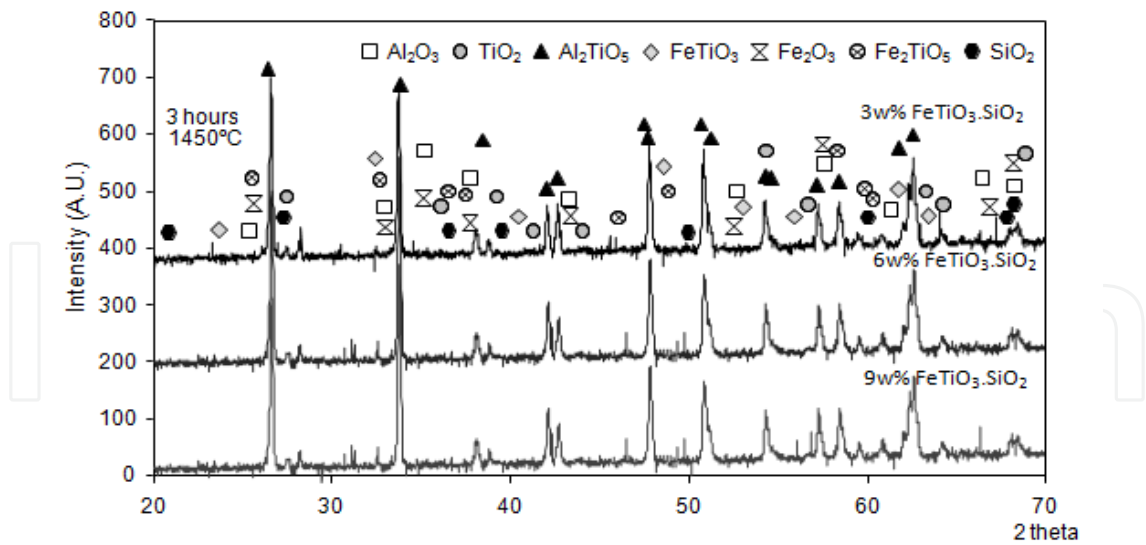


Fig. 9. X Ray Diffraction of equimolar mixture Al_2O_3 and TiO_2 with concentrated placer ilmenite ($\text{FeTiO}_3 \cdot \text{SiO}_2$): 3, 6 and 9 wt% addition, sintered at 1450°C for 3 hours.

6.2.1 Al_2TiO_5 formation phase quantification

The quantification of Al_2TiO_5 formed, was determined by the internal standard method; the achieved results are showed in Table 2.

AT+Additive (%)	%	% TiO_2 unreacted	% Al_2O_3 unreacted	% Al_2TiO_5 formed
V_2O_5	3	19.2	24.3	56.5
	6	20.1	25.5	54.4
	9	22.6	28.7	48.7
MnO	3	13.1	16.6	70.4
	6	11.2	14.1	74.7
	9	9.4	11.9	78.7
$\text{FeTiO}_3 \cdot \text{SiO}_2$ (mineral)	3	5.5	5.8	88.7
	6	5.1	6.4	88.5
	9	5.3	6.28	88.5
FeTiO_3 (pure)	3	1.9	2.7	95.0
	6	2.3	2.8	94.8
	9	2.1	2.0	96.0
FeSi_2	3	21.3	27.1	51.7
	6	19.8	25.1	55.1
	9	16.9	21.5	61.6

Table 2. Al_2TiO_5 % phase formation by sintering at $1450^\circ\text{C}/3$ hours.

It can be seen that the best results are obtained firstly for the pure ilmenite, secondly the mineral ilmenite, then the MnO, vanadium oxide and ferrosilicon additions respectively.

6.3 Microstructure analysis

The composition without addition (Fig. 10), shows the characteristic microstructure of the aluminum titanate: a porous and microcracked Al_2TiO_5 matrix phase and the presence of unreacted Al_2O_3 and TiO_2 , due to the formation reaction kinetics, which is a process led by nucleation and growth of Al_2TiO_5 grains and finally the diffusion of the reactants remnants through the matrix, this is controlled for a very slow reacting species diffusion, as it was found by: Wohlfromm et al., (1991).

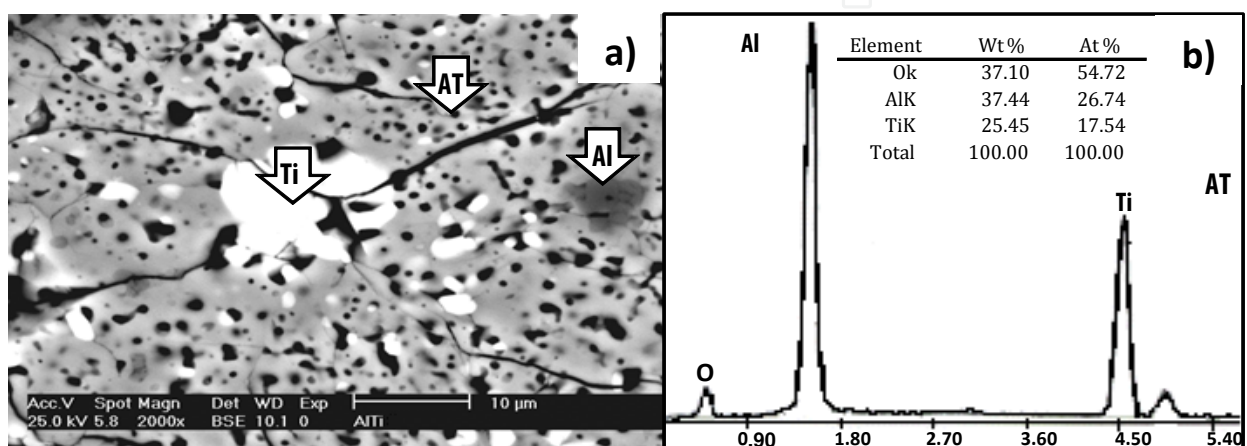


Fig. 10. a) BSE microstructure of Al_2O_3 and TiO_2 without addition, sintered at 1450°C for 3 hours, b) EDS of the matrix with an exact atomic relationship: 25 at% Al, 12.5 at% Ti and 62.5 at% O. (AT: Aluminum titanate; Ti: Titania; Al: Alumina).

The addition of the low melting point V_2O_5 (678°C) is evidenced in the microstructure with the presence of an abundant glassy intergranular phase, which constitutes a physical barrier between Al_2O_3 and TiO_2 , retarding the Al_2TiO_5 formation (Fig. 11a).

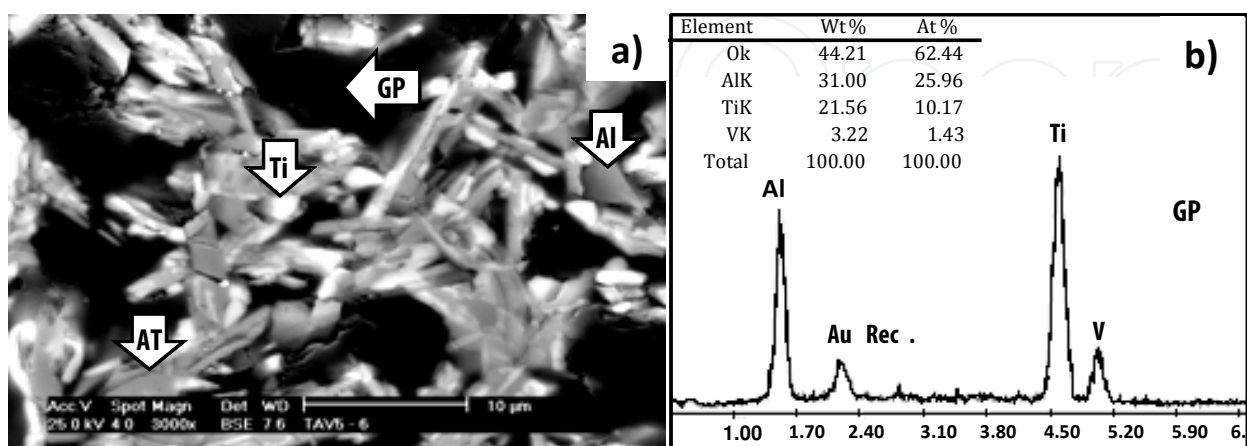


Fig. 11. a) BSE microstructure detail of Al_2O_3 and TiO_2 with V_2O_5 : 6 wt% addition, sintered at 1450°C for 3 hours. b) EDS of the intergranular glassy phase (GP), appearing due to V_2O_5 low melting point. (AT: Aluminum titanate; Ti: Titania; Al: Alumina).

Although localized EDS analysis on the glassy phase was carried out, the Al and Ti values obtained are due to the larger electron beam action volume compared to phase size (Fig. 11b).

The microstructure of MnO added samples shows extensive Al_2TiO_5 phase formation, with a minor presence of liquid phase, product of the two eutectic reactions, at 1290°C and 1330°C, between MnO and TiO_2 . However, opposite to the V_2O_5 added samples, the reacting species diffusion and Al_2TiO_5 formation is accelerated with the MnO contents and, unreacted TiO_2 is absent in the microstructure, due to the secondary reactions. EDS analysis identified the intergranular eutectic phase as $2\text{MnO}\cdot\text{TiO}_2$ and $\text{MnO}\cdot\text{TiO}_2$ (Fig. 12). Microstructure grain size decreased with MnO contents in the sintered bodies.

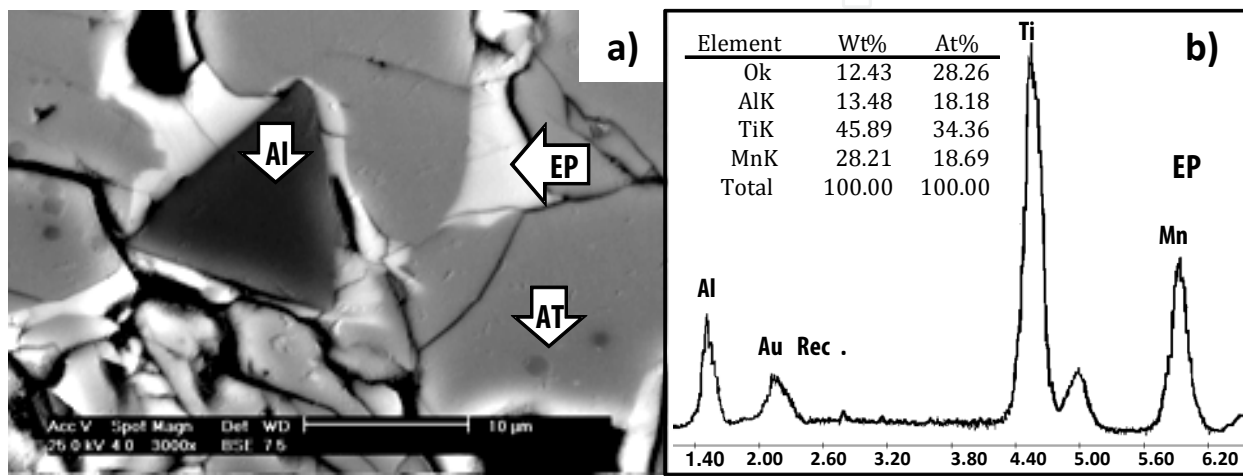


Fig. 12. a) BSE microstructural detail of Al_2O_3 and TiO_2 with MnO: 6 wt% addition, sintered at 1450°C for 3 hours. b) EDS of the intergranular MnTiO_3 eutectic phase (EP). (AT: Aluminum titanate; Al: Alumina).

The FeSi_2Si modified composition has a different microstructure to that obtained with other additives (Fig.13a-d).

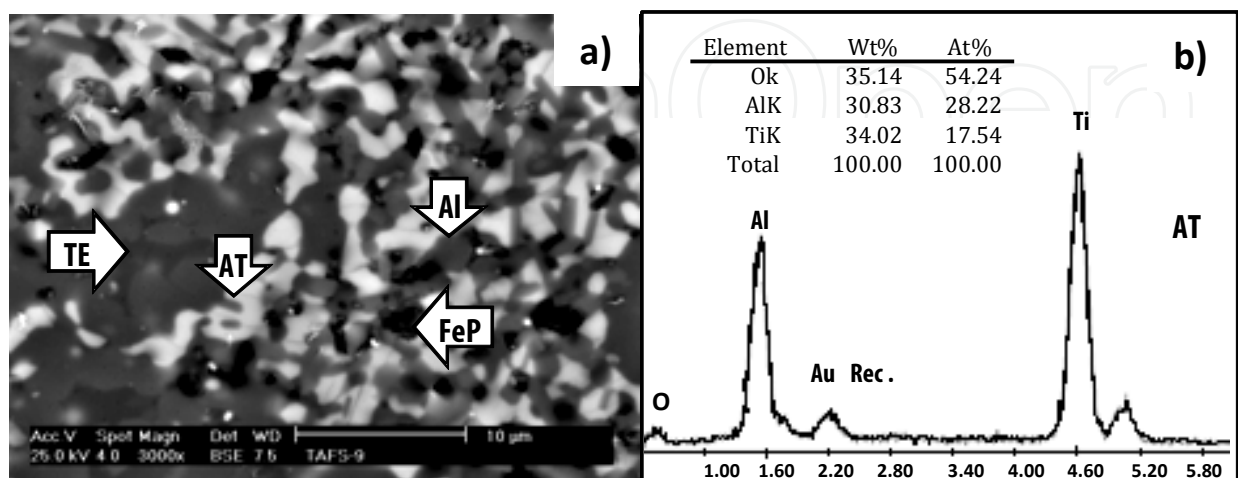


Fig. 13. a) BSE microstructural detail of Al_2O_3 and TiO_2 with FeSi_2Si : 6 wt% addition, sintered at 1450°C for 3 hours. b) EDS of the Al_2TiO_5 matrix.

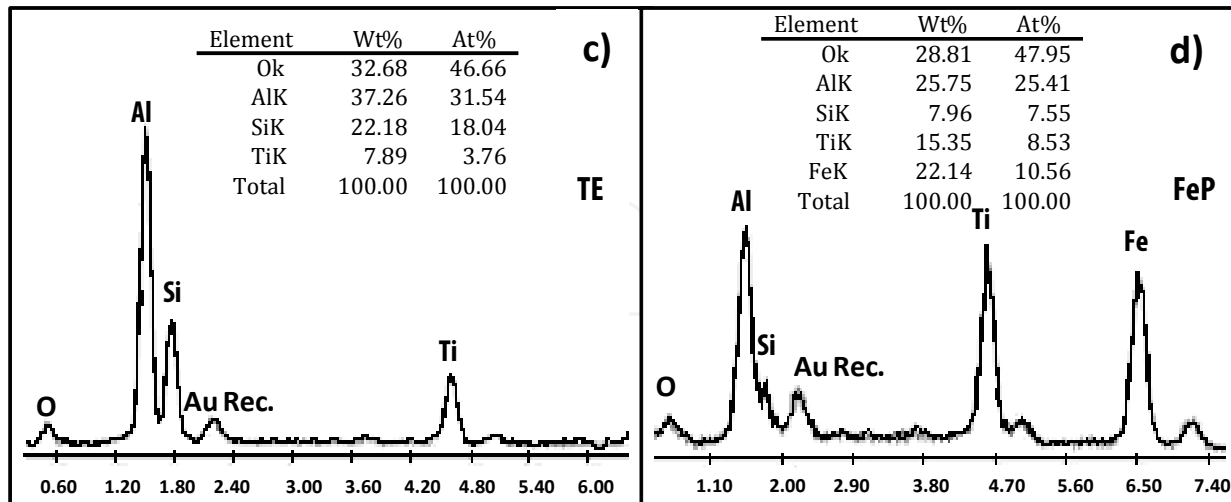


Fig. 13. c) EDS of ternary eutectic (TE) and d) EDS of the intergranular Fe rich phase (FeP).

A fraction of free Al_2O_3 remains as an intragranular phase, the TiO_2 reacts completely and besides the Al_2TiO_5 matrix phase a ternary eutectic reaction grainy phase is formed between the Al_2O_3 - SiO_2 - TiO_2 (as low as it is undetected by RXD), but increasing its quantity with the additive and, also a fourth intergranular phase rich in Fe is depicted (Fig.13d) (Arenas et al. 2011).

Pure and concentrated ilmenite (FeTiO_3), additions have a beneficial effect on grain growth control (Fig. 14 and 15). The SiO_2 left in the purified mineral promoted the formation of an intergranular liquid phase which could not be detected by XRD. Microstructures are practically free of unreacted original phases.

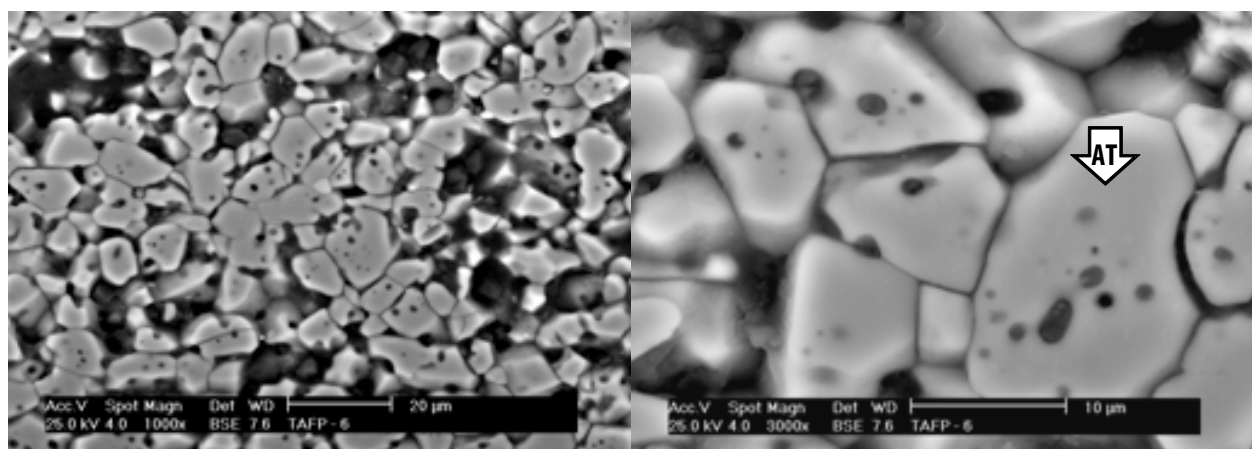


Fig. 14. a) BSE microstructural x1000 and a detail X3000, of Al_2O_3 and TiO_2 with pure FeTiO_3 : 6 wt% addition, sintered at 1450°C for 3 hours. Notice the grain growth control.

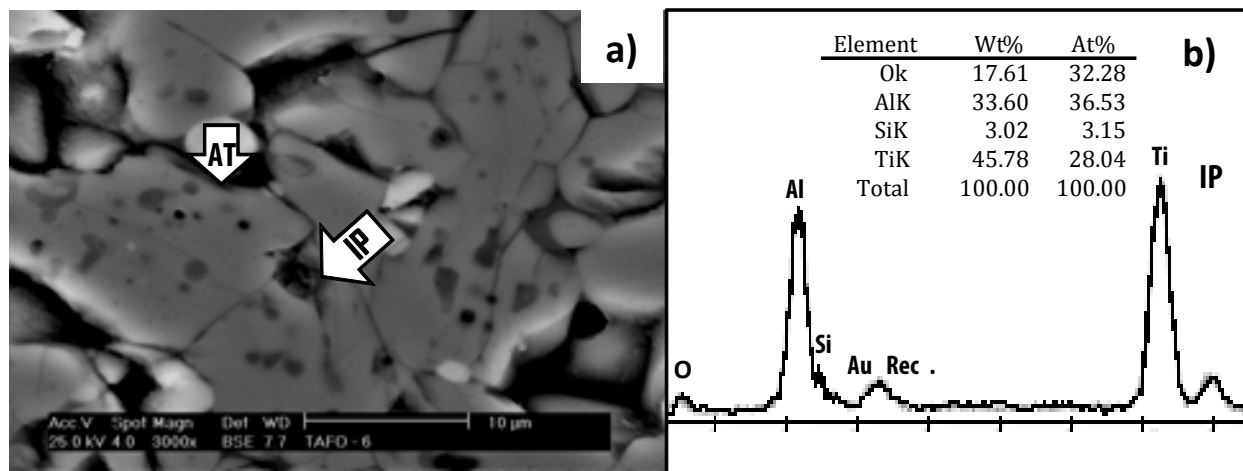


Fig. 15. a) BSE microstructural detail X3000, of Al₂O₃ and TiO₂ with with concentrated placer ilmenite (FeTiO₃·SiO₂): 6 wt% addition, sintered at 1450°C for 3 hours. b) EDS of intergranular phase due to SiO₂ presence.

6.4 Thermal stability

In order to determine the compositions stability, XRD analyses were performed on samples heat treated at 1100°C for 100 hours. The temperature selection is based on industrial applications working conditions and the maximum temperature for decomposition to occur.

The samples without additives and those with V₂O₅ and MnO showed a complete decomposition after heat treatment, as only the diffraction peaks of Al₂O₃ and TiO₂ were showed. (Fig. 16).

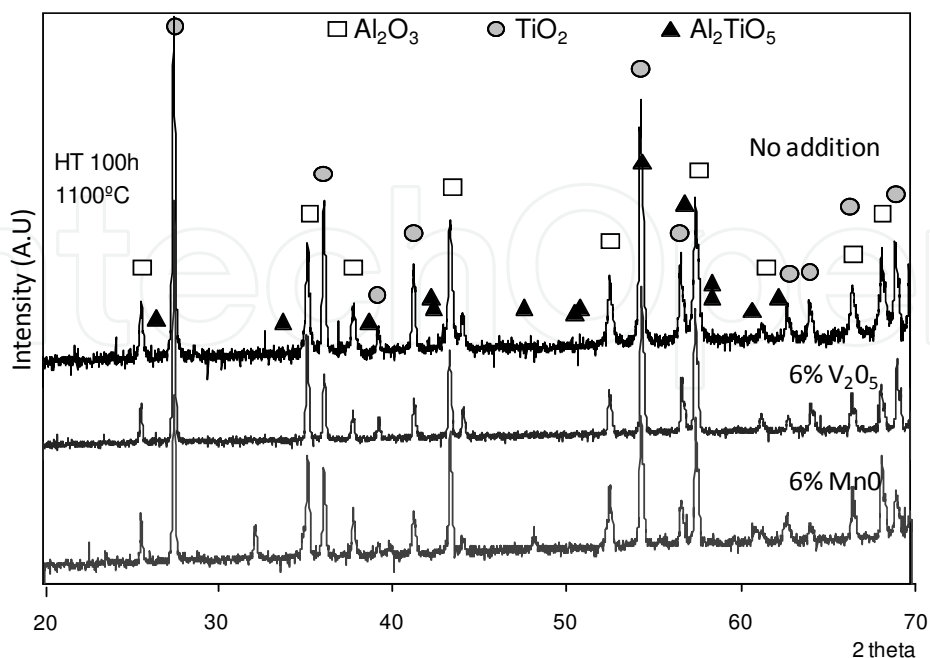


Fig. 16. X Ray Diffraction Al₂TiO₅ without addition, with 6wt% V₂O₅ and 6wt% MnO, heat treated for 100hours at 1100°C.

For $\text{FeSi}_2\text{.Si}$ added samples (Fig.17), although the presence of oxidizing atmosphere, leads to the oxidation of Si and subsequent formation of ternary liquid phase between Al_2O_3 , TiO_2 and SiO_2 which promotes a good densification, it has a minimal beneficial effect on stabilization. The presence of Al_2TiO_5 diffraction peaks is small if compared with that of Al_2O_3 and TiO_2 , product of decomposition. It might be explained as, that only a fraction of the Fe ions from the FeSi_2 react and substitute the Al^{+3} ions, stabilizing the material.

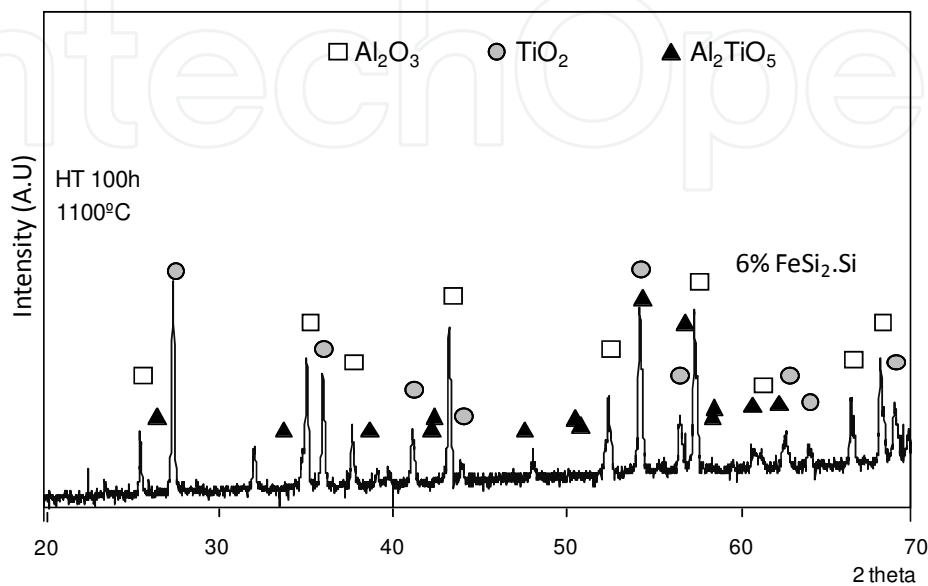


Fig. 17. X Ray Diffraction of Al_2TiO_5 with $\text{FeSi}_2\text{.Si}$ 6 wt% addition, heat treated for 100hours at 1100°C .

The addition of pure FeTiO_3 (ilmenite) clearly shows an increase in the aluminum titanate stabilization (Fig. 18).

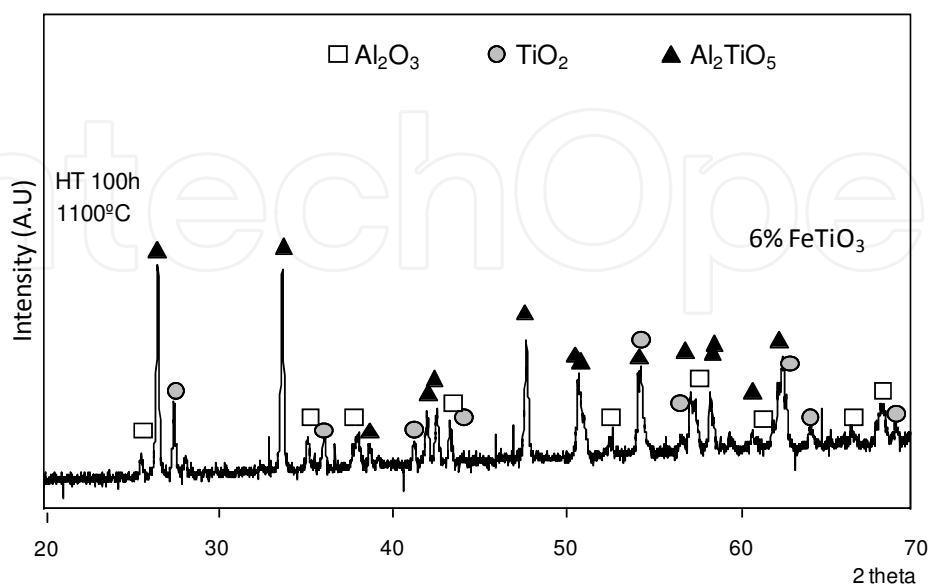


Fig. 18. X Ray Diffraction of Al_2TiO_5 with FeTiO_3 6wt% addition, heat treated for 100hours at 1100°C .

This behavior agrees with the expected solid solution formation between FeTiO_3 and Al_2TiO_5 , as depicted in the calorimetric studies (DSC). These experiments show the decomposition of ilmenite in air atmosphere to Fe_2O_3 and TiO_2 with the formation of Fe_2TiO_5 (Suresh et al.,1991), followed by Al_2TiO_5 reaction at higher temperature. This allows the possibility of a solid solution formation between Al_2TiO_5 and the isostructural Fe_2TiO_5 by a cation replacement mechanism.

From the ionic radii concept, the structural stabilization might be explained by the incorporation of Fe^{+3} ($r=0.67\text{\AA}$) which decreases the structure distortion, caused by the Ti^{+4} : Al^{+3} radii difference (Shannon, R., 1969).

In the case of ilmenite addition, but from the concentrated mineral (Fig.19), the structural stabilization effect is evidenced only for the composition with 9% addition. This behavior might be attributed to the SiO_2 contamination which, on the other hand, benefits body densification by a liquid phase sintering mechanism.

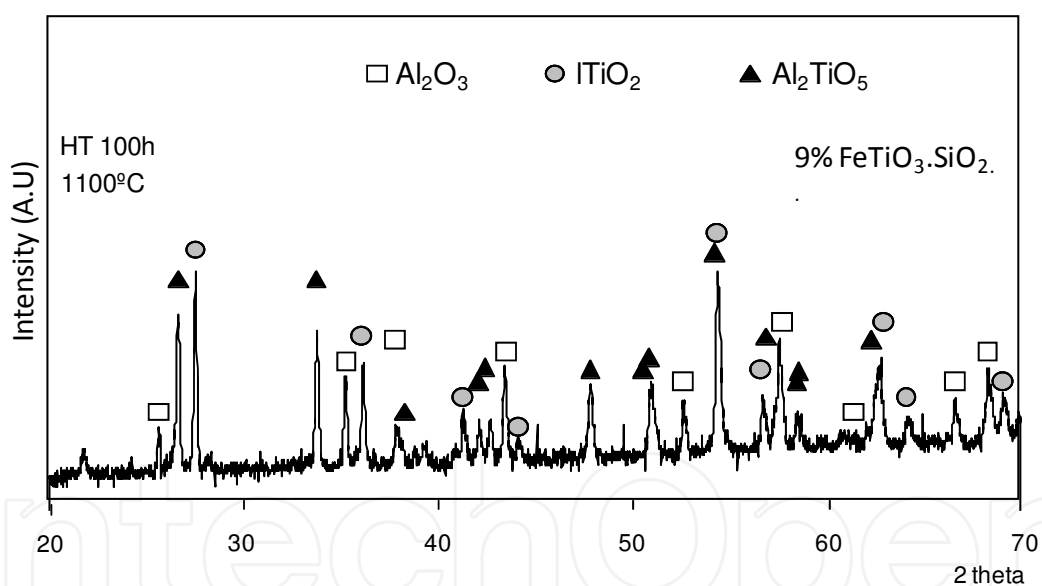


Fig. 19. X Ray Diffraction of Al_2TiO_5 with $\text{FeTiO}_3 \cdot \text{SiO}_2$ 9wt% addition, heat treated for 100hours at 1100°C .

The microstructure study by SEM-EDS corroborated the X-ray analysis evaluation (Fig. 20). All heat treated samples showed the characteristic elongated grain shape, typical of the Al_2TiO_5 decomposition into its original precursors raw materials Al_2O_3 and TiO_2 . However, stabilized Al_2TiO_5 phase is observed in the compositions with 6% pure ilmenite, 9% concentrated mineral and in lower proportion in the samples with 6% ferrosilicon.

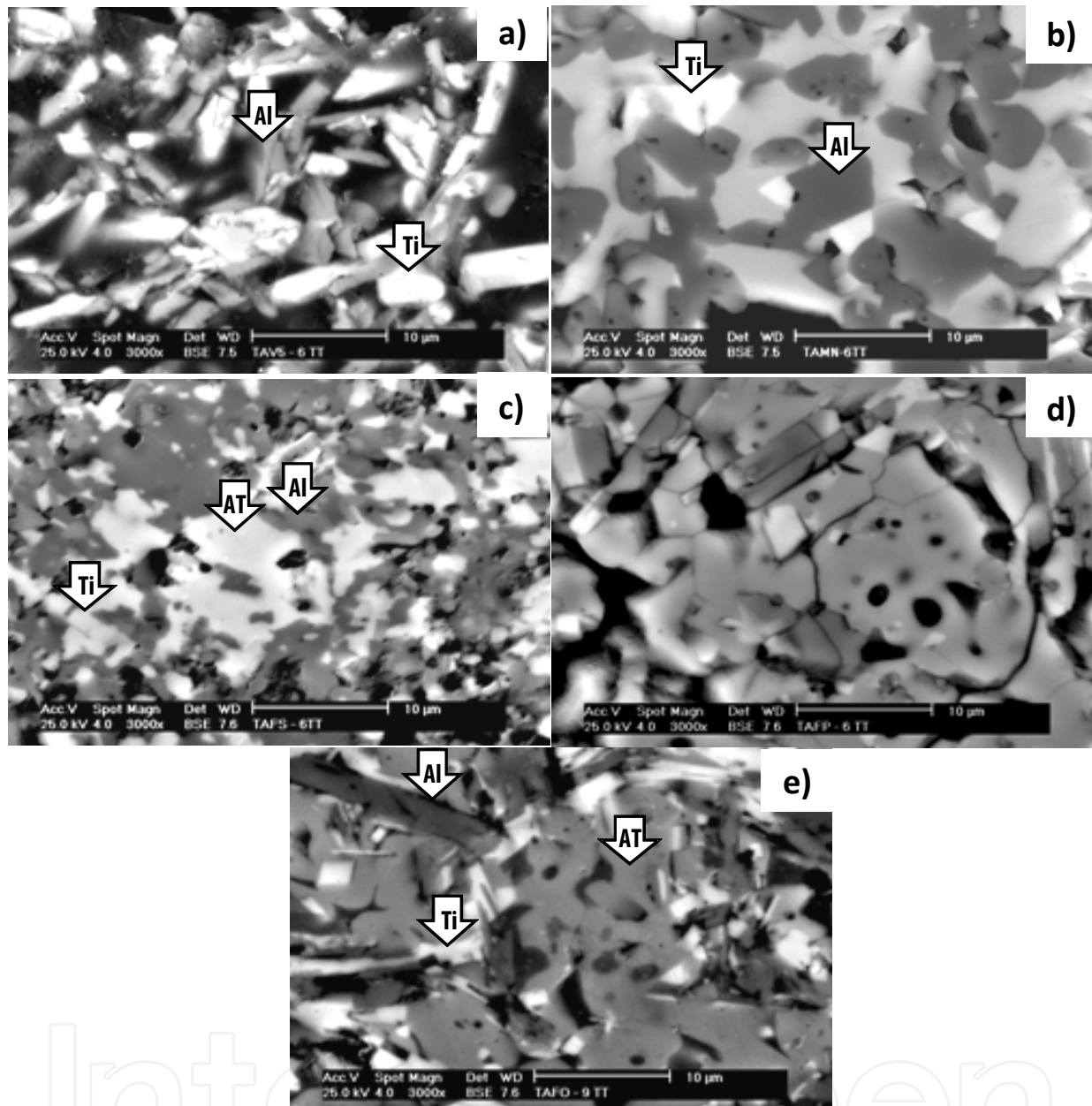


Fig. 20. a) BSE Microstructural detail x3000, Al_2TiO_5 with : a) V_2O_5 6wt% ; b) MnO 6wt%; c) $\text{FeSi}_2.\text{SiO}_2$ 6wt% d) FeTiO_3 6wt% and e) $\text{FeTiO}_3.\text{SiO}_2$ 9wt% addition, heat treated at 1100°C for 100 hours. (AT: Aluminum titanate; Ti: Titania; Al: Alumina).

6.5 Al_2TiO_5 decomposition phase quantification

The quantification of Al_2TiO_5 decomposed after heat treatment was also determined using the internal standard method being the results showed in Table 3. The values obtained corroborate BSE image analysis, observations. The stabilization addition effect being higher in the compositions with pure and concentrated mineral, the MnO and the ferrosilicon stabilize slightly while vanadium oxide have not any effect.

AT+Additive (%)	%	%TiO ₂ decomposed	% Al ₂ O ₃ decomposed	%Al ₂ TiO ₅ left
V ₂ O ₅	3	43.86	55.82	0.32
	6	43.29	55.10	1.61
	9	43.71	55.63	0.66
MnO	3	35.69	45.41	18.90
	6	32.40	41.22	26.38
	9	32.90	41.84	25.26
FeTiO ₃ .SiO ₂	3	26.53	26.97	46.50
	6	23.06	29.30	47,64
	9	23.10	30.12	48.78
FeTiO ₃	3	17.17	21.78	61.05
	6	10.28	13.01	76.71
	9	8.65	10.93	80.42
FeSi ₂	3	38.01	48.36	13.63
	6	36.26	46.13	17.61
	9	33.20	42.24	24.56

Table 3. Al₂TiO₅ % Phase decomposition after heat treatment at 1100°C/ 100hours, by internal standard quantification method.

6.6 Grain size

Since the best stabilizing behaviour after heat treatment was achieved by the ilmenites addition, these samples in the as sintered condition, were selected to determine the effect of additive contents on grains size. There were no significant variations in the grain size obtained with the two additives; however, there is a slight decrease in size as the percentage of the additive increases (in both cases), determined by image analysis of grain size (Table 4). The grain size varies between 9 and 12µm. In other words, the presence of second phase slightly inhibits the growth of grain.

FeTiO ₃ (%)	Tg (µm)	FeTiO ₃ .SiO ₂ (%)	Tg (µm)
3	11.96	3	11.18
6	11.60	6	10.52
9	9.18	9	9,31

Table 4. Effect of FeTiO₃ (pure and concentrated mineral) on the grain size of sintered samples at 1450°C/ 3hours.

6.7 Mössbauer spectroscopy

It has confirmed the presence of the Fe^{+3} ions in all compositions with Fe added, i.e., the ilmenites and ferrosilicon.

The Mössbauer spectrum for material with 6% of ferrosilicon addition can be adjusted to two doublets (Fig.21a). The first doublet corresponds to the ferrous cation (Fe^{+2}) with a resonance that fits the hyperfine splitting with an isomer shift: $\text{IS} = 1.01 \pm 0.002$ mm/s and a quadrupole splitting: $\text{QS} = 0.664 \pm 0.003$ mm/s. The second doublet corresponds to the resonance of the ferric cation (Fe^{+3}) with a $\text{IS} = 0.323 \pm 0.003$ mm/s and a $\text{QS} = 0.520 \pm 0.004$ mm/s. For composition with 6% pure ilmenite addition (Fig. 21b.), it is revealed a consistent doublet with the ferric state (Fe^{+3}), with a $\text{IS} = 0.323 \pm 0.003$ mm/s and $\text{QS} = 0.520 \pm 0.004$ mm/s. The spectrum for the sample with 6% of mineral ilmenite (Fig. 19c), one could guess the doublet corresponds to both states ferrous (Fe^{+2}) and ferric (Fe^{+3}), however should be noted that results are not accurate, with considerable dispersion.

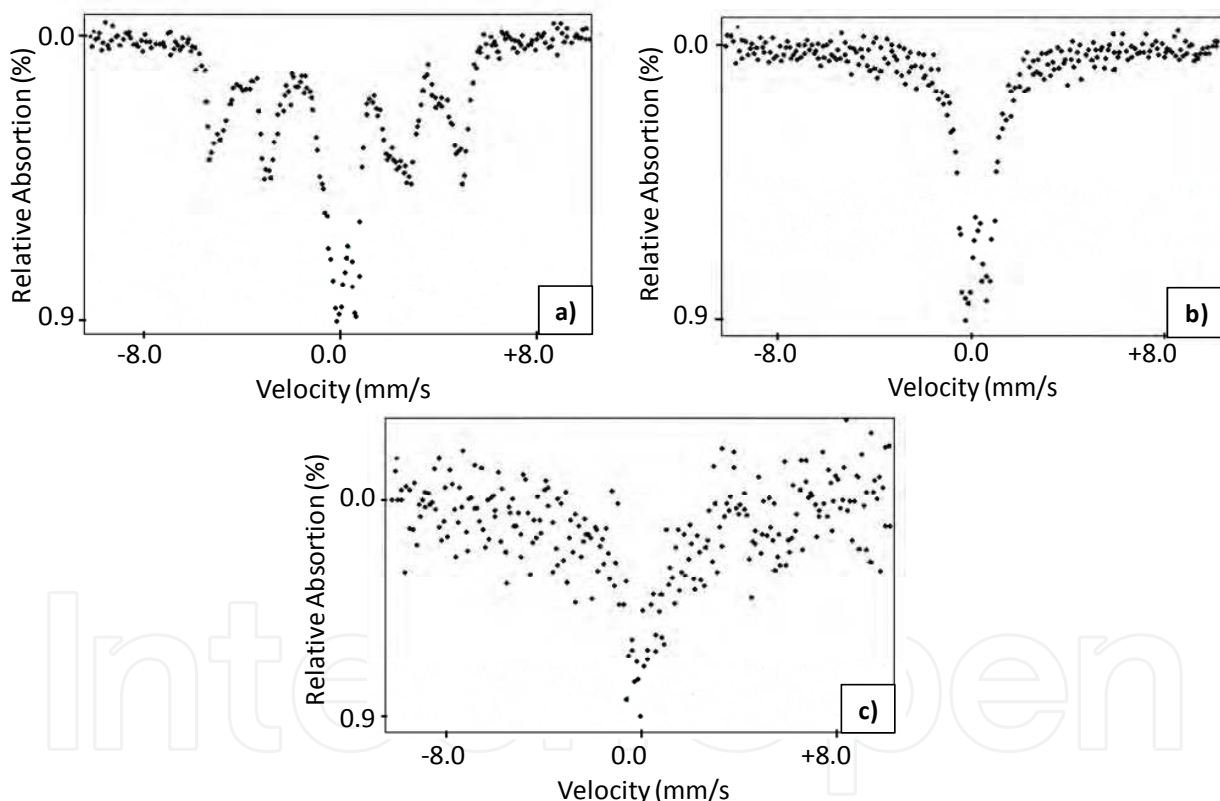


Fig. 21. Mössbauer Spectroscopy of Al_2TiO_5 with: a) 6wt% $\text{FeSi}_2 \cdot \text{SiO}_2$; b) 6wt% FeTiO_3 and c) 6wt% $\text{FeTiO}_3 \cdot \text{SiO}_2$ additions.

These results corroborate the possible replacement of the Al^{+3} ions by ion Fe^{+3} ; there is higher stabilization in samples with pure ilmenite addition, where all Fe ions are in ferric state. However, as it was found in previous research (Barrios de Arenas & Cho, 2010) , the presence of Fe^{+2} ions, also represent the possibility of Al^{+3} ions substitution, with the creation of defects, which in turn promotes the diffusion in solid state.

6.8 Thermal expansion

Some authors have directly related the area of hysteresis in thermal expansion curves to sample microcracks density (Lingenberg W. 1985). In this study, after comparison of the ilmenite added and pure Al_2TiO_5 materials results (Fig.22), show an evident reduction in the area of hysteresis in the formers, being even more important in the samples with concentrated mineral FeTiO_3 addition (fig.22 b).

In both FeTiO_3 -added samples, the property values are antagonist to addition contents.

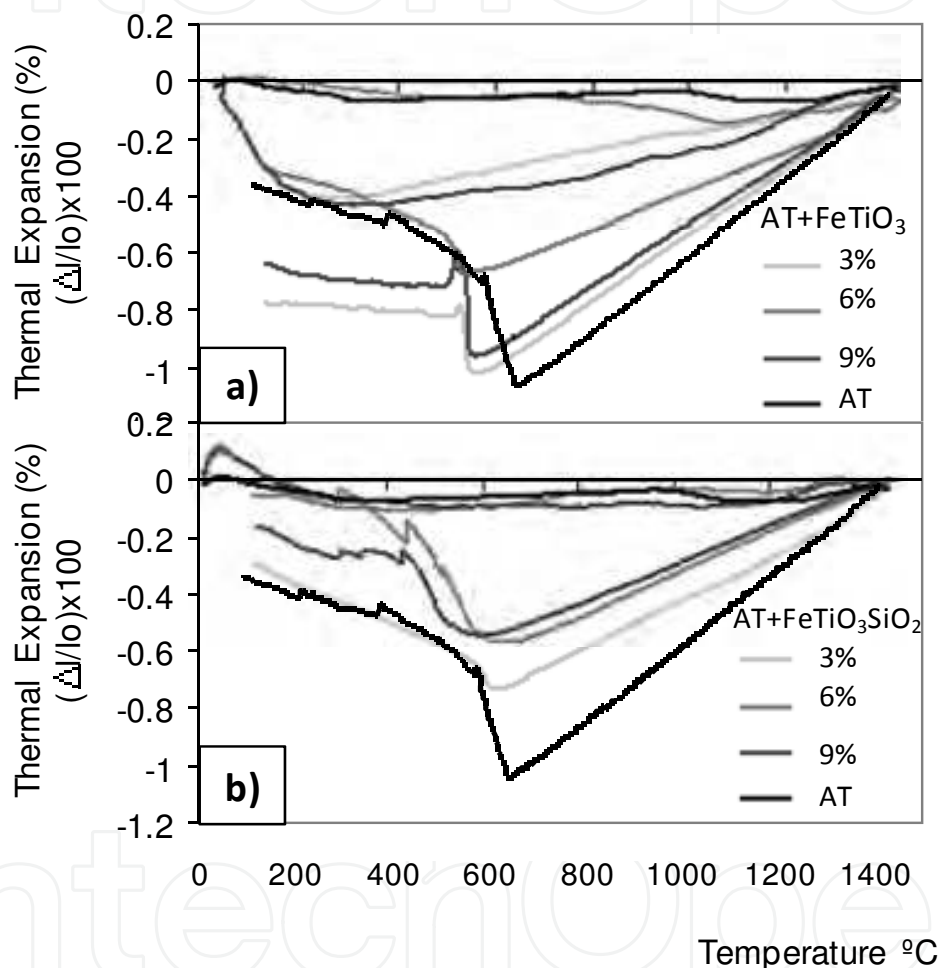


Fig. 22. Thermal Expansion between 20 -1450°C of Al_2TiO_5 with a) FeTiO_3 : 6wt% and b) $\text{FeTiO}_3\cdot\text{SiO}_2$ 6wt% addition.

Table 5, lists the experimental values obtained for the thermal expansion coefficients between 25-1000°C and 25-1450°C. The Al_2TiO_5 without additives has a value slightly negative $\alpha_{25-1000^\circ\text{C}} = -0.55 \times 10^{-6} \text{C}^{-1}$. Both additions turn this characteristic value into very low positive ones augmenting with the additive content in each case. This behavior could be explained by the reduction in grain size which causes a microcracking inhibition, as grains boundaries surface increases, i.e., it is necessary a higher energy for cracking (Yoleva et al. 2010).

Composition	$\alpha_{25-1000^{\circ}\text{C}} \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$	$\alpha_{25-1450^{\circ}\text{C}} \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$
Al_2TiO_5	-0.55	0.87
FeTiO_3 (%)		
3	0.62	0.96
6	0.76	1.05
9	0.86	1.16
$\text{FeTiO}_3 \cdot \text{SiO}_2$ (%)		
3	0.83	1.11
6	0.94	1.22
9	1.02	1.40

Table 5. Effect of FeTiO_3 (pure and concentrated mineral) addition, on Al_2TiO_5 Thermal Expansion.

7. Conclusion

The evident effect of FeTiO_3 additions on Al_2TiO_5 was established. An increase of the addition leads to a sensitive decrease on decomposition, this is due to the expected solid solution formed between Al_2TiO_5 and the isostructural Fe_2TiO_5 , the latter being the product of ilmenite decomposition in the oxidizing conditions used.

The ilmenites additions produce a slightly increase in the thermal expansion coefficients being more important with the concentrated mineral addition. Although the values remain acceptable.

The MnO increases densification by the presence of a localized liquid phase, which allows the rearrangement of particles in the first stage of sintering and also slightly decomposition controls. The ferrosilicon ($\text{FeSi}_2 \cdot \text{SiO}_2$), SiO_2 reacts with Al_2O_3 and the TiO_2 forming a liquid phase, allowing rearrangement of particles; however, it lacks of stabilizing effect.

8. References

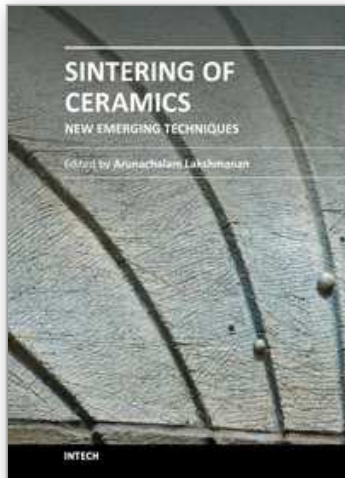
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