MICROSTRUCTURE CHARACTERISTIC OF ALUMINUM TITANATE SYNTHESIED BY BOTH SOLID- STATE AND SOL-GEL PROCESSES

M. Khosravi Saghezchi*, R. Ajami, M. Biazar Markie and H. Sarpoolaky

* *maryam_khosravi_s@yahoo.com*

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School of Metallurgy and Materials Engineering, Iran University of Science and Technology (IUST), Tehran, Iran.

Abstract: *A comparing study on formation and microstructure features of aluminum titanate is investigated through both solid-state and sol-gel processes. Aluminum titanate formed by firing at 1350ºC and 1450ºC for 4h in solid-state process. In the sol-gel process formation of submicron sized particles is followed by addition of sucrose into the transparent sol. XRD analysis was confirmed the formation of aluminum titanate at 1400ºC in lower duration of calcination (3h) without any additives in the sol-gel process. In this work 2wt% MgO is added to the samples as the additive for forming acceleration of aluminum titanate. The influence of MgO addition and heat treatment are studied on phase formation and microstructure development of aluminum titanate in both procedures. Additive optimizes aluminum titanate formation at lower temperatures (1300-1350ºC). Phase and microstructure studies of Mg containing samples optimally show significance in aluminum titanate formation.*

Keywords: *Aluminum titanate, Solid-state, Sol-gel, MgO, Sucrose.*

1. INTRODUCTION

Aluminum titanate (AT), Al_2TiO_5, is a promising candidate material for service conditions involving thermal shock resistance and good thermal insulation, such as thermal processing technology, automotive industry, refractory, combustion engines components and thermal barriers. Tialite is a suitable additive as a second phase that can improve the thermal properties of ceramic matrix composites as well [1]. AT exists in two allotropic forms α and ß which the low temperature phase is ß Al_2TiO_5 and it is a well-known ceramic used in metallurgical and glass industries [2]. It is also recognized as a second phase material for improving mechanical properties like toughness, and flaw tolerance behavior of many ceramic matrix composites [3]. AT is usually formed by several procedures. It is prepared by the solid-state reaction between alumina, Al_2O_3, and titania, TiO_2, above the eutectoid temperature 1280 ºC. It exhibits micro cracking during cooling from the sintering temperature due to the thermal expansion anisotropy [4]. The advantages of the traditional methods are low cost and none aggregation of powders, but it also has some drawbacks, because of the low purity, inhomogeneity and large particle size [5]. In addition of choosing a new and appropriate procedure, it is important to know how the properties of AT composite may be improved. AT has two major problems limiting its wider application [6], such as decomposition to alumina and titania in the range of 800 to 1280ºC and spontaneous micro cracking due to the thermal expansion anisotropy in the sintered ceramics which ultimately result in poor mechanical strength [7]. Additives such as Al_2O_3, MgO, SiO_2 and ZrO_2 are employed in AT precursor to suppress the decomposition [8]. Another approach is to control the sintered grain size to below1.5 μm, for which nano-size starting particle size is a possibility. Also, in some cases sintering aids and grain growth inhibitors are used [9]. Chemical techniques such as sol-gel, co-precipitation and combustion have also been reported for obtaining fine-grained AT, because by using these techniques one can control the initial particle size as well as the low temperature needed for densification [6]. Especially the sol-gel method is environmentally friendly and a water-based technique that does not require special or sophisticated equipments; it is characterized by flexibility, the usage of non-aggressive solutions, and relative simple reaction conditions. The sol-gel method is based on

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molecular synthesis of nano-particles [10]. Indeed sol-gel method is considered to have the advantages of mixing ions even on the atomic scale [11]. It is very well known that materials with nano-scale grain sizes show different properties from the same materials in bulk form. These unique properties are related to the large number of surface or interface atoms. Nano materials have good chemical resistance, mechanical resistance and hardness both at normal and high temperatures [12]. In synthesis of AT the usual aims are to reduce the formation temperature of aluminum titanate as well as to avoid micro cracking due to the thermal expansion anisotropy of AT. Synthesis of aluminum titanate in aqueous solution through a sol-gel method has shown to have advantages in particular with respect to controlled fine grain size, homogeneity of distributed of phases [4]. Aqueous sol-gel procedure owns simplicity, economic low cost, high separation efficiency (> 95 %) and by-product recycling [13]. The mechanical strength of Al_2TiO_5 ceramics also increased as the grain size decreased. The objective of present work is to investigate aluminum titanate characteristics with solid-state and aqueous sol-gel method. In sol-gel method the simplicity of synthesizing submicron AT has been followed with applying sucrose as the premium bonding agent, and as a result particles with average size of ~500 to 800 nm were produced. MgO is applied as the additive to the components in both procedures in order to observing its influence on formation, firing characteristics and microstructure features of aluminum titanate.

2. EXPRIMENTAL

2. 1. Solid-State Procedure

Aluminum titanate samples without any additive (AT), and samples containing 2 wt % of MgO (ATM) were obtained by reaction sintering of compacts prepared from mixtures of alumina, titania (Merck1008081000), and magnesia (Merck 1058621000). Aqueous suspension of each powder was prepared using alumina jar and balls. Dispersing conditions were optimized through rheological characterization of TiO_2 suspensions as a function of dispersant concentration (0.1–1.0wt. % on a dry solids basis) after 2h of ball milling. The obtained pressed samples were heated at 110ºC in an oven for 24h, and then were fired in the electrical furnace at the 900, 1100, 1300, 1350, 1400 and 1450ºC for 4h. Heating rate was 5ºC/min up to 500ºC and samples were kept in this temperature for half an hour and then the heating rate was increased to 10ºC/min.

2. 2. Aqueous Sol-Gel Process

For sol-gel route, titanium tetrachloride ($Ticl_4$, >99.9% Merck), aluminum nitrate ($Al(NO_3)_3.9H_2O$, >99.9% Merck), magnesium nitrate ($Mg(NO_3)_2.6H_2O$, >99.9% Merck), sucrose (Mw=342), nitric acid (HNO_3, 60%, Merck) and isopropyl alcohol (>99.9% Merck) were used as the starting materials. The molar ratio of Al and Ti was calculated as the stochiometric ratio for Al_2TiO_5 formation. 0.02 mole of aluminum nitrate was dissolved in 50 ml of distilled water and pH was adjusted to 1.0 by drop wise addition of nitric acid. Then, 0.01 mole of titanium tetrachloride was dissolved in 50 ml isopropyl alcohol under vacuum status and was dispersed very quickly into the aluminum nitrate containing solution with a syringe and under vigorously stirring. To prepare Mg containing samples 2% wt of magnesium nitrate was added to the sol and was stirred for 1h. Then the solution containing 1gr of sucrose solved in 50 ml distilled water was added drop wise and stirred for next 30 min(s). AT sol was aged for 24h and it remained homogeneous, stable, transparent, and colorless during the aging. The sol converted to a brown gel by adding NH_3 and thermal treatment was maintained at 100ºC in the oven, which resulted in getting a black foamed mass. This carbon- rich mass was grounded in to powders by a pestle and mortar and subsequently it was heat-treated in an electronic furnace at various temperatures: 1250, 1300, 1350 and 1400 ºC for 3h in order to get transformed carbon-free Al_2TiO_5 nano-particles.

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2. 3. Characterization Techniques

For phase analysis in both procedures, X-ray diffraction (XRD) patterns were recorded from 5 to 85° (2θ) by a Jeol 8030 diffractometer (Cu-Kα radiation). Scanning electron microscopy (SEM) was carried out for microstructure observation, and (LEO- 1455VP) model for sol-gel prepared samples and (XL30 –PHILIPS) model for the other samples were used. To prepare samples for SEM observing, the sol-gel derived powders were ultrasonically dispersed into ethanol, and the resultant suspension was spread on the surface of copper plates and were coated with a thin layer of platinum.

3. RESULTS AND DISCUSSION

3. 1. Solid- State Derived Aluminum Titanate

XRD patterns of AT samples derived with solid-state procedure fried at the 900, 1100, 1300, 1350 and 1450ºC for 4h are shown in Fig.1 (left). At 900ºC the main phases are anatase and corundum that implies no reaction is taken place between alumina and titania. By temperature increasing up to 1100ºC anatase converts to rutile phase and the phases are anatase, rutile and

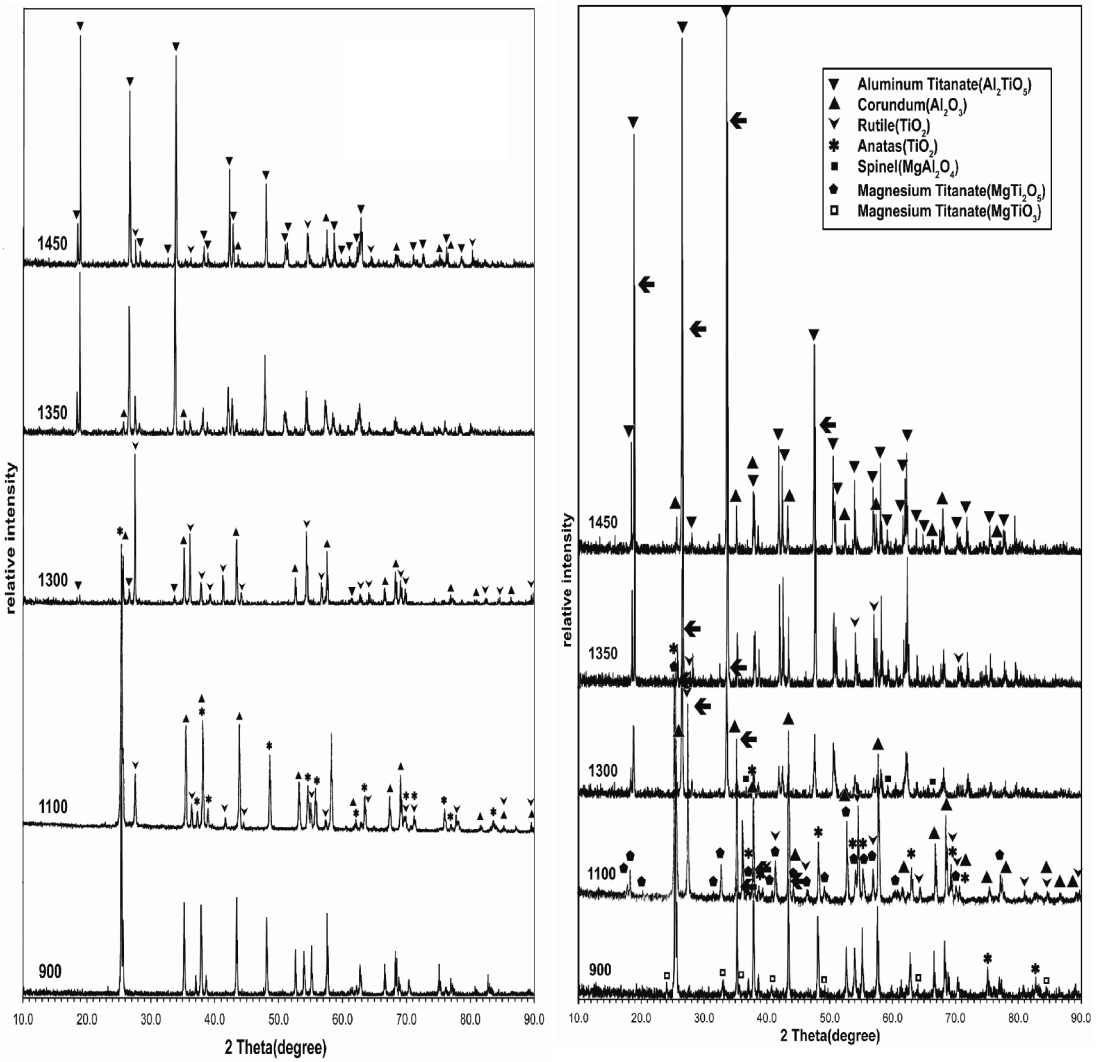

Fig.1. XRD patterns of AT (left) and ATM (right) in the various temperatures derived by solid-state procedure.

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corundum. In 1300ºC which is bit higher than normal forming equilibrium temperature of AT, a little amount of AT phase is formed. This means that reaction between the components begins, but existing of the major unreacted phases, rutile and corundum, proves that the reaction sintering does not take place completely and stops in the beginning stages. By increasing the temperature up to 1350ºC and 1450ºC the main phase is AT, but still rutile and corundum phases are observed due to remaining of unreacted starting materials. Aluminum titanate forming mechanism is nucleation and growth. Because of tialite's lower density (3.7 g/cm^3) than the mixture of equal moles of alumina (3.9 g/cm^3) and rutile(4.1 g/cm^3) [14] nucleation is started in limited spots during the firing process. These limited nucleuses of tialite were called "easy to nucleate "dots by Buscagilia et.al. [14], they also announced that the probable impure materials in initial reactants might be realized as nucleation spots as well. AT does not show much tendency to form because of its lower density as the product comparing to initial reactants. Due to free energy concepts for AT forming which is estimated as , the reaction between alumina and titania is nearly impossible in temperatures below 1280ºC [15]. Generally the transformation of anatase to rutile is taken place in the temperature ranges of 500ºC to 950ºC which is not reversible [16]. Also pure AT has the tendency to decompose to the initial oxides (TiO_2 and Al_2O_3) between 750 to 1280ºC [17]. Measurements carried out [15, 18] show that in final stages of reaction in high speeds only 60 percent of the product is aluminum titanate and the rest are unreacted corundum and rutile particle. About 8% of contraction, densification, and condensation have been taken place during phase transformation [19].

XRD patterns of samples containing 2 wt % MgO (ATM) presented in Fig.1 (right). In 900ºC the main phases are anatase, corundum, little amount of rutile and magnesium titanate ($MgTiO_3$). By increasing the temperature up to 1100ºC anatase converts to rutile. In 1300ºC aluminum titanate phase appear, but unreacted rutile and corundum phases still have been seen. In 1350ºC and 1450ºC the final intense peaks are aluminum titanate phase and still alumina and a little amount of rutile and corundum phases are remained. Additives like MgO [14, 20, 21], Fe_2O_3[22], TiO_2[23] are used, in order to improve thermal stability of aluminum titanate, and these additives are prone to form solid solutions with AT during the sintering process. Other additives such as SiO_2[24], ZrO_2[25], $ZrTiO_4$[26], SnO_2[27] BaO[28], Spodomen[29], Mulite[30], Spinel[31], Feldspar[32] are used as well for improving the stability of AT. The special properties of these additives lead mechanical properties of the final composition in an improved way; also they have significant impacts in order to limiting micro cracks. Forming of $MgTiO_3$ in 900ºC leads the reaction to form rutile. In 1100ºC some peaks show anatase and Mg_2TiO_5 phases. Also, the transformation of anatase into rutile is almost complete in this temperature. In 1100ºC the transition phase of Mg_2TiO_5 forms from reaction between $MgTiO_3$ and TiO_2. Al_2TiO_5 and $MgTi_2O_5$ have the same structure, this two solve in each other and form MgxAl2(1-x)Ti(1+x)O5 solid solution [14, 20], Therefore, it is to be expected that Al_2TiO_5 phase decreases while $MgTi_2O_5$ increases. In 1300ºC the transformation of anatase to rutile is terminated, unreacted rutile and corundum phases still appear, AT begins to form, and magnesium titante is converted to spinel phase. Spinel is more stable than the other unstable materials such as $MgTiO_3$, $MgTi_2O_5$, or Mg_2TiO_4 which all are formed from reaction between MgO, Al_2O_3 and TiO_2. Indeed, these unstable materials are exposed to extra alumina and finally they have been transformed to spinel phase. Buscagilia [14], describes that spinel is an appropriate phase as nuclea to start AT formation.At 1450ºC titania is consumed during the reaction and the good progress of AT forming is quite noticeable and becomes more obvious.

Fig. 2 shows the microstructure of solid-state aluminum titanate in 1350ºC (left) and 1450ºC (right) fired for 4h. From the left side, it can be observed that the aluminum titanate particles grow quickly in 1350 ºC because of the high speed nature of the reaction. Closed pores are distinguished in darker points in color. By increasing the temperature up to 1450ºC , energy for a more complete reaction sintering is

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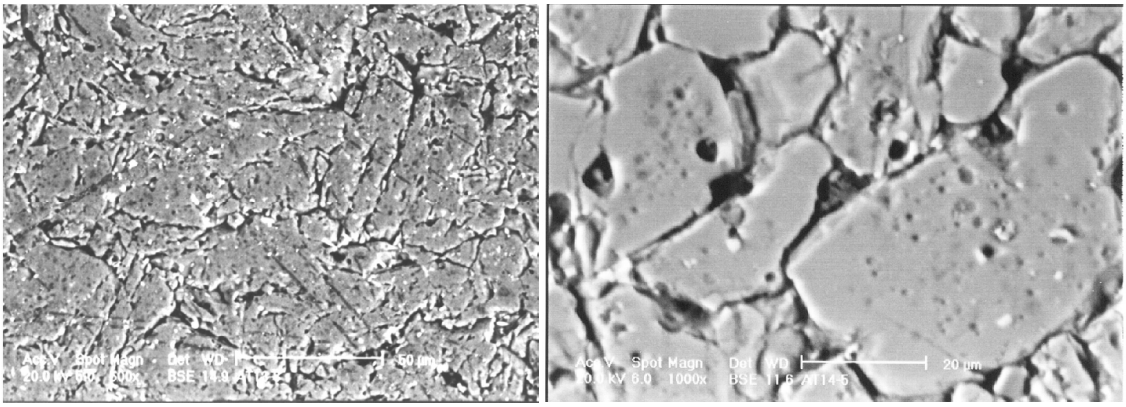

Fig. 2. SEM image of solid-state derived AT in 1350 ºC (left) and 1450 ºC (right) for 4h.

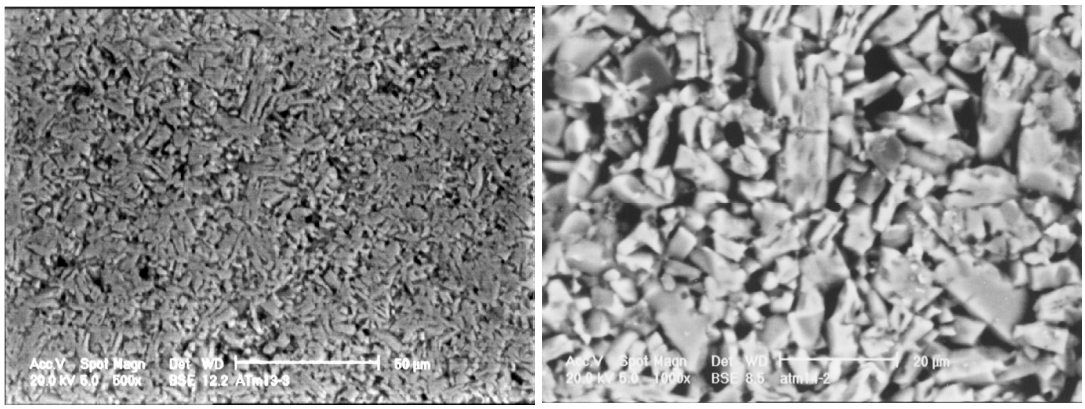

Fig. 3. SEM image of solid-state derived ATM in 1350ºC (left) and 1450ºC (right) for 4h.

provided, and therefore, formation of AT phase is improved, Fig. 2 (right). The particles are seen as grain shapes and the micro cracks are in the grain boundaries, because of anisotropy properties of the aluminum titanate. Reports indicated that exaggerated grain growth and micro cracking are very common phenomena in solid-state sintered AT ceramics [2].

SEM images of solid-state derived ATM calcined in 1350ºC (left) and 1450ºC (right) for 4h are shown in Fig. 3. It can be inferred that both particle size (~2-3μm) and micro cracks are decreased significantly for solid- state ATM samples, which proves that the additive has an influence on formation of aluminum titanate particles. The pores are limited and particles are seen as elongated grain shapes. In ATM samples, MgO converts the microstructure of AT from larger and equiaxed grain shapes into the elongated grains and smaller particle sizes. Density changes during heating treatment. Wohlfromn and et.al showed that increasing of the temperature increases the density because sintering process improves and pores eliminate as well [33]. Additives influence the density in two ways. First, additives own density influences the overall density. Second, the impact of additives on forming middle phases is a noticeable factor. So, with adding of MgO in different components, density increases significantly compared to the AT sample without any additive. MgO penetrates to composite's structure and decreases the diameter of particles and also the size of the pores. There are numerous researches that

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showed MgO has an influence on density behavior of aluminum titanate and as an additive, MgO increases its density [34, 14, 35]. Sarpoolaky and et.al. [36] examined various amount of MgO on aluminum tiatanate's sintering and the results showed that the additive with 5 % wt of MgO had the most impact on density in aluminum titanate formation. Perera and et.al [37] investigated that with higher amounts of MgO (10 %wt), spinel phase formed and as a result the density had been raised significantly.

3. 2. Sol-Gel Derived Aluminum Titanate

Al_2TiO_5 particles have been obtained through aqueous sol-gel method using commercial sucrose as the organic bonding precursor. XRD patterns of fired AT and ATM are indicated in Fig. 4. As it is seen in Fig. 4 (left) at 1250ºC major phase is not AT and corundum and anatase are seen. Al_2TiO_5 starts to crystallize at 1300ºC. XRD analysis of AT powder fired at 1350°C and 1400°C indicates the complete formation of AT phase. XRD patterns of ATM are compared to AT samples in two corresponded temperatures (1300ºC and 1350ºC) in Fig.4 (right). As it is shown for ATM samples the major phase in 1300ºC is significantly Al_2TiO_5. Also the XRD analysis of ATM in 1350ºC indicates complete forming of AT phase. Generally, In ATM samples, the additive accelerates the formation of AT phase in 1300ºC and1350ºC. Formation of ß-AT at low temperature is attributed to nucleation and growth process. Vasudevan and et al. reported that AT forms at 1362ºC from aluminium nitrate derived boehmite and titanium isopropoxide [38]. Sucrose is cheap, non-toxic, available in industrial scale, and easy to store [12]. Sucrose and sucrose degradation products act as chelating agents for metal cations and templating agents to form in a homogeneous solution with hydrogen bonding [39]. Sucrose is used in this work as well as a bonding agent. It makes a polymeric mediocre with other components in the sol, in where water breaks down sucrose by hydrolysis. The hydrolysis stage is similar in all of the sol-gel methods [40]. With assistance of sucrose the precursors react in molecular scale, and the transparent sol contains the initial homogeneous precursor of Al_2TiO_5.

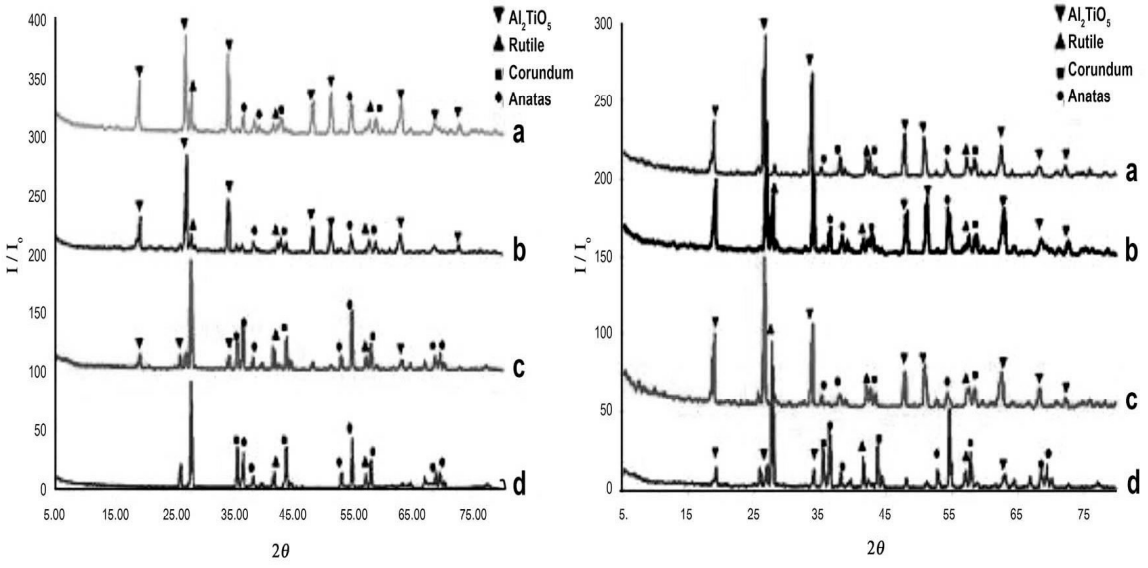

Fig. 4. XRD analysis of sol-gel derived samples calcined in various temperatures for 3h. AT samples without any additive (left): a; 1400ºC, b; 1350ºC, c; 1300ºC, d; 1250ºC. AT and ATM samples (right): a; ATM 1350ºC, b; AT 1350ºC, c; ATM 1300ºC, d; AT 1300ºC.

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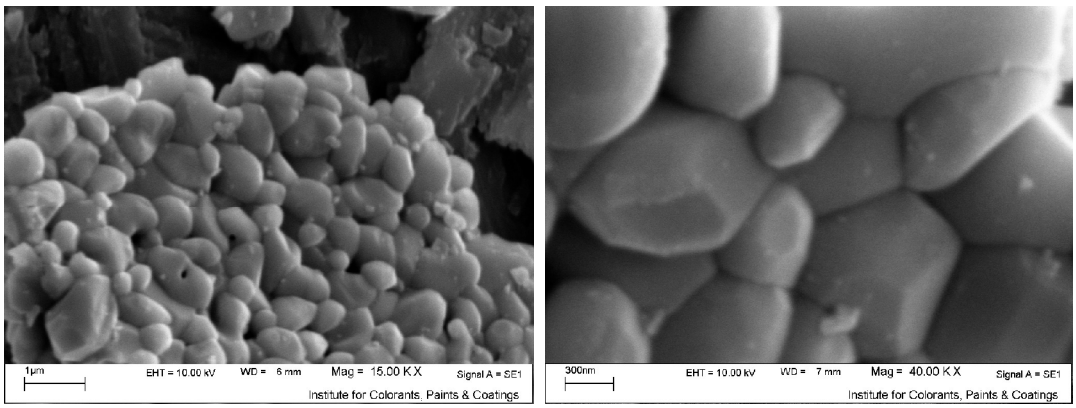

Fig. 5. SEM image of sol-gel derived AT in 1300ºC (left) and1400ºC (right) for 3h.

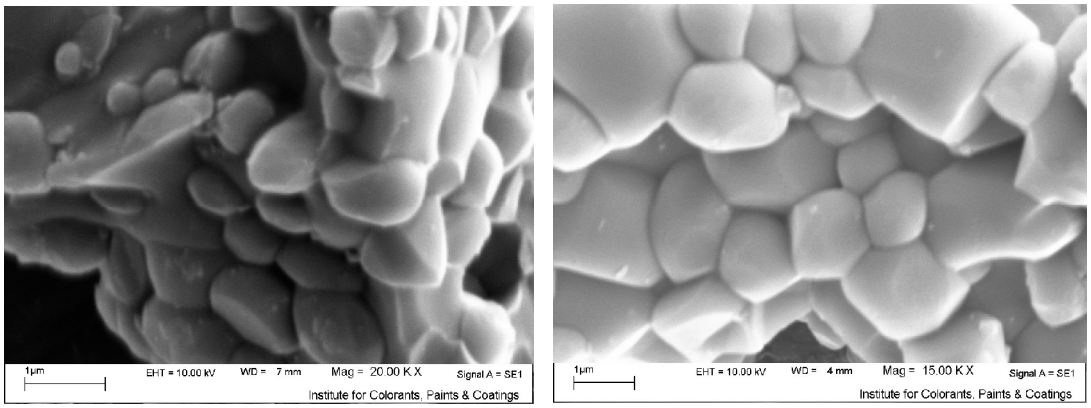

Fig. 6. SEM image of sol-gel derived ATM in 1300ºC (left) and 1350ºC (right) for 3h.

Micrograph of sol-gel synthesized AT fired at 1300ºC for 3h is presented in Fig. 5 (left). It shows that size of particles decreased very significantly comparing to the size of particles in solid-state samples, where in the particles, after thermal treatment at 1300ºC for 3h are less than 800 nm with no crack association. Indeed, synthesized AT particles prepared with assistance of sucrose are uniformed with medium particle size of 500 to 800 nm. Titania-alumina reaction might be preferentially enhanced due to high reactivity of the homogenous distribution. Isolated closed pores are seen due to specific firing schedule in presence of no liquid phase. In sol-gel samples adopted without any additives direct sintering forms distinguished particles and the fine grain size should be due to enhanced reactivity of the precursors. Also, the development of micro cracks is minimized and the sample is highly densified with little amount of closed pores within the grains. Fig. 5 (right) shows the SEM image of AT sol-gel derived in 1400ºC for 3h. As it is shown sintering of AT takes place very fully, grains are side to side, and no pore is seen. This prime image shows that despite the fact that temperature raises up from 1300ºC to 1400ºC, the particle size of AT is still below 800nm (about 300-800 nm).

Fig. 6 (left) shows sol-gel derived ATM sample sintered in 1300ºC for 3h. As it is seen there is a diffused background with sporadic particles. Particles on the balk surface seem to be in the middle of adhering process. As described, MgO forces the sintering of AT in order to form middle

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solid solutions during the reaction. On the other hand, the samples calcined with MgO appear to be well-sintered with lower micro/nano distributed pores. The role of additives can be rationalized in terms of promotion of firing process of AT ceramics [41]. Addition of Mg has good effects on density and optimizing of the microstructure and as a result, synthesized composite has more excellent mechanical properties [1]. This is enhanced by more homogenously distribution of Al_2TiO_5 grains and is attributed to the finer and more homogenous microstructure of aluminum titanate. Fig. 6 (right) shows the SEM image of sol-gel derived ATM in 1350ºC for 3h. The whole background shows particles which are packed side to side and sintered very fully. Size of grains is still incredibly below ~800 nm in this temperature and these grains nucleate from the homogeneous bed of sintered ATM. This image proves that sintering process occurred more completely comparing to the samples with no additive.

4. CONCLUSION

Formation of aluminum titanate fired for 4h takes place at 1350ºC and 1450ºC in solid-state process. In sol-gel process formation of tialite particles takes place in lower duration of firing time (3h) at 1400ºC. Sol-gel precursors synthesized with assistant of sucrose are very homogeneous and well sintered. Adding 2wt% of MgO accelerates and optimizes phase formation and microstructure development of aluminum titanate in both procedures, indeed, enriched crystals of MgO are the original sites for nucleation. Middle phases such as magnesium titanate and spinel form in lower temperatures in the presence of MgO and, accordingly the temperature reduces in which sintering of AT starts.

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