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Eutectoid decomposition of aluminum titanate (Al_2TiO_5) ceramics under Spark Plasma (SPS) and Conventional (CRH) thermal treatments

R. Papitha[a], M. Buchi Suresh[a], D. Chakravarty[a], A. Swarnakar[b], Dibakar Das[c], Roy Johnson[a,*]

[a]International Advanced Research Centre for Powder Metallurgy and New Materials, Balapur, Hyderabad-500 005, India
[b]Department of Metallurgy and Materials Engineering (MTM), KU Leuven, Belgium
[c]School of Engineering Sciences & Technology (SEST), University of Hyderabad, Hyderabad-500046, India

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Abstract

Eutectoid decomposition of Al_2TiO_5, is a strong function of heating rate, exposure temperature and dopant chemistry. Al_2TiO_5 specimens formed from the mixture of precursor oxides were subjected to Spark Plasma (SPS) and Conventional Ramp and Hold (CRH) heating to evaluate the extent of decomposition. SPS with heating rates of 200 °C/min and 50 °C/min under 50 MPa have shown phase decomposition of 95% and 74%, respectively. CRH conditions have resulted only in low phase decomposition of 11% however, substantial decomposition of 54% is observed on thermal cycling. A major slope change in dilatometric curve along with appearance of a new XRD peak at 40° in similar temperature regimes close to 1000 °C complements initiation of decomposition. A significant retardation in eutectoid decomposition of Al_2TiO_5 phase could be achieved by doping with 5 wt% of magnesium silicate as revealed by studies under identical conditions including SPS. Thermo-mechanical properties also correlated well with the observed decomposition profiles.

Keywords: A. Powders: solid state reaction; B. X-ray methods; C. Mechanical properties; C. Thermal properties; Al_2TiO_5

1. Introduction

Aluminum titanate due to its inherent low thermal expansion, high thermal shock resistance and low thermal conductivity is emerging as a candidate material for potential applications such as diesel carbon soot traps [1], raiser tubes and nozzles for handling liquid aluminum melts [2] and not wetting surfaces for molten non ferrous melts [3]. Aluminum titanate (Al_2TiO_5) is a mixture of Al_2O_3 and TiO_2 which forms the solid solution in stoichiometric proportion while heating in air above 1280 °C [4]. Al_2TiO_5 in pure form is not stable in the temperature range of 800–1200 °C where the solid solution decomposes into two phases α-Al_2O_3 and TiO_2 due to the eutectoid reaction. This decomposition occurs when the adjacent Al^{3+} (0.54 Å) and Ti^{4+} (0.67 Å) octahedra collapse because the lattice site occupied by the Al^{3+} ion is too large [4–6]. The thermal energy available from this collapse allows Al^{3+} to migrate from its position and causes structural disintegration to rutile (TiO_2) and corundum (Al_2O_3). The maximum decomposition is observed between 1100 and 1150 °C and the reaction completes between 5 and 50 h. The decomposition process is very slow below 900 °C [7]. Decomposition of Al_2TiO_5 leads to increase in the coefficient of thermal expansion of the material, so it affects thermal shock properties deleteriously. This eutectoid reaction (decomposition reaction) is reported to be a function of processing conditions, heating rate, presence of dopants etc [7,8–12]. Decomposition of Al_2TiO_5 is susceptible to environmental attack or sensitive to the variations in oxygen partial pressure during aging [13–15].

Recently, formation of porous alumina/aluminum titanate composite was prepared by Spark Plasma Sintering (SPS) using nano-structured powders and their properties were investigated [16]. These composites were reported to exhibited excellent mechanical properties due to the unique mechanism involved in

*Corresponding author. Tel.: +914024443169; fax: +914024442699.
E-mail address: royjohnson@arci.res.in (R. Johnson).

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SPS condition. In addition, in-situ formation of Al_2TiO_5 have also studied from precursor oxides through SPS [17] however, to best of our knowledge a systematic study on preformed Al_2TiO_5 under SPS conditions are not reported in open literature.

In the present study, two post formation thermal treatments were carried out under Spark Plasma Sintering (SPS) and Conventional Ramp and Hold (CRH) heating techniques for the phase pure aluminum titanate (designated as, ALT) and $Mg_3Si_4O_{10}(OH)_2$ doped ALT samples (designated as, MSALT). Decomposition studies with respect to thermal cycling (CRH) were also carried out through thermal cycling of ALT and MSALT samples for 10, 20 and 30 cycles from 28 to 1200 °C at a heating rate of 100 °C/min.

It is observed that on exposure to the sensitive temperature regime (1000–1200 °C) in combination with pressure of 50 MPa and particle plasma interaction resulted in the spontaneous dissociation of Al_2TiO_5 phase to an extent of 95% and 74% heated at 200 °C/min and 50 °C/min respectively. However, MSALT samples retarded decomposition close to 30%, signifying the effect of ($Mg_3Si_4O_{10}(OH)_2$) doping. Under CRH conditions samples of ALT and MSALT shows significantly low decomposition of 11% and no decomposition, respectively when heated at 50 °C/min under pressureless conditions. Slope change associated with dilatometric curve in the temperature regime of 1000–1200 °C and appearance of high intensity peaks at around $2\theta = 40°$ in the high temperature XRD (1000 °C) also correlates well in the present study.

ALT samples exhibited a unique microstructure as result of the non equilibrium heating and cooling underwent by sample in the presence of plasma. The samples have shown a porous matrix with aggregates and elongated grains exhibiting a unique texture. There are some anomalies in density and porosity values of ALT samples due to the extreme experimental conditions experienced by the samples during processing. Decomposition studies with respect to thermal cycling were also carried out through thermal cycling of ALT and MSALT samples for 10, 20 and 30 cycles from 28 to 1200 °C at a heating rate of 100 °C/min. CRH-ALT samples have shown the microstructures retaining Al_2TiO_5 matrix with precipitates of Al_2O_3 and TiO_2 as identified by EDS analysis showing elemental enrichments. The unique texture observed with ALT-SPS samples is absent in CRH samples even after 30 cycles which have undergone close to 54% decomposition. The ALT samples also shown no significant microstructural changes or texture formation as a result of thermal cycling however, precipitation of precursor oxides along the grain and grain boundaries. MSALT samples, cycled under CRH condition even after 30 cycles which shown no significance decomposition and correspondingly no microstructural changes is observed.

2. Experimental procedure

2.1. Sample preparation and post thermal treatments

Raw material specifications and process parameters employed for the formation of stoichiometric ALT and MSALT formulations are described elsewhere [11,12]. Extents of ALT phase formation in both samples were evaluated by X-Ray Diffraction studies (D8 advanced, Bruker, Germany). Further, the particle size of the powders was analyzed using Dynamic Light Scattering (DLS) technique (Nanosizer, Malvern Inst ltd, UK). SPS of the samples (ALT/MSALT) were carried out using SPS facility (Dr. Sinter 1050) under identical conditions. For SPS, 8–10 g of (ALT/MSALT) samples were loaded in a graphite die with an internal diameter of 20 mm and thermal treatments were carried out in two heating rates of 50 °C/min and 200 °C/min to the temperature of 1400 °C, under a pressure of 50 MPa with a holding time of 3 min. CRH treatments were carried out on compacts of preformed Al_2TiO_5 of same batch. The compacts were conventionally heated to 1300 °C for 10 min in a PID controlled thermal cycling furnace (CMC, Industries) at two heating rates of 50–100 °C/min (maximum allowable heating rate) to 1300 °C in-order to compare with SPS conditions.

Thermal cycling experiments of both ALT and MSALT formulations were carried out in a thermal cycling furnace from 28 to 1200 °C under a maximum heating rate of 100 °C/min. Cycling were carried out for 10, 20 and 30 cycles under identical conditions along with samples for flexure strength ($45 \times 4 \times 3$ mm^3, as per type ASTM C 1161) and CTE measurements ($25 \times 4 \times 4$ mm^3).

2.2. Characterization of samples

Extent of decomposition of the samples subjected to post thermal treatments was evaluated using X-ray Diffraction technique. The polished samples were thermally etched and micro-structural investigations were carried out using a scanning electron microscope (S-4300SE/N, Hitachi, Tokyo, Japan). All the samples were subjected to hardness measurement using a Vickers hardness tester.

Thermally cycled ALT and MSALT samples were evaluated for mechanical properties, namely the strength using 3-point bend loading (ASTM C-1161-02C). The samples were also subjected to CTE measurements following ASTM-E-339.

3. Results and discussion

3.1. Characterization of preformed ALT and MSALT formulations

The XRD patterns of the preformed ALT and MSALT powders are presented in Fig. 1(a) and (b). XRD pattern of the ALT sample has shown Al_2TiO_5 as the prominent phase with minor quantities of α-Al_2O_3 and TiO_2. An increase in the Al_2TiO_5 phase content from ~92% (in case of ALT) to ~96% (in case of MSALT samples) was observed with addition of 5% magnesium silicate [12]. It is well known that magnesium silicate ($Mg_3Si_4O_{10}(OH)_2$) transform to clinoenstatite ($MgSiO_3$) at 1100 °C and decomposes to MgO and SiO_2 at high temperatures. In the case of MSALT formulation, Mg^{2+} (MgO-1.5%) and Si^{4+} (SiO_2-3%) ions undergo simultaneous lattice substitutions for Al^{3+} to stabilize Al_2TiO_5 stoichiometry [18].

The DLS pattern of the ALT and MSALT powders are shown in Fig. 2(a) and (b) respectively. The average particle

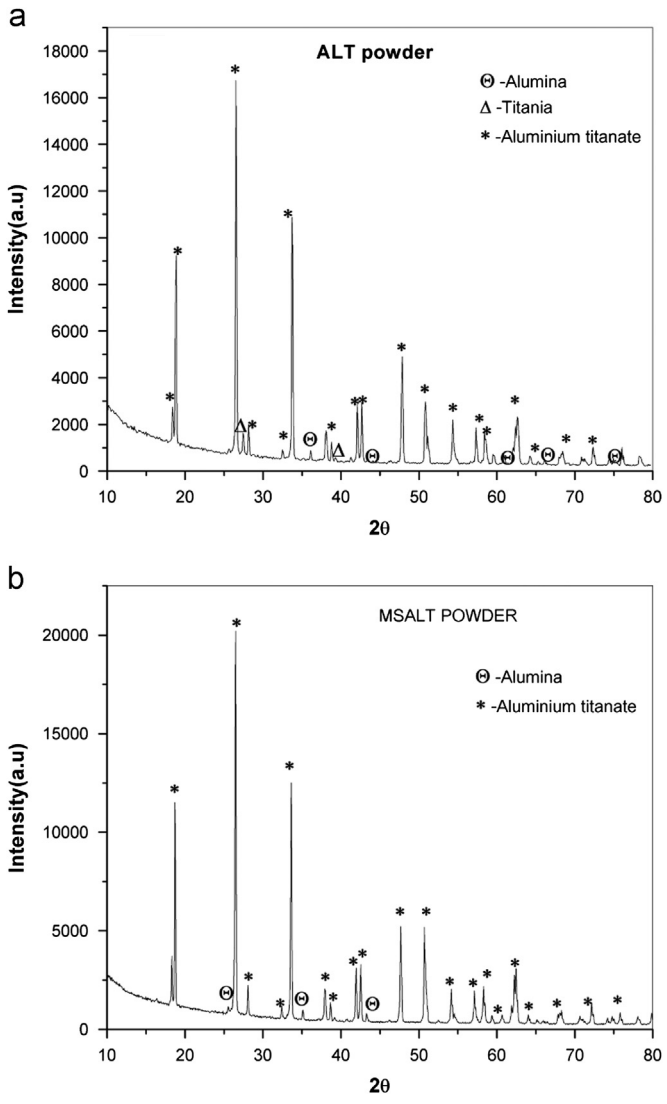

Fig. 1. X-ray diffraction patterns of (a) ALT powder and (b) MSALT powder.

size of the ALT sample was found to be ~505 nm with distribution in the range of 300–800 nm in comparison to MSALT samples exhibiting ~410 nm with a distribution in the range of 230–700 nm.

3.2. Post thermal treatments and estimation of phase concentrations

X- ray diffraction patterns of the ALT and MSALT samples recorded for SPS and CRH under various processing conditions are shown in Fig. 3(a) to (c). Phase concentrations of Al_2TiO_5 (AT), α-Al_2O_3 (A) and TiO_2 (T) were calculated using the peak height intensity ratios/area under the peaks and presented along with the XRD data. It is evident from the XRD pattern (Fig. 3(a)) that ALT samples on exposure to spark plasma conditions at a heating rate of 200 °C/min undergone maximum decomposition retaining only ~5% of the Al_2TiO_5 phase. A corresponding increase in precursor oxide phases of α-Al_2O_3 and TiO_2 are also evident from the XRD patterns. On decreasing the heating rate to 50 °C/min (minimum possible

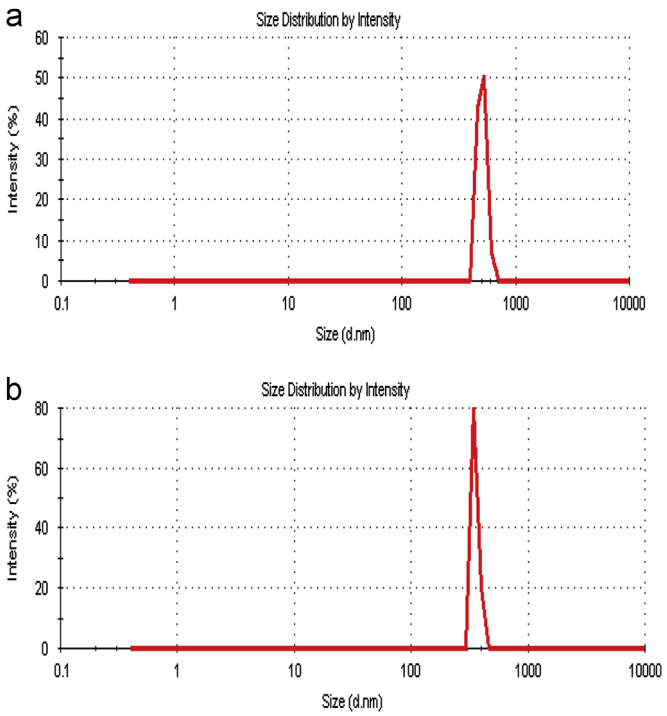

Fig. 2. DLS patterns of (a) ALT powder and (b) MSALT powder.

with SPS facility), Al_2TiO_5 phase retention increased to ~26% revealing that heating rate plays a major role in dictating the eutectoid decomposition.

Under SPS conditions, during the initial stage of sintering as the powders are loosely packed the plasma will be generated between the graphite electrodes through the gaps in loose powders. This plasma generated in the initial stages is expected to clean the particle surfaces and results in nascent reactive surfaces leading to enhanced reactivity and more decomposition. It is important to point out that the decomposition of Al_2TiO_5 phase is strongly dependent on the reacting environment. As the SPS conditions are under argon, the reduced partial pressure of oxygen also contributes to the enhanced decomposition. Decreased partial pressure of oxygen will increase the Ti^{3+} state which in turn increases Ti^{3+}/Ti^{4+} ratio and exchange of Al^{3+} against Ti^{3+} enhancing the Al_2TiO_5 phase decomposition(β-$Al_2TiO_5 \rightleftharpoons \alpha$-$Al_2O_3$+$TiO_2$) [20].

However, magnesium silicate doping in ALT (MSALT) could substantially minimize the decomposition to ~36% even with 200 °C/min heating rate under SPS conditions against 5% retained with undoped ALT samples(Fig. 3(b)). XRD pattern of the sample exposed to CRH conditions of 50 °C/min has indicated significant retention of ~85% Al_2TiO_5 phase (Fig. 3(c)).Unlike ALT samples, a complete elimination of decomposition under CRH condition is also evident from the XRD studies with MSALT samples under identical conditions.

In order to elucidate the decomposition, dilatometric measurements were carried out for ALT and MSALT samples (Fig. 4(a)). Dilatometric curve of ALT samples exhibited a slope change indicating that the decomposition proceeds with a decrease in dimension. In Al_2TiO_5 structure, each Al^{3+} and Ti^{4+} cations are surrounded by six oxygen ions forming distorted AlO_6 or TiO_6

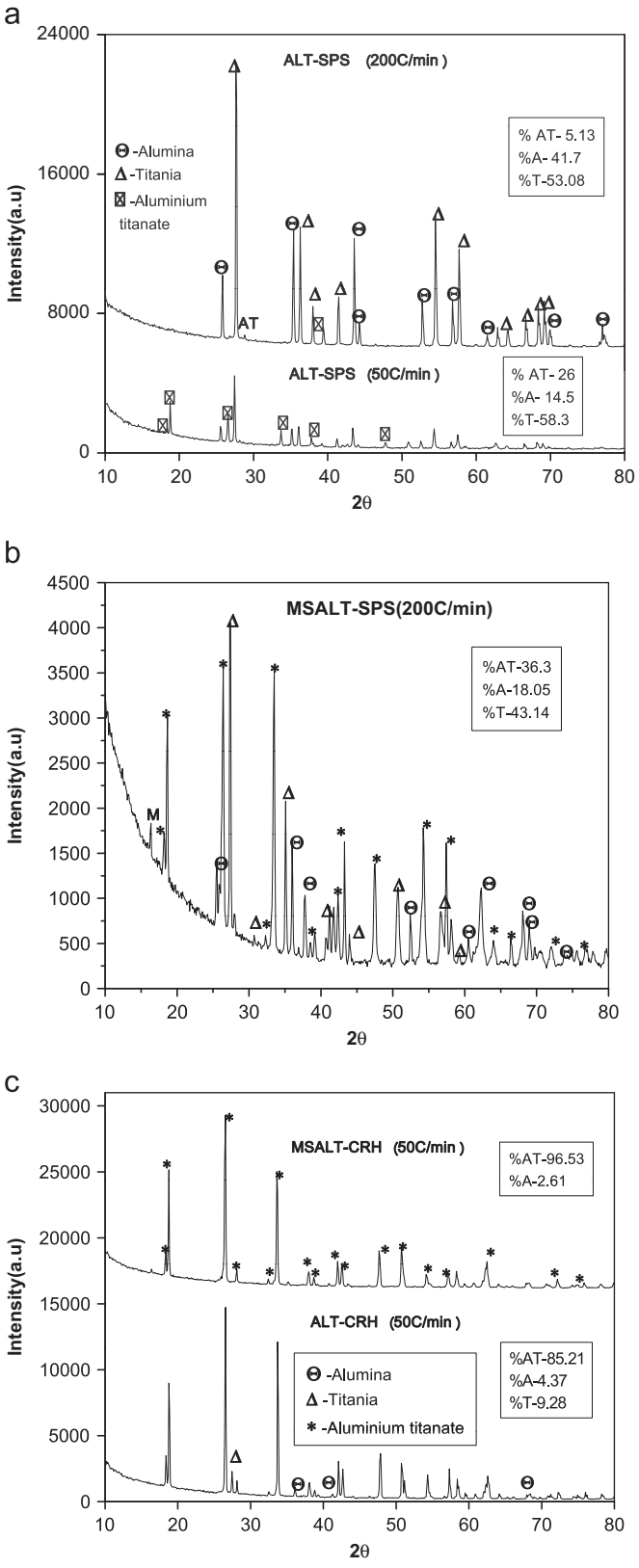

Fig. 3. XRD pattern of the (a) SPS ALT samples, (b) SPS MSALT sample and (c) CRH treated ALT and MSALT samples.

octahedra forming (001) oriented double chains. The dimensional decrease observed during dilatometric studies may impart elastic strain in the matrix. When the elastic strain energy surpasses, the chemical driving force of decomposition, it results in dissociation into precursor oxides and is also dictated by the temperature ranges. It may be regarded as the strain energy surpassess the chemical driving force in the temperature regime of 1000–1200 °C leading to spontaneous decomposition [4,19,20]. High temperature XRD studies (at 1000 °C) under CRH condition shown in Fig. 4(b) also exhibited the appearance of high intensity peaks corresponding to a metastable phase at around $2\theta = 40^{\circ}$, which in turn decomposes into α-Al_2O_3 and TiO_2.

Further, it is evident from the micrograph of Fig. 5(a) that in the case of partially decomposed SPS-ALT samples, there are particles on the surface of the grain and grain boundaries which are confirmed as alumina enriched composition through EDS analysis. These particles act as the nucleation sites resulting in the growth into elongated grains with average grain size of 0.8 μm with distributed porosity throughout the bulk in the case of fully decomposed (Fig. 5(b)) samples. Beyond 1200 °C, Al_2TiO_5 phase get stabilized entropically (ΔS), which can be attributed to the minimization of cation disorder as result of decomposition [11,19,20].

The crystallographic stabilization is responsible for the retention of ~36% Al_2TiO_5 phase even with a heating rate of 200 °C/min under SPS conditions. Due to the simultaneous doping of MgO and SiO_2 through magnesium silicate ($Mg_3Si_4O_{10}(OH)_2$), decomposition may result in crystallographic stabilization. XRD unit cell parameters and cell volume of ALT sample were a=9.4315 Å, b=9.6385 Å, c=3.590 Å and 326.35 Å^3 which have increased to a=9.4651 Å, b=9.6715 Å, c=3.5981 Å and 329.37 Å^3 respectively in case of MSALT samples [2,11]. Microstructure of MSALT sample (Fig. 5(c)) shows the evidence of decomposition complimenting the XRD observations.

3.3. Post thermal CRH cycling

The effect of thermal cycling is studied in order to elucidate its practical application point of view. ALT samples on application as particulate trap practically undergoes several thermal cycles during the regeneration process. Incineration of carbon soot in presence of air may often results in temperature shoot up in combination with hot spot formation and the temperature is reported to reach beyond 1200 °C [1]. The XRD pattern of ALT and MSALT samples after subjecting to thermal cycling are depicted in Fig. 6(a) and (b). Further, a plot of retained Al_2TiO_5 phase and corresponding increase in precursor oxides against number of thermal cycles is shown in Fig. 6(c). It is evident from the XRD patterns that in case of MSALT samples, the decomposition is completely eliminated which are also evident from the dilatometric curve (Fig. 4(a)) showing no significant slope change imparting elastic strain in the matrix leading to decomposition. It is interesting to note that the decomposition rate is maximum for initial 10 cycles. Further, increase in number of cycles exhibiting a non linear behavior of the curves beyond 10 cycles with decrease in decomposition rate (Fig. 6(c)). This can be attributed to the fact that unlike non equilibrium SPS conditions, CRH conditions follows chemical equilibrium and limits further decomposition of the bulk Al_2TiO_5 phase.

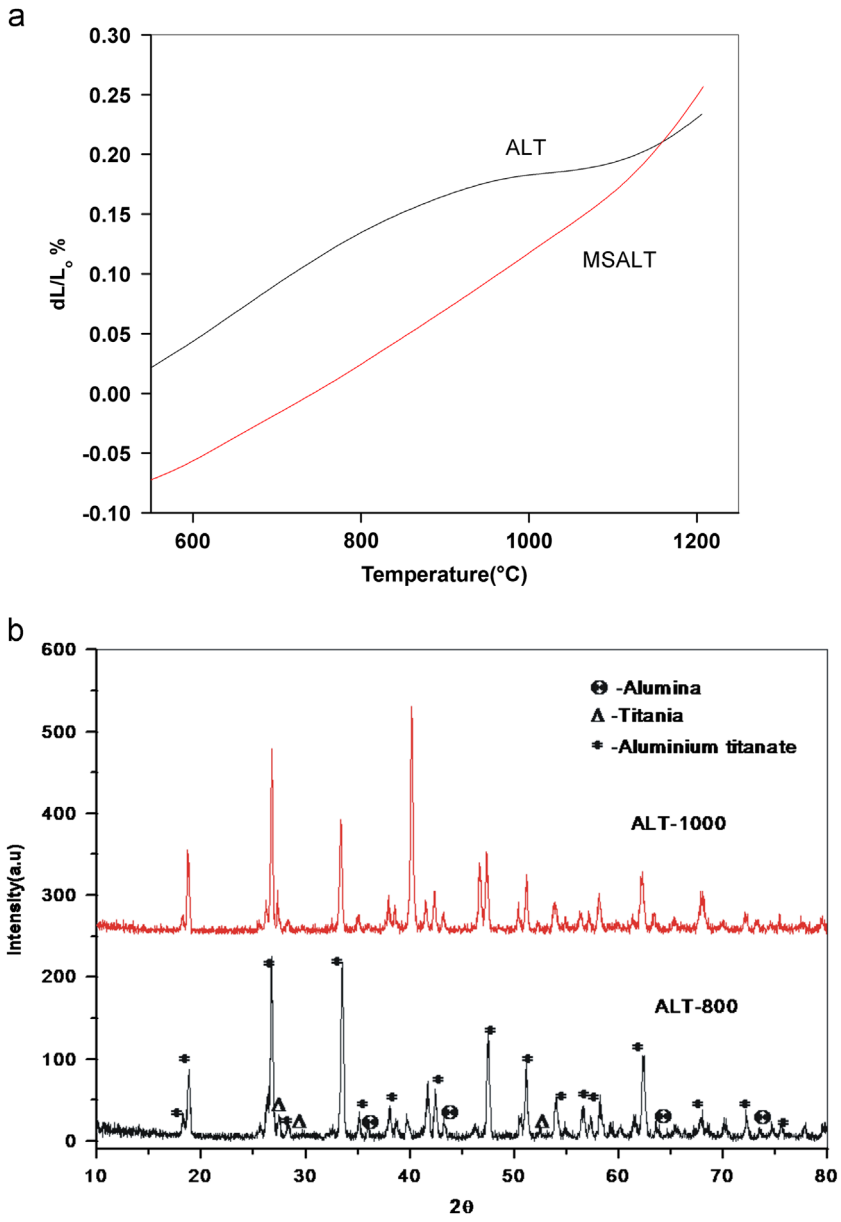

Fig. 4. (a) Dilatometric curve of ALT and MSALT samples upto 1200 °C and (b) high temperature XRD pattern of the ALT samples at 800 and 1000 °C.

A comparison of the microstructures of CRH treated ALT and MSALT samples exposed to 30 thermal cycles are also shown in Fig. 7(a) and (b). The CRH-ALT samples have shown a different microstructure in comparison to SPS-ALT (Fig. 5(a)) with irregular grains and precursors precipitated occasionally. MSALT samples under CRH conditions have shown occasional micro-cracking and no precipitations of precursor oxides could be seen. Fig. 7(b) shows the microstructure of the MSALT sample without decomposition complementing XRD data.

3.4. Thermo-mechanical properties

Flexural strength, hardness and CTE values are measured for ALT and MSALT samples, under various CRH cycling conditions and are depicted in Table 1. Flexural strength of SPS samples is not measured because of the sample size limitations however; hardness and CTE of the samples in both ALT and MSALT subjected to SPS conditions were measured. SPS samples have shown a hardness values of 906 ± 35 HV and 638 ± 25 HV for ALT and MSALT respectively in comparison to 170 ± 20 and 310 ± 30 HV observed for ALT and MSALT samples as prepared with $> 90\%$ Al_2TiO_5 phase. The increase in hardness of SPS samples by 432% and 105.43% in both ALT and MSALT samples can be attributed to the presence of high hardness corundum (α-Al_2O_3) phase and titania providing a composite effect. Further, it is evident from the microstructure that decomposed phases show an average grain size of less than 1 μm enhancing the mechanical properties.

Thermal cycled samples have shown an increase in the hardness from 170 ± 20 to 370 ± 35 HV for ALT samples. However, the hardness values of the MSALT samples do not show a significant change (as the phase composition remains

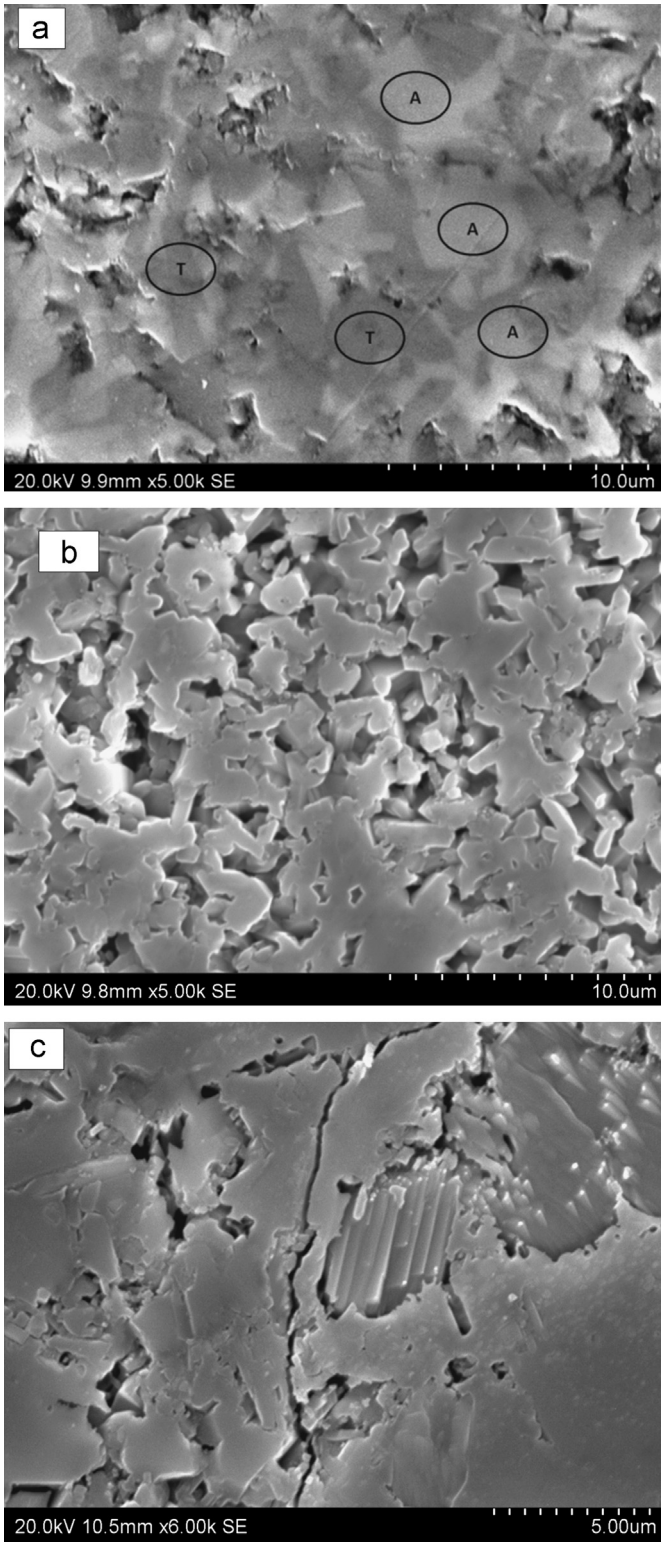

Fig. 5. Microstructure of (a) partially decomposed ALT samples, (b) fully decomposed ALT samples and (c) MSALT samples under SPS conditions.

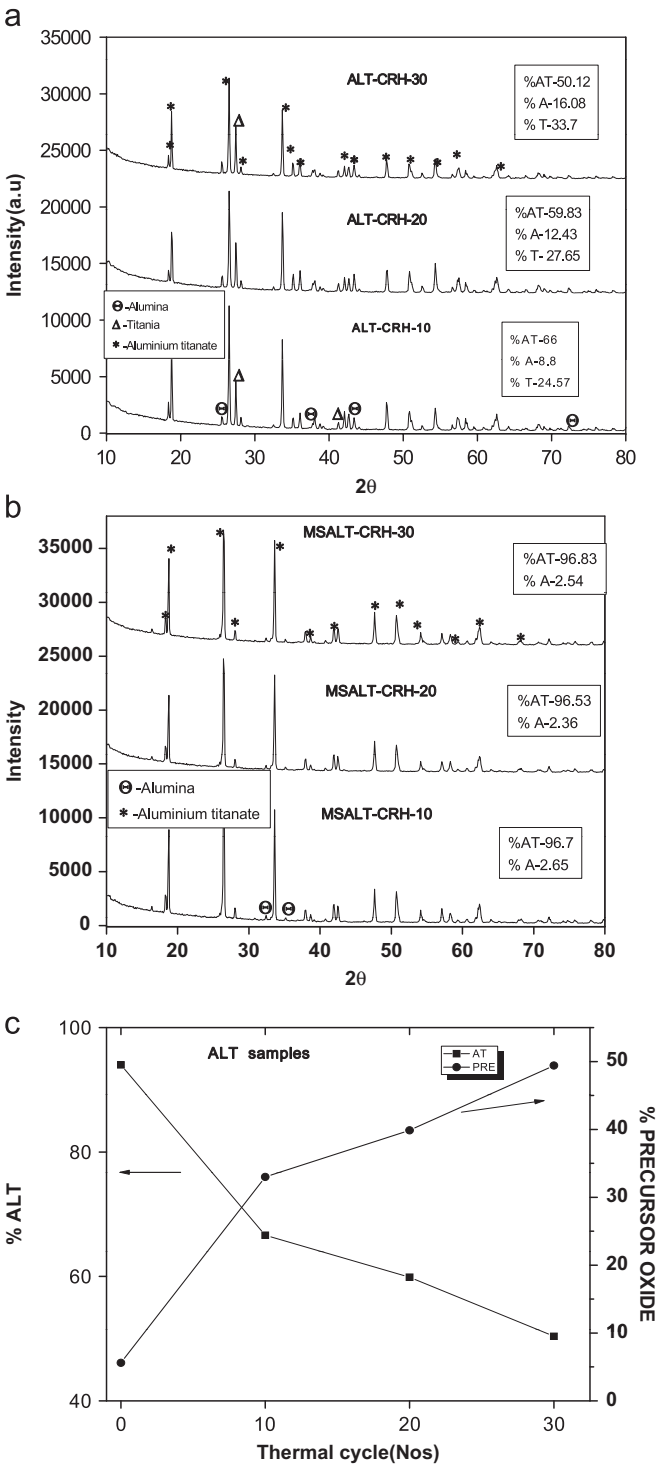

Fig. 6. XRD pattern of the thermal cycled (10, 20 and 30) (a) ALT samples, (b) MSALT samples and (c) Plot of % Al_2TiO_5 phase retained and % of precursor oxide under thermal cycles of 10, 20 and 30 of ALT samples.

unchanged) as a result of thermal cycling. Increase in flexural strength was ~54% for ALT samples signifying the composite effect as discussed in the case of hardness and a marginal increase in flexural strength by ~15% is observed for MSALT samples.

Coefficient of thermal expansion (CTE) of the samples is found to be complementary with the decomposition patterns observed with SPS and CRH samples. CTE values of 7.62×10^{-6}/°C and 5.5×10^{-6}/°C are observed with SPS samples with ~5% and 36% residual Al_2TiO_5 phase. Enhancement of CTE values from 1.09×10^{-6}/°C (for ALT-CRH) to higher values can be attributed

Table 1
Flexural strength, hardness and CTE of ALT and MSALT samples subjected to SPS and CRH thermal cycling conditions.

Sample	Flexural strength (MPa)[a]	Hardness (HV)[*]	CTE($\times 10^{-6}$/°C, 30–1000 °C)
ALT (SPS-200 °C/min)	–	906 ± 35	7.63
MSALT (SPS-200 °C/min)	–	638 ± 25	5.51
ALT-0cycling	11.83 ± 1.5	170 ± 20	1.09
ALT-30cycling	18.13 ± 0.7	370 ± 35	2.53
MSALT-0cycling	26.53 ± 0.5	310 ± 30	0.42
MSALT-30cycling	30.57 ± 2	330 ± 30	0.47

[a]Average of 5 readings.
*Average of 10 readings.

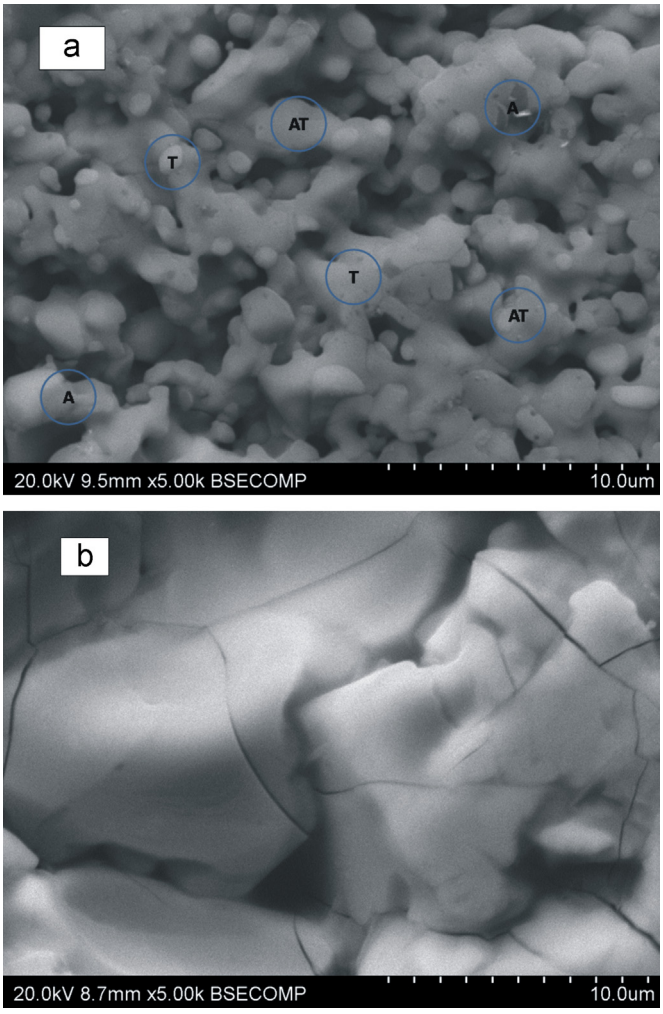

Fig. 7. Microstructures of (a) ALT sample and (b) MSALT sample exposed to 30 thermal cycles.

to the presence of precursor oxides of α-Al_2O_3 (8.0×10^{-6}/°C) and TiO_2 (9.0×10^{-6}/°C) respectively in the temperature range of measurements. A similar enhancement in CTE value of 30 thermal cycled ALT sample (2.5×10^{-6}/°C) is observed, it can be attributed to the residual Al_2TiO_5 phase (46%). In the case of MSALT samples CTE values remained almost same further confirming the elimination of Al_2TiO_5 phase decomposition.

4. Conclusion

Eutectoid phase decomposition of Al_2TiO_5 is studied under Spark Plasma (SPS) and Conventional Ramp and Hold (CRH) conditions. Decomposition of Al_2TiO_5 phase close to 100% in the case of SPS conditions can possibly be attributed to plasma generated in the initial stages of SPS condition that produces the reactive surfaces, extremely high heating rate of 200 °C/min and argon atmosphere. $Mg_3Si_4O_{10}(OH)_2$ doping in Al_2TiO_5 suppressed the decomposition to considerable extent retaining by 36% of the residual Al_2TiO_5 phase due to the crystallographic stabilization. Conventional Ramp and Hold (CRH) with a heating rate of 100 °C/min under pressure-less condition of ALT samples lead to Al_2TiO_5 phase decomposition to an extent of 54% after 30 thermal cycles and complete suppression of phase decomposition in $Mg_3Si_4O_{10}(OH)_2$ doped Al_2TiO_5 (MSALT) samples. A comparative evaluation of microstructure of samples is found to correlate well with the Al_2TiO_5 phase decomposition elucidated through XRD measurements. Further, dilatometric measurements indicated slope changes in the temperature regime of decomposition. A significant increase in hardness and thermal expansion values are observed with ALT samples treated under SPS condition. However, the enhancement of hardness and thermal expansion values were moderate in CRH thermally cycled ALT samples. Flexural strength values of CRH thermally cycled ALT samples also exhibited a marginal increase due to the composite matrix formation. No significant change in thermo-mechanical properties of $Mg_3Si_4O_{10}(OH)_2$ doped Al_2TiO_5 (MSALT) samples were observed in the present study confirming the observed trends in decomposition.

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