Application of two-step sintering cycle to 20–30 nm $\alpha$-Al$_2$O$_3$ compacts to investigate microstructure, hardness, strength, density and corundum formation

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Abstract

This paper discusses the hardness variation of 20–30 nm $\alpha$-Al$_2$O$_3$ sintered specimens by two-step sintering (TSS) technique. This study mainly reveals the microstructure transformation of nano alumina into corundum grains. The uniaxial load of 4 ton was used to produce alumina compacts of 11 mm diameter and 3.5 mm thickness. In this two-step sintering process, high step temperature ($T_1$) varied from 1250 °C to 1600 °C, whereas low step temperature ($T_2$) was between 1100 °C to 1550 °C. To achieve hard nano alumina structure, seven TSS cycles were designed by varying the heating rate ($°\text{C/}\text{min}$), high step temperature ($T_1$), high step temperature holding time ($t_1$), low step temperature ($T_2$), and low step temperature holding time ($t_2$). After each TSS cycle, the Vickers hardness (HV), load-bearing capacity (KN), ultimate compressive strength (MPa), yield strength (MPa) and density (g cm$^{-3}$) measurements of each sintered sample were carried out. The significant improvements observed in hardness and density of sintered nano alumina with an increase in high step temperature ($T_1$) as well as holding time ($t_1$). The microstructure transformation and material elemental analysis were carried out using Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) respectively. It is evident from SEM micrographs that the specimen of sintered samples with high step temperature ($T_1$) 1600 °C with 2 h holding time, low step temperature ($T_2$) 1550 °C with 10 h holding time produced corundum grain transformation in the nano alumina matrix.

1. Introduction

Alpha-Alumina ($\alpha$-Al$_2$O$_3$) is a potential ceramic to meet dry cutting conditions in machining industries at ultra-high-speed. It possesses a combination of outstanding mechanical properties like high hot hardness, high compression strength, excellent abrasion resistance, superior refractory characteristics, chemical stability and retention of strength at high temperatures [1, 2]. The fabrication of alumina sintered products with nanopowder produced excellent mechanical properties. This is due to the distribution of smaller size particles of powder in green compacts leads to higher green density and enhances sintering rate [3]. Nanopowder under consideration in this study consists of high specific surface area (m$^2$ g$^{-1}$) and high surface energy which leads to the development of higher interfacial particle bonding strength during sintering. Moreover, it exhibits good flexural strength and fracture toughness as compared to micro-scale alumina [4].

Earlier, literature has focused on sintered alumina nanoparticles using different methods like conventional sintering [5–7], conventional sintering by chemical synthesis [8], gel-casting shaping method [9], hot isostatic pressing [10, 11], microwave sintering [12, 13], spark plasma sintering [14] etc.

Two-step sintering (TSS) is one of the simplest and cost-effective methods which controls the sintering process in two steps to achieve high-density ceramic through controlled grain growth without adding any
dopants\cite{15}. TSS was first reported by Chen and Wang\cite{16} and successfully applied on yttrium oxide (Y$_2$O$_3$) to achieve high densified Y$_2$O$_3$ ceramic bulk. It is a modified form of the conventional sintering process.

Later on, more studies have emphasized the TSS processing method with distinct particle size and various two-step temperature ranges. Jiangong Li and Yinping Yez\cite{17} worked with a distinct range of nano alumina particles in conventional as well as two-step sintering technique. These authors concluded that in TSS the densification and grain growth occurred at different temperature limits. Bodisova K et al\cite{18} worked on the elimination of porosity in densified alumina having sub-micrometre particle size of 150–200 nm. They used the two-step sintering method with $T_1$ and $T_2$ in the range of 1330 °C–1450 °C and 1100 °C–1160 °C respectively. Jen wang et al\cite{19} also used the TSS method to produce high densification alumina-zirconia composite. Zirconia induced pinning effect on grain boundary migration of alumina. Moreover, sintering temperature $T_1$ and $T_2$ were optimized and it was suggested that $T_1$ should be above 1450 °C and $T_2$ in the range of 1350 °C–1400 °C. Loh et al\cite{20} recently applied TSS technique to achieve high densified commercial alumina–92%, 96% and 99% purity and found better properties in alumina–99% amongst them. The best combination of $T_1$ and $T_2$ was 1550 °C with 5 min holding time and 1500 °C with 4 h respectively. Finally, they achieved 96% relative density with 0.94 ± 0.15 μm grain growth suppression. Narutaki et al\cite{21} compacted pure alumina (99.99%) of 0.22-micron particle size using high-speed centrifugal forces and reported that a sintering temperature of 1230 °C produced a new ceramic cutting tool suitable for dry machining operations.

Majority of previous studies suggests that sintering temperatures and corresponding relative densities vary with alumina particle size\cite{18}. Furthermore, we experimented the TSS cycles from previous studies on 20–30 nm particles, but results were not encouraging to get the hard alumina structure.

As per best of the authors’ knowledge, the two-step sintering of pure alumina of 20–30 nm and its microstructure transformation has not been explored much up till now. Hence, seven new TSS cycles were designed to sinter this 20–30 nm alumina green compacts which were compacted by laboratory uni-axial press. The method for achieving a high hardness of nano α-Al$_2$O$_3$ pellets is discussed using TSS cycles in the results and discussion section of this study; it also includes microstructural characterization, ultimate compressive strength and its fracture for TSS-5, TSS-6 and TSS-7 in order to examine structural changes and failure behaviour under compression.

### 2. Materials

The Alpha-alumina (α-Al$_2$O$_3$) powder having 20–30 nm particle size and 99.9% chemical purity was procured from Sisco Research Laboratories, India. The x-ray Diffractometer (XRD) (Make: X’pertPRO PANalytical) analysis is used to confirm the α-alumina phase in the powder substrate under consideration as shown in Figure 1(a). Further, the particle size in the range of 20–30 nm was confirmed by ImageJ software tool using micrographs produced by Field Emission Scanning Electron Microscope (FESEM) (Make: JEOL JSM7610F) shown in Figure 1(b). Poly Vinyl Alcohol (PVA)\cite{22} was used as a temporary binder in alumina powder to obtain strengthened green compacts which generally gets evaporated approximately about 600 °C. Agate Mortar and Pestle set was used to prepare the homogeneous mixture of alumina nanopowder and PVA binder.
3. Experimental methods

The homogeneous mixture of nano alumina powder and PVA binder was pressed to make round green pellets by laboratory uniaxial hydraulic press (Make: KIMAYA Engineers, Capacity: 15 Ton). The alumina green compacts of 11 mm diameter and 3.5 mm thickness were produced as shown in the Figure 2. The two-step sintering (TSS) was carried out using a Muffle furnace (Make: Dass & co) (1600 °C) equipped with a microprocessor which automatically controls sintering parameters like heating rate, holding time, and cooling rate. The surface hardness of the sintered sample after each TSS cycle was measured using Vickers hardness tester (Make: Mitutoyo, Model: HM200). Likewise, after each TSS cycle, the density of sintered samples was also measured using setup arranged with the Archimedes principle.

Servo hydraulic universal testing machine (Make: Instron, Model: 8802, Capacity: 250 KN) was used to measure the compressive strength of sintered alumina pellets with a constant crosshead speed of 0.05 mm min \(^{-1}\). The compression tests were carried out at atmospheric conditions and averaged results of three iterations are reported.

Finally, these samples were characterized by Scanning Electron Microscope (SEM, JEOL, 6380 A) to analyse the transformation of microstructures.

The Two-Step Sintering method consists of high step temperature (T1) and low step temperature (T2) with their corresponding holding times t1 and t2 respectively. The high step temperature T1 leads to the formation of the kinetics of grain boundary migration while low step temperature T2 produces grain boundary diffusion. Designing of these TSS cycles according to particle size is vital. Hence, to begin with, few existed alumina TSS cycles from literature along with employed compacting methods were summarised in table 1.

Table 1. Summary of existing TSS cycles used for α-Alumina.

| Year   | Particle size (nm) | T1 (°C) | T2 (°C) | Compaction method                                      |
|--------|--------------------|---------|---------|--------------------------------------------------------|
| 1997 [21] | 220                | 1230    | —       | Sol-gel technique                                      |
| 2006 [17] | 10, 15, 48, and 80 nm | 1380–1450 | 1330–1350, 1500 | Uni-axial pressing at different pressures (300 to 1500 MPa) |
| 2007 [18] | 150–200            | 1330–1450 | 1100–1160 | Uni-axially pressed at 100 MPa.                        |
| 2009 [19] | 150                | 1400–1450 | 1350–1400 | Uni-axially pressed at 150 MPa.                        |
| 2017 [20] | 730–2160           | 1550    | 1500    | Uni-axially pressed at 140 MPa.                        |

It was observed from table 1 [17] that researchers have applied TSS method to mean particle size of 10, 15, 48 and 80 nm particles only, so the range chosen for current study is 20–30 nm which falls between 15 to 48 nm particle range, where, there was a scope to investigate furthermore by TSS method.

Therefore, the sinterability of 20–30 nm alumina was intended to be verified using the TSS cycles listed in table 1. However, these TSS cycles were found to be incompatible for 20–30 nm α-alumina as those cycles produced very low hardness and density as compared to published literature.

Therefore, the applicability of new TSS cycles was verified to find the sinterability of 20–30 nm alumina particles. Seven new TSS cycles were designed based on trial and error method by varying the sintering...
parameters like heating rate, T₁, t₁, T₂ and t₂ as given in table 2. The detailed schematics of these TSS temperature flow curves are represented in figure 3. The temperature ranges opted for designing TSS cycles were 1250 °C–1600 °C and 1100 °C to 1550 °C for T₁ and T₂ respectively.

4. Results and discussion

4.1. Density, hardness and corundum formation

Samples of sintered alumina were produced by using seven two-step sintering cycles and those samples were analysed for hardness, density and microstructural characteristics. In each TSS cycle heating rate (°C min⁻¹), step temperatures (T₁, T₂) and their corresponding holding times (t₁, t₂) were controlled to obtain the final quality of the sinter.

More the high step temperature (HST) holding time (t₁) which leads to rapid grain growth and ultimately it results in grain boundary migration [15, 23]. Low step temperature (LST) holding time (t₂) promotes grain boundary diffusion which results in grain growth suppression and densification. Over-growth of grain causes coarsening in the sinter, therefore controlling the ‘t₁’ is necessary to obtain effective grain growth and ‘t₂’ is for grain growth suppression.

### Table 2. Experimental runs for Two-step sintering (TSS).

| Sintering cycle | H.R | T₁ (°C) | t₁ (h) | CR₁ | T₂ (°C) | t₂ (h) | CR₂ | Total CT (h) |
|----------------|-----|---------|--------|-----|---------|--------|-----|--------------|
| TSS-1          | 10 °C/min | 1250 °C  | 0      | FC  | 1100 °C  | 5      | FC  | 7.083        |
| TSS-2          | 10 °C/min | 1250 °C  | 2      | FC  | 1100 °C  | 5      | FC  | 9.083        |
| TSS-3          | 5 °C/min  | 1400 °C  | 0      | FC  | 1100 °C  | 5      | FC  | 9.667        |
| TSS-4          | 5 °C/min  | 1400 °C  | 2      | FC  | 1350 °C  | 5      | FC  | 11.667       |
| TSS-5          | 5 °C/min  | 1400 °C  | 3      | FC  | 1350 °C  | 10     | FC  | 17.667       |
| TSS-6          | 5 °C/min  | 1600 °C  | 0      | FC  | 1550 °C  | 10     | FC  | 15.333       |
| TSS-7          | 5 °C/min  | 1600 °C  | 2      | FC  | 1550 °C  | 10     | FC  | 17.333       |

H. R = Heating rate, T₁ = High step temperature, t₁ = holding time for T₁, T₂ = Low step temperature, t₂ = holding time for T₂, CR₁, CR₂ = cooling rates, FC = Furnace cooling, CT = Cycle time.

Figure 3. Designed two-step sintering curves for: (a) TSS-1, (b) TSS-2, (c) TSS-3, (d) TSS-4, (e) TSS-5, (f) TSS-6 and (g) TSS-7.
In this experimentation, seven experiments from TSS-1 to TSS-7 were performed as shown in Table 2. The microstructure and its corresponding results of each TSS cycle are briefly discussed from TSS-1 to TSS-7 to understand the method of obtaining hard nano alumina sinter.

TSS-1 and TSS-2 were designed and executed on alumina powder (20–30 nm, 99.9% purity) based on the study of Narutaki et al.\(^\text{[21]}\), who had sintered hard alumina for dry-machining purpose using sol-gel compaction method and conventional sintering at 1230 °C with 1.5 h.

The TSS-1 had no holding time \((t_1)\), whereas TSS-2 had a two-hour holding time \((t_1)\) at high step temperature \((T_1)\). The TSS-1 produced hardness of 180 HV with loosely packed alumina and powdery surface. Whereas, TSS-2 with higher holding times \(t_1 2 \text{ h}\) and \(t_2 5 \text{ h}\) exhibited improvement in hardness i.e. 250 HV. Still, it is observed that the nano alumina sinter was loosely packed which ascertain the fact that 1250 °C was insufficient to form hard ceramic solid. So few more TSS cycles were designed with higher step temperatures and by varying holding times.

TSS-3, TSS-4 and TSS-5 cycles as shown in Table 2 were designed and experimented with temperatures between 1400 °C and 1100 °C based on the study of Bodisova K et al.\(^\text{[18]}\) and experimental results were discussed in the subsequent part of this study.

Bodisova K et al. executed a two-step sintering technique with \(T_1 = 1330 °\text{C–1450 °C}\), \(T_2 = 1100–1160 °\text{C}\) and achieved high relative density and grain growth suppression in the final stage. In TSS-3, the high step temperature was raised to 1400 °C with a heating rate of 5 °C/min without any holding time. This raised temperature \(T_1\) and reduced heating rate somewhat improved hardness and density to 320 HV and 3.012 g cm\(^{-3}\) respectively as compared to TSS-2. These results revealed that the compacts were not showing significant hardness of alumina. But, the lower heating rate (5 °C min\(^{-1}\)) improved the sinter properties due to slow and steady heating and therefore, remaining TSS cycles were executed with a lowered heating rate of 5 °C min\(^{-1}\).

Similarly, TSS cycle—4 executed with a heating rate of 5 °C min\(^{-1}\), \(T_1 = 1400 °\text{C}, t_1 = 2 \text{ h}, T_2 = 1350 °\text{C}, t_2 = 5 \text{ h}\) and using a higher low step temperature \((T_2)\) of 1350 °C. In this cycle, the holding time of 2 h at 1400 °C resulted in higher hardness and density of 450 HV and 3.12 g cm\(^{-3}\), respectively.

In general, micrographs of hard alumina would produce crystalline (hexagonal or rhombic) grain structures which are also called corundum with well-grown grain boundaries\(^\text{[23]}\). But any notable grown crystalline structure was not observed in the micrographs of the initial four cycles (TSS-1–TSS-4). Hence the further detailed microstructural analysis of previously mentioned cycles is not discussed here.

To investigate further, the effect of higher holding time \(t_1\) at a higher temperature TSS-5 cycle was designed with 3 h holding time at 1400 °C. This exhibited 44% increase in hardness to 800 HV along with 2.5% increase in density, still, it was observed that microstructure with irregular pattern (vermicular shaped) of sintered alumina in TSS-5 as revealed by SEM micrographs shown in Figure 4.

The similar microstructures were observed by Brosnan et al.\(^\text{[12]}\) and Azar et al.\(^\text{[24]}\) in their conventional sintering of alpha alumina and transition alumina respectively at 1400 °C. The observed vermicular microstructure reflected the inability of TSS-5 cycle to produce substantial grain growth for obtaining desired

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**Figure 4.** SEM observations showing the vermicular microstructure of sintered alpha-alumina by TSS cycle-5: (a) 5000 magnified and (b) 10000 magnified images.
hardness. Therefore, it is concluded that the 1400 °C temperature was inadequate to transform vermicular microstructure into polycrystalline or orderly form. Hence, supplying more heat at higher temperature was desirable to form the orderly crystalline structure of 20–30 nm alumina.

The TSS-6 cycle as given in table 2 was executed with elevated step temperatures $T_1 = 1600 ^\circ C$ and $T_2 = 1550 ^\circ C$ with a corresponding holding time of $t_1 = 0$ h and $t_2 = 10$ h, respectively. The hardness obtained was nearly 1200 HV and density was 3.38 g cm$^{-3}$. The elevated high step temperature resulted in disappearing the vermicular microstructure and had coalesced the grains together as shown in figure 5(a). The similar microstructures were observed from the literature of Loh et al who sintered pure alumina at 1500 °C with 4 h holding time, further Hotto and Goto [25] also observed same microstructure in spark plasma sintering of pure alumina at 1200 °C.

The grain development and neck formation had observed in various regions in the magnified (10000x) image due to grain boundary diffusion as shown in figure 5(b). In addition to this, hardness and density were proportionally increased; the percentage increase in Vickers hardness is by 52.50% and density is by 5.62% compared to TSS-5. Here, few regions of the orderly structure were observed in TSS-6 due to the effect of grain boundary diffusion. To get this orderly structure throughout the matrix uniformly, the substantial holding time is desirable at $T_1$.

Finally, the TSS-7 cycle was designed with slightly modified parameters in TSS-6 with high step temperature holding time $(t_1)$ 2 h and the rest of the parameters were similar to TSS-6 as shown in table 2. The use of even higher holding times (like conventional sintering) reduces the hardness due to grain boundary migration which results coarsening effect in grain structure [15]. Therefore controlling the high step holding time $t_1$ is crucial in two-step sintering cycles [15]. With this view, the holding time for $T_1$ temperature has selected within the time span of 2 h. The TSS-7 produced Vickers hardness of 1420 HV and 3.56 g cm$^{-3}$ density as shown in figures 7(a) and (b). The obtained grain structure as shown in figure 6(a) explains that the formation of polycrystalline structure in a sintered alumina matrix, which also looks like rock structured shapes with hexagonal or rhombic crystal shape. These grains are called corundum grains, which are very hard and abrasive in nature.

These microstructures were comparable with published literature of Legros et al [26] who observed in transition alumina conventional sintering at 1450 °C with 4 h holding time, Hotto and Goto [25] also observed this rock structured alumina in spark plasma sintering at 1400 °C, Azar et al [24] reported similar microstructure in nano-structured transition alumina sintering at 1700 °C with slip casting compaction method, Dey and Biswas [27] identified similar micrographs with zirconia distribution in sintering of zirconia-toughened alumina with various metal oxides and Broniszewski et al [28] also reported corundum grains in alumina-vanadium composite with hot pressing method with parameters $T = 1350 ^\circ C$, $t = 1$ h, $p = 20$ MPa, argon atmosphere.

The neck formation was observed in the developed corundum grains as shown in figure 6(b). This might be due to the collective effect of evaporation-condensation, volume diffusion of a particle, grain boundary diffusion, surface diffusion etc, due to elevated temperatures.

All the seven TSS cycles’ mean values of three iterations of hardness and density are graphically represented in figures 7(a) and (b). The final sinter obtained from TSS-7 shows the transformation of microstructure to corundum which exhibits high hardness and density.
Overall, the hardness and density properties experienced an upward trend from TSS-1 to TSS-7. Initially, the relative increase in hardness was less till TSS-4 followed by a sudden growth in TSS-5 and TSS-6 as shown in 7(a). Whereas, the density growth was bit higher initially; afterwards, the relative growth was less beyond TSS-5 as shown in figure 7(b).

The hardness versus density graph figure 8, which gives a correlation of the hardness variation to densification.

Overall, the hardness of the nano alumina showed a steady and significant rise over densification. The growth of the hardness was steady from density value 2.5 to 3.1 g cm$^{-3}$. Afterwards, it increased sharply with small variation in density and finally reached 1420 HV with a density of 3.56 g cm$^{-3}$.

Energy Dispersive x-ray Spectroscopy (EDS) analysis was also carried out to identify the composition of material elements in sintered alumina ($\alpha$-Al$_2$O$_3$). The alumina ($\alpha$-Al$_2$O$_3$) sintered samples were free from any foreign elements as observed from the EDS graphs of TSS-5 to TSS-7 as shown in figures 9(a)–(c). Hence, it is concluded that the atmospheric two-step sintering was appropriate for pure alumina without any oxidation or undesirable chemical reactions.

4.2. Comparison of the obtained results with other alumina nanoparticles $<100$ nm
Researchers have reported difficulties in sintering pure Al$_2$O$_3$ with less than 100 nm particle size using hot pressing [29], spark plasma sintering [30], pulse electric current sintering [31] etc. They reported comparatively lower relative density with finer grains. However, Li et al [17] studied the effect of $<100$ nm mean particle size of alumina (10, 15, 48, and 80 nm) by TSS method for the relative density of alumina sinter at elevated
Figure 8. Shows hardness variation of nano alumina with densification.

Figure 9. EDS analysis of alumina ($\alpha$-Al$_2$O$_3$) for (a) TSS-5 (b) TSS-6 and (c) TSS-7 cycle.
temperatures. Relevant findings reported by the authors are (i) The relative density of sintered samples increases with increase in applied compaction pressure, (ii) At a particular compaction pressure and sintering temperature, the increase in particle size resulted to gradual decrease in relative density, (iii) For 48 nm mean particle size, the relative density was increased with increase in sintering temperatures, (iv) The rock structured grains or corundum grains were reported for 10 nm alpha-alumina mean particle size compacted at 1120 MPa and sintered at 1500 °C with 5 h holding time.

Similarly, in the current investigation for alumina 20–30 nm sintering by different TSS cycles, a directly proportional relationship was observed between temperature & density (figure 7(b)). Also, temperature & hardness (figure 7(a)), density & hardness (figure 8) are having a directly proportional relationship.

The results in this study revealed that 20–30 nm alumina is not sintered properly by low temperatures below 1400 °C to produce the polycrystalline structure.

The samples fabricated by the TSS cycle—7 having T₁ is 1600 °C with 2 h holding time produced a polycrystalline structure having high hardness and high density. It is also important to study of other mechanical properties like ultimate compressive strength, yield strength, load-bearing capacity (KN) and fracture behaviour on these samples to confirm the applicability of TSS cycle - 7 and which are compared with published literature in the subsequent section.

4.3. Compressive mechanical properties of TSS-5, 6, and 7

Experiments carried out on sintered alumina pellets to explore compressive mechanical properties like load carrying capacity (KN), Yield Strength (MPa), Ultimate strength (MPa), fracture behaviour for TSS-5,6 &7. As the objective of this investigation is to develop hard sinter to be used as dry machining tool, the sintering cycles TSS-1 to TSS-4 are not considered for detailed analysis as they failed to satisfy the hardness criteria.

In general, the alumina compressive strength was strain rate insensitive in low strain rate range of 10⁻⁵ to 10⁻¹/s but it is sensitive in the high strain rate range of 10⁻¹ to 10⁶/s [32, 33].

It is observed from the studies of Manjima Bhattacharya et al [33], alumina ceramic mean compressive strength is constant in low strain rates of 10⁻⁵ to 10⁻¹/s, and beyond the strain rates of 10⁻¹ to 10⁶/s the compressive strength is higher and more sensitive.

Hence, the constant strength which lies in the low strain rates of 10⁻⁵ to 10⁻¹/s reported by Manjima Bhattacharya, is considered as a basic strength or effective strength of the alumina ceramic which is approximately 1 GPa or 1000 MPa.

The experiments were conducted as per the published literature [33] to find the compression strength of sintered nano alumina samples of TSS-5, TSS-6 and TSS-7. The strain rate under consideration is 0.05/min or 8.33 × 10⁻⁴/s throughout the experiments. The samples are kept between anvils as shown in figure 10(a). The experiments were conducted until the fracture failure of samples. The corresponding compressive load (KN) and compressive extension (mm) were recorded as shown in figure 10(b) by a data acquisition system (DAQ). These load Versus compressive extension data converted into the stress-strain values to identify the strength of sintered alumina for TSS 5,6,7.

The obtained stress-strain curves of TSS 5, TSS 6, TSS 7 shows significant variation in strength. Among the three TSS cycles, the samples from TSS 7 exhibits more strain and strength. The TSS-5 and TSS-6 are showing less load carrying capacity and less compressive strength. The compressive strength of TSS-7 approximately 50% higher than the TSS-5 and TSS-6 and the strain value achieved in TSS-7 is approximately 30% and the
corresponding strength value is around 808 MPa, which are shown in Figure 11. This strength value is comparable with the study of Yoshihiro Hirata et al who reported effective strength of alumina is 827 MPa.[34]. Earlier studies also reported the compressive strength of alumina-based in low strain rates approximately 800 to 2200 MPa[33], which depends on the relative density of sintered alumina.

Finally, the achieved properties of TSS-7 sintered alumina are tabulated in table 3.

4.4. Fracture behaviour
In the previous literature, the failure mechanism of ceramics has been reported in two ways: (1). The nucleation of microcracks, its growth and coalesce together make the sudden failure of the ceramic and (2). The ceramic damaging route including macro-cracking and fragmentation [35–40].

Based on the above failure mechanisms the fractured surface of TSS-7 sintered alumina has been studied. The Nucleation of Macro and micro localized cracks were observed from the fractured surfaces, which are illustrated with SEM micrograph as shown in figures 12(a) and (b).

The style of global brittle failure into the fragmentation of alumina samples (TSS-5, TSS-6 and TSS-7) under compression forces was also reported in figures 13(a)–(c). TSS-5 sample showing major crushed and more edge damage representing loosely sintered alumina as shown in figure 13(a). TSS-6 and TSS-7 showing less edge damage with an in-plane fracture into longitudinal direction under compression load as shown in the figure b and c respectively.

5. Conclusions
The two-step sintering method was optimized for 20–30 nm alpha-alumina to obtain hard sintered samples of 1420 HV. The vermicular microstructure which existed at 1400 °C was transformed into polycrystalline structure or corundum in cycle TSS-7 which consists \( T_1 = 1600 \degree \text{C}, t_1 = 2 \text{ h}, T_2 = 1550 \) and \( t_2 = 10 \text{ h} \). The high step temperature holding time \( t_1 \) in TSS cycle-7 was 2 h to avoid coarsening of grain structure. The obtained corundum in TSS-7 has the hardness of 1420 HV and density 3.56 g cm\(^{-3}\).

The TSS-7 sintered alumina exhibits excellent compressive mechanical properties compared to TSS-5 and TSS-6. The achieved load carrying capacity, ultimate compressive strength and yield strength of TSS-7 sintered alumina was 107 KN, 808 MPa and 529 MPa respectively. The global brittle fracture of TSS-7 sintered sample have very few fragments with less edge damage revealed that pellets were well sintered.

From all the obtained results and analysis, it is concluded that nano alpha alumina with polycrystalline or corundum structure, high hardness and high strength could be produced by using uni-axial compaction with

![Figure 11. Experimentally measured stress-strain curves of TSS-5, TSS-6 and TSS-7 sintered alumina at the low strain rate of 0.05/min or 8.33 \( \times 10^{-4} \)/s.](image)

| TSS cycle | Density (g cm\(^{-3}\)) | Vickers hardness (HV) | Load bearing capacity (KN) | Yield strength (MPa) | Compressive strength (MPa) |
|-----------|--------------------------|-----------------------|-----------------------------|---------------------|--------------------------|
| TSS-7     | 3.56                     | 1420                  | 107                         | 529                 | 808                      |
two-step sintering with Heating rate of 5 °C/min, sintering temperature of T$_1$ = 1600 °C and T$_2$ = 1550 °C with corresponding holding times t$_1$ = 2 h and t$_2$ = 10 h, which are used in TSS-7 without inert atmosphere.

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