EFFECT OF WASHING ON STRENGTH AND WEIBULL MODULUS OF ALUMINA

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CERTIFICATE

This is certified that the work contained in the project entitled “EFFECT OF WASHING ON THE STRENGTH AND WEIBULL MODULUSOF ALUMINA” by Taniya Biswas (Roll 10508010), have been carried out under my supervision and that this work has not been submitted elsewhere for a degree.

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ABSTRACT

Reactive alumina from NALCO was washed and aged in DI water for 3, 6 and 10 days. The as received as well as the aged powders were uniaxially compacted and sintered at 1500?C for different holding periods (2, 4, and 6 hours). The density and biaxial flexure strength carried out on different sintered samples show a strong effect of washing on the above property. Weibull modulus was found to be higher for longer ageing periods indicating a possibility of particle size redistribution during washing and ageing. The highest Weibull modulus of 6.57 was obtained.

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CHAPTER 1 ~ INTRODUCTION

Aluminium oxide is an amphoteric oxide of aluminium with the chemical formula Al_2O_3. It is also commonly referred to as **alumina** or **aloxite** in the mining, ceramic and materials science communities. It is produced by the Bayer process from bauxite. Its most significant use is in the production of aluminium metal, although it is also used as an abrasive due to its hardness and as a refractory material due to its high melting point.

1.1 Natural Occurrence

Corundum is the naturally occurring crystalline form of aluminium oxide. Rubies and sapphires are gem-quality forms of corundum with their characteristic colors due to trace impurities in the corundum structure.

1.2 Properties

Aluminium oxide is an electrical insulator but has a relatively high thermal conductivity (40 W/m K). In its most commonly occurring crystalline form, called corundum or ?-aluminium oxide, its hardness makes it suitable for use as an abrasive and as a component in cutting tools.

Aluminium oxide is responsible for metallic aluminium's resistance to weathering. Metallic aluminium is very reactive with atmospheric oxygen, and a thin passivation layer of alumina quickly forms on any exposed aluminium surface. This layer protects the metal from further oxidation. The thickness and properties of this oxide layer can be enhanced using a process called anodising. A number of alloys, such as aluminium bronzes, exploit this property by including a proportion of aluminium in the alloy to enhance corrosion resistance. The alumina generated by anodising is typically amorphous, but discharge assisted oxidation processes such as plasma electrolytic oxidation result in a significant proportion of crystalline alumina in the coating, enhancing its hardness.

1.3 Typical Characteristics

Fig 1.1 Typical characteristics of alumina

1.4 Crystal Structure

The most common form of crystalline alumina, ?-aluminium oxide, is known as corundum. Corundum has a trigonal Bravaice lattice. Each unit cell contains six formula units of aluminium oxide. The oxygen ions nearly form a hexagonal close-packed structure with aluminium ions filling two-thirds of the octahedral interstices.

Fig 1.2 Schematic drawing of the first two layers in alumina structure. Octahedral Al ions are black, tetrahedral are grey

1.5 Application

. The major uses of speciality aluminium oxides are in refractories, ceramics, polishing and abrasive applications.

. Large tonnages are also used in the manufacture of zeolites, coating titania pigments and as a fire retardant/smoke suppressant.

. In lighting and photography, alumina is a medium for chromatography, available in basic (pH 9.5), acidic (pH 4.5 when in water) and neutral formulations. Aluminium oxide is also used in preparation of coating suspensions in compact fluorescent lamps.

. Health and medical applications include it as a material in load bearing hip prostheses, and dental implants because of its combination of excellent corrosion, good biocompatibility , high wear resistance and high strength.

. It finds use in water filters (derived water treatment chemicals such as aluminium sulfate, aluminium chlorohydrate and sodium aluminate, are one of the few methods available to filter water-soluble fluorides out of water), and even in toothpaste formulations.

. Aluminium oxide is also used for its strength. Most pre-finished wood flooring now uses aluminium oxide as a hard protective coating. Alumina can be grown as a coating on aluminium by anodising or by plasma electrolytic oxidation. Both its strength and abrasive characteristics are due to aluminium oxide’s great hardness It is widely used as a coarse or fine abrasive, including as a much less expensive substitute for industrial diamond.

. Many types of sandpaper use aluminium oxide crystals.

. Its low heat retention and low specific heat make it widely used in grinding operations, particularly cutoff tools. As the powdery abrasive mineral aloxite, it is a major component, along with silica, of the cue tip "chalk" used in billiards.

. Its polishing qualities are also behind its use in toothpaste.

. Aluminium oxide is widely used in the fabrication of superconducting devices

. Aluminium oxide is considered a Welsbach material. It has been suggested that this chemical could be sprayed into the upper atmosphere to reflex sunlight and thus lower the global temperature

1.6 Typical Uses

Fig 1.3 Typical uses of alumina

1.7 Weibull Modulus

The nature of flaws in most ceramics are statistical in nature.As such, the strength of ceramics is not one specific value, but a distribution of strengths. The Weibull modulus is a measure of the distribution o flaws, usually for a brittle material. The modulus is a dimensionless number corresponding to the variability in measured strength and reflects the distribution of flaws in the material. For brittle materials , the maximum strength (stress that a sample can withstand) varies unpredictably from specimen to specimen – even under identical testing conditions. The strength of a brittle material is thus more completely described with a statistical measure of this variability, eg. The Weibull modulus. For example, consider strength measurements made on many small samples of a brittle material such as ceramic. If the measurements show little variation from sample to sample , the Weibull modulus will be high and the average strength of the material would be a good representation of the potential of sample-to-sample performance of the material. The material is consistent and flaws—due to the material itself and or the manufacturing process—are distributed uniformly and finely throughout the material. A low Weibull modulus reflects a high variation in measured strengths and an increase in the likelihood that the flaws will tend to congregate and produce a weaker material. A material with low Weibull modulus will likely produce products where the strength is substantially below the average and show greater inconsistency of strength. Such products will exhibit greater variation in strength performance and will probably be less reliable.

CHAPTER 2~ LITERATURE REVIEW

2.1 Reliability Of Alumina Ceramics :Effect Of Processing

The mechanical properties that can currently be achieved with the best alumina powders available by utilizing a series of simple processing strategies and surface preparation techniques. The processing techniques include cold-isostatic pressing, slip casting, settling and centrifuging as well as hot-isostatic pressing of as-sintered alumina samples. the fracture strength can be increased from 460 MPa for cold-isostatic pressing to 925 MPa for a centrifuged and cast alumina slip, the Weibull modulus remained between 8 and 12 for most processing strategies.

The reliability of Alumina, is mainly determined by two factors: fracture strength and Weibull modulus (m). These are primarily governed by the processing technology, especially the consolidation technique. Weibull modulus (m) of 24 for alumina can be achieved by consolidating concentrated slurry obtained by centrifuging. But it was concluded that the mean size of the defects (pores due to soft or hard agglomerates) was reduced by the colloidal consolidation technique, but that the distribution of the defects and thus m remains unaffected. The larger pores, which are responsible for failure and determine the Weibull modulus, however, remained unaffected.

The strength of the hot-isostatically pressed samples is reduced when compared with the sintered samples. This strength degradation can be correlated with an increased average grain size, which leads to a reduction in strength. When optimization by hot-isostatic pressing is contemplated, a balance between pore closure and grain growth therefore is required. The strength may still be affected by residual stresses introduced during machining. The Weibull modulus remained unchanged if comparing samples with ground and polished surfaces (G = 1.4 pm) and if comparing samples having ultrasonically treated surfaces with ground and finally polished surfaces (G = 1.3 pm). Grinding and polishing leads to an increase in strength with some specimens due to an alleviation of edge flaws and introduction of a compressive surface stress.” At the same time, coarse-grained alumina is more susceptible to severe surface degradation and wear-induced microfracture associated with pull-out of large grains. These grains in combination with neighbouring pores can lead to an additional failure population, reducing the strength of some of the specimens and in the last consequence changing the Weibull modulus. This low Weibull modulus of $m = 5$ is omparable to the lowest Weibull modulus of the largest grain size alumina of $m = 5.8$, where abnormal grain growth and localized grain pull-out were suggested to lead to an additional failure origin.

2.2 Effect of Grain Size on Reliability of Alumina

The grain size dependence of fracture strength and Weibull modulus of alumina using a high purity, commercial starting powder was investigated. In the regime of an average grain size between 1.7 and 11, fracture strength increases with decreasing grain size. No dependence of Weibull modulus on average grain size and hence on R-curve behaviour could be observed. The reliability of ceramics is governed by two factors; the strength and the variability of strength. An approach to increase the strength of polycrystalline ceramics is afforded by the reduction of the average grain size.The relationship between strength and grain size has generally been described using a (T versus G’/2 (strength versus inverse square root of the average grain size) diagram. With large grain sizes, an increase in strength with decreasing grain size is observed. In the fine grain size regime the size of the processing defects is larger than the grain size.Weibull modulus doesn’t depend on grain size.

2.3 Processing Defects and their Relevance to Strength in Alumina Ceramics Made by Slip Casting

Al_2O_3 made by slip casting inherently contained the elongated and the spherical shaped defects. The pores of elongated shape were formed through the liquid flow during the casting process, since they were found in all slip cast specimens and not found in the spontaneously dried specimen where no rigorous flow of water happened. The origin of spherical pores was likely due to the entrapped air bubbles during de-airing procedure. Their removal by de-airing as easy for a dispersed slurry having a low viscosity, but difficult for a flocculated slurry of high viscosity. The Weibull’s plots for the Flexural strengths are essentially the same in the region of high strengths. Specimens made from the flocculated slurry contain a higher concentration of the spherical pores, and some of the resultant specimens have low strength. The lower strength of those ceramics has been ascribed to more detrimental defects, i.e. the spherical ones. Clearly, there are two types of defects in the ceramics made by the slip casting. One is of elongated shape and the other of a spherical shape. The former and the latter defects govern the fracture of ceramics in the regions of high strengths and low strengths, respectively. The characteristics of the latter defects are sensitive to the slurry properties, contrary to those of the former which appear not to be susceptible. Specimens made from the flocculated slurry contain a higher concentration of these spherical pores, and some of the resultant specimens have low strength. Clearly, the pores of elongated shape are formed through the liquid flow during the casting process. They are found in all slip cast specimens in this study, and are not found in the specimen made by a spontaneous drying where no rigorous flow occurred or capillary suction of water to the mold is present. Their characteristics are rather insensitive to the slurry properties in the present system.

This is interesting, since it was believed that they affected the structure and properties of ceramics at the start of the present study. Their strong effect on the pore size distribution has been well known for sintered bodies made through the powder compaction method. The Weibull's plots for the flexural strengths are essentially the same in the region of high strengths.

2.4 Pore Defect Related to Slurry Character and their Relevance to Strength Distribution in Alumina Ceramics

A significant difference of strength distribution was noted for alumina ceramics made through powder granule compaction starting with dispersed and flocculated slurry. To investigate the origin of the change, the structures of both intermediate and sintered bodies were examined. Dimples and large pores were observed in the granules made from the dispersed slurry, whereas no dimple structure was obtained from flocculated slurry. The granule defects were responsible for major defects in powder compacts, which were not eliminated during sintering. As a result, the slurry character strongly influenced the defect structure in the final sintered parts, which governed the strength distribution of ceramicThe slurry flocculation affects the granule structure, which in turn determines the defect structure in the green body and the sintered body, thus governing their strength distribution According to the strength–density relationship, these ceramics are expected to have nearly the same fracture strength, provided that they are prepared from the same powders through a similar production route. Difference in strength and its distribution for the present sintered alumina ceramics should, therefore, be explained in terms of pore size distribution. The average pore sizes for sintered body made from the dispersed slurry and that from the flocculated slurry are 26 and 19 micro-m respectively. This difference accounts for the change of the average fracture strength observed in the present ceramics, since the strength decreases as the defect size increases, the fracture toughness being nearly the same for both ceramics. Higher Weibull modulus exceeding 20 is obtained for the sample made from the dispersed slurry. This can be explained by the uniform pore size distribution as fracture origins. On the other hand, the broad pore size distribution observed for the sample made from the flocculated slurry results in lower Weibull modulus.

2.5 Effect of heat treatment of alumina granules on the compaction behavior and properties of green and sintered bodies

Variation of the strength of Al_2O_3 ceramics fabricated through the granular compaction route is governed by the pore-size distribution in the sintered bodies, especially that of large pores, measuring nearly 100 mm. Potential flaws are first introduced into the green compacts during forming, then persist or develop to strength-limiting large pore defects during the subsequent densification process. For processing using powder granules, major potential flaws in the green compacts are the residual pores or cracks in and between the trace granules, caused by the incomplete deformation and fracture of the granules. Incomplete deformation and fracture of the granules during compaction are also responsible for the density distribution in the pressed compacts, because of nonuniform powder packing between the packed layer of powder particles near the surface of the trace granules and internal less-dense regions in which pores or cracks exist. Non uniform powder packing results in differential densification between the highly packed and the less-dense regions with sintering, causing the development of void spaces in the less-dense regions to form large pore defects.[24,25] Introduction of these heterogeneities into the green compacts is related to the characteristics of the granules, as well as to the compaction behavior, so that soft and deformable granules are favored to promote uniform powder packing with better joining at their boundaries and to achieve high green density with small pore size. The present results indicate that even hard and less deformable granules can result in enhanced powder packing during compaction and in improved fracture strength of the sintered body.

Thus, the heat treatment of Al_2O_3 granules effectively reduced the size and population of potential flaws in the green bodies during compaction under a high applied pressure. Heat treatment resulted in high strength for Al_2O_3 ceramics after sintering. The hard and brittle characteristics of the heat-treated granules contributed to achieving a uniform packing structure in the green body, because the spaces between granules were efficiently filled with primary powder particles caused by fracture of the granules. On the other hand, the asspray- dried granules preserved more clear interfaces between granules and internal pores in the green compacts, resulting in the development of large pores, and, thus, a decrease in fracture strength for the sintered body.

2.6 Structure of strength-limiting flaws in alumina ceramics made by the powder granule compaction process

The powder compaction process is widely used for producing a variety of ceramic parts. The powder granules used in this process tend to form large strength-limiting flaws. The authors have examined the formation of the flaws over the entire processing stages with optical microscopy operating in the transmission mode.Formation of strength-limiting flaws was examined for alumina ceramics made through powder granule compaction process. The flaws observed as black circle dots in transmission optical microscopy were found to be cracks rather than pores. The flaws are formed due to incompletely joined granules during compaction. These flaws located near the tensile surface actually governed the strength of the specimen. The results indicate that the packing structure of granules in the green compact is critical for formation of the strength-limiting flaws and must be carefully controlled for better ceramics.

CHAPTER 3~ EXPERIMENTAL

3.1 Ageing of powders

As received powder of reactive alumina was taken and it was aged in distilled water. the ageing was carried out in a 1 liter plastic vessel. The pH was maintained between 5-6 with 1:1 HCl. After the alumina powder is settled, the water is replaced with deionized water and the pH is maintained again. This was done after every 3 days. Ageing was done for 3, 6 and 10 days. Then the settled powder is dried in oven.

3.2 Compaction and sintering

For compacting the pellets, the dried powder is mixed with calculated amount of (PVA) binder. 3% PVA solution was added. PVA was about 2.5% o the powder. It was mixed thoroughly and was scrapped out with the help of a spatula. It was then weighed and was compacted into pellets with the help of die and punch in a hydraulic press at a load of 3.5 Ton.

The green pellets were then sintered with 2 hours, 4 hours and 6 hours of soaking time, 40 pellets at each soaking time. Sintering was carried out in raising hearth furnace.

3.3 Density Measurement

Density of the sintered pellets were measured by using Archemede's principle. Kerosene was used. The dry, suspended and soaked weights of each pellet were measured to calculate the bulk density.

3.4 Strength Measurement

The strength of the sintered samples were measured in a Universal Testing Machine (UTM, Model HK10S TINIUS OLSEN). The strength was measured in Biaxial Flexural Mode on cylindrical samples, diameter 12.5 mm.The strength was calculated from the relation

$$S=2P/?dt$$

Where,

P is the breaking load in Newton

d is the diameter of the pellet in cm

t is the thickness of the pellet in cm

The failure probability was calculated.

3.5 Weibull Modulus

The most popular means of characterizing the flaw distribution is by the Weibull approach. It is based on the weakest link theory, which assumes that a given volume of ceramic under a uniform stress will fail at the most severe flaw. It thus presents the data in a format of probability of failure F versus applied stress ?, where F is a function of the stress and the volume V or area, S under stress

$$F=f\ (?, V, S)$$

Weibull proposed the following relationship for ceramics:

$$f\ (?)=(?-?_{\mu})/??)^{m}$$

Fig 3.1 Failure probability vs strength

where ? is the applied stress, $?_{\mu}$ the threshold stress (stress below which the probability of failure is zero) , ?? a normalizing parameter(often selected as the characteristics stress, at which the probability of failure is 0.632) and m is the Weibull modulus, which describes the flaw size distribution (and thus the data scatter). The probability of failure as a function of volume is

$$F = 1- exp(-?((?-?_{\mu})/??)^{m} dV)$$

Fig 3.2 Plot for calculating Weibull modulus

This results in an S shape which can be seen from Fig 3.1. Such a curve can easily be plotted from experimental data by estimating F by n/(N+1),where n is the ranking of the sample and N the total number of samples. The curve provides only an approximation of the probability of failure and does not yield the m value. Plotting ln ln (1/(1-F)), calculated using the above equation, versus ln ? results in a straight line of slope m as shown in the Fig 3.2. This form of the Weibull curve is used extensively in depicting reliability or predicted reliability of materials or components. The above two equations represent three parameter Weibull functions where ?µ, ??, and m are the three parameters. Usually, a two parameter form is used for ceramics, where the threshold stress ?µ is set equal to zero. Thus, the equation becomes

$$F = 1- exp(-?(?/??)^{m} dV)$$

Cracks initiate and propagate in ceramics under tensile loading rather than compressive loading, so that only the volume or area of material under tension is of concern in the Weibull equation. Therefore, if the full volume is under uniform uniaxial tension, the two-parameter equation becomes

$$F = 1- exp(-V(?/??)^{m})$$

If the loading is three-point bending, the effective volume under tensile stress is substantially lower.

CHAPTER 4 ~ RESULTS AND DISCUSSIONS

4.1 Relative Density of Sintered Samples

Table 4.1 Relative density of sintered samples for different soaking period

4.2 Strength as a Function of Soaking Time

Fig.4.1(a-d) shows the fracture strength against soaking time at the sintering temperature of 1500?C for as received powder as well as for samples prepared from powder which has been aged in water for 3, 6, and 10 days.

Fig 4.1(a) Fracture Strength vs Soaking Time for 3 days aged powders

Fig 4.1(a) shows the strength of sintered alumina prepared from powders which has been aged for 3 days in DI water. The strength value shows an increase with an increase in soaking time from 2 to 4 hours followed by a decrease for 6 hours soaking period. The decrease in strength at longer soaking time may be due to the grain growth which cause a drop in strength. However if we see the scattering in the strength data, it is noticed that the 6 hours strength is more reliable than strength value at 4 hours soaking because of the higher scattering for the later case.

Fig 4.1(b) Fracture Strength vs Soaking Time for 6 days aged powders

The variation in strength value with soaking time prepared from 6 days aged powder show an altogether different trend. It shows an increase in the strength value with soaking time. This indicates that during ageing there is a possibility of deagglomeration, change in particle size distribution as well as removal of water soluble impurities (Na^{+}) which alters the densification behavior and or grain size distribution of the sintered body. This probably increases the density of the sintered body. The relatively lower scattering of the strength data hints at better reliability of this sample.

Fig.4.1(c) Fracture Strength vs Soaking Time for 10 days aged powders

Fig 4.1(c) is the fracture strength versus soaking time for 10 days aged alumina powder. The variation is similar to that of 3 days ageing. A lower value of scattering for 2 and 6 hours soaking time shows reliability of strength of those two data points.

Fig 4.1(d) Fracture Strength vs Soaking Time for as received powder

Fig 4.1(d) shows Fracture strength versus soaking time for as received powder for sintered alumina prepared from as received powder. Two points are interesting in this figure, firstly the strength decreases with increase in soaking time and the value of scattering is high for all the three soaking times. This indicates that the as received powder may have impurities, agglomeration and wide distribution of particle size which causes a localized variation in density, improper densification and probably grain growth (for longer soaking times). All these may cause a decrease in strength with increase in soaking time as well as large value of scattered data.

4.3 Probability of Failure as a Function of Sintering Time

Fig 4.2 (a) F vs ? for Pellets made from Alumina powder aged for 3days (sintered at 1500°C for 2 hours)

Fig 4.2 (b) F vs ? for Pellets made from Alumina powder aged for 3days (sintered at 1500°C for 4hours)

Fig 4.2 (c) F vs ? for Pellets made from Alumina powder aged for 3days (sintered at 1500°C for 6hours)

Fig 4.3 (a) F vs ? for Pellets made from Alumina powder aged for 6days (sintered at 1500°C for 2hours)

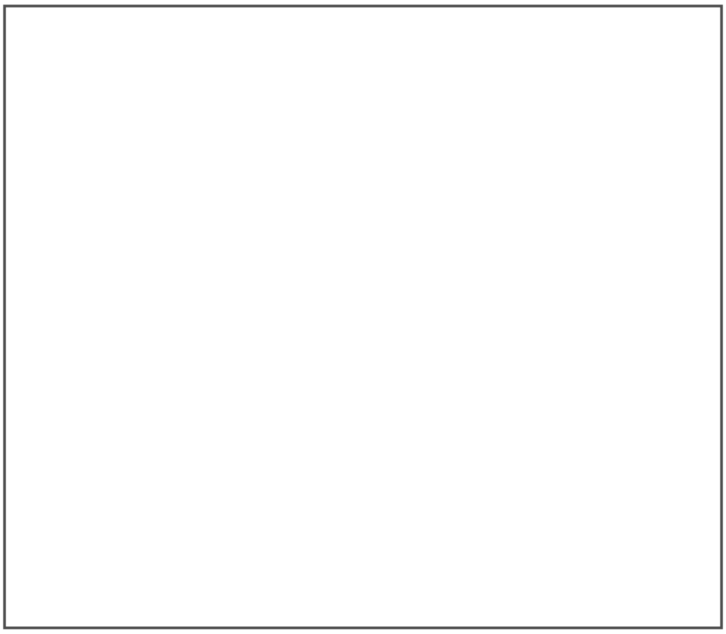

Fig 4.3 (b) F vs ? for Pellets made from Alumina powder aged for 6days (sintered at 1500°C for 4hours)

Fig 4.3 (c) F vs ? for Pellets made from Alumina powder aged for 6days (sintered at 1500°C for 6hours)

Fig 4.4 (a) F vs ? for Pellets made from Alumina powder aged for 10days (sintered at 1500°C for 2hours)

Fig 4.4 (b) F vs ? for Pellets made from Alumina powder aged for 10days (sintered at 1500°C for 4hours)

Fig 4.4 (c) F vs ? for Pellets made from Alumina powder aged for 10days (sintered at 1500°C for 6hours)

Fig 4.5 (a) F vs ? for Pellets made from As Received Alumina powder (sintered at 1500°C for 2hours)

Fig 4.5 (b) F vs ? for Pellets made from As Received Alumina powder (sintered at 1500°C for 4hours)

Fig 4.5 (c) F vs ? for Pellets made from As Received Alumina powder (sintered at 1500°C for 6hours)

All the graphs of Probability of Failure versus Fracture Strength shows an S-shape nature. At low load the probability of failure is low, increases at the intermediate stage because in this range an increase in strength increases the crack propagation probability from the cracks even with smaller flaw size. At very high load, the probability of failure is high and it remains unchanged with load because at this stage, even a very small flaw size can cause failure to the material.

4.4 Weibull Modulus as a Function of Sintering Time

Fig 4.6 (a) ln ln(1/1-F) vs ln? for pellets made from alumina powders aged for 3 days(sintered for 2 hours at 1500?C)

Fig 4.6 (b) ln ln(1/1-F) vs ln? for pellets made from alumina powders aged for 3 days(sintered for 4 hours at 1500?C)

Fig 4.6 (c) ln ln(1/1-F) vs ln? for pellets made from alumina powders aged for 3 days(sintered for 6 hours at 1500?C)

WEIBULL MODULUS OF 3 DAYS AGED POWDER

Although the fracture strength value or sintered alumina increases significantly after 3 days ageing for all soaking period, a study of the Weibull modulus for these samples (Fig.4.6(a-c)) indicates that Weibull modulus is low for 2 hours and 4 hours sintering time (in fact the Weibull modulus decreases for 4 hours sintering time although it shows the highest strength). Thus, for these two holding time, although the strength value is high, reliability of the alumina ceramic is poor. However, for samples sintered for 6 hours at 1500?C the Weibull modulus is close to 6 indicating a low scattering of strength value and thus this sample has a better reliability in comparison to the previous one.

Fig 4.7 (a) ln ln(1/1-F) vs ln? for pellets made from alumina powders aged for 6 days(sintered for 2 hours at 1500?C)

Fig 4.7 (b) ln ln(1/1-F) vs ln? for pellets made from alumina powders aged for 6 days(sintered for 4 hours at 1500?C)

Fig 4.7 (c) ln ln(1/1-F) vs ln? for pellets made from alumina powders aged for 6 days(sintered for 6 hours at 1500?C)

WEIBULL MODULUS OF 6 DAYS AGED POWDER

The weibull modulus or 6 days aged samples shows that weibull modulus is high for 4 hours and 6 hours in comparison to 2 hours sintered sample which has a low weibull modulus. Thus, it can be said that longer soaking period at the sintering temperature helps to remove the pores and flaws which not only increases the density but also improves the reliability of the material.

Fig 4.8 (a) ln ln(1/1-F) vs ln? for pellets made from alumina powders aged for 10 days(sintered for 2 hours at 1500?C)

Fig 4.8 (b) ln ln(1/1-F) vs ln? for pellets made from alumina powders aged for 10 days(sintered for 4 hours at 1500?C)

Fig 4.8 (c) ln ln(1/1-F) vs ln? for pellets made from alumina powders aged for 10 days(sintered for 6 hours at 1500?C)

WEIBULL MODULUS OF 10 DAYS AGED POWDER

The fracture strength of 10 days aged sample (Fig.4(a-c)) shows a variation with soaking period, reaching a maximum after 4 hours soaking and then decreases again. The strength is lower than that of the peak strength of 3 days aged sample. However, if we consider the reliability of the 10 days aged sample by studying the weibull modulus, it can be seen that the weibull modulus of 10 days aged samples is higher than that of the 3 days aged sample or any soaking period. Thus, although the material shows a lower strength value, a higher value of weibull modulus indicates better reliability.

Fig 4.9 (a) ln ln(1/1-F) vs ln? for pellets made from as received alumina powders (sintered for 2 hours at 1500?C)

Fig 4.9 (b) ln ln(1/1-F) vs ln? for pellets made from as received alumina powders (sintered for 4 hours at 1500?C)

Fig 4.9 (c) ln ln(1/1-F) vs ln? for pellets made from as received alumina powders (sintered for 6 hours at 1500?C)

WEIBULL MODULUS FOR AS RECEIVEID POWDER

The fracture strength of this powder is lowest. Weibull modulus is not different for soaking periods of 2 and 4 hours i.e. 3.28 and 3.8. However, ater 6 hours holding, weibull modulus drops to 2.6. The low value of these samples suggests that the sintered samples have large population of strength limiting flaws which probably arises out of improper sintering of as received agglomerated powder.

Thus, from the study it can be concluded that ageing improves reliability of these ceramics by reducing the scattering in the strength data. However, the detailed microstructure analysis, particle size distribution and sintering behavior needs to be studied to validate this point.

Table 4.2 Weibull Modulus of the aged samples for different soaking period

SOAKING TIME AGEING TIME	3 DAYS	6 DAYS	10 DAYS	AS RECEIVED POWDER
2 HOURS	3.88	3.67	5.93	3.28
4 HOURS	2.64	5.93	4.53	3.8
6 HOURS	5.93	5.92	6.57	2.67

Table 4.2 shows the Weibull Modulus for alumina samples sintered at 1500?C for different holding time prepared for powders aged in DI water for different periods (3, 6 and 10 days)

4.5 Effect of Ageing and Soaking Time on the Reliability of Alumina Ceramics

The improvement in fracture strength with ageing is due to the possible removal of Na? from alumina which is a usual source of impurity in the Bayer's process. It has been well documented in the literature that the presence of Na? results in the formation of sodium-?-alumina which hampers densification, strength and other properties. From the figure, it appears that the best combination of strength is obtained after 4 hours of soaking time at 1500C? on the 3 days aged powder. However, in the subsequent section, the strength results will be discussed in the light of Weibull modulus in order to comment on the reliability of different sintered samples.

CHAPTER 5~ CONCLUSION

The aged powders were uniaxially compacted and sintered at 1500?C for different holding period (2, 4 and 6 hours). The sintered density shows a strong effect on washing. The sintered density was high (0.97-1) for all the samples. Weibull modulus was found to be higher for longer ageing period (for 10 days aged powder and sintered for 6 hours, m = 6.57) indicating a possibility of particle size redistribution during washing and ageing. The biaxial flexure strength carried out on different sintered samples also shows a strong effect on washing. The highest strength was 370 KPa for 3 days aged alumina powder sintered at 1500?C for 4 hours.

CHAPTER 6 ~ SCOPE FOR FURTHER WORK

1. Instead of only ageing, washing and ageing may cause better effect on densification.
2. The effect of washing and ageing on particle size distribution should be studied and it should be correlated with green density and sintered density.
3. Microstructural study should be carried out.
4. The effect of washing on the removal of Na^{+} should be checked.

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