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- as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii))
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(54) Title: METHOD AND APPARATUS FOR PREPARING ALPHA ALUMINA SEED SUSPENSION

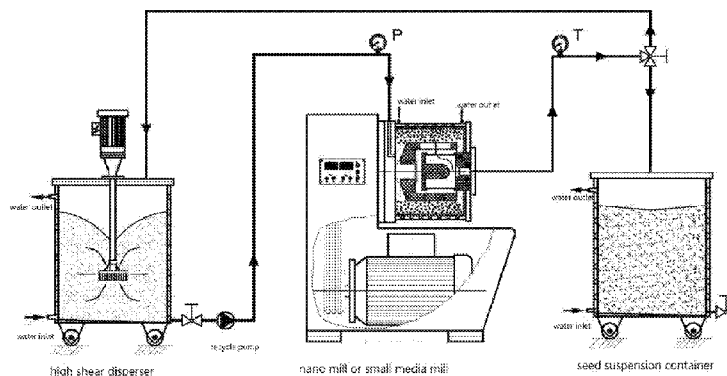


Fig. 1

(57) Abstract: A method and apparatus is applied to prepare alpha alumina seed suspension for ceramic abrasive grain manufacture. Nano mill or small media mill is used to prepare alpha alumina seed suspension with very small dispersed particle size, narrow particle size distribution and high productivity.

WO 2016/141577 A1

METHOD AND APPARATUS FOR PREPARING ALPHA ALUMINA SEED SUSPENSIONField of Technology

The invention relates to a method and apparatus to prepare alpha alumina seed suspension used in sol gel abrasive grain manufacture. Nano mill or small media mill is used to prepare alpha alumina seed suspension with very small dispersed particle size, narrow particle size distribution and high productivity, which are very critical to control the product performance and increase the yield of sol gel abrasive grain.

Description of Related Arts

Since the early 1980's, sol-gel technology has been used to improve the performance of alumina abrasive and has had a major impact on both the coated and bonded abrasive business. Sol-gel processing permits the microstructure of the alumina to be controlled to a much greater extent than is possible by the fusion process. Consequently, the sol-gel abrasive has a crystal size several orders of magnitude smaller than that of the fused abrasive and exhibit a corresponding increase in toughness and abrasive performance.

There are 2 key players in sol gel abrasive industry: 3M and Saint Gobain. Saint Gobain used nano-sized alpha alumina as seed to improve the densification during sintering process of sol gel abrasive grain, while 3M used various modifiers such as magnesia, yttria, lanthana, and neodymia to control microstructural strength and surface morphology upon subsequent sintering. For example, one of the most popular materials, Cubitron 321, has a microstructure containing submicron platelet inclusions which act as reinforcements somewhat similar to a whisker-reinforced ceramic.

Because of the high cost of rare earth oxides such as yttria, lanthana, and neodymia and high nitrogen oxide emission from rare earth nitrate salts during calcining stage, Seeded gel abrasive grain is preferred for mass production of ceramic abrasive grain, and one of the critical process steps of seeded gel abrasive grain production is to prepare nano-size dispersed alpha alumina seed suspension.

US 4,623,364 describes seeded sol gel abrasive material and method for preparing the same. The hardness and microstructure of alumina abrasives produced from alumina gels are enhanced by introduction of seed material as by wet vibratory milling of the material with alumina media, or by the direct addition of very fine alpha alumina particles in the amount of 1% or less. This patent also discloses the apparatus to prepare the seed alpha alumina suspension. A suitable vibratory mill is shown in US patent No. 3,100,088. The alumina media composition was about 90% alpha alumina

with silica as the main impurity, as shown in example II, the main impurities were MgO 1.74%, SiO₂ 8.9%, Fe₂O₃ 0.18%, TiO₂ 0.2%, CaO 0.8%, Na₂O 0.34%. Because the seed material was from the milling debris of alumina media, these impurities would be remained in sintered abrasive grain and have negative effect on grinding performance. Furthermore, the particle size and its distribution of seed material from milling debris is not in good control, which will impact the quality stability of sol gel abrasive grain, not suitable for mass production.

US 4,744,802 describes a process for durable sol-gel produced alumina-based ceramics, abrasive grain and abrasive products. A sol gel process of producing durable alpha alumina-based ceramic particularly useful as abrasive grain from alpha alumina monohydrate is improved by the addition of a nucleating agent (seed material). In examples 15-17, this patent disclosed the process and apparatus to prepare alpha alumina seed suspension. 2.5 liters of deionized water, 2.5 kilograms of Sumitomo AKP-50 alpha alumina powder and 9 g of 15N reagent grade nitric acid were dispersed at high speed for 3 minutes in the 6 liter stainless steel vessel of the "Waring" blender. The resultant suspension was centrifuged at 1000 times the force of gravity for 50 minutes and the supernatant removed. That supernatant was centrifuged at 1300 times the force of gravity for 50 minutes and its supernatant removed. The resultant supernatant was again centrifuged at 1300 times the force of gravity for 50 minutes. The process is very complicated and the productivity is low, needs 3 centrifuge process. Furthermore, the dispersed alpha alumina particle size in the seed suspension from this process is not so small, as shown in example 5, 280 nanometers. Usually the smaller the dispersed alpha alumina seed particle size, the smaller the crystal size of sintered abrasive grain and the higher grinding performance.

So, there is a need to design a method and apparatus to prepare high purity alpha alumina seed suspension with very small dispersed particle size, narrow particle size distribution and high productivity

Summary of the Invention

It is an object of the invention to provide a method and apparatus to prepare high purity alpha alumina seed suspension with very small dispersed particle size, narrow particle size distribution and high productivity for sol gel abrasive grain manufacture.

In this invention, the method and apparatus for preparing alpha alumina seed suspension are described as follows:

Nano-sized alpha alumina powder is mixed with deionized water and nitric acid in a high shear mixer to form slurry; then the slurry is pumped into a nano mill or small media mill. Under highly

intensive mechanical energy, the alpha alumina powder is ground to highly dispersed state with very small particle size and narrow particle size distribution. The slurry is recycled between high shear mixer and nano mill or small media mill until it reaches the target particle size, it can be ground to the designed particle size within a short time, so the productivity of this process is very high, compared with the process described in US 4,623,364 and US 4,744,802.

The nano mill in the above mentioned process is available from the market, for example, nano mill ZETA[®] RS from NETZSCH (Shanghai) Machinery and Instruments Co., Ltd., or nano mill from other suppliers in China. Preferably the nano mill is lined with zirconia or polyurethane to avoid contamination.

The small media mill is also available from the market, for example, the DMQ attritor from Union Process Inc. The DMQ attritor is the newest member of the small media mill family. It is the hybrid of Deltamill and the QC mills. Preferably, the DMQ attritor is also lined with zirconia or polyurethane to avoid contamination.

Brief Description of the Drawings

Fig. 1 is apparatus for preparing alpha alumina seed suspension

Fig. 2 is process to make seed sol gel abrasive grain.

Detailed Description of the Preferred Embodiment

The invented apparatus and process to prepare alpha alumina seed suspension and seeded sol gel abrasive grain is shown in Fig. 1 and 2. The detailed description of the invention is illustrated in the following example.

1.6 liters of deionized water is acidified to a PH of 2.7 with nitric acid, 400 grams of alpha alumina powder with 99.99% purity, 25 m²/g BET surface area and 30 nm primary particle size is mixed into the acidified water by a high shear disperser to form slurry in a 3 liter stainless steel vessel. Then the slurry is pumped into a nano mill (lined with zirconia) with a 0.5 liter grinding chamber volume. The slurry is recycled between the nano mill and high shear disperser. After 60 minutes, the dispersed alpha alumina particle size in acidified water is $D_{50} = 150$ nm and $D_{90} = 350$ nm as determined by dynamic light scattering by using a Laser Particle Sizer. It clearly shows the invented process and apparatus can prepare alpha alumina seed suspension with very small particle size and narrow particle size distribution. The processing time is also very short (within 60 minutes), suitable for mass production. For mass production, the stainless steel vessel volume of high shear disperser is more than 1000 liters and the grinding chamber volume of nano mill is above 25 liters.

The above-mentioned seed suspension is mixed with sol of alpha alumina monohydrate (boehmite), the seed alumina is 1 wt% of final alpha alumina content in the sintered seeded sol gel abrasive grain. The mixed dispersion is then gelled, dried, calcined, and sintered to abrasive grain, the Vickers hardness is 21 GPa at 100 gram load and the density is 3.90, suitable for abrasive applications.

Claims

1. A method to prepare alpha alumina seed suspension for sol gel abrasive grain manufacture, characterized in that, the alpha alumina seed is dispersed into HNO₃ acidified deionized water in a high shear mixer, then milled in a nano mill or small media mill to particle size < 500 nanometers, preferably < 300 nanometers, most preferably < 200 nanometers.
2. A method as in claim 1, in which A nano mill or small media mill is used to prepare alpha alumina seed suspension, preferably the nano mill or small media mill is lined with polymers including but not limited to polyurethane, PTFE and ceramics including but not limited to alumina and zirconia to avoid contaminations.
3. A sol gel abrasive grain, characterized in that, its alpha alumina seed suspension is prepared as described in claim 1 and 2.
4. A method to prepare sintered alumina abrasive, characterized in that, the alumina or alumina precursor is ground to particle size below 1 micron by nano mill or small media mill.
5. A sintered abrasive grain, characterized in that, it is prepared as described in claim 4.
6. A coated abrasive product, characterized in that, its grain is prepared as described in claim 3 and 5.
7. A bonded abrasive product, characterized in that, its grain is prepared as described in claim 3 and 5.

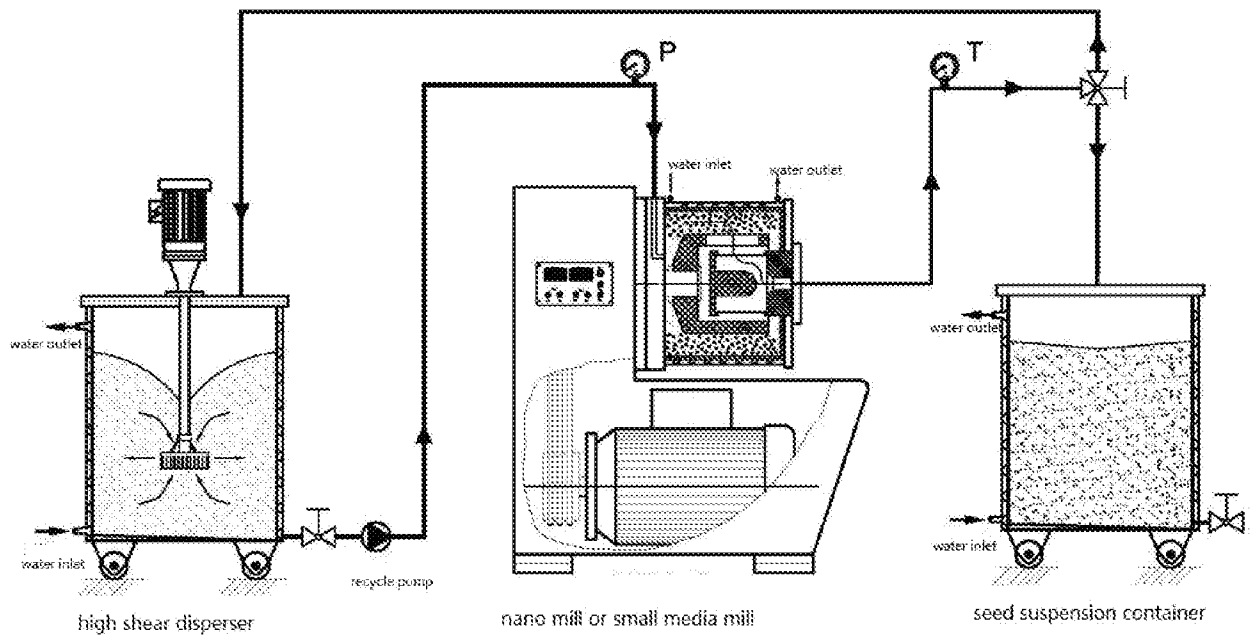


Fig. 1

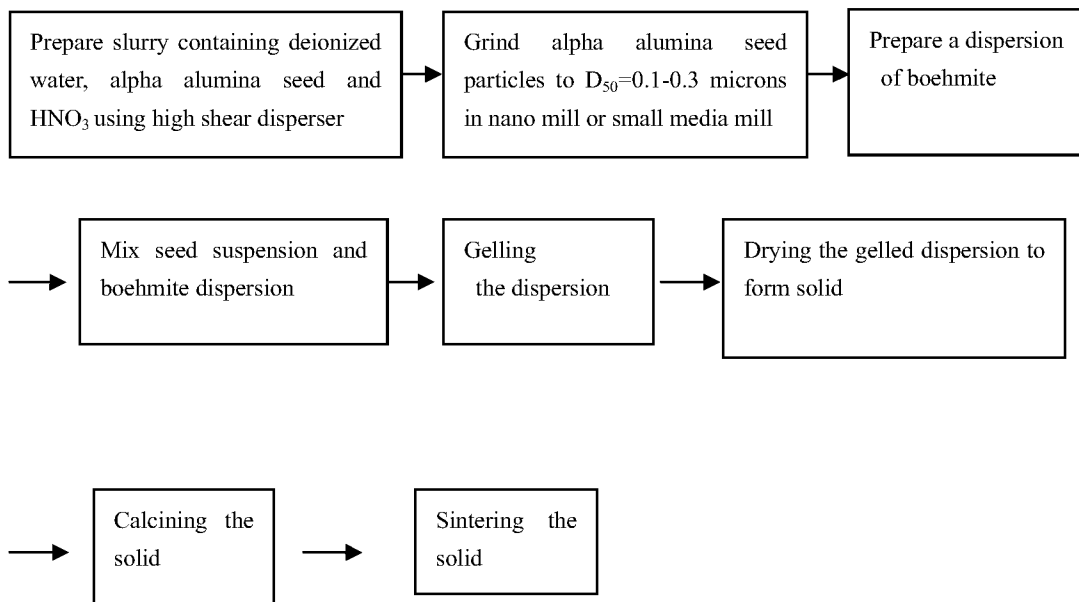


Fig. 2

INTERNATIONAL SEARCH REPORT

International application No.

PCT/CN2015/074051

A. CLASSIFICATION OF SUBJECT MATTER		
C01F 7/02(2006.01)i; B02C 19/00(2006.01)i; B24D 3/00(2006.01)i		
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED		
Minimum documentation searched (classification system followed by classification symbols) C01F 7/- B02C 19/- B02C 21/- B24D 3/-		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) VEN, CNABS, CNTXT, CNKI, ISI WEB OF KNOWLEDGE: alumina?, aluminum oxide?, aluminium oxide?, Al2O3, nano mill?, small media mill?, sol gel, abrasive, ruby, sapphire		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 5453104 A (MINNESOTA MINING & MFG) 26 September 1995 (1995-09-26) description, column 1, lines 5-10	1, 2, 4-7
Y	US 7347393 B2 (TECHWORLD CO LTD) 25 March 2008 (2008-03-25) description, column 2, lines 13-47	1, 2, 4-7
X	US 5453104 A (MINNESOTA MINING & MFG) 26 September 1995 (1995-09-26) description, column 1, lines 5-10	3
<input type="checkbox"/> Further documents are listed in the continuation of Box C. <input checked="" type="checkbox"/> See patent family annex.		
* Special categories of cited documents: "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier application or patent but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art "&" document member of the same patent family		
Date of the actual completion of the international search 04 December 2015		Date of mailing of the international search report 23 December 2015
Name and mailing address of the ISA/CN STATE INTELLECTUAL PROPERTY OFFICE OF THE P.R.CHINA 6, Xitucheng Rd., Jimen Bridge, Haidian District, Beijing 100088, China		Authorized officer ZHOU,Quan
Facsimile No. (86-10)62019451		Telephone No. (86-10)62084698

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

- [1] Because claims 1-7 are not novel or lack an inventive step according to search, same technical features in these claims are not so linked as form a single general inventive concept according to PCT Rule 13.

1. As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.
2. As all searchable claims could be searched without effort justifying additional fees, this Authority did not invite payment of additional fees.
3. As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:
4. No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

- Remark on Protest**
- The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.
 - The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.
 - No protest accompanied the payment of additional search fees.

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.

PCT/CN2015/074051

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