

# HIGHLY POROUS GRINDING WHEELS BY SELECTIVE EXTRACTION OF PORE INDUCERS WITH DENSE CARBON DIOXIDE

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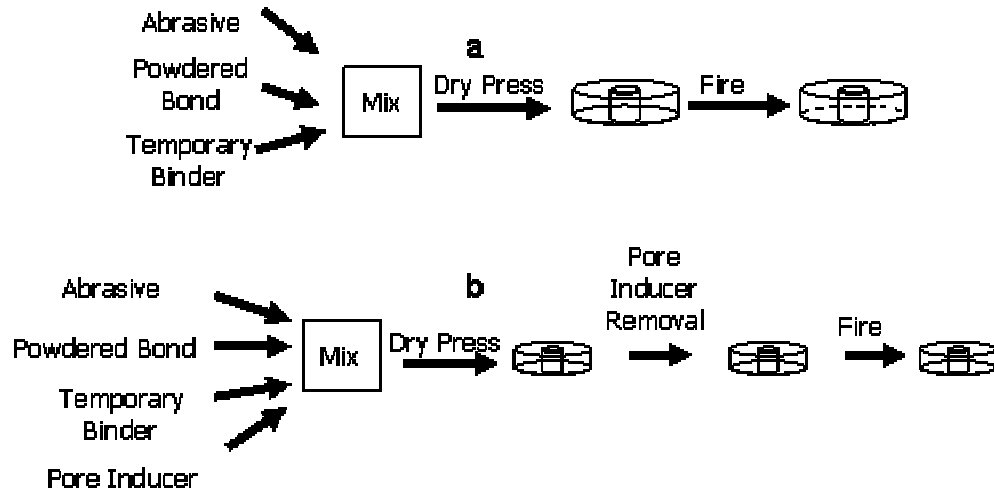
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A novel route for the production of highly porous vitrified grinding wheels was developed via selective extraction of pore inducers with dense CO<sub>2</sub>. Butyl carbamate and biphenyl were used as pore inducers. The extraction was performed with liquid and supercritical CO<sub>2</sub> at temperatures ranging from 295 to 338 K, pressures from 8.8 to 27.6 MPa and flow rates of 3.4x10<sup>-5</sup> and 7.5x10<sup>-5</sup> kg s<sup>-1</sup> CO<sub>2</sub>. The extraction rate was a strong function of temperature, flow rate, and flow direction, while unaffected by particle size of the pore inducer and pressure. The extraction had no detrimental effect on the green wheel's microstructure. Optical microscopy and mercury intrusion porosimetry (MIP) were performed on the green, extracted wheels to quantify the effect of the different particle size pore inducers on the pore structure. The induced pores in the wheel were clearly evident and these pores could be tailored by the size of particles used as pore inducer. The pores induced in the 715µm sample were on the order of 700µm in diameter and the pore sizes decreased with decreasing inducer particle size. In addition, the pores appeared well dispersed and isolated, suggesting the biphenyl and butyl carbamate did not agglomerate during mixing. Grinding tests were performed on the CO<sub>2</sub> extracted pore induced wheels and results were compared to those from a conventionally manufactured pore induced grinding wheel. The extracted grinding wheels performed similarly to the conventional wheels. At high metal removal rates, the extracted wheels with large pore sizes outperformed the wheels with smaller particle sizes as well as the conventional wheel.

## INTRODUCTION :

Vitrified bonded grinding wheels are essential tools in the manufacturing and finishing of countless products used in everyday life. Porosity in vitrified bonded grinding wheels is necessary to provide adequate lubrication to the workzone, the proper bond strength to maintain sharp abrasive grains, and sufficient clearance for the removal of chips. In general, the porosity is controlled by the packing of the abrasive grains. In some high performance grinding applications, in which low temperatures, large chip sizes, or high metal removal rates are needed, a larger amount of porosity than that produced by natural packing of the abrasive particles is needed. Such applications include, but are not limited to, aerospace, high performance alloys, and parts with complex shapes. A technique that is used in modern grinding wheel processing to increase the porosity of the vitrified product is the use of pore inducers. A pore inducer is a material that is added to the mix, along with the bond, abrasive, and binder, and later removed leaving a porous structure. A diagram of the pore induction process is given in Figure 1. In general, the pore inducers must be removed by thermal means

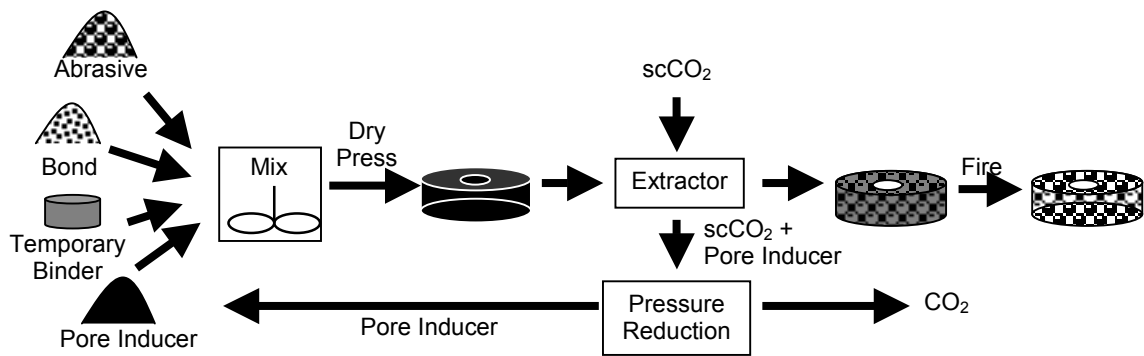
in order to minimize the effect on the pore structure. Other technologies which use pore inducers take advantage of solvent leaching, usually with water<sup>1</sup>. However, due to the fact that much of the temporary binder is partially soluble or swollen in water, this technique cannot be used.



**Figure 1-** Vitrified bonded grinding wheel processing, a) conventional vitrified bonded abrasive b) pore induced vitrified bonded grinding wheel.

Several materials have been investigated as pore inducers. Naphthalene has been found to be a suitable pore inducer. Its removal by sublimation enables the production of high quality porous vitrified bonded abrasive products. However, the use of polyaromatic hydrocarbons (PAHs) requires strict and costly processing controls to provide protection of operators and the environment. Other materials investigated include sugars, starches, and polymeric materials, and they are removed by thermal means. However, there are problems associated with their removal, including carbon residues and product defects due to swelling and slumping.

The pore inducer of choice today is nut shell powder. The material is inexpensive and is removed by thermal means, however it has disadvantages. The elastic nature of the shells causes spring-back, which weakens the green structure and makes it difficult in predicting the part geometry after firing. In addition, thermal removal of the pore inducer can take on the order of days since the process is carried out at a relatively low temperature of approximately 700 K. Furthermore, off gassing of these pore inducers causes structural changes and cracking, which leads to rejection. In general, the products produced are inferior to those produced by naphthalene sublimation. Therefore, there is a need to investigate alternative technologies which can produce high quality porous vitrified bonded abrasives, while avoiding the pitfalls of the conventional technique. One possible route to avoid the problems of conventional pore inducers is the use of supercritical CO<sub>2</sub> (scCO<sub>2</sub>) or liquid CO<sub>2</sub> as a solvent to remove pore inducers. A schematic diagram of the proposed process is given in Figure 2.



**Figure 2-** Schematic of scCO<sub>2</sub> extraction process.

## EXPERIMENTAL

An important consideration in development of this technology is the selection of an appropriate pore inducer. A suitable pore inducer should have the following characteristics. First, the pore inducer must be soluble in scCO<sub>2</sub> at conditions that are not detrimental to the wheel's integrity. The extraction temperature should be relatively low to prevent softening of the temporary binder which would lead to deformation of the green wheel. The pressure should not be excessive since very high pressures may result in deformation or cracking of the wheels. Furthermore, excessive pressures lead to high capital costs. The pore inducer needs to be in the form of a solid at conditions above room temperature in order for it to induce the desired pore structure during mixing and pressing. Furthermore, it may be important that the pore inducer does not liquefy at extraction conditions. Liquefied inducer may lead to structural damage of the wheel due to swelling by dissolved CO<sub>2</sub>. The pore inducer should not be environmentally hazardous nor a severe health hazard. In addition, it must be obtainable in a particle size range that is suitable for creating pores of the desired size. The average particle size should be on the order of 100 to 1000 μm, depending on the porosity requirements of the wheels. Finally, the pore inducer must not be elastic in nature. Under applied stress, the inducer must either remain rigid, or plastically deform. Elasticity leads to spring-back in dry pressing, which decreases part quality.

Two pore inducers were chosen for this study, biphenyl and butyl carbamate. Biphenyl has a high solubility in scCO<sub>2</sub> (MW 154.21), can be screened into appropriate particle size range, and has an appropriate melting point of 343 K. In addition, the phase behavior and solubility of biphenyl in scCO<sub>2</sub> is well known. However, biphenyl is not an industrially acceptable material for pore induction, as it suffers the same drawbacks at the original naphthalene process. Yet, biphenyl was used as a model pore inducer for this investigation, as the system was modeled to determine the phenomena governing the system, and the quality of the parts produced. Subsequent to the investigation of biphenyl pore induced wheels, an appropriate pore inducer was used.

Butyl carbamate, which is not a PAH, has the desirable physical properties required for a pore inducer. It is non elastic, has melting point of 327 K, and can easily be sieved into the appropriate particle size range. The solubility of butyl carbamate in scCO<sub>2</sub> was not available, however expected to be relatively high. In addition, the investigators believed butyl

carbamate would be an industrially suitable pore inducer. The toxicological effects of butyl carbamate are not fully known. However, studies have indicated it is a relatively benign chemical<sup>2</sup>.

Biphenyl, purchased from Aldrich, was sieved into three fractions with nominal sizes of 715, 360, and 194  $\mu\text{m}$  by passing through -20/+30, -40/+50, and -70/+80 sieves, respectively. Each fraction was mixed with bond, abrasive, and a temporary binder and then pressed into 0.0508 x 0.0159 x 0.0127 m (OD x H x ID) wheels. The composition of the wheels is given in Table 4 and the pore inducer volume of each wheel was kept constant at 15%. The mass of each green grinding wheel was approximately 0.062 kg. Butyl carbamate wheels were produced in the same method as previously. However, the butyl carbamate, purchased from Aldrich, was sieved through -70/+100 mesh for a nominal particle size of 127  $\mu\text{m}$ .

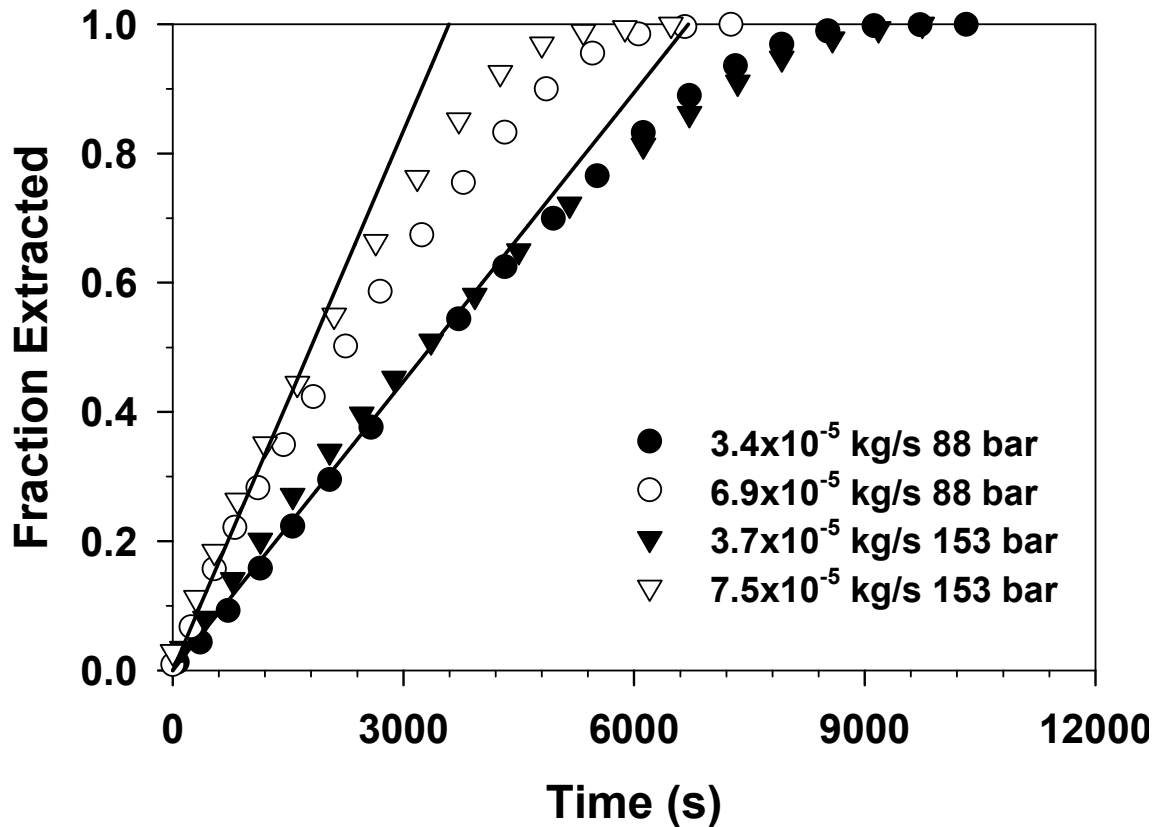
The extraction methods for both butyl carbamate and biphenyl pore induced wheels were similar.  $\text{CO}_2$  was transferred from a gas cylinder (Airgas, research grade 4.8) into the high pressure syringe pump (ISCO, model 260D). From the syringe pump,  $\text{CO}_2$  was pumped through a preheating coil and then through a custom made 0.051 m ID, 0.0635 m L,  $1.3 \times 10^{-4}$   $\text{m}^3$  stainless steel extraction vessel, which was placed inside an oven (Thelco, model 2DG), accurate to 0.1 K. The green ceramic wheel was placed in the extraction vessel, and the void space was filled with sand, sieved through 50/+70 mesh screens. The bed void fraction,  $\epsilon_{\text{bed}}$ , was determined to be 0.41 experimentally. The wheel was made to just fit into the vessel, as to avoid  $\text{CO}_2$  bypass around the outside diameter of the wheel. Bulk flow was through the hole in the center of the wheel. The extractor effluent was mixed with a cosolvent delivered by a reciprocating pump (Thermo Separation Products Minipump) to prevent plugging during expansion. The solvent for biphenyl experiments was acetone, while ethanol for butyl carbamate. The pressure was maintained by a back pressure regulator (Tescom, model 26-1722-24-194). Sampling was performed by collecting the effluent for one minute, and these were analyzed via gas chromatography (HP 6890, with 6' Alltech 10% Carbowax packed column) with 100 mg 1-nonanol as an external standard. With a known  $\text{CO}_2$  flow rate and density, the concentration of pore inducer in  $\text{CO}_2$  was calculated. The mass removed was calculated by numerical integration of the concentration profiles as a function of time.

## RESULTS

Optical microscopy and mercury intrusion porosimetry (MIP) were performed on the green, extracted wheels to quantify the effect of the different particle size pore inducers on the pore structure. The images that were taken at 42X magnification of the cross section of each of the three extracted wheels showed that the biphenyl induced pores in the wheel, and these pores could be tailored by changing the size of particles used as pore inducer. The pores induced in the 715 $\mu\text{m}$  sample were on the order of 700 $\mu\text{m}$  in diameter and the pore sizes decreased with decreasing inducer particle size. In addition, the pores appeared well dispersed and isolated, suggesting the biphenyl did not agglomerate during mixing.

Extraction was performed on wheels prepared with 360  $\mu\text{m}$  particles and the flow rate of  $\text{CO}_2$  was varied from  $3.4 \times 10^{-5}$  to  $7.5 \times 10^{-5}$   $\text{kg s}^{-1}$  and pressure from 88 to 153 bar. As shown in Figure 3, there was no effect of pressure on the extraction profiles at these conditions. As the flow rate was doubled at constant pressure, the rate of extraction increased. At 88 bar, the flow rate was doubled from 3.4 to  $6.9 \times 10^{-5}$   $\text{kg s}^{-1}$ , and the extraction time decreased from

7800 to 5520 s. The lines in Figure 3 are the profiles one would observe if the effluent stream was saturated with biphenyl. The lines are provided for only extractions at 88 bar for clarity. Considering the initial portion of the extraction was at saturated conditions, the effect of flow rate becomes trivial.



**Figure 3-** Effect of pressure and flow rate, (—) Extraction at saturation at 88 bar, (295 K, pore inducer particle size 360  $\mu\text{m}$ , gravity assisted).

Extracted wheels were then fired and prepared for grinding tests. Results were compared to those of conventionally manufactured wheels, with pores induced by nut shells. The nut shells used to make the conventional wheels had a particle size between 419-590  $\mu\text{m}$  (-30/+40). Wheels were tested in wet internal plunge grinding of 52100 bearing steel at metal removal rates,  $Q'$ , of 1.8, 2.6 and 3.0  $\text{mm}^3/\text{s},\text{mm}$ . The surface finish,  $R_a$ , gives a measure of the average roughness of the surface. The total waviness height,  $W_t$ , describes the undulations or waves across the surface of the metal, caused by either non-uniform wear of the wheel, or machine vibration. The  $R_a$  of all of the wheels was very similar. There was a slight increase in  $R_a$  as  $Q'$  was increased, which was expected, as increased feed rates lead to rougher grinding and rougher surfaces. However, there was minimal deviation in roughness between the conventionally pore induced wheel and the  $\text{CO}_2$  extracted wheels. Furthermore, no variation could be seen between wheels made of different particle size. However, there were variations in the  $W_t$  of the part. At  $Q'$  of 1.8  $\text{mm}^3/\text{s},\text{mm}$ , all four wheels behaved similarly. As  $Q'$  was increased to 2.6  $\text{mm}^3/\text{s},\text{mm}$ , the conventional and 715  $\mu\text{m}$  wheels led to a decrease in waviness of the part, while the 360  $\mu\text{m}$  and 194  $\mu\text{m}$  wheels' rise slightly. As  $Q'$

was increased to 3.0 mm<sup>3</sup>/s,mm, waviness increased for the conventional and 194 μm wheels. This trend generally indicates a need for redressing. However, the waviness for the 360 μm and 715 μm wheels did not change, and the 715 μm wheel outperformed all of the wheels. This data indicate that the larger biphenyl particle size pore induced wheel is outperforming the conventional wheels, potentially allowing more parts to be ground at a faster rate before redressing is required.

## CONCLUSIONS

The technical feasibility of a process for pore induction in vitrified bonded wheels was demonstrated using scCO<sub>2</sub> as a solvent for selective extraction of pore inducers. The CO<sub>2</sub> extraction of pore inducers appears to be a promising alternative to the conventional technique of inducing pores in a ceramic body. With the CO<sub>2</sub> process, pore inducers may be extracted in a fraction of an hour, enabling the complete production of the final wheel in a matter of hours, as opposed to the conventional technique, which can take on the order of days. This process also avoids some of the drawbacks of the conventional thermal removal of pore inducers, including swelling, cracking by gas pressure build-up, formation of residues, and springback. In addition, the pore structure can be tailored since the size of the induced pores is controlled by the size of the particle sizes used.

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