Summer 2009

CISP

Center for Innovative Sintered Products

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Director's Message

CISP continues to maintain a strong position in particulate processing R&D. Recent activities include the 2009 Industrial Member's Meeting on April 15-16 and an invited presentation to the MPIF Refractory Metal's Association Meeting in Annapolis, MD on April 28th. Multiple research programs in microforming techniques and refractory and hard materials are underway. Horiba Instruments has just consigned an LA950 particle size analyzer to the laboratory.

The 2009 CISP Industrial Member's Meeting was a success. Our attendance records indicate that 15 different companies and 35 participants were present. Highlights of the meeting included a review of Penn State capabilities in sintering densification, a review of the capabilities of the Horiba LA950 particle size analyzer, and an invited presentation on Tomolithography Techniques for forming mesoscale components using particulate materials. We at CISP hope to see you at our next meeting in April 2010. We look forward to your future involvement.

CISP continues its effort in refractory and hard materials. The Kennametal Foundation has funded a one-year effort to assess the workforce and educational needs for the refractory and hard materials industries. A survey to determine the industrial needs has been developed and is being formatted by the Penn State survey center for electronic presentation and compilation. We have a list of potential candidates, and if you would like to participate, please contact us. As a bonus to the participants, we will provide the results, so you should seriously consider participation in this confidential survey. With the addition of a new Spark Plasma Sintering unit and the already in place infrastructure focused on metal particulate processing, CISP is ideally suited to serve the refractory and hard metal industry.

CISP continues to support our North Central conventional powder metallurgy industry. We are participating in the P/M Initiative Project which is being driven by the Pennsylvania Technical Assistance Program. In this program, we are looking into methods for the conventional powder metallurgy industry to become more competitive through technical innovations in low cost high temperature sintering and to obtain higher green densities during compaction.

CISP plans on participating in the upcoming MPIF PM short course being held in State College on July 27–29. During this event, CISP will lecture on Spark Plasma and vacuum sintering, testing of P/M products, and provide a tour of the CISP laboratory. *For more information on how you can be more involved with participating in CISP and maintaining this academic focused effort at Penn State, please contact us at cisp@psu.edu. Donald F. Heaney, dfh100@psu.edu, 814-865-7346.*

Members' Insider

Portions of this newsletter are distributed to members only:

- ***Powder-Based Stainless Steel Microcomponents Fabricated by Utilizing Lithographic Featured Tooling***
- ***Effect of Sintering Temperature on Micro Feature Resolution***
- ***Financial Update***
- ***Student and Staff Contact information***

For more information on becoming a member, visit our web site at www.cisp.psu.edu or send an email to cisp@psu.edu.

Inside This Edition

Upcoming Events

July 27-29, 2009
Basic PM Short Course
Penn State Conference Center Hotel
University Park, PA
www.mpif.org

August 29-September 2, 2009
30th Intnl. Thermal Conductivity Conf. & 18th Intnl. Thermal Expansion Symposium
Seven Springs Mountain Resort
Pittsburgh, PA
www.thermalconductivity.org

October 13-16, 2009
11th Biennial Unified International Technical Conference on Refractories (UNITECR '09)
Pestana Bahia Hotel
Salvador, Brazil
www.unitecr2009.org

October 25-29, 2009
Materials Science & Technology 2009 Conference & Exhibition (MS&T'09)
David L. Lawrence Convetion Center
Pittsburgh, PA
http://matscitech.org/

January 24-29, 2010
34th International Conf. & Exh. on Adv. Ceramics & Comp.
Daytona Beach, FL
www.ceramics.org/daytona2010/

April 2010
Industrial Members' Meeting
University Park, PA
www.cisp.psu.edu

Vacuum Sintering

Vacuum is often used for sintering metals and less often used for sintering ceramics. The reasons for the use of vacuum are to provide a clean atmosphere and in many cases to evaporate impurities from the high surface area powder starting material. The concern with using a vacuum is the potential to evaporate the metal at elevated temperatures when the vacuum pressure approaches the vapor pressure of the metal. Vacuum has an affect on the purification of metal powders, reduction of metal powders, evaporation of metals, and the densification of metal powder. Also, equipment configurations, such as vacuum level requirements, binder removal, and partial pressure sintering need consideration. These configuration topics are important since modern vacuum sinter processing often occurs under multiple steps. One example is the use of high vacuum during initial processing to remove impurities followed by a partial pressure sintering at elevated temperatures to prevent metal evaporation. Other advanced processing techniques such as the use of reducing atmospheres at partial pressures at low temperatures followed by high vacuum at elevated temperatures are utilized when the metal is difficult to reduce and not susceptible to evaporation at elevated temperatures. Applications and starting point process conditions are available for many common materials that are typically processed using a vacuum configuration. *For more information, contact Donald Heaney at 814-865-7346 or dfh100@psu.edu.*

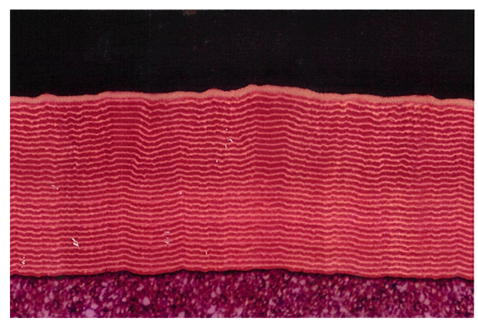
Photo courtesy of Centorr Vacuum Industries

Probabilistic Fracture Assessments of the Response of Ceramic Cannon Barrels Including the Influence of Alternate Rifling Geometries

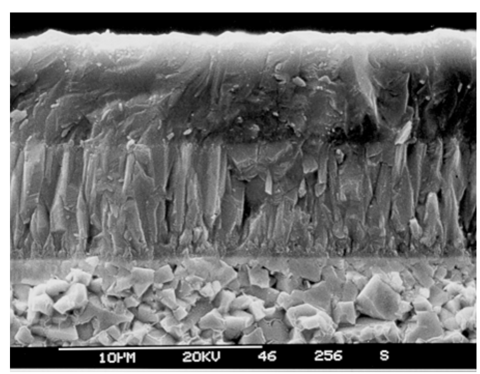
Given their resistance to wear, thermal shock, and thermochemical degradation, ceramics make sense for gun barrel applications. However, their brittle nature and the severe stresses caused by intense pressure and temperature transients must be factored into the design process. Given these concerns, a 3-D finite-element model was used to simulate the severe and localized thermal/pressure transients and the resulting stresses experienced by a rifled ceramic-barrel with a steel outer-liner; the focus of the simulations was on the ceramic materials response, as well as the influence of non-traditional rifling geometries on the thermoelastic- and pressure-stresses generated during a single firing event. In order to minimize computational requirements, a twisted segment of the barrel length based on rotational symmetry was used. Using this simplification, the model utilized uniform heating and pressure across the ID surface via a time-dependent convective coefficient and pressure generated by the propellant gasses. Results indicated that the unique rifling geometries and refractory ceramic had a significant influence on the maximum circumferential (hoop) stresses and temperatures when compared with more traditional rifling configurations. As with earlier studies, it was also found that increasing fillet radii could lower the stresses and failure probabilities over time. *For more information, contact A. E. Segall at 814-865-7829 or aesegall@psu.edu. (R. Carter, contributing author.)*

Contemporary Hardmetals

Hardmetals, or cemented carbides, are hard wear-resistant, refractory materials in which the hard carbide particles are bound together, or cemented, by a ductile metal binder. Such a high hardness and high toughness can be combined. The main constituents of a hardmetal are tungsten carbide and titanium carbonitride, cemented with a cobalt or cobalt-nickel binder. Since their development eighty years ago hardmetals are the dominating tool material in machining and metal forming. Coatings were later developed, mainly TiCN and Al_2O_3 via chemical vapor deposition, to increase the performance in metal cutting. More recently ultrafine powders were introduced to increase the strength and wear resistance. A review of the properties, production and applications was presented at the CISP annual Industrial Members Meeting. *For more information, contact Ivi Smid at 814-863-8208 or smid@psu.edu.*



Functional Multi-Layer Al_2O_3



TiN/TiC Coatings on Hardmetal Tools

(Kennametal/Widia)

Innovative Hot Pressing Technology

Hot pressing is a well known technique in the metal powder field. It is one of the sintering methods to solidify cold pressed (metal) powders. However, for mass production, free sintering is preferred. With free sintering, the manual work to fill the pressing moulds can be saved, resulting in cheaper final products. Another competing technology for traditional hot pressing is the high isostatic pressure or HIP process. This process puts Pascal's Law - "pressure is transmitted equally and in all directions through a liquid or a gas" to work to produce high quality parts. While HIPping also needs preparation of the prepressed powder parts, it gives high final density and good structural proportion to the parts. The costs of a HIP press, is however, substantial. Uni-axial hot pressing is mainly used in the diamond tool business although there are also some other applications. Here, a green density of only approximately 65 per cent is used because of the abrasive character of the powders. Higher green density would inevitably result in poor tool life of the cold press die. To achieve higher green density, a higher pressing force would be needed. This would mean more friction and - due to the use of diamond grit - more wear, and therefore lower tool life. This low green density needs substantial stroke length for sintering. Sintering temperatures of 650-1200°C are normally used. Within hot pressing technology, three distinctly different types of heating can be found in use:

- Inductive heating
- Indirect resistance heating
- Direct resistance heating

1. Inductive heating technology. It works by heating with high-frequency coils that can induce temperature rises. The mould is made out of graphite or steel, and pressure is applied by one or two cylinders onto the punches. The mould is positioned in an induction coil. During sintering a high frequency generator and the induction coil generates heat in the mould. The advantage is that the pressure and the inductive power are completely independent. Powders with a liquid phase are suitable and low pressures are possible. Among the disadvantages are the expense of a high-frequency generator and the need for proper alignment. If the mould is placed off centre, the heat distribution is uneven. But the main disadvantage is the dependence of the process on good heat conductibility. The magnetic field can penetrate the mould only 0.5mm to 3mm. From there on, the heat has to be "transported" into the mould by the thermal conductivity of the mould material. Even heating is much more difficult if the mould is off-centre. Another potential problem is heating rate. Too high a heat up rate will therefore result in high temperature differences that can destroy the mould.

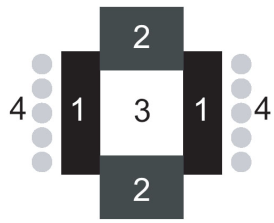


Figure 1. Inductive heating
(1) Ring mould
(2) Top/bottom punch
(3) Powder part
(4) Induction coil

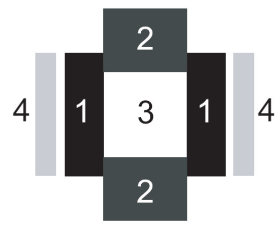


Figure 2. Indirect resistance heating
(1) Ring mould
(2) Top/bottom punch
(3) Powder part
(4) Graphite Heating Element

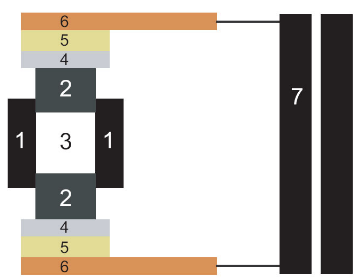


Figure 3. Direct resistance heating
(1) Ring mould
(2) Top/bottom Punch
(3) Powder Part
(4) Graphite Electrode
(5) Brass Electrode
(6) Copper Plate
(7) Transformer

2. Indirect resistance heating. With indirect resistance heating technology, the mould is placed in a heating chamber. The chamber is heated by graphite heating elements. These elements are heated by electrical current. The heat is then transferred into the mould by convection. As the electrical energy heats the heating elements that then heat the mould in a second state, the process is called indirect resistance heating. Advantages are high achievable temperatures, independent from the conductibility of the mould and independent from heat and pressure. The main disadvantage is the time that it takes to heat up the mould. It takes a relatively long time to bring the heat from the outside of the mould to the center and to distribute the heat evenly.

3. Dr. Fritsch direct resistance heating. With direct resistance heating, the mould is directly connected to electrical power. The resistance of the mould and the powder part leads to heat that is generated directly in the mould. This results in very high heating speed. The previous two methods are both somehow related to the physical properties of thermal conductivity. However, with direct resistance heating, the heat is generated where it is needed. Up to now, this technology has been used mainly in the metal powder field with typical maximum temperatures of 1,400°C. Using metal powder, the conductivity of the mould is ideal for fast heating of the work-piece. Moulds that have a big diameter and relatively small height can be heated up very fast. Since 2005, Dr. Fritsch has been offering

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Testing Services

The Center for Innovative Sintered Products is proud to announce the arrival of new scientific equipment for particle sizing. In a collaboration between CISP's director Donald Heaney and Keith Swain of Horiba Instruments, Inc. (and Penn State alumnus); the newest particle size analyzer is installed and operating at CISP in 136 Research West. The Horiba LA-950 extends our testing capabilities down to fine particles in the 10 nanometer (0.01 micrometer) range all the way up to 3.0 mm (3000 micrometers). Particles are initially dispersed in an aqueous or organic fluid, then introduced into the circulation system. The proper refractive index is selected and the dispersion streamed between two light sources and ring, side, and rear-mounted detectors which collect scattering data. Mie theory analyzes and interprets the scattered light patterns, reporting information about the size and distribution. Many results are available including average (mean), mode, but typically D_{10}, D_{50}, D_{90} values are reported. If a D_{10} and D_{90} were 20 and 80 micrometers (µm) respectively, this indicates 10% of the distribution is 20 µm or less, and 90% is 80 µm or less. This kind of reporting provides greater in-depth statistics regarding the overall distribution.

The Horiba LA-950 was checked to be in calibration with a sample of 97 nanometer monodisperse polystyrene latex beads. The average particle size should have been 97 nm, and our Horiba LA950 showed the beads on target at 95 nm (just 0.002 microns variation!).

We can test metals, ceramics, pharmaceuticals and other powders in a variety of fluids. Most recently we have successfully tested tungsten, aluminum, and silicon carbide powders. *If you want to have your powders tested or have a question about the specifications on a powder you own, please contact the CISP lab or Kristina Cowan-Giger at 814-865-1393 or kcc126@psu.edu. You can also visit our testing services website at http://www.cisp.psu.edu/testserv.*

Innovative Hot Pressing Technology

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a high temperature sintering press. This machine has reached 2,400°C and combines high heating rates with high temperatures and precise temperature control. The workpiece is directly measured by means of a pyroscope. Typically, maximum pressing power is up to 600kN (60 tonnes), heating power is 150kVA at 4.5V to 6V secondary voltage. The machine can operate at vacuum or under protective atmosphere; overpressure can be realised as an option. The machine has been successfully tested for metal and ceramic work pieces such as boron carbide at temperatures up to 2400°C. An optional magazine is available which can load the workpieces automatically. Up to 54 pieces can be stocked. Process control and off-line programming via PC is also an option. The machine is especially suitable for processes that need high heating rates, e.g. for materials that should not be kept at high temperatures too long or for processes that require fast heating rates for high productivity. *For further information go to www.directhotpressing.com or contact Dr. Fritsch Sondermaschinen GmbH, Jens Huber, Business Unit Powder Shaping Technologies, Dieselstrasse 8, D-70736 Fellbach, +49-711-51832-221, jens.huber@dr-fritsch.de.*

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