Fall 2004

CISP

Center for Innovative Sintered Products

The Pennsylvania State University
CISP Lab, 147 Research West
University Park, PA 16802-6809

Phone: (814) 865-2121 • Fax: (814) 863-8211 • E-mail: CISP@psu.edu • Web: http://www.cisp.psu.edu

Who's on the Team

Sharon Elder – Executive Director

Research techniques continue to grow in complexity and sophistication. Whether it is long-, intermediate-, or shorter-term research, assembling a high-performance project team is critical. Geographic borders and time zones mean little as industry competes for market share and mind share. A team can commonly consist of industrial, academic, and government partners. Our example of a long-term effort is in response to a recent NASA call. At the invitation of the Kennedy Space Center a group of industrial, academic, government, and NASA partners assembled to file a NASA notice of intent to develop a modular system to mine regolith on the Moon and manufacture structural construction elements *in situ* - such as slabs, blocks, beams, columns, and pipes. The team includes some cold cement investigators inside Cold Regions Research and Engineering Laboratory (CRREL), Caterpillar for vehicles, NASA, an unnamed nuclear reactor supplier, CISP in sintering, Carnegie Mellon University in robotics, Colorado School of Mines in powder fragments-particle control, University of California San Diego for composites, and two small companies for traction and manipulators.

The idea is to build a caterpillar tractor train that includes nuclear power source, powder scoop and particle size separation, shaping stage, sintering stage, and so on. The target is to produce simple shapes for direct use on the moon, such as bricks, tubes, and plates. Early uses will be 30- to 90-day missions to demonstrate technical feasibility, but eventually to harvest local materials to build rocket launch and landing pads, emergency shelters, and possible tubes for hydrogen or water transport (highly speculative). The CISP role would be in determining the sintering cycle, furnace design, determination on shaping technologies, and testing of sintered products for density, strength, permeability, and other important features. The system will contribute to meeting the challenges of the extended "logistics tail" long-term ambitious activities on the Moon and Mars. This is a low-technical-feasibility effort and must move to show all of the pieces exist and can work together.

An example of an intermediate-term research team is the Multiscale Virtual Manufacturing and Design Center for Advanced Vehicular Systems (CAVS) at Mississippi State University. CAVS is partnering with several schools with the ultimate goal of establishing an Engineering Research Center from the National Science Foundation. MSU had an ERC from 1990 to 2002.

continued inside

PENNSTATE
Center for Innovative Sintered Products

For Members-Only

Special insert page...

- Die-compaction of injection molding powders
- Accelerated debinding in large IM bodies
- Hardcoating of P/M parts by electrophoretic deposition

Portions of this newsletter are distributed to members-only. For more information on becoming a member visit our web site at *www.cisp.psu.edu* or contact Sharon Elder: *cisp@psu.edu*

Upcoming Events

November 16-17, 2004
PIM Tutorial
State College, PA

October 17-21, 2004
PM2004 World Congress
Vienna, Austria

March 20-23, 2005
Int. Conf. on Injection Molding of Metals, Ceramics & Carbides
San Diego, CA

April 4-6, 2005
PM Asia 2005
Shanghai, China

April 11-12, 2005
CISP IMM
University Park, PA

May 9, 2005
Metal & Ceramic Injection Molding Process, Design, Applications
Erie, PA

Aug 29-Sept. 1, 2005
Sintering'05
Grenoble, France

Processing with Nanoscale Powders

A new project began in July termed "Press and Sinter Processing Realities with Nanoscale Powders" has generated a computer model to predict the processing and properties of powder compacts over a broad range of particle sizes. The idea is to train the model to material-specific attributes, such as the interplay between grain growth and densification in sintering, using existing data. Then the model is used to extrapolate into the nanoscale size range. The first efforts were on tungsten, which surprisingly is one of the most popular materials in powder metallurgy. Just as an aside, 10% of all sintering literature deals with tungsten-based materials (tungsten, tungsten-copper, tungsten heavy alloy, and tungsten carbide are dominant). From the data and classic models for packing, compaction, green strength, sintering shrinkage, densification, and grain growth, the computer model was able to reasonably predict the sintered density, grain size, strength, hardness, ductile-brittle transition temperature, and wear abrasion rate for sintered tungsten. One of the surprises was the early data on nanoscale tungsten powder. As far back as1963 there were data on 20 to 50 nm tungsten powders, their packing, sintering, and such. By 1976, nanoscale tungsten was a standard product and was treated in the textbooks. Clearly, nanoscale is not new in the refractory metals, but it is not just hype since our efforts show some amazing property combinations possible if the processing can be tailored to preserve the microstructure.

Two major findings so far are worth attention. Nanoscale powders are typically dirty, with lots of oxygen, carbon, water, and other absorbed contaminations. Thermodynamic calculations largely show these contaminants cannot be extracted cleanly below about 1100 C in hydrogen, and possibly even higher temperatures are required in vacuum. In the computer model this immediately became a barrier to processing nanoscale powders. It proved impossible to press to a minimum green strength for handling, sinter to at least 96% density, and still preserve anything of a small grain size. The sintered properties, if 1100 C or higher is required, are not different from plain old garden variety tungsten. Further, the model showed several handling problems. Nanoscale tungsten with a 20 nm particle size (sold commercially in the 1970s) has an apparent density of about 6% of theoretical. Thus, compaction tooling requires considerable punch motion to press this powder to a reasonable green strength. The bottom line was an immediate incompatibility between the small powders and conventional press-sinter technology. Simply put, conventional tooling and compaction pressures do not provide sufficient green density nor green strength conventional sintering cycles cannot preserve the small grain sizes.

What has happened as a consequence of the modeling is construction of two different tool sets. The first tooling went to compaction pressures of 2000 MPa or 150 tsi and was used to compact various powders and to explore tool materials, tool design for high pressures, buckling, and other problems. A subsequent carbide tool set has been fabricated and used in the last few days and has been taken to 4500 MPa or 330 tsi. It is doing exactly as the model predicted. The refractory powders are pressing to high densities and have excellent green strength. So this means progress is in hand on the compaction part of the problem. The next difficulty is keeping the powder clean so that sintering temperatures can be reduced. Right now impurity evaporation is inherently part of the sintering cycle. The model shows a negative consequence on the ductile-brittle transition temperature, so the pending question is how to bring sintering down to 800 C and still remove (formerly evaporate) impurities. Glove box handling would be one option.

One of the consequences of the tungsten modeling is the prediction of some extraordinary properties. For example, strengths over 2000 MPa (almost 300 ksi) are predicted for high pressure compaction and low temperature sintering of tungsten. Experimental plans are being formulated to try out some of these predictions. One consequence is a prediction of significant improvements in wear resistance.

In some first experiments, molybdenum powder has been pressed to 90% of theoretical density and is being sintered in hydrogen over a range of temperatures and times to provide data for extension of the model to a second metal. Meanwhile for molybdenum a total of 150 prior experiments on the compaction and sintering response have been coded into the model to extract the material-specific exponents, coefficients, and other parameters. The same model developed for tungsten is initially being extended to molybdenum, with supplemental data from our lab experiments.

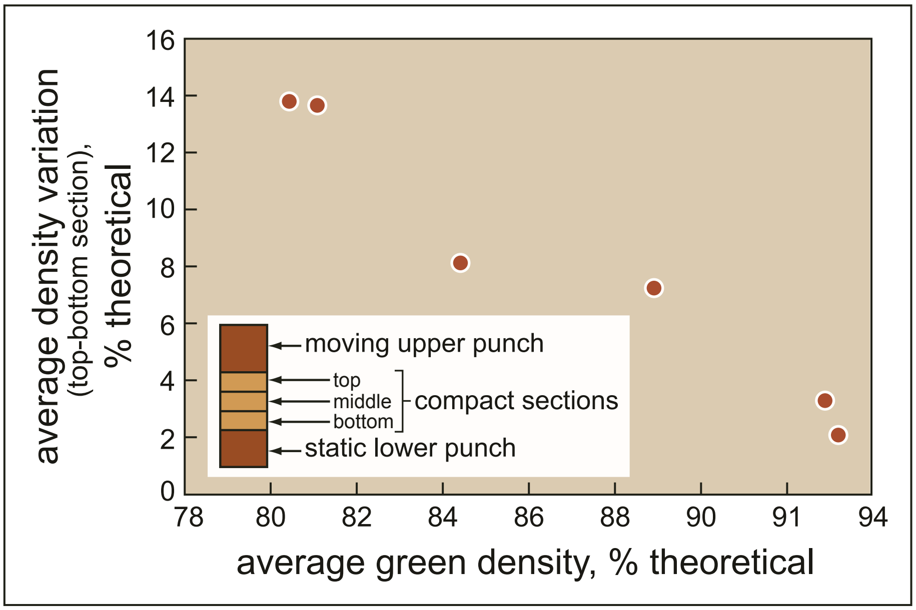
This project has achieved creation of a first model for press-sinter processing applicable to nanoscale powders, guided the experimental program into some new tooling and tool designs, suggests that inert handling will be mandatory, and shows that what seems new is actually old. Consequently, the team is going back to the literature of the 1960s and critically examining prior decisions on powder handling, compaction, and sintering cycles to really move forward and not just repeat the errors of the past. Stay with us on this one, it could be fun.

Currently the team consists of Rand German, Tracy Potter, and Pranav Garg, but we expect a few more people to become involved as the efforts ramp up this fall.

Rand German: rmg4@psu.edu

Green Density Gradients

The effect of green density gradients on the sintered dimensional capability of a P/M product is significant. A variation in green density results in inhomogeneous sintering shrinkage that may result in cracks or warpage. Also, green body gradients lead to property gradients in the sintered components. Hence, there have been many efforts to characterize and control the formation of green density gradients. As part of the CISP study on high density P/M, rods from water-atomized iron powder were pressed to different average densities, and the densities of the top, middle, and bottom sections were measured. The study showed that if the average density of the green compact is increased, the total variation in the green density is decreased, within the density range studied. This indicates that higher green density not only results in a higher sintered density, but also improved dimensional capability for a given sintering temperature.



The figure plots the variation of the green density across ferrous compacts cut into a top, middle, and bottom sections at different compact green densities. Guneet Sethi: gsethi@psu.edu

Is it Time for Titanium P/M ?

Discovered in 1790 and only in commercial production since the 1940's, titanium is a young metal as compared to bronze and steel. The workhorse alloy, Ti-6Al-4V, was only patented and in commercial production in the late 1950's. Is titanium truly the "wonder metal", as proclaimed in a 1952 Time magazine article? DARPA thinks so; they have recently started a titanium initiative. DARPA has awarded $12.5 million to Timet to optimize the FCC Cambridge Process and $1.2 million to International Titanium Powders for optimization of the Armstrong Process for Ti alloys, to name a few. The unique opportunity for the powder metal community is that titanium goes from ore or chemical to powder before any other geometry such as billet or sheets are made; thus, once a low cost powder is available, a low cost shaping technique can further keep the final component's cost competitive. However, the problem to solve is the same as it was in the 1950's. Can we make and process low oxygen content titanium economically? Hopefully, the scientific and engineering communities have advanced to make this a reality. CISP has had some success in producing and welding titanium P/M components with decent ductility, provided the starting powder was low in oxygen and minimal time elapsed between powder exposure to atmospheric conditions and pressing and sintering.

The commercial market for titanium could be huge due to its high strength to weight ratio and excellent corrosion resistance. The majority of current applications are in commercial and military aerospace applications, however, the motor sports industries, such as motocross and sprint cars, have embraced titanium for over 40 years. With this in mind, one can speculate that titanium motor sports applications should be good candidates for the commercial auto industry provided a low cost processing route is found. Some of these applications being exhaust systems, connecting rods, and cam belt wheels. Three production automobiles feature titanium connecting rods, the Acura NSX, the Ferrari F355, and the Ferrari F50. The Chevy Z06 Corvette features one of the first commercial titanium exhaust systems. Weighing in at just 26-pounds, this exhaust system weighs 44 percent less than the standard 46-pounds stainless steel system. Titanium is making inroads into the commercial automobile market; however, how powder processing will capitalize on this will be determined by the production of a low cost, high purity powder and the determination of viable and economical forming and sintering operations. Donald F. Heaney, dfh100@psu.edu

Industry Member Meeting

4-5 October 2004

Nittany Lion Inn
State College, PA

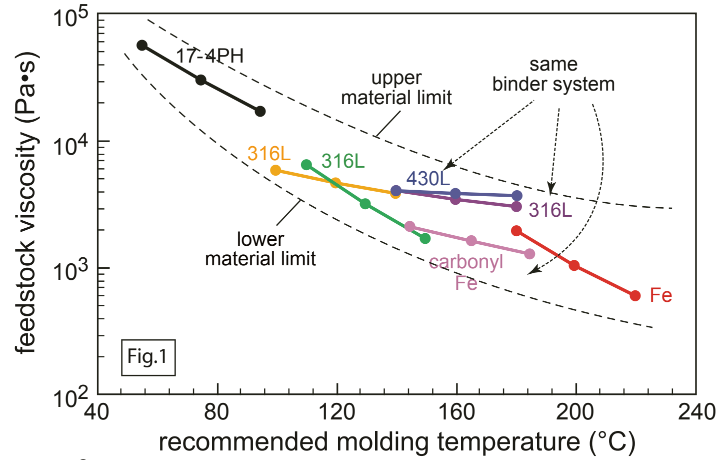
Registrater on-line at:
http://www.cisp.psu.edu/events/IMMreg.html

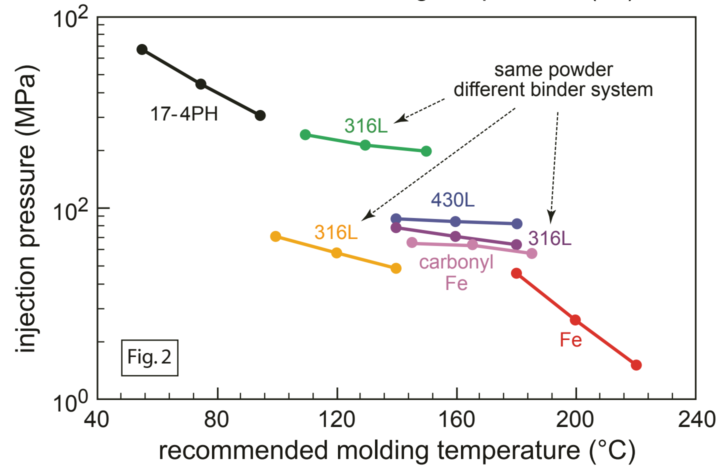
Powder Injection Molding Simulation

The CISP Modeling and Simulation Group has established a feedstock database with corresponding models for powder injection molding. The database has material properties for 16 different feedstocks with a combination of 12 different powders and 7 different binder systems. The database includes rheological and thermal properties. These can be used for flow and heat transfer simulation of the injection molding process. The variations of feedstock viscosity and simulated injection pressure of a tensile bar, with recommended molding temperatures for several feedstocks of iron and steel powders, are shown in Figures 1 and 2. These figures indicate a wide range of injection pressures, depending on powder and binder system, as well as molding temperature. This simulation helps define robust processing windows for high quality green parts. Contact: Seong Jin Park sup13@psu.edu and Debby Blaine dcb193@psu.edu

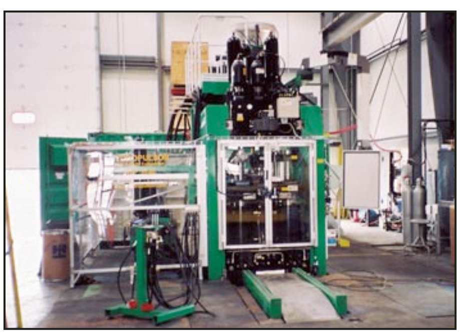
Fig. 1 Feedstock viscosity for various feedstocks.

Fig. 2 Simulated injection pressure for various feedstocks





Hydropulser High Velocity Compacting Press Installed



Gasbarre Products, Inc. announces it has recently completed installation of a Hydropulsor 2000 ton high velocity compacting press at its DuBois, Pennsylvania facility. CISP is using this press for the dynamic compaction of ferrous powders in the pre-competitive Fundamental Limitations and Capabilities of High Density PM project.

Gasbarre Products, is the North American distributor for the Hydropulsor high velocity compacting press product line. The Hydropulsor company is located in Karlskoga, Sweden.

Although this product line is similar to conventional powder compaction presses in many ways, after pre-compaction, the high speed entry of the top punch into the die cavity and powder results in the ability to produce higher density components than produced in the conventional compacting process. Higher densities generally mean higher performance. Powder metallurgy, magnetics, and ferrite components are some of the market segments that will benefit from the high velocity compaction process states Robert McKotch, President of the Gasbarre Press Division.

The press and future such presses at the DuBois facilty will be utilized in working with customers in development and trial runs of specific components for higher densities and conversion to the high velocity compaction process. Contact Guneet Sethi: gsethi@psu.edu

Pictured bottom left: John Fink, Gasbarre press trainer.

Metal Injection Molding of Co-Cr-Mo

Cobalt-chromium alloys are commonly used for surgical implants because of their high strength, superior corrosion resistance, non-magnetic behavior, and biocompatibility. Material properties for various compositions and processing routes are covered by a number of ASTM specifications. Co-28Cr-6Mo is a common composition and can be cast (ASTM F75), wrought (ASTM F1537), or forged (ASTM F799). Due to the shape complexity of surgical implants, casting is often the selected processing route. For higher volume applications, metal injection molding (MIM) can compete favorably with castings on cost.

High sintered densities can be achieved by sintering MIM Co-28Cr-6Mo slightly above its solidus temperature. The solidus temperature varies from about 1350 to 1370°C depending on composition, especially carbon content, so carbon control is very important. The ASTM F75 chemistry specification permits carbon levels of Co-28Cr-6Mo to vary from 0 to 0.35 wt.%. Carbon variation within the specification can have large effects on both the sintering response and mechanical properties. Both the starting carbon content of the powder and the debinding and sintering atmosphere affect the final carbon content.

Debinding and sintering in hydrogen give carbon levels less than 0.01 wt.%, which increase the solidus temperature. Low carbon levels result in higher fatigue strength, but have poor wear resistance. Atmospheres that are less decarburizing give densities above 96% of wrought or forged material at lower sintering temperatures than 100% hydrogen. The mechanical properties compare very favorably with the requirements for cast material, but many Co-28Cr-6Mo components also have fatigue property requirements. In these cases, both MIM and cast components must be hot isostatically pressed to full density, since fatigue properties are highly sensitive to porosity. John Johnson: jlj120@psu.edu

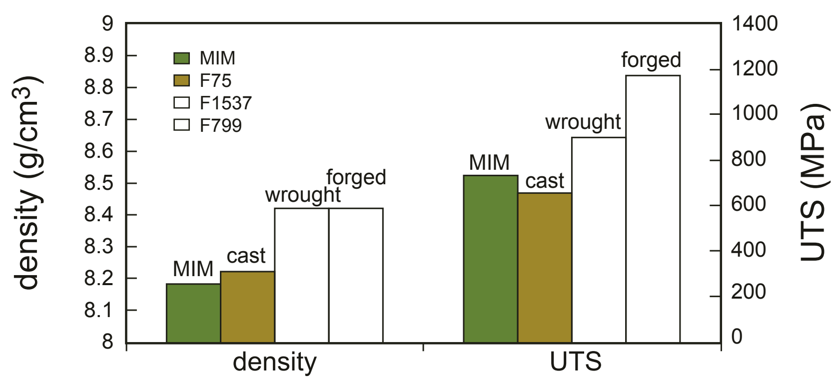


Fig. 1. Comparison of the sintered density and ultimate tensile strength of Co-28Cr-6Mo in comparison to ASTM specifications for cast, wrought, and forged material.

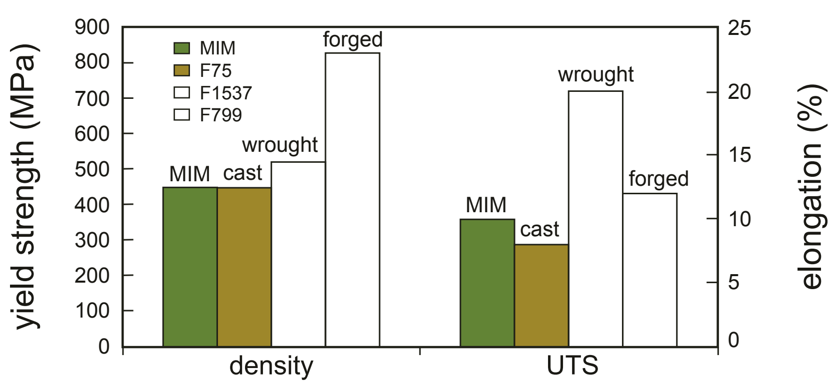


Fig. 2. Comparison of the sintered density and ultimate tensile strength of Co-28Cr-6Mo in comparison to ASTM specifications for cast, wrought, and forged material.

Who's on the Team

(continued from page 1)

CAVS is a $35-million state-funded effort in collaboration with Nissan in the university research park. For the current effort, Northwestern is the key partner, with CISP, Georgia Tech, Washington State, Clark Atlanta, Morehouse/Spelman, and Tuskegee, along with several industrial relations. Currently most links are in thermomechanical processing. In sintered materials they have already linked to Hoeganaes, General Motors, Eaton, Ford, ABAQUS (only in the current software), USCAR, and Center for Powder Metallurgy Technology.

A shorter-term research partnership is looking at titanium for automotive applications. This effort is investigating furnaces needed, sintering technologies, whole design, powder synthesis, and so on. Regardless of the team, all these activities integrate sophisticated technology into products that require collaboration between many individuals. Sharon Elder: cisp@psu.edu

Unveiling the Computational Modeling Center

CISP will showcase the ability to deliver seminars to remote sites at the Industry Member Meeting in October. Changes in traveling habits and a hesitancy of industry representatives to take time from their busy schedules has lead CISP to consider alternative education and communication methods.

Dr. Debby Blaine will unveil the new Computational Modeling Center with a virtual live demonstration of the simulation capabilities in areas such as injection molding, compaction, sintering, and furnace control. Examples of procedures from experimental characterization of models to prediction of properties will be showcased.

Student Profile

Ravi K. Enneti

Ravi K. Enneti (Kumar) is a Ph.D student at CISP. He graduated with a masters degree in Materials Science from Indian Institute of Technology (IIT) Bombay. After finishing his masters, he worked as senior engineer in the Engineering Research Centre at Tata Motors Ltd, India. During his first year at CISP, he worked on hard facing of 15-5PH stainless steel. A part of his work on this project was recognized with a grand prize in the metallography competition at 2002 PM^2TEC. His doctoral thesis is focused on the polymer burnout process with an emphasis on the polymer-metal interactions and shape loss phenomena. His work will result in a better understanding of the polymer burnout process which in turn will help in designing rapid polymer burnout cycles. During research for his thesis work, Kumar developed an innovative process to die compact gas atomized powders. His interests include designing and developing novel materials and processes and working on metallurgy related problems. He will be graduating in spring 2005 and is seeking a job in the industry. Ravi Kumar Enneti (rke104@psu.edu).

Winning Poster

The entry "Observation of Microstructural Changes During Polymer Burnout Process" authored by Ravi K. Enneti, Matthew J. Kelly, Sundar V. Atre and Randall M. German to 2004 International Metallographic Contest was awarded third prize in the scanning electron microscopy category. This work will be on display at annual ASM events and at the ASM headquarters. Congradulations to all on your international accomplishments.

PM Asia 2005 Conference

To create a platform for information exchange and trading between the Chinese PM industry and its international counterparts, Metal Powder Report (UK), will offer a new conference and trade exhibition dedicated to advancing PM technology in Asia. PM Asia 2005 will be held in Shanghai from 4 - 6 April 2005. This event has been created to provide component producers, suppliers, and end-users in a rapidly growing Asian market with the latest information on global PM technology trends. It aims to strengthen links between the growing number of PM companies in Asia and the rest of the world, and to promote the exchange of PM technology and information. Richard Felton, Editor Metal Powder Report http://www.pmasia2005.com/

CISP Conferences & Publications 2004

"Realtime Sintering Observations in W-Cu System: Accelerated Rearrangement Densification via Copper Coated Tungsten Powders Approach" Burak Özkal, Anish Upadhyaya, M.L. Öveçoğlu and Randall M. German
To be presented at PM2004 Vienna conference in Oct.

"Strength Predictions for Bulk Structures Fabricated from Nanoscale Tungsten Powders" Randall German, Eugene Olevsky
Submitted to the International Journal of Refractory Metals and Hard Materials.

"Green Body Homogeneity Effects on Sintered Tolerances" Randall M. German
To be presented at the PM2004 Vienna conference in Oct.

"Supersolidus Sintering of Boron Doped Stainless Steel Powder Compacts" Ravi Bollina & Randall M.German
To be presented at the PM2004 Vienna conference in Oct.

"*In situ* Evaluation of Viscosity During Sintering of Boron Doped Stainless Steel Using Bending Beam Technique" Ravi Bollina & Randall M.German
To be presented at the PM2004 Vienna conference in Oct.

"Master Sintering Curve Concepts as Applied to the Sintering of Molybdenum" Deborah Blaine, John D. Gurosik, Seong Jin Park, Donald Heaney, Randall M. German
Submitted for publication to Metallurgical and Materials Transactions A.

"Quantitative Microstructure Analysis of Tungsten Heavy Alloys (W-Ni-Cu) During Initial Stage Liquid Phase Sintering" J. Shen, L. Campbell, P. Suri, & Randall M. German
Submitted to the International Journal of Refractory Metals and Hard Materials.

"Development Progress: Sintered Tough-Coated Hard Powders (TCHPs)"
R.E. Toth, I. Smid, J. Keane, R.M. German, P. Ettmayer
To be presented at the PM2004 Vienna conference in Oct.

"Influence of Mixing and Effect of Agglomerates on the Debinding and Sintering of 97W-2.1Ni-0.9Fe Heavy Alloys" Pavan Suri, Randall M. German, Justin J. Brezovsky, and Jupiter P. deSouza
Submitted to Materials Science and Engineering A

"International Research Collaboration: An Empirical Assessment" Sharon L. Elder
Submitted to Metal Powder Report

"Mechanical Properties and Corrosion Resistance of MIM Ni-Based Superalloys," J.L. Johnson, L.K. Tan, P. Suri, and R.M. German, presented at PM2Tec 2004, Chicago, IL (June 14-17, 2004).

"Processing of Biocompatible Materials via Metal and Ceramic Injection Molding," J.L. Johnson and D.F Heaney, presented at ASM Materials & Processes for Medical Devices Conference, St. Paul, MN (Aug.2004)

"Metal Injection Molding of Heat Sinks," J.L. Johnson and L.K. Tan, Electronics Cooling, accepted for publication.

"Fabrication of Heat Transfer Devices by Metal Injection Molding," J.L. Johnson and L.K. Tan, to be presented at PM2004, Vienna, Austria.

"Evaluation of Copper Powders for Processing Heat Sinks by Metal Injection Molding, J.L. Johnson, L.K. Tan, R. Bollina, P. Suri, R.M German, submitted to Powder Metallurgy.

Center for Innovative Sintered Products
The Pennsylvania State University
CISP Lab, 147 Research West
University Park, PA 16802-6809
visit us on the web at: *www.cisp.psu.edu*
Phone: (814) 865-2121 • Fax: (814) 863-8211 • E-mail: cisp@psu.edu

Randall M. German, Director
Phone: (814) 863-8025
E-mail: rmg4@psu.edu

Sharon L. Elder, Exec. Director
Phone: (814) 865-1914
E-mail: cisp@psu.edu

CISP Newsletter
Published four times per year

Managing Editor
Sharon L. Elder

Graphic Designer
Rick Sharbaugh