Summer 2002

The Pennsylvania State University

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Center for Innovative Sintered Products

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Medical Field Opens Doors

Randall M. German-Center Director

In keeping with our strategic replanning, which began at the beginning of the year, we recently visited the medical devices show in New York City. We see the PM industry showing up as this field is open for

enabling technologies for stainless steel and titanium into medical and surgical applications. As the trend moves towards non-invasive surgery, one sees that although expensive, there are many opportunities in design process and surface treatment. New evolutions in surface treatment will give PM options not realized before. With our special skill sets, it is our role to guide sponsorship into areas such as medical devices, filtration, and temporary components such as dental orthodontics where success is measured in dollars rather than tons.

Micro-partnerships with CISP Continue to Evolve

Forming micro-partnerships with CISP offers additional opportunities.

ARCiNA (Austrian Research Centers in North America) opened its Materials & Production Technology Center just under a year ago. According to Researcher Rudi Zauner, "Working with CISP is a real benefit to us, giving our start-up company a "kick-start". The benefits are mutual and we are proud of having brought a number of long-term projects to CISP."

Another partnership was formed with CetaTech, CAE software and consulting company, for the powder metallurgy processes including the powder injection molding process. President Young-Sam Kwon maintains "CISP is the most suitable research center because CISP already has active research work with many P/M companies and an enthusiasm for new technologies in P/M processes."

John Johnson, R&D Manager AMTellect said "Having local access to the CISP labs enables AMTellect to explore new technologies with world-class equipment that we could not justify the expense of purchasing on our own. We are able to participate in experiments and see the results as they happen to compress development cycle times."

Aesthetic Materials was created to simplify the production of bronze art via use of hybrid polymer-powder forming and sintering technologies. The company provides a variety of replication technologies for artists, designers and engineers; capitalizing on advantages in surface finish, lead times, costs, dimensional control, and detail preservation.





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Numerical Modeling the Sintering Process

Year three goal of the Numerical Modeling project is to develop and verify a software simulation tool that captures the evolution of dimensional changes of a compact during the sintering process. The densification and creep of cylindrical, die-compacted stainless steel parts during free sintering is the primary focus.

The model starts with an initial density distribution found using results from both previous die compaction research and current work on simulation of compact ejection from the compaction press. The ejection process is complicated by friction and changing compression conditions and preliminary results are encouraging, but contact-release problems are not totally resolved. Consequently, post-sintering simulation results agree in average magnitude, but not in profile with experimentally determined dimensional changes. Sintering simulation includes porosity evolution, grain growth, and the effects of gravity, thermal expansion, and thermal conductivity. Once the ejection problem is solved, the plan is to:

- (1) incorporate the computed shape & green density distribution after ejection directly into the sintering simulation model
- (2) treat complex shapes, specifically flanged compacts
- (3) investigate the applicability of the compaction and sintering model to other material systems, such as bronze and nickel.

Nick Salamon (njsalamon@psu.edu)

Exploration of Net-shaping and Consolidation Techniques for Refractory Metals

A rather limited number of metals and alloys are suitable for application at very high temperatures, such as tungsten, tantalum, rhenium, niobium and molybdenum. Net-shaping options and dimensional control will be compared and qualified for each method in this new project. To produce partially and/or fully dense parts sintering in H2, HIP, hot pressing, MW sintering, SPS, and laser assisted buildup will be applied.

To establish the routines needed to handle, characterize, produce and qualify compacts of this class of materials, a new project on Net-Shaping and Consolidation of Techniques is now underway. Ivi Smid (smid@psu.edu)

Potential applications:

- lighting, wiring, electronic, power engineering
- furnaces, other high temperature applications
- thermal management, contact materials
- catalysts
- sputtering targets
- chemical apparatus engineering,
- process equipment, welding electrodes
- medical
- space
- defense

Qualification of High Strength P/M Alloys

Now in its second year, this project continues to examine the effect of alloying additions on the mechanical properties of P/M components for high performance capabilities. Microalloy additions of niobium, vanadium, chromium, nickel, molybdenum, and manganese have been developed for cast and wrought alloys, showing large improvements in alloy performance. Furthermore, microalloy additions of boron and phosphorus will be made for enhanced sintering capabilities. The effects of these additions to P/M alloys are being studied as a function of processing conditions. In particular, the effect of these alloy additions on mechanical properties, including fatigue response, will be evaluated and compared to standard high-strength nickel/molybdenum alloys processed during year one of this study. Don Heaney (dfh100@psu.edu)

With 13 new or continuing projects

56% of the CISP budget is earmarked for research.

Laminated Structures for High Performance Cutting Tools

Research on this project is now entering the 3rd year. Instrumented cutting tests on Ti-6Al-4V alloys are currently underway, and indicate that laminated tools experience significantly lower machining forces than monolithic tools during machining, and comparable tool wear behavior.

Tool failure correlates with the occurrence of free carbon (sometimes referred to as C-porosity) in the hot pressed laminated tools. However, recent enhancements in our pressureless sintering methodology have enabled us to eliminate C-porosity, and a new generation of laminated tools is currently being processed for instrumented machining tests.

Work on spark plasma sintering (SPS) of laminated, nano-grained tools will begin in July at Nihon University in Japan, in cooperation with Sumitomo Coal Mining Co. Kevin Fox, a CISP graduate student will focus on identifying the suite of processing parameters necessary for densifying laminated composites with functionally graded, nanograin size prototype tools for evaluation at CISP Labs. John Hellmann (jrh3@psu.edu)

Dimensional Control of Stainless Steel

This research aims at understanding the relationship between powder chemistry, oxide species, sintering atmosphere and sintering response in compaction grade 316L stainless steel. This will lead to the development of a process control tool to obtain higher dimensional precision and broaden the market for P/M stainless steels. To date, six different lots of 316L stainless steel were compacted, sintered, and measured for dimensional change. Correlation values were drawn between the dimensional change and bulk chemistry, surface chemistry, and particle size. Surface chemistry was measured by X-ray photospectroscopy. The data indicates a strong influence of surface chromium content, bulk manganese content, bulk carbon content, and D_{10} particle size on shrinkage. In recent months, efforts have been focused toward better powder sampling for improved isolation of variables. Eight new 316L powder lots are currently in process. One lot has been sieved and re-blended to create five different particle size distributions with one chemistry. Carbon will be added to selected lots to study the effect of carbon to oxygen ratio on deoxidation. A 400 series grade will be processed in the next year, as well as powder injection molding grade stainless powders. Stepwise regression analysis will be used to identify the effects of each variable. Information will be used in the development of a sintering model to aid parts makers in selection of furnace parameters for a given powder lot. Neal Myers (nsm104@psu.edu)

Multiple Axis In Situ Monitoring of Dimensional Changes

During the first year of the project, the laser dilatometer was developed, calibrated and coupled with a data analysis system. The dilatometer was used to monitor component dimensional change during the solvent debinding step of the powder injection molding process. The effects of soluble binder content, soluble binder density, particle size, solids loading, and solvent temperature were analyzed. These results were compared with soluble binder mass loss observations to investigate the mechanisms of solvent debinding.

In year two, a furnace allowing laser measurement will be integrated with the dilatometer to record *in situ* dimensional changes during thermal debinding, with future observations during sintering. A high precision motor will be incorporated with the sample stage to allow vertical and rotational movement of the sample during heating which will result in multi-dimensional measurements. *In situ* monitoring of thermal debinding and sintering will provide guidance for sintering cycle design, component geometry modification, and forming process improvement. The connection between microstructural variations and macroscopic dimensional changes will also be established. This will provide important information for the differential shrinkage, distortion, and cracking commonly observed in sintered products.

The final year of the project will introduce higher temperatures to allow direct observations in high temperature sintering and heat treatment to allow full tracking of dimensional change without sample contact. Rand German (rmg4@psu.edu)

KYK Furnace Open for Business

Trial sintering runs in the recently installed KYK "Oxynon" furnace will begin in late July and early August. This furnace is currently booked for Toll Sintering until late August. The Center is offering sintering trials for a daily charge. The features of this furnace are 2000°C belt sintering in an inert atmosphere (nitrogen or argon). The furnace functions by reducing the partial pressure of oxygen in the furnace to promote the dissociation of oxides. We envision this furnace to provide good carbon control in ferrous materials, inert gas sintering of titanium, and the densification of other difficult to sinter materials.



For additional information about the furnace or to schedule toll sintering, contact: Don Heaney (dfh100@psu.edu) phone: (814)-865-7346.

Numerical Simulation of Binder Burnout & Stress Formation

The goal of this new project is to simulate the debinding and delubrication steps of powder processes that utilize a binder for shape retention. Final model simulations will allow the user to numerically evaluate the effect of various processing parameters on the debinding rate and stress formation of a PM component. Therefore, the results from the simulations will help the part producers to understand and optimize the debinding or delubrication steps of their PM processing. Chantal Binet (cub9@psu.edu)





During year one 3 design of the experiments were completed for the Six Sigma project. Variables examined have been powder type, powder size, powder loading, switchover technique, compounding method, sintering temperature, and powder lot. A tremendous amount of dimensional data has been obtained. This year, the focus will include organizing and analyzing this data and continue laboratory experiments to validate findings. We also will generate an "input/response" model for modeling dimensional variation of a PIM process. Don Heaney (dfh100@psu.edu)



To better utilize the research and equipment capabilities of CISP, AMTellect, Inc. started an operation in State College in April as a subsidiary of Advanced Materials Technologies (AMT), Singapore. AMTellect will handle marketing and development activities in North America. AMT, Singapore specializes in high volume metal and ceramic injection molding.

AMTellect represents a strategic move by AMT to capitalize on the rapid growth of the powder injection molding (PIM) market and applications. AMTellect's focus is customer and technology development. This includes PIM innovations in the creation of assembly integration, hollow components, and controlled porosity using AMT's patented technologies for producing anisotropic materials and two-material systems. The use of computer simulations and process modeling for PIM will further enhance customer satisfaction through shorter response times and lower cost. Mike Sherwin (contact@amtellect.com)

MPIF Awards

CISP researchers, Ravi K. Enneti (PH.D. candidate), Kristina Cowan (Research Technologist), and Neal Myers (Project Manager) claimed the Grand Prize in the Metallography Competition at the MPIF World Conference in Orlando, Florida.

Additional CISP winners: Lou Campbell (Sr. Research Technologist), 2nd place in Technical Service category & 3rd place in Method Development and Debby Blaine (PH.D. candidate) recipient of the CPMT Axel Madsen Award. Congratulations to all for a job well done.



Pictured: 1st place in the Research & Development category leading to the Grand Prize in the Metallography Competition for the use of failure analysis of carbide coated stainless steel. The brazed carbide layer caused a carburization of the stainless steel, leading to intergranular fracture in fatigue loading.

Students Corner

From Classroom to Industrial Application

Two CISP students are furthering their training on summer internships.

Ravi Bollina is on an internship at Osram Sylvania in Towanda PA. He is working as a process engineer and developing press and sinter parts in the TMM department (Tungsten Moly Metals). TMM manufactures tungsten and molybdenum refractory metal parts which include rods, slabs, wire and other shapes. John Gurosik is also at Osram Sylvania at the chemical & metallurgical products plant. He is acting as a process engineer in the wire drawing area where he is creating defined procedures for the reworking of existing carbide wire drawing dies and overseeing the training of employees on these machines.

NSF Research Experience for Undergraduates (REU)

Enrichment Training for Undergraduates

Outstanding students from throughout the US are now participating in the NSF Research Experience for Undergraduate (REU) program. Now in our second summer, the program continues to generate widespread interest. Over eighty applicants expressed an interest in participating with thirteen students selected representing 10 Universities. The ten-week program provides each REU student with approximately 32 hours a week in research experience on a CISP project, with an additional eight hours allocated for sintering and research seminars. The students will also be touring five CISP companies for first hand exposure to see how industrial applications fits into the "big picture" and how industry and societal needs drive basic and applied research.

Dimensional Producibility of High Precision Sintered Components

Work is on-going with five CISP companies to fully characterize the dimensional capabilities of conventional press and sinter metal parts in both the green and sintered conditions. To date, more than 130,000 production part features have been evaluated from production P/M components at member companies for this project.

On-going work has clearly shown that feature centering errors contribute significantly to overall part dimensional errors. Centering errors are often as large as, or are even larger than, part-topart dimensional variation for a given production run. Additional data analysis is on-going in order to develop specific strategies that can be used by sintered material producers to promote component dimensional conformance. Upcoming plant trials will also carefully examine dimensional centering "drift" issues using robust design of experiment techniques. Experiments monitoring hydraulic press repeatability and dimensional "drift" both within a run and from run-to-run are planned. Measurement system analysis techniques will also be used to evaluate the adequacy of measurement systems commonly used for dimensional inspection on the shop floor. Bob Voigt (rcv2@psu.edu)

INDUSTRY MEMBER MEETING 9 & 10 OCT 2002



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