Winter 2006

Center for Innovative Sintered Products

CISP

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Director's Message

Dr. Judith A. Todd

Seasons greetings to all our CISP members. Save the date for the next CISP meeting which will be held on Monday April 9th and Tuesday April 10th, 2007. Our meeting will be held in conjunction with "Materials Day," which commences at 3.00 p.m. on Tuesday April 10th and continues through Wednesday April 11th. There will also be an opportunity to visit the new Center for Multiscale Wave-Materials Interactions facility, which will open in January, 2007. In addition to updates on our CISP research programs, our next meeting proposes to feature an Industry Forum on "Industrial Innovations in Powder Materials Processing and Products."

Associate Directors, Ivi Smid and Don Heaney, together with colleagues and students, have been very active this year in presenting their research at conferences including: the International Conference on Tungsten, Refractory and Hardmetals VI; TMS 2006; the International Thermal Spray Conference; the International Conference on Powder Metallurgy and Particulate Materials; the First International Conference on Micromanufacturing (ICOMM 2006); ASME Manufacturing Science and Engineering Conference; and MS&T 2006.

Ivi has been elected to the Powder Materials Committee of TMS and will organize sessions on "Cross Cutting Issues in Particulate Consolidation" at MS&T'08 in Baltimore.

Congratulations to Don Heaney on his appointment to the dual titles of Senior Research Associate and Associate Professor of Engineering Science and Mechanics. Congratulations also to CISP students Lou Campbell and Parul Walia on completing their M.S. degrees.

As we approach 2007, we should like to wish you all very happy holidays and a Happy New Year. We look forward to seeing you in April.

Judith A. Todd <jtodd@psu.edu>

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 Diane K. Bierly at <cisp@psu.edu>

Inside This Edition

Upcoming Events

February 25 – March 1, 2007
TMS 2007
Orlando, Florida, USA

April 2-4, 2007
PMAsia2007
Shanghai, China

April 9-10, 2007
Industry Member Meeting
University Park, Pennsylvania, USA

April 11, 2007
Materials Day
University Park, Pennsylvania, USA

Featured Research

Microwave Processing of Metallic Materials

Microwave energy has been in use for over 50 years in a variety of applications such as communications, food processing, rubber vulcanization, textile and wood products, and drying of ceramic powders. Microwave technology is attractive because it has many advantages when compared with conventional methods, such as very short cycle time resulting in energy savings as high as 90% over conventional methods, finer microstructures, and hence, improved mechanical properties and environmental friendliness.

Microwaves are electromagnetic radiation with wavelengths ranging from about 1 mm to 1 m in free space and frequencies between 300 GHz to 300 MHz, respectively. However, only very few frequency bands in this range (2.45 GHz, 28 GHz and 915 MHz) are allowed for research and industrial applications to avoid interference with communication with 2.45 GHz, the frequency at which kitchen microwaves operate, is the most common frequency for research and industrial applications.

Microwave processing of materials was mostly confined, until 2000, to ceramics, semiconductors, inorganic and polymeric materials. There have been very few detailed reports on microwave processing of metals. The main reason for this lack of work in microwave heating/sintering of metals was due to the misconception that all metals reflect microwave and/or cause plasma formation, and hence cannot be heated, except exhibiting surface heating due to limited penetration of the microwave radiation. This observation is evident from Figure 1 which shows microwave absorption in solid materials versus electrical conductivity. [1] Figure 1 indicates that only semiconductors are good microwave absorbers, ceramics/insulators are transparent to microwaves, and metals should reflect microwaves. However, this relation is valid only for sintered or bulk materials at room temperature, and not for powdered materials and/or at higher temperatures. Which it has now been proved that all metallic materials in powder form do absorb microwaves, the cause for this phenomenon is not yet well explained.

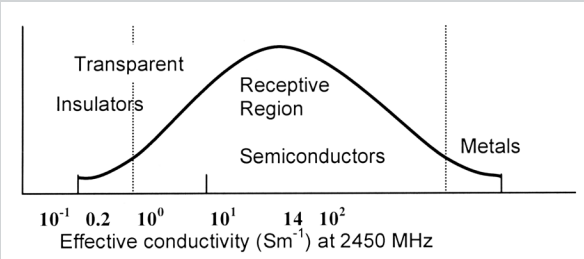

Figure 1. Microwave energy absorption as a function of electrical conductivity

At 2.45 GHz it is observed that the skin depth in the bulk metals is very low (of the order of a few microns), and hence very little penetration of microwaves takes place. However, in the case of fine metal powders rapid heating can occur. A theoretical model predicted that if the metal powder particle size is less than 100µm, it will absorb microwaves at 2.45 GHz. It was further observed that the degree of microwave absorption depends upon the electrical conductivity, temperature and the frequency. In magnetic materials other manifestations of the microwave coupling include hysteresis losses, dimensional resonances, and magnetic resonances due to unpaired electrons. [2]

The earliest work of microwave interaction with metallic powders is reported by Nishitani, [3] who reported that by adding a few percent of

Accomplishments

Gaurav Aggarwal has taken a position as Applications Engineer with Diamond Innovations (formerly GE Superabrasives) in Worthington, Ohio, while he continues to work on his doctoral thesis. He is working in Micron Technology.

Lou Campbell successfully defended his thesis "Gravitational Effects on Mechanical and Microstructural Properties of Tungsten Heavy Alloys." He has taken a position as a Metallurgical Engineer with Eaton Electrical in Horseheads, New York.

Donald F. Heaney, Associate Director for CISP, has been appointed Senior Research Associate and Associate Professor of Engineering Science and Mechanics. Don came to Penn State in 2000 after working for both 3M and Bristol Meyer Squibb. He is also a registered Professional Engineer in Metallurgy. His current areas of interest are in micro-miniature device fabrication utilizing micro EDM and lithography techniques, titanium P/M processing, utilization/understanding of polymers for net shape fabrication, and allow development for predictable fracture behavior and process independent strengthening.

Ivi Smid, Associate Director for CISP, has been elected to the Powder Materials Committee of the Minerals, Metals and Materials Society (TMS) and will organize a session on "Cross Cutting Issues in Particulate Consolidation" at MS&T 2008 in Baltimore, Maryland.

Judith A. Todd was the Conference Chair of the 2006 ASME (American Society of Mechanical Engineers) Pressure Vessels and Piping (PVP) Conference in Vancouver, British Columbia, July 23-27, 2006. This year's technical program of the PVP Conference included over 700 technical papers, panel and plenary sessions, and forums organized into more than 210 technical sessions, tutorials, workshops, and a student paper competition. The ASME Pressure Vessels and Piping Division sponsored this Conference jointly with the International Council on Pressure Vessel Technology (ICPVT), and in collaboration with the ASME NDE and Pipeline Systems Divisions and the Canadian Society of Mechanical Engineers. With participation from the Americas, Canada, Europe, and Asia; the conference was able to address current and emerging pressure vessel technologies in a global context.

Parul Walia successfully defended her thesis, "Development of Ni-Based Self-Lubricating Composite Coatings for Ti-6Al-4V Dovetail Joints Using the Cold Spray Process." She has taken a position as a Research Engineer with Saint Gobain Abrasives, Inc. (also known as Norton Abrasives) in Worcester, Massachusetts.

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Featured Research

…continued from page 2

electrically conducting powders such as aluminum, the heating rates of the refractory ceramics is considerably enhanced. Walkiewicz et al. [4] likewise simply exposed a range of materials, including six metals to a 2.45 GHz field, and reported modest heating (but not sintering) in the range from 120°C (Mg) to 768°C (Fe). Whittaker and Mingos [5] used the high exothermic reaction rates of metal powders with sulfur for the microwave-induced synthesis of metal sulphides. Sheinberg et al. [6] heated Cu powders coated with CuO to 650°C but did not report any sintering. Narsimhan et al. [7] succeeded in heating Fe alloys in a microwave oven only up to 370°C in 30 minutes. But in all these studies no sintering of pure metal or alloy powders was reported. It was only in 1998 in this laboratory that the first attempt at microwave sintering of powder metals took place, [8] and since then many other researchers have reported successful sintering of many metallic materials. [9-12]

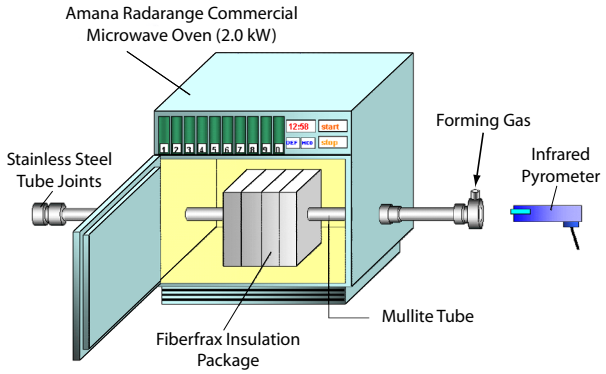

Figure 2. Schematic of a multimode, 2 kW and 2.45 GHz microwave sintering system for metals

Figure 2 illustrates a schematic of a typical microwave system used for the processing of metallic materials.

Figure 3 shows some commercial steel products sintered in a microwave field. Many commercial powder-metal components of various alloy compositions including iron and steel, Cu, Al, Ni, Mo, Co, Ti, W, Sn, etc. have also been sintered in microwaves successfully.

The microwave sintering of PM green bodies comprising various metals, steels and metal alloys produced highly sintered bodies in a very short period of time.[11] Typically the total cycle time was about 90 minutes, sintering temperature ranges between 1100°C to 1300°C and soaking time of 5 to 60 minutes. The mechanical properties such as the modulus of rupture (MOR) and hardness of microwave-processed samples were much higher than the conventional samples. As an example, microwave sintering of copper steel

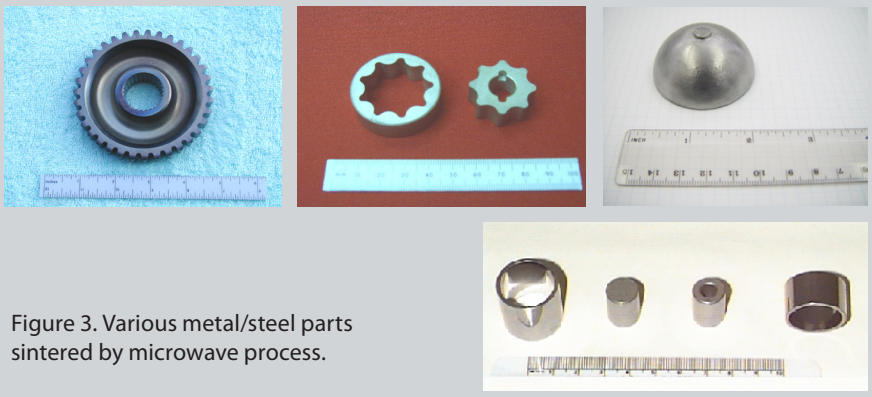

Figure 3. Various metal/steel parts sintered by microwave process.

Accomplishments

Professor Dinesh K. Agrawal, director of the Penn State Microwave Processing and Engineering Center, Professor of Engineering Science and Mechanics and Professor of Materials Science and Engineering, has been invited to join the World Academy of Ceramics as a Professional Member (Academician) of the Class "Science." Professional Members of the Academy are individuals who have made an international noteworthy contribution to the advancement of ceramics.

Dr. Agrawal gave a keynote lecture at the Fifth International Symposium on Electromagnetic Processing of Materials, organized by The Iron and Steel Institute of Japan, held at Sendai, Japan. His talk was titled "Electromagnetic Field Processing of Materials at Microwave Frequency (2.45 GHz)." He also gave a plenary talk on "Application of Microwave Energy for Materials Processing: Global Trends and Commercialization" at the Sixth Symposium on Microwave Science and Applications to Related Fields in Ogaki-City, Gifu Prefecture, Japan.

Tony Jun Huang, James Henderson Assistant Professor at the ESM Department, along with Prof. James Connor in the Department of Neurosurgery at the Penn State Hershey Medical Center, were awarded the Grace Woodward Grant for Collaborative Research in Engineering and Medicine for their proposal titled "Nanoporous Polymer Structure-based Biosensor Array for the Detection of RNA-Protein Interactions". The Grace Woodward Grants for Collaborative Research in Engineering and Medicine are an outgrowth of the Grace Woodward Endowments to Penn State. These merit-based awards are presented to PSU faculty on a competitive basis for creating or capitalizing on opportunities to use engineering to solve problems in the life sciences and medicine.

Dr. Huang along with Prof. Chung-Chiun Liu at Case Western Reserve University, Prof. Fraser Stoddart at UCLA, and Prof. William Goddard at Caltech, will receive $1.3 million over the next four years for their joint proposal "NanoElectroMechanical Systems (NEMS) using Light-Driven Molecular Shuttles as Active Nanostructures". This project is one of 25 out of 386 original proposals selected by the National Science Foundation (NSF) to be funded as part of its highly competitive Nanoscale: Interdisciplinary Research Teams (NIRT) grants.

Bernhard R. Tittmann, Schell Professor of Engineering Science and Mechanics, was elected Fellow of ASM International "For contributions to materials characterization and remote sensing in high temperature materials processing." Dr. Tittmann is a long-term member of ASM International, Secretary of the Penn State Chapter, and has been very active with the Electronic Device Failure Analysis Society (EDFAS).

Dr. Tittmann was recognized by the Acoustical Society of America in recognition of 25 years of continuous membership in which he increased and diffused the knowledge of acoustics and promoted its practical applications. Dr. Tittmann has been a Fellow of the Acoustical Society of America since 2000.

Publications/Presentations

Ivi Smid, Don Sampson, Lou Campbell, Rick Toth, and John Keane

Manufacturing and Machining Performance of Tough Coated Hard Particles and Products Thereof
Presented at 2006 International Conference on Tungsten, Refractory & Hardmetals VI, February 7–8, 2006, Orlando, Florida

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Ivi Smid, Gaurav Aggarwal and Albert Segall

Modeling of Deformation and Bonding of Composite Particle during Cold-Spray Deposition
TMS 2006, March 12-16, 2006, San Antonio, Texas

Parul Walia, Gaurav Aggarwal, Albert E. Segall, Ivi Smid, Timothy J. Eden

Development of Self-Lubricationg Coatings for Ti-6A1-4V Dovetails using a High-Velocity-Particle Consolidation (HVPC) Process
International Thermal Spray Conference 2006, May 14 - 17, 2006, Seattle, Washington

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Gaurav Aggarwal, Seong Jin Park, and Ivi Smid

Development of Niobium Powder Injection Molding: Part I. Feedstock and Injection Molding
International Journal of Refractory Metals and Hard Materials, Vol. 24, Issue 3, pp. 253-262, May 2006

Rick Toth, John Keane, and Ivi Smid

Manufacturing and Machining Performance of Tough-Coated Hard Particles and Products Thereof
2006 International Conference on Powder Metallurgy & Particulate Materials, June 18–21, 2006, San Diego, California

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Ivi Smid, Erich Neubauer, Paul Angerer, Guillermo C. Requena, and Hans-Peter Degischer

Thermophysical Properties of Copper Composites Reinforced with Negative CTE Fillers
2006 International Conference on Powder Metallurgy & Particulate Materials, June 18–21, 2006, San Diego, California

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J. M. Martin, John L. Johnson, Randall M. German, and F. Castro

Microstructural Evolution of Tungsten Heavy Alloys during Heating to the Sintering Temperature
2006 International Conference on Powder Metallurgy & Particulate Materials, June 18–21, 2006, San Diego, California

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Featured Research

…continued from page 3

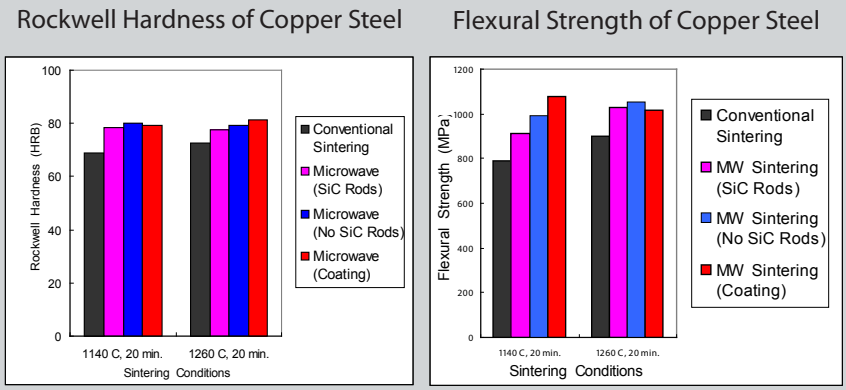

Figure 4. Comparison of hardness and flexural strength of microwave and conventionally sintered FC-0208 samples

(MPIF FC-0208) produced good sintered density, hardness, flexural strength, and near net dimensions, thus yielding equivalent or even sometimes superior mechanical properties than conventional sintering (Figure 4).

Some refractory metals such as W and Mo were also microwave sintered at much lower temperatures and sintering times than normally used in a conventional process. Figure 5 exhibits microstructures of microwave sintered nano-W powders which have been doped either with Y_2O_3 or with HfO_2 as grain growth inhibitors. It is to be noted that microwave sintering at 1400°C for 20 minutes produced submicron size microstructures and densities in the order of 95+%.

Figure 6 *(following page)* shows a typical microstructure of Mo sample sintered in microwave at 1600°C for 1 minute. The average grain size in this sample is also submicron and density approximately 98%.

The application of microwaves to metallic materials has been extended from sintering to melting, brazing and joining of bulk metals.[13-15] Figure 7 shows some bulk metals that have been melted in a microwave field using a

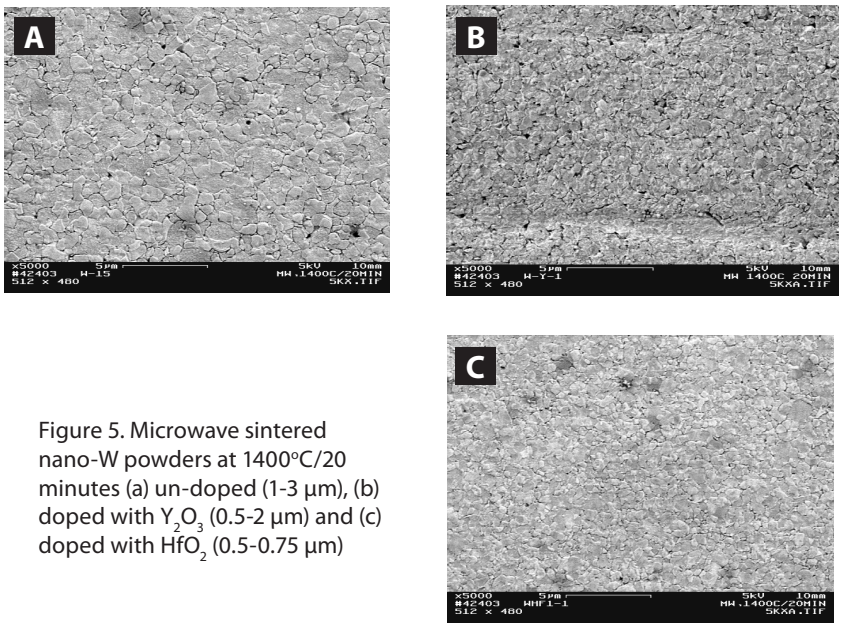

Figure 5. Microwave sintered nano-W powders at 1400°C/20 minutes (a) un-doped (1-3 µm), (b) doped with Y_2O_3 (0.5-2 µm) and (c) doped with HfO_2 (0.5-0.75 µm)

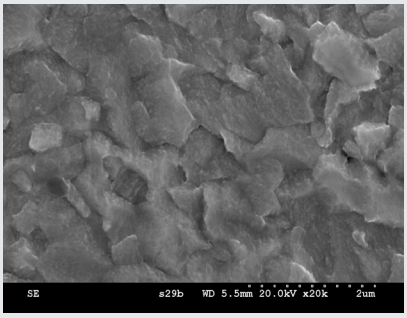

Figure 6. Typical microstructure of microwave sintered nano-Mo powder (1600°C for 1 minute, H2) showing average grain size of ~0.55 µm

special insulation package with susceptors. Now at Oak Ridge National Lab's Y-12 complex, this technology has been scaled up for commercial use for metal melting and casting into useful products including recycling of aluminum cans.

As mentioned earlier, microwave selectively heats powder metals and reflects bulk metals at room temperature. This feature has been exploited to braze and join bulk metals using powdered metal/alloy braze materials. We have successfully joined steels, W bulk metals, and also brazed super alloy components. An example is shown in Figure 8 in which regular steel and cast iron parts have been joined in the microwave field in 2-3 minutes using a braze powder. The joint is almost perfect, as indicated by the microstructural examination of the sample. This work can be extended to join metal to a ceramic, and also to develop ceramic coatings on steels or metal coatings on ceramics.

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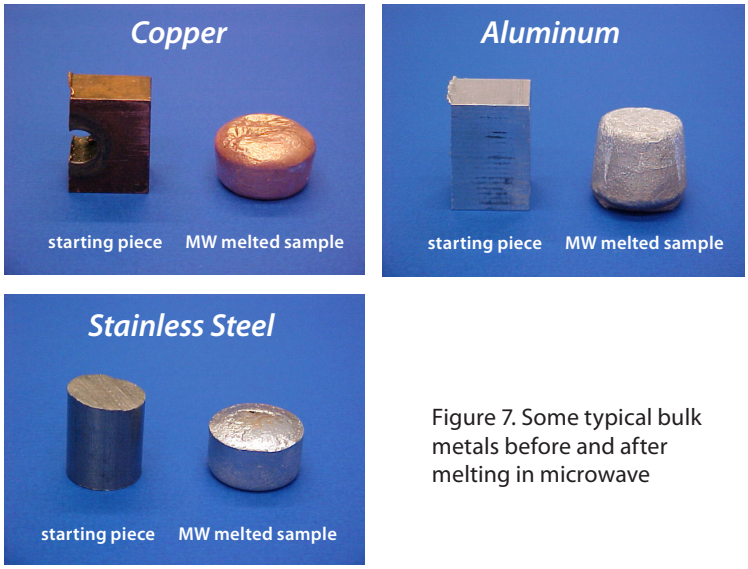

Figure 7. Some typical bulk metals before and after melting in microwave

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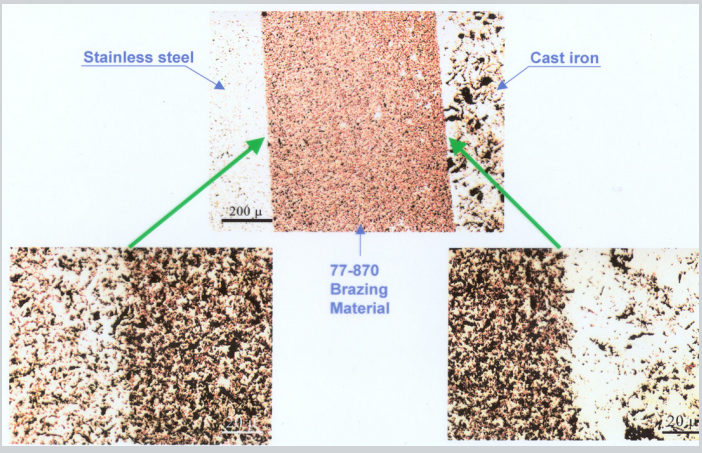

Figure 8. Microwave joining of stainless steel and cast iron using braze powder

Exchange of Students Continues

Three CISP students spent part of this past summer in Spain while one PhD candidate from Spain spent time doing research in the CISP lab. This is the fourth year that we have exchanged students with the Universidad Carlos III de Madrid, Spain.

Don Sampson, a graduate student in Engineering Mechanics, participated in the International Research Experience for Students in Innovative Sintered Materials (IRESISM) with the University Carlos III de Madrid in June and July. While in Spain, Don conducted research on sintering problems in titanium alloys. Efforts were made to sinter titanium alloys without reaction in varying atmospheres and on varying substrate or support materials. Different experiments were performed sintering titanium alloys in air while sealing the alloys in ceramic materials such as yttria, alumina, and cubic boronitride. Similarly, supports were created of titanium oxide, yttria, cubic boronitride, alumina, graphite, and titanium oxide; these supports were then used in sintering varying titanium alloys in inert gas environments. The resulting samples were then evaluated with x-ray diffraction and scanning electron microscopy to try and ascertain the level of contamination or reaction of the sintered parts.

Erik Byrne, an undergraduate student in Aerospace Engineering, also participated in the International Research Experience for Students in Innovative Sintered Materials (IRESISM) with the University Carlos III de Madrid in June and July. Erik studied metal injection molding, specifically working with superalloy-718, while in Spain. The goal was to find the right amount of binder to mix with the powder. He used a torque rheometer to mix the powder and binder (paraffin wax, polyethanol, acetic acid) and then the feedstock was put into a grinder to make it into pellets. Afterwards, a twin-screw extruder was used to further homogenize the mixture. A capillary rheometer was used to determine the viscosity of different feedstocks. Mixtures with a higher percentage of powder had higher viscosity. Also, the addition of more acetic acid lowered the viscosity. The low viscosity is desirable for the actual molding of a part in the injection molding machine.

Roderick Reber, an undergraduate student in Earth and Mineral Sciences, also participated in the International Research Experience for Students in Innovative Sintered Materials (IRESISM) with the University Carlos III de Madrid in June and July. Rod conducted research on materials characterization.

Maria Luisa Delgado, a PhD student in Chemistry at the Universidad Carlos III de Madrid, visited CISP in July and August to obtain more data for her doctoral thesis on the liquid phase sintering of aluminum alloys. This was the third annual trip that Maria has made to Penn State to study the sintering process and the atmospheric conditions to eject during the manufacturing process of her material.

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Maria Luisa Delgado <mdtienda@ing.uc3m.es>

Student Update

Summer Researchers

Four undergraduate students took advantage of various summer research programs and worked in the CISP P/M Lab this past summer.

Erik Byrne, an undergraduate student in Aerospace Engineering at Penn State, worked on sample preparation for coated hard powders and an SLS alloy

Cynthia Lin, an undergraduate student in Biological Sciences at the University of Chicago, worked on the encapsulation of tungsten carbide particles with nickel using an electroless plating procedure.

Iris Noriega, an undergraduate student in Aerospace Engineering at Penn State, studied lubricants. She evaluated six mixtures produced from different lubricants mixed with A1000 metal powder at 0.7wt% content for powder characteristics, ejection force, green strength, and sinter strength.

Benjamin Risser, an undergraduate in Aerospace Engineering at Penn State, worked on a testing procedure for estimating the fracture toughness of a sintered composite psuedoalloy.

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Debinding Research

Binder Removal in N_2/H_2 Atmospheric Mixtures

The choice of atmosphere during debinding is important for optimum processing of press/sinter and injection molded components. High purity hydrogen and nitrogen gases generate straightforward and well studied degradation curves during thermal gravimetric analysis. However when mixtures of these gases are used, mass changes during heating become more complex; and chemistry of the debound part can show undesired carburization or oxidation. Mapping of the generated gases from the debinding process is of great interest in understanding and controlling the final powder metallurgy product.

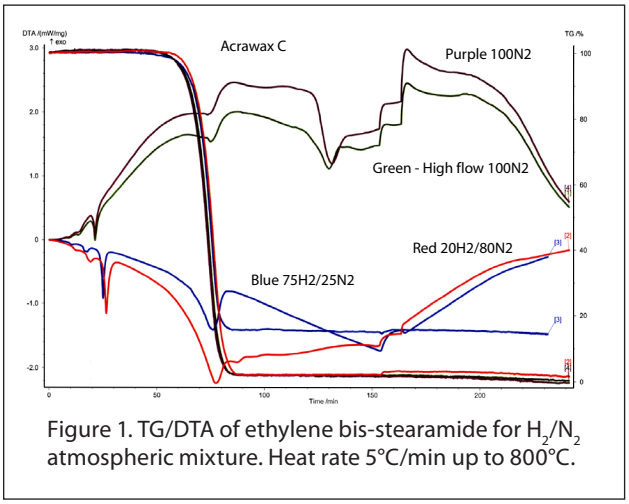


Figure 1. TG/DTA of ethylene bis-stearamide for H_2/N_2 atmospheric mixture. Heat rate 5°C/min up to 800°C.

Tests are complete on polymers commonly used in press and sinter and injection molded components including ethylene bis-stearamide, paraffin wax (PW), polyethylene glycol (PEG), stearic acid (SA), polypropylene (PP) and a general mixture of PP/PW/SA (50/45/5wt%). They were tested using a using a TG/DTA analyzer coupled with a FTIR that allows evaluation of the thermal degradation pattern and characterization of the gases formed during heating (Figure 1).The atmospheres chosen were pure nitrogen and mixtures of hydrogen and nitrogen (20/80 and 75/25 respectively).

Analysis of the resulting data is the next step in this project to evaluate powder-binder interactions during debinding. This will be followed by testing mixtures of powder with binder. Evaluation of thermal degradation together with gas evolution characterization on binder and powder-binder mixes will give a detailed understanding of the overall debinding process.

Chantal Binet <cub9@psu.edu>

Moses Awarded Scholarship

James Moses, a junior majoring in Engineering Science and Mechanics, has been awarded the 2006-2007 CPMT AMETEK scholarship.

James is an honors student transferring from the Penn State York campus to the University Park campus for the fall 2006 semester. He has held leadership positions and is very familiar with industrial processes and manufacturing needs. His outstanding academic record combined with his strong background in industrial applications give us confidence that he has a high potential for success.

Powder Metals Fundamentals Course

Don Heaney presented a course on Powder Metals Fundamentals on November 16 at Penn State DuBois. He reviewed all aspects of powder metal processing, component design, powder selection, powder pressing, sintering, and component characterization.

Debinding Research

Gaurav Aggarwal, Ivi Smid, Seong Jin Park, and Randall M. German

Development of Niobium Powder Injection Molding: Part II. Debinding and Sintering
International Journal of Refractory Metals and Hard Materials, in press, available online July 10, 2006

Samerjit Homrossukon, Sheldon Mostovoy, and Judith A. Todd

Investigation of Hydrogen Assisted Cracking in Pressure Vessel Steels
2006 ASME Pressure Vessels and Piping Division Conference, July 23-27, 2006, Vancouver, British Columbia, Canada

Donald F. Heaney

Mass Production of Micro Components Utilizing Lithographic Tooling and Injection Molding Technologies
ICOMM 2006, First International Conference on Micromanufacturing, September 13-15, 2006, Urbana, Illinois

Donald F. Heaney

Powder Injection Molding of Implantable Grade Materials
Proceedings of MSEC: 2006 ASME International Conference on Manufacturing Science and Engineering October 8-11, 2006, Ypsilanti, Michigan

E. Neubauer, Ivi Smid, G. Requena, H. P. Degischer, and P. Angerer

Thermophysical Properties of Copper Composites Reinforced with Negative CTE Fillers
2006 International Conference on Powder Metallurgy & Particulate Materials, June 18–21, 2006, San Diego, California

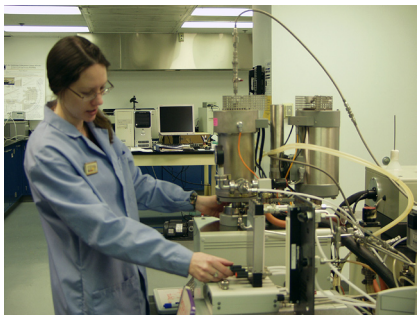
In Memoriam

CISP honors the memory of **Trista Danielle Martin**, age 22, a graduate student in the InterCollege Graduate Degree Program in Materials, who had just begun conducting research with Professors Ivi Smid and Al Segall. Trista and her friend, Kristy Murray, were tragically killed on Nov 17, when their car swerved to avoid a deer as they traveled home for Thanksgiving. We shall remember Trista's warm smile, enthusiasm for her research and lively personality.

Testing and Services

Testing & Services (Lab Services) was developed as a non-profit way to offer technical testing for industry and the research community, especially within the state of Pennsylvania. Inside CISP, Lab Services provides funding for basic supplies, equipment maintenance, and student training.

Thermal analysis, particle size and scanning electron microscopy are the three most frequently requested services in the past six months. Such techniques give insight into dimensional; phase and density changes, and size, shape and condition of parts and powders.



Most recently we have been able to identify phase changes in precious metal and steel alloys with greater than five constituents, and to identify crack origin and progression in hard, wear resistant materials.

Requested work is usually one week turnaround due to straightforward reporting of results without consulting or recommendations. CISP members receive member discounts for work coming through Testing and Services.

Contact Kristina Cowan at <kcc126@psu.edu> or call 814.865.1393 to schedule or discuss specifications, requirements and number of samples.

Student Update

Students Continue P/M Research

The following students are working in the CISP P/M Lab at Penn State:

Graduate Students:

Gaurav Aggarwal, *Engineering Science & Mechanics, May 2007 <aggarwal@psu.edu>*

Ramprasad Chandrasekharan, *Engineering Mechanics, May 2007 <rxc306@psu.edu>*

Vani Ramabhatt, *Industrial Engineering, May 2007 <vxr151@psu.edu>*

Don Sampson, *Engineering Science & Mechanics, May 2007 <das343@psu.edu>*

Jens E. Weyant, *Engineering Science, May 2008 <jew238@psu.edu>*

Undergradaute Students:

Shaun Campbell, *Engineering Science & Mechanics, May 2007 <smc981@psu.edu>*

Dan Cunningham, *Aerospace Engineering, May 2007 <djc292@psu.edu>*

Eric M. Gift, *Mechanical Engineering, May 2008 <emg5013@psu.edu>*

James Moses, *Engineering Science and Mechanics, May 2008 < jmm741@psu.edu>*

Rod Reber, *Earth & Mineral Sciences, May 2008 <ryr5000@psu.edu>*

Tyler Wise, *Materials Science & Engineering, May 2007 <tmw214@psu.edu>*

Celebrating 100 years ESM engineering science & mechanics

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