Center for Innovative Sintered Products

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Director's Message

PENNSTATE



CISP has increased in membership revenue under the new structure which concentrates on refractory and hard materials. Precompetitive revenue has grown by 40 percent. This revenue has primarily been used for CISP operational expenses and support for graduate students that may lack a semester of stipend and tuition to finalize their degree. Current member's include:

- Global Tungsten and Powders
- Plansee
- Kennametal, Inc.
- Dennis Tool Company Inc.
- MPIF
- Horiba
- Anter

Other companies have expressed interest and plan to join shortly. We are in the process of putting together prospective precompetitive projects for voting. Our industrial council has suggested a focus in hard metals. This hard metal project should begin at the beginning of the fall 2011 semester. We will be looking to both members and nonmembers to evaluate these prospective projects.

CISP participated in the Metal Powders Industry Federation (MPIF) Sintering seminar, Cleveland, OH, in November 2010. Donald Heaney, associate professor of engineering science and mechanics, lectured on vacuum sintering. CISP also plans to participate at the MPIF powder metallurgy short course in State College, PA, during July 2011. Heaney plans to lecture on refractory and hard metals – applications, properties, and processing, and powder metal testing and characterization. An update on the center was also given at the spring meeting of the Refractory Materials Association in Alexandria, VA.

Our research activities over the last six months have focused on the following projects:

- 1. Microforming Federal
- 2. SPS (or FAST) of refractory metals Industrial
- 3. Bonding of metals and ceramics Industrial
- 4. Copper diamond composites via metal injection molding- Industrial
- 5. Final stage sintering CISP
- 6. Refractory metal ordinance components Federal

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.

Member's Insider

Portions of this newsletter are distributed to members, only:

- · Laser-Sustained Plasma Deposition of Titanium Nitride Nanopowder
- Electroless Nickel Deposition on Aluminum Powder as Feedstock Preparation for Cold Spray Processing of Ni-Al Alloys
- The Effect of Vacuum on Final Stage Sintering
- Self-Lubricating Coating Utilizing Cold Spray Technology

For more information on becoming a member, visit our website at www.cisp.psu.edu or send an e-mail to cisp@psu.edu.

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- Tooling for Spark Plasma Sintering Technology
- Thermal-Mechanical Hysteresis in Shape Memory Alloys (SMAs)
- Nanodiamonds: The Magic Dust
- Abrasion Wear Testing

Upcoming Events

October 9-12, 2011 **Euro PM2011 Congress & Exhibit** Barcelona, Spain www.epma.com/pm_2011

October 16-20, 2011 **Materials Science & Tehcnology 2011 Conference & Exhibition (MS&T'11)** Columbus, OH www.matscitech.org

January 22-27, 2012 36th International Conference and Expo on Advanced Ceramics and Composites (ICACC'12) Daytona Beach, FL http://ceramics.org/tag/icacc12

April 2012 (TBD) Industrial Members' Meeting University Park, PA www.cisp.psu.edu

June 10-13, 2012 MPIF/APMI International Conference on Powder Metallurgy & Particulate Materials (PowderMet2012) Nashville, TN www.mpif.org



Tooling for Spark Plasma Sintering Technology

Spark Plasma Sintering (SPS) is an emerging technology for fabrication of metals, intermetallics, ceramics, and their composites starting from powder forms. A number of university-based and government research institutions including RDECOM-ARDEC at Picatinny Arsenal and Penn State's Department of Engineering Science and Mechanics have purchased a SPS system for research, development, and prototyping of armament components. Industrial Graphite Sales (IGS) will jointly develop a cooperative road map from R&D to commercial exploitation of graphite tooling design, specification and manufacture for SPS technology for military, dual use and strictly commercial applications.

Presently, there are more than 100 powder metallurgy companies located within Pennsylvania alone and more than 500 powder metallurgy companies in the tri-states New Jersey, New York and Delaware. Many of these companies will benefit from the commercialization of SPS capability. The primary emphasis of IGS will be to optimize the use of the tooling for already developed component production SPS technology and combine it with the gained knowledge to vastly improve its performance on existing and new applications. Benefits to Department of Defense (DOD) and commercial applications are currently being explored and numerous high value payoffs have been identified, including significant cost savings, higher reliability and lower manufacturing costs of failure-prone components, and totally new material that will enhance war-fighting capabilities.

THE SPS PROCESS: SPS is a one-step process that produces ~100 percent dense components. It is robust and has the potential to be approximately three- to four times more productive than current single unit fabrication methods. The SPS process is also a significant energy savings due to shorter processing cycles and lower required temperatures. In the SPS process, powder is simultaneously compacted within a graphite die under high density current load. Through this high current density, a spark is generated at the particle-particle contacts resulting in substantial localized heat generated in the form of plasma that directly produces a high diffusion rate and results in a stronger particle bonding. This technique results in very limited grain growth and therefore, fine particle material structures or microstructures. In contrast, conventional sintering techniques produce coarse grained and subsequently weaker microstructures. Current estimates and case histories from Germany and Japan suggest that fully dense parts are produced with SPS with a total process reduction time of approximately 75 percent. Most importantly, these described successful "production-izing" of SPS will create one of only two commercially available single press/full density powder metal production processes in the world.

BENEFITS OF SPS: The SPS process is expected to result in numerous advantages for military components made of metal and other hard materials that will offer performance improvements for a wide range of applications. SPS technology offers considerable advantage over current powdered metal material pressing and sintering techniques: Density, Tool cost, Repeatability, Cycle length/control.

The search for full density represents the Holy Grail of structural iron based PM manufacture. Of the \$150 million annual estimated amount of R&D spent annually by commercial R&D component and powder producers, more than 70 percent is focused on increasing component density. The term of this development stretched over a half a century with a robust process to create full density still being sought. Because the global PM industry has cultivated the majority of its low hanging fruit in product conversion and application development, increased density processes have become even more important if the industry is to continue to grow. Because powder metal and ceramic manufacturing represents a relatively high fixed cost process, the impact of tooling on its overall cost structure is marked. SPS has yet to launch a highly productive manufacturing option and the roll of tooling in this development will be vital.

MARKETS: SPS technology is in an early stage of introduction in North America and exists only as an R&D process at the present time. Primary benefits of this technology to U.S. DOD and commercial manufacturing include:

- · An extensive class of new materials with potential for many unique properties
- Substantially lower manufacturing costs as compared to the next available full density manufacturing process alternatives
- More uniform and refined metallurgical microstructures that result in greater physical and mechanical properties and resulting component performance
- Newly refined robust and productive manufacturing best practice producing enhanced reliability
- 100 percent dense nanoparticle and conventional particle structure components for enhanced penetrators, cutting tools, more reliable rotating machine parts, etc.

GOALS AND OBJECTIVES OF PROCESS DEVELOPMENT: The goals and objectives of this effort contain the following focus:

- 1. Introduction of new and significantly advanced tooling materials and design concepts and their application to a myriad of SPS needs, including war fighting, logistics, maintenance, medical, automotive, aerospace and others
- 2. Increased effectiveness and reliability of tooling and its inter-relationship with SPS systems
- 3. Reduced overall SPS tooling build, repair and maintenance costs through the development of an understanding of the following factors: tool material, component geometry, tool geometry, SPS operating conditions, component material of fabrication.

For more information, contact Rocco Petrilli at 814-590-6972 or rpetrilli@ppspi.com.

Thermal-Mechanical Hysteresis in Shape Memory Alloys (SMA)

Shape Memory Alloys (SMA) undergo a reversible solid-solid phase transformation that enables the recovery of large deformations, albeit a hysteresis exists. The phase transformation is a martensitic transformation (MT). Figure 3.1 illustrates the fundamental characteristic SMA material responses. The high-temperature parent phase (austenite) transforms to a low-temperature product phase (martensite), which exhibits a different crystal structure. The characteristic temperatures Ms/Mf and As/Af, respectively, define the start and finish temperatures for the austenite-to-martensite (exothermic reaction) transformation and the back transformation martensite-toaustenite (endothermic reaction). The hysteresis is characterized by the temperature differential between Ms and Af. When the temperature is cycled under a constant load, large strains can be fully recovered upon heating. This behavior is referred to as the Shape Memory Effect (SME). Furthermore, mechanical deforma-



Figure 3.1: Schematic of fundamental SMA behavior

tion occurring below the Ms temperature can be recovered via heating above the Af temperature due to SME. Deformation at a constant temperature above the Af temperature can be recovered during unloading and this response is referred to as the pseudoelastic effect. The austenite-to-martensite is stress-induced at a critical stress level and the back transformation takes place at a lower stress level. In this case, the hysteresis is characterized by the stress differential.

Shape Memory Alloy	Lattice Invariant Shear	Temperature Hysteresis (°C)	A _f Tempera- ture (°C)	PE Criti- cal Stress (MPa)	Maximum Strain (Tension)	Transformation (Compression)
NiTi (cubic to monoclinic)	0.13	~30	-10	700-900 (C)	0.105	-0.064
NiTi (cubic to orthorombic R)	0.026	~2	-	-	0.0017 to 0.0051	-0.0026 to -0.0052
FeNiGa (cubic to tetragonal)	0.1267	~2	30	900 (T)	0.14	-0.062
CoNiAl (cubic to tetragonal)	0.1137	~10	40	1000- 1700 (C)	0.06	-0.033

Table 3.1:	Characteristics	of different	classes	of SMA

The fundamental response is dependent on the SMA chemistry. Representative properties of different classes of SMAs are included in Table 3.1. The underlying transformation mechanism is a lattice invariant shear, which is identified by the magnitude of the Burgers vector. This shear is associated with a martensite variant and the martensite microstructure comprises multiple variants. The number of variants depends on the alloy chemistry, and thus the martensite morphology can differ for different SMAs. Consequently, at the macroscale, stark contrasts are observed in transformation hysteresis, critical stress, and transformation strain. A comprehensive experimental program, including stress-free thermal cycling, constant load thermal cycling, and constant temperature stress/strain cycling, has been

undertaken for NiTi, CoNiAl, and NiFeGa SMAs in the aged and unaged states. The NiFeGa class of SMAs undergo martensite to martensite (or inter-martensitic) transformations. The experimental findings characterize the effect of second-phase particles and inter-martensitic transformations on the level of hysteresis. For more information, contact Reginald Hamilton at 814-865-7684 or rfhamilton@psu.edu.

Reginald F. Hamilton, assistant professor, Engineering Science and Mechanics



Nanodiamonds: The Magic Dust

Detonated nanodiamonds (DND) are a bulk powder material, that when individually looked at, have the same atomic configuration as a diamond at a size under 100nm. To give perspective, the width of a DNA strand is 1.4nm in diameter. DND can have sizes that range from 3-40nm. The DND NanoBlox produces have an average size distribution of 3-5nm. Nanotechnology has become a novel and exciting field due to the change in properties of normally familiar materials due to size. In the case of diamonds, what had been thought of as an abrasive, in bulk powder on the nano-scale becomes a lubricant. Also, more importantly in our discussion, nanodiamonds demonstrate significant heat transfer and conductivity capabilities. Preliminary data suggests that the addition of nanodiamonds to motor oil dissipates heat more efficiently resulting in lower engine temperatures, increased fuel efficiency by approximately 4 percent and prolonged oil life by at least four-fold. Current sponsored projects for nanodiamonds include sintered coatings, alloys, hardening agents, dendrimers for drug delivery, nanodiamond modified biodegradable and bioinert scaffolds, water purification, heat transfer fluids and alloys. Military applications include lighter and harder armor, plating techniques, oil additives, and dry lubricants. *For more information, contact Ben Legum at 814-205-3393 or blegum@clarion.edu*.



Figure 4.1: Unrefined and refined detonated nanodiamond powder with average particle size of 3-5nm

Abrasion Wear Testing



ASTM G-65 measures the wear resistance of a material against abrasion. The test method utilizes an apparatus that forces a sample against a rubber wheel while it spins (Figure 5.1). At the same time, a well controlled sand flows between the sample and the wheel. This combination of rotating wheel and sand wears away the sample material depending on its resistance to abrasion. The method allows for modifications to the force and "distance" traveled by the wheel to adjust the test to the necessary aggression level for various materials.

Ben Legum, assistant professor, Clarion University

Like all ASTM standards, the apparatus and conditions of the consumables are controlled to different tolerances to provide for consistent and comparable results. The most critical factor in generating repeatable results is the wheel's sur-

Figure 5.1: CISP's abrasion wear testing apparatus.

face. The rubber surface degrades with use and it is a qualitative decision on when the wheel needs to be redressed to prevent poor wear problems. If the edge of the rubber wheel is not square or the test is performed incorrectly a two step wear pattern develops as shown on the right in Figure 5.2. The left image demonstrates a proper wear pattern that has straight edges, smooth base, and no "two step" pattern.

After testing is complete, the samples are weighed and the mass loss due to abrasion is determined. The density of the sample is measured and utilized to convert mass loss into a volume loss. Samples can then be ranked according to their volume loss, if they used the same testing conditions. This ranking provides a qualitative comparison between the samples and gives an indication which material would provide better wear resistance. For more information on this technique or characterization of your materials, please contact Michael Disabb-Miller at 814-865-1393 or mjd39@engr.psu.edu. You can also visit our testing services price list at http://www.cisp.psu.edu/ testserv/pricelist.htm.

Consistent Wear

Inconsistent Wear





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