PENNSTATE 1855

# CISP

## Center for Innovative Sintered Products

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## Director's Message

CISP's recent activities include finalizing the new structure of CISP with a concentration on refractory and hard materials. Revenues for this pre-competitive effort are up 20 percent; however, we would like to have more companies participate in this effort. Your support will support an academic institute that provides employees, testing, services, and research for our industry. CISP participated in the Metal Powders Industry Federation (MPIF) PM short course held here in State College on July 26-28, 2010. During this event, CISP lectured on refractory and hardmetals – applications, properties, and processing, and testing of P/M products. CISP also participated in the MPIF PM Sintering Seminar held in Cleveland on December 7-8, 2010. During this event, CISP lectured on vacuum sintering processing and equipment configurations.

Our research activities over the last 6 months have focused on the following projects:

1. Microforming – funded by the National Science Foundation
2. Spark Plasma Sintering (SPS) or Field Assisted Sintering Technology (FAST) of refractory metals – industrial
3. Bonding of metals and ceramics – industrial
4. Wear testing of P/M products – industrial
5. Final stage sintering – internal

Please join us at our next members' meeting being held on April 13-14, 2011, in State College. Contatct Renee Lindenberg at 814-865-2121 or cisp@psu.edu for more information on this upcoming event.

*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:*

- Engineered Self-Lubricating Coatings Utilizing Cold Spray Technology
- Photoresist Micro Components
- Electroless Nickel Deposition onto Aluminum Powder
- Particle Sizing Analysis
- Student and Staff Contact Information

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

## Inside This Edition



## Upcoming Events

January 23-28, 2011
**35th International Conference & Exhibition on Advanced Ceramics and Composites**
Daytona Beach, FL
http://ceramics.org/icacc-11

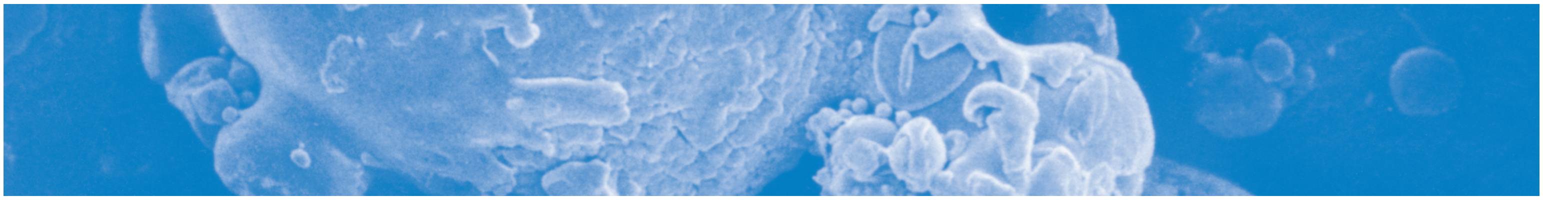
March 14-16, 2011
**MIM 2011**
Orlando, FL
www.mpif.org

April 13-14, 2011
**Industrial Members' Meeting**
University Park, PA
www.cisp.psu.edu

May 18-21, 2011
**PowderMet 2011 (with) Tungsten, Refractory & Hardmaterials VIII**
San Francisco, CA
www.mpif.org

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

October 16-20, 2011
**MS&T'11**
Columbus, OH
www.matscitech.org



# Overview of the Cobalt and Tungsten Markets

*This article is an updated summary of a presentation given at the April 28, 2009, Refractory Metals Association members meeting in Annapolis, MD. The U.S. Geological Survey's (USGS) National Minerals Information Center collects, analyzes, and distributes statistics and information on mineral and metal commodities. Our products are available from the USGS minerals information Web page at http://minerals.usgs.gov/minerals/.*

Major similarities between the markets for cobalt and tungsten are shown in figure 1.

**Figure 1: Major Similarities in 2008**

- World Market Size
  - Cobalt ~63,000 tons
  - Tungsten ~66,000 tons
- U.S. Supply
  - No significant U.S. mine production
  - High import reliance
    - ❑ Cobalt 81%
    - ❑ Tungsten 60%
  - Scrap recycling
  - Shipments from the National Defense Stockpile
- China
  - Major producer
  - Major consumer
  - Investing overseas
- Some common end uses

**Figure 2: Chinese Policies (Relative to Tungsten)**

- Control of production & exports
  - Quotas
  - Export tax rebates—>export taxes
  - Production constraints
- Investment in foreign mine projects
  - Australia
    - ❑ King Island Scheelite Ltd. / King Island Mine
    - ❑ Queensland Ores Ltd. / Wolfram Camp Mine
    - ❑ Thor Mining PLC / Molyhill Mine
- Stockpiles
  - National strategic stockpile
  - Local government stockpiles of tungsten concentrates

## Tungsten

In 2008, global tungsten consumption was distributed as follows: cemented carbides, 57 percent; steels and other alloys, 23 percent; mill products, 14 percent; and chemical uses, 6 percent. In the past decade or so, China's economic and industrial growth has resulted in an increase in consumption of many raw materials, and by 2008 China consumed nearly 40 percent of the world's annual tungsten supply.

For much of the past century, China has been the dominant producer of tungsten concentrates, so it has had a significant influence on the global market. As Chinese internal consumption has grown, the Chinese government has added controls to tungsten mining, processing, and exports in an effort to conserve its tungsten supplies, reduce energy consumption and pollution, and stabilize prices. China has begun to invest in new mine projects outside of its borders to gain access to additional tungsten supply. There have been reports that the Chinese government planned to add tungsten to its strategic materials stockpile, and some local governments stockpiled tungsten concentrates to help mines in their areas remain in production during the economic downturn that began in late 2008 (figure 2).

Since 1999, the U.S. government has contributed to world supply through its sales of tungsten from the National Defense Stockpile (NDS). At the end of 2008, a significant amount of tungsten concentrates (19,700 metric tons of contained tungsten) and a lesser amount of tungsten metal powder (183 tons) remained in the NDS.

## Cobalt

Historically, superalloys were the leading use of cobalt on a global basis. Since the early 1990s, cobalt use in rechargeable batteries has grown very rapidly, and now the battery industry is the leading consumer of cobalt (figure 3). In the

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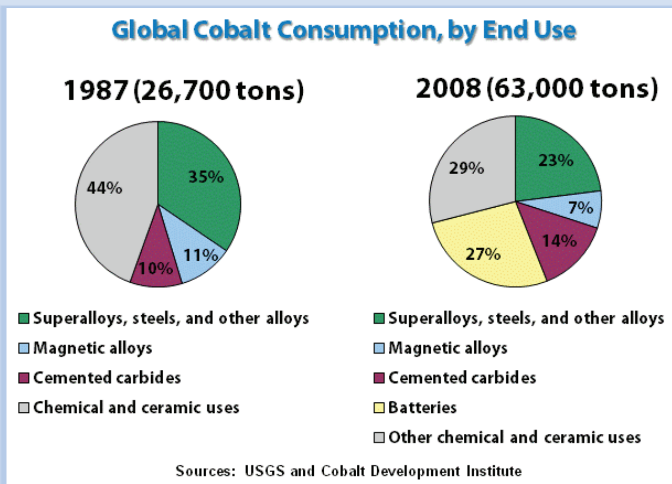


**Figure 3**: Global cobalt consumption by end use

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past decade, Chinese cobalt consumption has increased from insignificant to almost one-quarter of world consumption. Most of this growth has been brought about by the needs of the battery industry, which now represents more than one-half of the cobalt consumed in China.

In 2008, more than 40 percent of world cobalt mine production came from the Democratic Republic of the Congo (DRC); other significant sources included Australia, Canada, China, Russia, and Zambia. Cobalt is often a byproduct of mining other, more abundant metals, such as nickel or copper. This limits the flexibility in adjusting cobalt mine production to match demand.

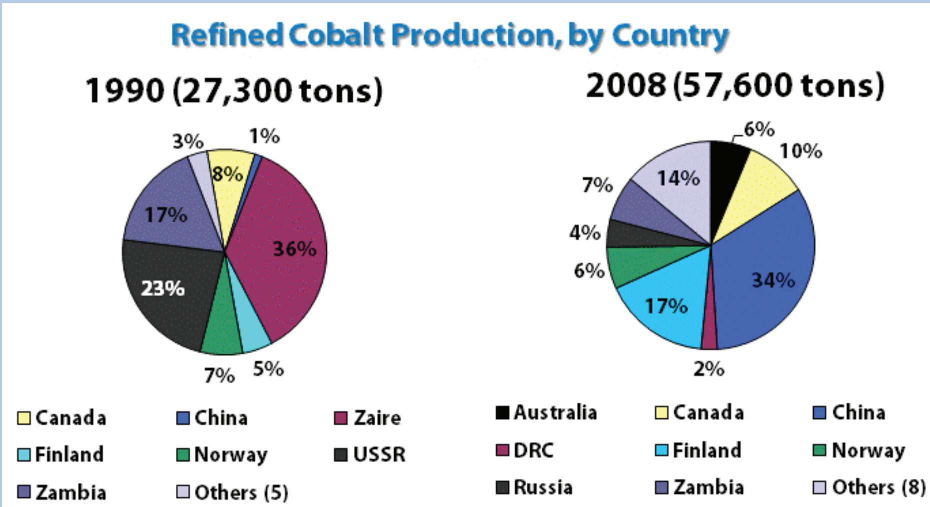


**Figure 4**: Refined cobalt production by country

**Figure 5: Summary**

| Common Factors | Differences |
|---|---|
| ■ Global economy | ■ Main ore source<br>• Cobalt - DRC<br>• Tungsten - China |
| ■ Market size | ■ Leading end uses<br>• Cobalt - batteries and superalloys<br>• Tungsten - cemented carbides |
| ■ High U.S. dependence on imports | ■ Unique end uses |
| ■ Importance of recycling | ■ National Defense Stockpile inventories<br>• Cobalt - almost gone<br>• Tungsten - more than five years of supply |
| ■ China as leading consumer | ■ Cobalt mine production less flexible with respect to demand |
| ■ China as leading producer | ■ Cobalt futures trading |
| ■ Some end uses<br>• Cemented carbides<br>• Specialty steels<br>• Wear- and corrosion-resistant alloys<br>• Catalysts | |

Since 1990, world production of refined cobalt has more than doubled (figure 4). The DRC (formerly Zaire) has shifted from being the leading producer of refined cobalt to producing an insignificant amount. In recent years, large amounts of ores, concentrates, and semirefined alloys and compounds have been exported from the DRC to China for refining. Chinese refinery production has increased to about one-third of world production.

Similar to tungsten, scrap recycling and the NDS are significant sources of cobalt supply. The U.S. government began selling cobalt in 1993, and by the end of 2008, the NDS held less than 500 tons of cobalt.

In summary, the similarities and differences in the cobalt and tungsten markets are presented in Figure 5.

*Kim B. Shedd,*
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# Spark Plasma Sintering of Tungsten Powder

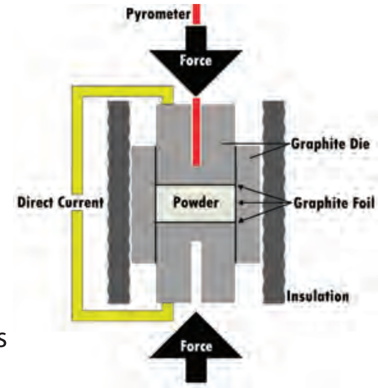


**Figure 1**: Schematic of SPS

Spark Plasma Sintering (SPS) or Field Assisted Sintering Technique (FAST) is a sintering method that can produce near 100 percent dense materials without the use of binder or lubricants. The powder is held in a die while direct current and pressure are applied simultaneously by rams. Within SPS, there are many different factors that can be explored which affect the density and microstructure of a sintered material. High pressures and heating/cooling rates allow for quick sample production and control of the microstructure that would not be possible through traditional sintering techniques. To this end, tungsten powder was sintered in the SPS at CISP with varying heating rates and hold times to explore the relationship between processing conditions and final density.

The samples were produced with a 20 mm diameter die and approximately 25 grams of Global Tungsten and Powders (GTP) M55 tungsten powder. Figure 1 is a schematic of the SPS components contained within a vacuum chamber. The main body of the die was graphite, and graphite foil was used to protect the die from direct contact with the powder. Load was applied uniaxially, direct current heated the sample, and carbon fiber insulation was used to maintain the temperature. At 4 mm from the top edge of the powder, a pyrometer measured the temperature of the die. The constants through the tungsten sintering were 65 MPa of maximum pressure, a heating rate of 100°C/min to reach 1300°C, and maximum pyrometer temperature of 1900°C.

**Table 1**: SPS of tungsten powder with varied heating rates.

| *Heating Rate (°C/min)* | *Heating Time (min)* | *Hold Time (min)* | *Total Time (min)* | *Density (g/cc)* | *Theoretical Density %* |
|---|---|---|---|---|---|
| 50 | 12 | 1 | 13 | 18.131 | 94.2% |
| 150 | 4 | 9 | 13 | 18.42 | 95.7% |
| 250 | 2.4 | 10.6 | 13 | 18.402 | 95.6% |
| 150 | 4 | 1 | 5 | 18.136 | 94.2% |
| 250 | 2.4 | 1 | 3.4 | 18.305 | 95.1% |

The heating rate from 1300°C to 1900°C was varied between 50, 150, and 250°C/min. To compare the 50°C/min sample to subsequent runs, all other samples were held for one minute at 1900°C or had a total run time of 13 minutes. The results from these tests are contained in Table 1 and demonstrate little to no effect of heating rate on the final density of this tungsten powder. (The powder had a starting density of 19.03 g/cc, as determined by pycnometery, and tungsten has theoretical density of 19.25 g/cc.)

The sintered samples did not achieve the theoretical density of tungsten indicating the presence of porosity, which is visible in the polished cross section of the sample created with a 250°C/min rate and 10.6 min hold shown in Figure 2. The final density could have also been reduced by the production of tungsten carbide from the interaction between the tungsten and graphite foil at the elevated processing temperature. The heating rate did not significantly affect the density in these tests. This result demonstrates that instead of focusing on density, SPS processing conditions could be tuned to achieve other important material characteristics, such as grain size and microstructure. *For more information on this technique or characterization of your materials, please contact Michael Disabb-Miller at 814-865-1393 or mjd39@psu.edu. You can also visit our testing services price list at http://www.cisp.psu.edu/testserv/pricelist.htm.*

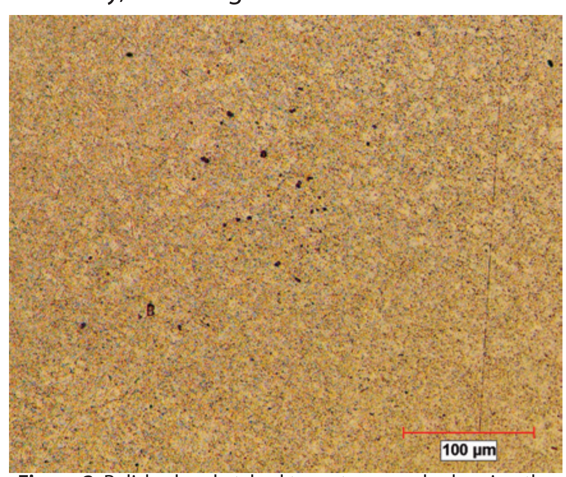


**Figure 2**: Polished and etched tungsten sample showing the porosity of the sample.

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