ADMINISTRATIVE INFORMATION

1. **Project Name:** Development of Bulk Nanocrystalline Cemented Tungsten Carbide for Industrial Applications

2. **Lead Organization:** University of Utah
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3. **Principal Investigator:** Zhigang Zak Fang & H. Y. Sohn
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4. **Project Partners:** Idaho National Lab, (Dr. Peter Kong, Peter.kong@inl.gov, (208)526-7579)
Kennametal Inc. (Dr. Shivanand Majagi) shivanand.majagi@kennametal.com, (479) 636-1515)
Smith International, Inc. (Dr. Anthony Griffo, agriffo@smith.com, (281)443-3370)

5. **Date Project Initiated:** April 1 2004

6. **Expected Completion Date:** March 31, 2007

PROJECT RATIONALE AND STRATEGY

7. **Project Objective:**

The project has the objective to investigate, develop and deploy processes for the synthesis of nanocrystalline tungsten carbide and cobalt composite powder and the consolidation of nanocrystalline WC/Co powders into bulk nanocrystalline materials. The overall goal is to develop a process that can produce bulk materials with true nanoscale grain sizes (<100 nm) and realize the potential of mechanical properties of cermet materials with a microstructure scale that has never been achieved before in a consolidated bulk state.

8. **Technical Barrier(s) Being Addressed:**

Nanocrystalline WC-Co was among the first group of materials that demonstrated the potential of nanocrystalline materials. The advances in nanoscale powder producing technologies raised hopes for dramatically improving mechanical properties. However, further development of nanocrystalline WC-Co materials are currently stalled because of the lack of sintering technologies that could produce *bulk* WC-Co materials with true nanometer grain sizes (<100nm). The lack of success with the sintered materials affects adversely further development of the powder synthesis processes. Consequently, the cost/benefit ratios of nanocrystalline WC-Co powders produced by existing technologies are still unacceptable to the industry.

9. **Project Pathway:**

The project pathway consists of three steps. The first step is to develop a new powder synthesis process based on a chemical vapor synthesis method that holds promise to produce uniformly mixed nanocrystalline composite powders with less energy and more control. The second step of project which is carried out in parallel with the first step focuses on developing an ultrahigh pressure

sintering technology that can produce fully consolidated bulk nanocrystalline materials. During the last portion of the project, mechanical properties of the cermet material with true nanoscale grain sizes will be characterized. Engineering components will be manufactured and tested in real industrial applications.

10. **Critical Metrics:**

- Produce nano WC/Co composite powder with particle sizes below 50 nm via the CVS process,
- Produce fully consolidated WC-Co materials with grain sizes below 100 nm,
- Achieve 15% improvement with respect to hardness and toughness combination of cemented tungsten carbide.

PROJECT PLANS AND PROGRESS

11. **Past Accomplishments:**

1. Powder synthesis process - CVS

- Several different types of reactors have been designed, built, and tested to perform the Chemical Vapor Synthesis (CVS) of nanoscaled WC/Co composite powders. These different types of reactors include: a vertical high temperature tube reactor, a flame reactor, and a plasma reactor. The flame reactor method has been determined to be not suitable due to controllability issues.
- The vertical high temperature tube reactor system has been successful for the synthesis of nanoscaled WC/Co powders. Considerable studies have been completed on the dependence of the chemical and phase compositions of the powder on process variables including temperature, time, reactant gas ratios and reactor configuration.
- The vertical high temperature tube reactor has been chosen as the method for the production of powders for sintering experiments.

2. Powder synthesis – Plasma Reactor

- A plasma reactor has been designed and built. This reactor system is being tested and fine tuned for the synthesis of nano WC/Co powders. It is expected that by applying the knowledge learned from the vertical tube reactor and the advantages of the plasma process, increased production of the nanosized WC/Co composite powders will be possible.

3. Powder synthesis – High Energy Milling

- A high energy dual drive planetary mill has been utilized as an alternative for sintering and consolidation of nano WC/Co powders. Batches of 400 grams of 10 nm powders are produced routinely.

4. Ultrahigh Pressure Rapid Heating and Consolidation Process development

- An ultrahigh pressure (1 GPa) rapid heating and rapid hot consolidation press system has been designed, built, and successfully tested,
- Preliminary samples produced by using the UPRC system demonstrated very encouraging results. The grain sizes of the samples consolidated using the UPRC system are less than 100nm at greater than 99% density, meeting the goal of the project,
- Continued efforts are being made to fully characterize the preliminary samples.
- Process studies of the dependence of the densification and grain growth on process variables namely, temperature, pressure, and time are under way.

5. Comprehensive analytical evaluations and process optimization

- Comprehensive pressure-less sintering experiments were completed. Detailed knowledge on the mechanisms of grain growth has been established. The knowledge is expected to be very useful for process optimization,
- Process optimization will be carried out in parallel with the fabrication samples for the evaluation of mechanical properties.

6. Characterization and study of mechanical properties

This task has been initiated in the second quarter of 2006.

7. Component fabrication, functional testing, and field tests

This task is expected to start at the beginning of the fourth year.

12. **Future Plans:**

Task / Activities	**Responsible Partners**	Expected completion date
Development of CVS process in a tube reactor	University of Utah	Completed
Produce powders using vertical tube reactor CVS system	University of Utah	March 2008
Complete development of plasma synthesis process	University of Utah, Idaho National Lab	December 2006
UPRC process adjustments/fine tune for making specimens for evaluation of mechanical properties.	University of Utah, Idaho National Lab,	December 2006
Optimize UPRC process variables	U of Utah	December 2006
Consolidate nano WC/Co powder using the UPRC and produce specimens for property evaluations	University of Utah, Kennametal	December 2006
Demonstrate mechanical properties of WC-Co with true nanometer grain size	University of Utah, Kennametal, Smith International	March 2007

The overall project milestones and partner responsibilities are listed as follows.

Date	**Milestone/Deliverable**	**Partner Activities**
Completed	Successful development of nano WC/Co powder synthesis technique	University of Utah
Completed	Fabrication of a plasma unit for nano WC/Co powder synthesis	University of Utah, Idaho National Lab
March 08	Complete plasma synthesis process and production of larger amounts of nano WC/Co powder	University of Utah
September, 2006	Successful consolidation of nano WC-Co with<100 nm grain size at as-sintered state	University of Utah, Idaho National Lab, Kennametal,
December, 2006	Demonstrate of mechanical properties of WC-Co with nanometer grain size	University of Utah, Kennametal, Smith International
March, 2008	Proof-of-concept test of the material in industrial field applications	Smith International, University of Utah, Kennametal,

13. **Project Changes**

The technical scope of the project is expanded to include the sintering of functionally graded WC-Co materials using nanocrystalline WC-Co powders. This is a promising avenue by which the nanocrystalline cemented tungsten carbide can be commercialized.

14. **Commercialization Potential, Plans, and Activities:**

Efforts and activities toward the technology transfer are planned throughout duration of the project. This task will be accomplished by

- Communications with industrial partners through project reviews and technical collaborations,
- Disseminate research results through website as well as professional technical conferences and publication in technical journals,
- Proper management of intellectual properties.

15. **Patents, Publications, Presentations:**

Patent Application: A provisional patent application has been filed on our WC-Co nanocomposite synthesis. “Methods for Making Refractory Carbide Nanocomposite Powders”.

Publications:

1) X. Wang, Z. Zak Fang, and H.Y. Sohn, “Grain Growth during the Early Stage of Sintering of Nanocrystalline Cemented Tungsten Carbide”, to be submitted. J. of American Ceramic Society, 2006
2) Eso, Z. Zak Fang, and A. Griffo, “Kinetics of the Sintering of Functionally Graded WC-Co”, accepted, Int. J. of Refractory Metals and Hard Materials, 2006
3) Peng Fan, Oladapo O. Eso, Z. Z. Fang, and H. Y. Sohn, “Liquid Phase Migration during the Sintering of Functionally Graded WC-Co,” The Sohn International Symposium, TMS, 2006.
4) Manolete Mena, Taegong Ryu, Hong Yong Sohn, Gilsoo Han, Young-Ugk Kim and Zhigang Zak Fang, “Chemical Vapor Synthesis of WC-Co Nnanocomposite Powder,” The Sohn International Symposium, TMS, 2006.
5) M. Mena, Chemical Vapor Synthesis of WC-Co Nanoparticles, M.S. Thesis, University of Utah, 2006.

Presentations

6) X. Wang, Z. Zak Fang, and H. Y. Sohn, “Grain Growth during the Early Stage of Sintering of Nanocrystaline WC-Co Powders”, 2006 MPIF/APMI International Conference on Powder Metallurgy & Particulate Materials, June 18-21, San Diego, California
7) H. Wang and Z. Zak Fang, “A Review of Nano Sintering: Fundamentals and Technologies”, 2006 MPIF/APMI International Conference on Powder Metallurgy & Particulate Materials, June 18-21, San Diego, California,
8) Eso and Z. Zak Fang, “Kinetics of Sintering of Functionally Graded WC-Co”, 2006 MPIF/APMI International Conference on Powder Metallurgy & Particulate Materials, June 18-21, San Diego, California.
9) S. Johnson, J. Lu, Z. Zak Fang, R. Riley, and T. Webb, “Study of Nanosized WC-Co Powder during Heating Using Differential Scanning Calorimetry (DSC)”, ”, 2006 MPIF/APMI International Conference on Powder Metallurgy & Particulate Materials, June 18-21, San Diego, California.