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A Final Report
Grant No. DAAL03-91-G-0092

June 1, 1991 - August 15, 1992

**MECHANICAL ALLOYING PROCESSING AND APPLICATION TO
DEVELOPMENT OF STRUCTURAL MATERIALS**

Submitted to:

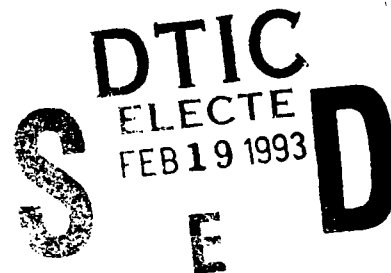
U. S. Army Research Office
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Attention:

Dr. Andrew Crowson
Materials Science Division

Submitted by:

Thomas H. Courtney
Professor



SEAS Report No. UVA/525272/MSE93/101
October 1992

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13. ABSTRACT (Maximum 200 words) This report summarizes results of several areas of research dealing with mechanical alloying of materials having potential structural use. The areas involve modeling of the process, synthesis of unique materials via mechanical alloying and consolidation of mechanically alloyed products. The latter area is one receiving most current attention as consolidation involves high temperatures and (usually) pressures. This can lead to a "loss" of the unique structures produced by mechanical alloying. Thus, consolidation studies attempt to produce fully dense material maintaining many of the desirable properties manifested by mechanically alloyed powders.				
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SYNOPSIS OF RESEARCH FINDINGS AND ACCOMPLISHMENTS FOR PERIOD JUNE 1, 1991-AUGUST 15, 1992.

This report summarizes research conducted under this program for the period specified above. We initiate our discussions by noting that our work under mechanical alloying (MA) has progressed through several stages. It initiated with modeling of the process (as exemplified in the publications associated with David Maurice and Beverly Aikin), proceeded with some synthesis studies (e.g., the publications of Zhongang Wang, Alex Aning and Tomasz Kosmac) and is now concentrating on consolidation studies (as an example, see the publication of Robert Vance listed below). There is a simple reason for this changed emphasis. In particular, the unique structures (e.g. amorphous materials, non-equilibrium structures, nanocrystalline structures) generated by MA in particulates must be subsequently consolidated to render them useful engineering solids. Consolidation requires application of pressure and temperature (usually simultaneously) and these "forces" tend to promote the transformation from non-equilibrium structures to the thermodynamically equilibrium ones, thereby negating (at least in part) the desirable properties associated with the non-equilibrium manifestations of MA processing.

Our modeling studies have progressed in two ways. One (Aikin's) is a phenomenological approach in which empirical rate constants for fracture and welding events in MA are deduced; these then have some limited use in extrapolation to other processing conditions (in particular, the effect of mill power and charge ratio can be "predicted"; see thesis of Aikin, a paper is currently being prepared on this effect). Maurice, who received partial support from ARO, attempted a "first principles" approach to modeling. Equations were developed for the deformation per collision event in MA and the fracture and welding probabilities of the process. From this particle hardness, size and shape can be followed during processing. The programs used to describe the process have been made available; ALCOA laboratories has made use of them already. A synopsis of the approach has been presented in the JOM article cited below; several articles are now in preparation. In addition to explaining previously observed phenomena, the effect of process variables such as grinding media velocity and properties, starting particle size etc. on times required for processing can be "predicted" by these models.

Much current work on MA is related to synthesis of "unique" microstructures; examples have been provided above. We have made some modest contribution to this area. Amorphous Ni-W alloys have been generated in our laboratory by MA. This system is not a prime candidate for amorphization based on thermodynamic and kinetic arguments, but we have shown that amorphization in it takes place when powders are milled for fairly long times in a high energy mill such as a SPEX mill. We have also shown that various sulfides, some having fairly high melting temperatures, can be synthesized by milling of the elemental powders. And in related work, sponsored by DARPA/NASA, we amorphized elemental sulfur and some forms of graphite by high energy milling of them as well as instigating a number of chemical reactions in ceramic solids.

We are now concentrating on consolidation studies and assessment of microstructure and properties of consolidated products as affected by processing variables. Some work has already been accomplished. Robert Vance's M.S. thesis showed that models of hot isostatic pressing could be applied to densification of mechanically alloyed Cu-Nb powders, provided the appropriate properties of these materials were used in the programs now used for mimicking densification. Mr. Zhongang Wang conducted some preliminary densification studies of amorphous Ni-W alloys. He found that the amorphous phase could not be retained during hot isostatic pressing at temperatures for which full densification could be approached. Nonetheless, the resulting microstructure was much finer than the heavy metal alloys prepared conventionally; i.e. by liquid phase sintering. Mr. Charles Mukira is pursuing his Ph.D. thesis along these same lines, starting with a Ni₅₀-W₅₀ alloy. He is to determine microstructure-density relationships as they are affected by temperature and pressure during hot isostatic pressing. He will then assess pertinent properties (e.g. tensile behavior and

fracture toughness) of the most promising structures. Mr. Robert Comstock is investigating the thermal stability of mechanically alloyed Cu-Nb mixtures. He finds that structures milled for lesser times maintain their fine structure for longer times of exposure than do powders milled for longer times. Most interesting, though, is that during the thermal treatments, conducted in hydrogen, a Nb layer forms on the surface of the particles. This layer surrounds the fine Cu-Nb mixture. The effect is caused by the lower hydrogen-niobium surface energy in comparison to that of the corresponding hydrogen-copper one. We expect similar behavior would be observed in other Cu-transition metal alloys (e.g. Cu-W, Cu-Cr etc.). These are intriguing structures for potential high temperature use. In particular, their consolidation would result in a continuous Nb cage surrounding tough and ductile composite Cu-Nb particles. The Nb cage could provide high temperature strength; the particles, good fracture toughness. Mr. Brian Murphy's Ph.D. thesis deals with the issue. He is preparing and consolidating structures like this. Assessment of creep properties and fracture toughness will be made.

The appendices below list degrees granted and papers published under the grant during the stipulated period. Note that the papers involving David Maurice list joint sponsorship. Mr. Maurice was supported for two years of his Ph.D. work by DARPA/NASA; the remainder of his study was supported by ARO.

APPENDIX A: DEGREES AWARDED,

JUNE 1, 1991-AUGUST 15, 1992

Robert. R. Vance, M.S., June, 1991
 Zhongang Wang, M. S., February, 1992
 Beverly J. M. Aikin, December, 1991
 David Maurice, May, 1992*

*- Partial support from DARPA/NASA

APPENDIX B: MANUSCRIPTS SUBMITTED OR PUBLISHED, JUNE 1, 1991-AUGUST 15, 1992

A. O. Aning, Z. Wang and T. H. Courtney, "Tungsten Solution Kinetics and Amorphization of Nickel in Mechanically Alloyed Ni-W Alloys", accepted for publication in *Acta Metall. et Mater.**

R. R. Vance and T. H. Courtney, "Hot Isostatic Pressing of Mechanically Alloyed Cu-Nb Powders", *Scripta Metal. et Mater.*, 26, 1435, (1992).

T. H. Courtney and Z. Wang, "Grinding Media Wear During Mechanical Alloying of Ni-W Alloys in a SPEX Mill", *Scripta Metal. et Mater.*, 27, 777, (1992).

T. Kosmac, D. Maurice and T. H. Courtney, "Synthesis of Nickel Sulfides by Mechanical Alloying", submitted for publication*

R. W. Rydin, D. Maurice and T. H. Courtney, "Milling Dynamics: I: Attritor Dynamics: Results of a Cinematographic Study", accepted for publication in *Metall. Trans. A*.*

T. H. Courtney, "Elevated Temperature Morphological Instabilities in Metal Matrix Composites", Metal Matrix Composites-Processing, Microstructure and Properties, Proc. 12th Riso International Symposium on Materials Science, N. Hansen, D. Juul Jensen, T. Leffers, H. Lilholt, T. Lorentzen, A. S. Pedersen, O. B. Pedersen and B. Ralph, Eds., Riso National Laboratory, Roskilde, Denmark, 17, (1991). (Invited paper)

D. Maurice and T. H. Courtney, "Modeling of Mechanical Alloying", JOM, 44, No. 8, 10, (1992)*
(Invited paper)

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