Nanocrystalline Materials by Machining

Kevin Trumble
Srinivasan Chandrasekar
W. Dale Compton

Purdue University
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Project ID
PM_10460

Nanocrystalline Materials by Machining

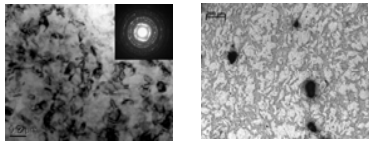
21CTP Technical Goal: **Develop and demonstrate an emissions compliant engine system for Class 7-8 highway trucks that improves the engine system efficiency from ~42% today to 50% by 2010.**

Project Objectives
Develop bulk alloys with nanocrystalline/ultra-fine grained microstructure and enhanced mechanical properties by chip formation

FY 2005 Focus
- TEM studies of nanostructure development in machining
- Low-temperature consolidation routes for Al 6061-T6

Planned Duration
October 2004 to April 2007

DOE Funding/Industry Cost Share
FY04: $75K; FY05: $110K



Left: Bright-field TEM showing sub-100 nm grains in Al 6061-T6 chip. **Right:** Optical micrograph showing 1:1 composite of 6061-T6 chip particulate and pure Al produced by powder extrusion at room temperature (~99% dense).

Principal Investigator(s)
Srinivasan Chandrasekar, Purdue University
(765) 494-3623; chandy@purdue.edu

Kevin Trumble, Purdue University
(765) 494-4114; driscol@ecn.purdue.edu

W. Dale Compton, Purdue University
(765) 494-0828; dcomptonl@ecn.purdue.edu

Technology Development Manager
Sid Diamond, DOE/OFCVT
(202) 586-8032; sid.diamond@ee.doe.gov

Accomplishments
- Several nanocrystalline alloys (e.g. Al alloys, superalloys, titanium, steels and copper alloys) created by machining
- Initial low-temperature extrusion and pressing methods for consolidation of these alloys into monolithic/composite materials showing promise

Significant Future Milestones
Creation of fully dense and bonded bulk alloy prototypes -July 2006

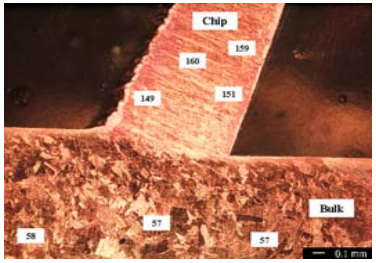
Project ID/Agreement ID	Program Structure	Sub-Program Element	R&D Phase	Date
PM_10460	Materials Technology	HV Propulsion Materials	Exploratory R&D	8-05

Objectives

- Develop bulk alloys with nanocrystalline/ultra-fine grained microstructure and enhanced mechanical properties by chip formation
  - High-strength and/or light-weight alloys
  - Fully densified and well-bonded bulk forms with ultra-fine grained microstructure
- FY2005 Progress
  - TEM studies of nanostructure development in machining chips
  - Low-temperature consolidation routes for making Al 6061-T6 bulk forms

Approach

- Nanocrystalline materials by machining
  - Chip formation
  - Production of nanocrystalline particulate
  - Production of nanocrystalline bulk forms
- Production of Bulk Forms
  - Powder processing
  - Infiltration processing
  - Direct production of bulk forms
- Characterization
  - Microstructure
  - Mechanical properties



High strain (1-10) introduced in one machining pass drives the microstructure refinement

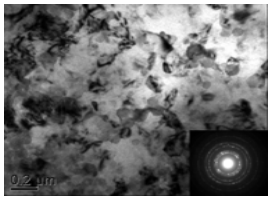
Relevance to 21 CT Goals

- Design and demonstrate emissions compliant engine
  - Class 7-8 highway trucks efficiency from ~42% to 50% by 2010
- Project success
  - High-performance structural materials and/or wear-resistant coatings
  - Weight reduction
  - Low-cost processes for nanomaterials
- DOE providing facilities and technical capabilities in addition to funding
- Other technology benefits beyond 21 CT
  - Particulate production and processes
  - Tools/Dies
  - Aerospace components
  - Agriculture/Construction components
  - Metal- and polymer-matrix composites
  - Concrete reinforcements

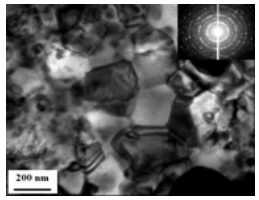
Technical Accomplishments/ Progress/Results

- Nanostructured chips from a variety of alloys and metals
  - Al alloys, titanium, superalloys, steels, and copper alloys
  - Verified chip grain sizes as 50-700nm with TEM
  - Nanostructured chips 2-3 times as hard as bulk material
  - Direct production of nanostructured particulate
  - Thermal stability of nanostructured chips for process windows
- Bulk forms with ultra-fine grained microstructure
  - Low temperature powder extrusion
  - Infiltration processing
  - Direct production of nanocrystalline bulk samples
- Bulk forms key milestone for overall approach
  - Characterize mechanical properties and performance

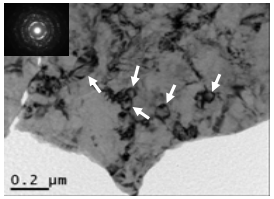
Nanocrystalline Alloy Microstructures



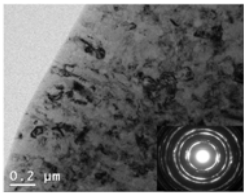
Al6061-T6, grain size: 75 nm



52100 steel, grain size: 330 nm



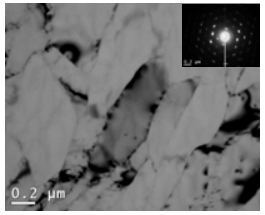
Ti, grain size: 50 nm



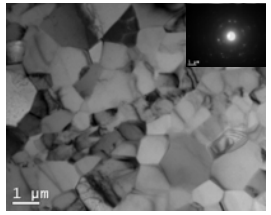
Inconel-718, grain size: 100 nm

Strain Effects on Microstructure

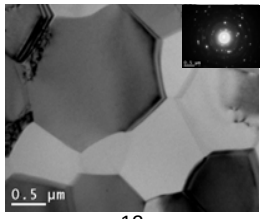
Al 1100



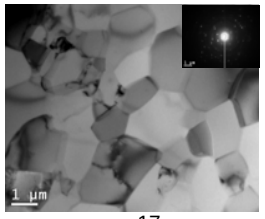
γ=6



γ=11

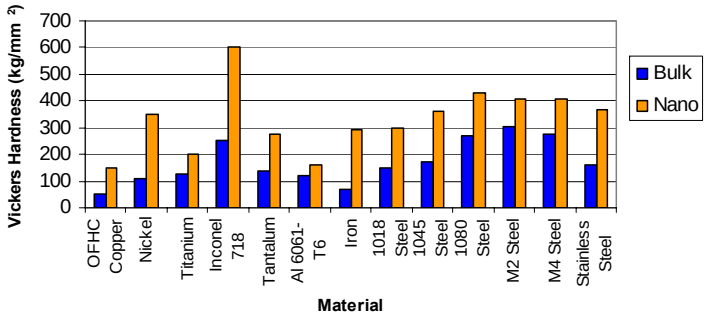


γ=12



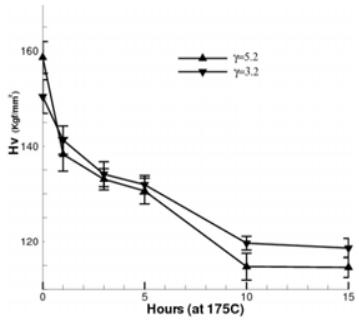
γ=17

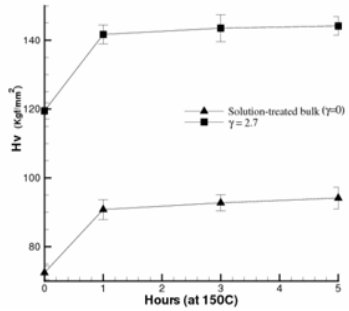
Mechanical Properties



Nanostructured chips are typically 2-3 times as hard as bulk material

Annealing of Chips



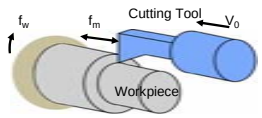


Peak-aged 6061-T6

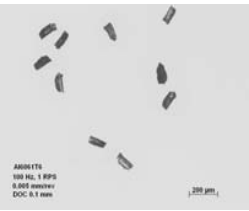
Solution-treated 6061

Thermal stability suggests a new route => Solution-treat, machine, age

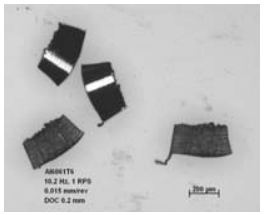
Production of Nanocrystalline Particulate



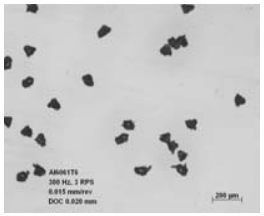
- Modulation-assisted machining
  - Size control (~10µm – 2mm)
  - Shape control (platelet, ribbon, equi-axed, needle)



Platelet ~40x100x50 µm



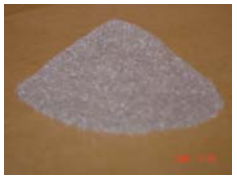
Ribbon ~500x200x20 µm



Equi-axed ~25x25x25 µm

Particulate Consolidation

Nanocrystalline particulate

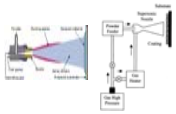


Compaction

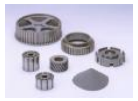
Extrusion

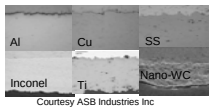
Injection molding

Powder spraying



Monolithic bulk forms
Composite bulk forms
Free forms
Films and coatings

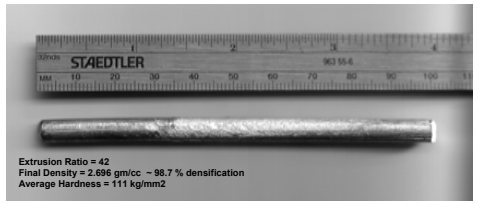


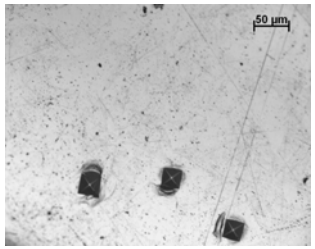


Courtesy ASB Industries Inc

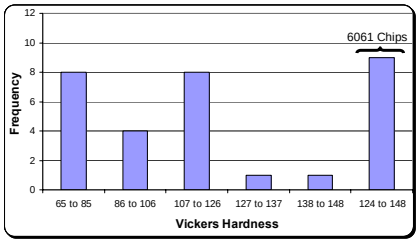
Powder Extrusion

- Gas atomized Al blended with Al 6061-T6 nanostructured particulate



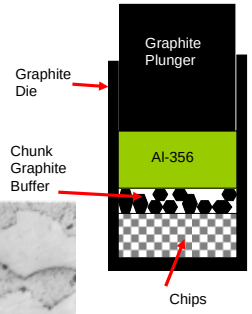


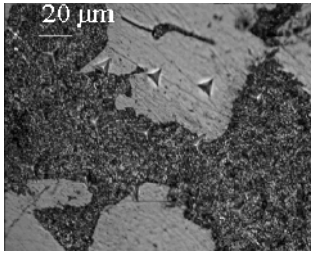
Microstructure



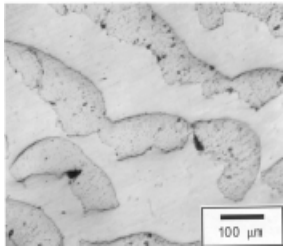
MMCs by Infiltration Processing

- Metal Matrix Composites (MMCs)
  - –Nanostructured steel chips
  - –Al or Bronze matrix





M2 Steel Chips in A356 Matrix
1atm LPI at 750°C/7 min



M2 Steel Chips in Bronze Matrix
Spontaneous Infiltration at ~1100°C

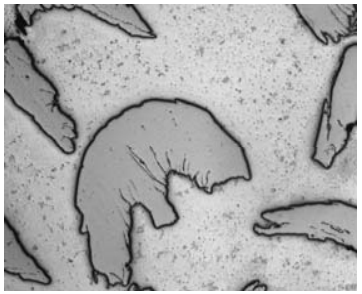
MMCs by Infiltration Processing

- **Kirksite: zinc- based alloy for stamping die applications**

  Composite of 1080 nanocrystalline steel chips in Kirksite matrix

  –60% increase in hardness compared to bulk Kirksite

  –30% lower wear rate compared to bulk Kirksite

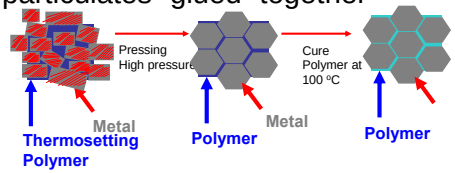


Bulk Kirksite: 104 ± 6 Kg/mm^2
1080 Steel chips: 340 ± 15 Kg/mm^2

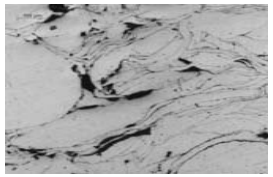
Composite hardness: 168 ± 93 Kg/mm^2

Epoxy-Bonded Chip Particulate

- Die press
  - –High metal fraction composite
  - –Chip particulates "glued" together



Al 6061-T6/M bond Epoxy 500MPa



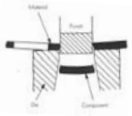
Al 6061-T6/M bond Epoxy 1000MPa

Bulk Forms by Extrusion Cutting

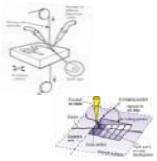
Extrusion cut bulk form

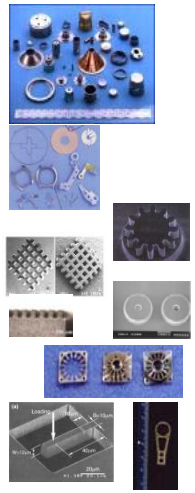
Roll straightening
Roll forming
Thermal processing

Stamping



Energy beam processing





Technology Transfer

- Tech transfer activities/path
  - Consolidation routes and properties/performance database for the bulk ultra-fine grained alloys through collaborations
  - Interaction (e.g., licensing) with primary materials suppliers (e.g., NanoDynamics) and end users (e.g., Ford)
- Collaborators
  - Extrusion and Vibratory compaction (ORNL)
  - Cold spray and Dynamic compaction (Sandia)
  - Wire encapsulation/thermal spray (Ford)
  - Powder compaction/sintering (NanoDynamics)
  - Corrosion (Boeing Phantom Works)
- Component design and manufacturing
  - Reduced weight and/or increased operating life with minimal re-design
  - Enhanced performance via direct material substitution (no re-design)
- Methodologies for bulk form prototypes Fall 2006 - Fall 2007

Future Work

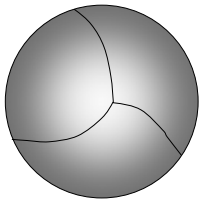
- FY2006 Plan
  - Creation of bulk form prototypes with nanocrystalline microstructure (July 2006)
  - Explore various low-temperature consolidation routes through collaborations
  - Characterize stability of the ultra-fine grained microstructure
- Project overall objective
  - Creation of fully dense and bonded bulk alloy prototypes

Supplemental Slides

Nanocrystalline Particulate

Conventional particle

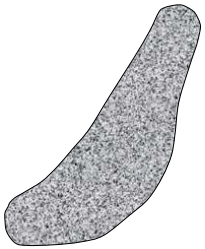
Grain size ≈ Particle size



50 μm

Chip particle

Grain size << Particle size



Much higher strength

Consolidation Challenges

- Maintain nanostructure in thermal processing
  - Low processing temperature
  - Short times at temperature
- Coarsening resistance of nanostructures

Grand Challenge:
Bulk alloys with nanocrystalline microstructure that are fully densified and well-bonded

Publications, Presentations, Patents

1. S. Swaminathan, T.L. Brown , M. R. Shankar, B. C. Rao, K.P. Trumble, W. D. Compton, S. Chandrasekar, Large Scale Manufacturing of Ultra-fine Grained Materials, Symposium Proceedings , *Ultrafine Grained Materials, TMS (The Minerals, Metals and Materials Society)* 2004, pp. 161-166.
2. S. Chandrasekar, W.D. Compton, T.N. Farris, and K.P. Trumble, Method of Forming Nanocrystalline Structures and Product Formed Thereof, U.S. Patent 6,706324 B2, Mar 16, 2004.
3. T.L. Brown, S. Swaminathan, B.C. Rao, R.F. Kezar, S. Chandrasekar, W.D. Compton, K.P. Trumble and A.H. King, Machining As A Method for Studying Effects of Very Large Strain Deformation, *Ultrafine Grained Materials, TMS (The Minerals, Metals and Materials Society)* 2004, pp. 167-172.
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6. M. Ravi Shankar, S. Chandrasekar, W.D. Compton and A.H. King, Characteristics of Aluminum 6061-T6 Deformed to Large Strains by Machining, *Materials Science and Engineering A*, in press.
7. M. Sevier, S. Lee, M. Ravi Shankar, H.T.Y. Yang, S. Chandrasekar and W.D. Compton, Deformation Mechanics Associated with Formation of Ultra-Fine Grained Chips in Machining, *Materials Science Forum*, in press.
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9. J. B. Mann, S. Chandrasekar, and W.D. Compton, Tool Holder Mechanism for Applying Modulation in Machining, US Patent Pending Ser. No. 60/677,43, May 3, 2005.
10. Y. Uluca, B.C. Rao, M. Ravi Shankar, T.L. Brown, J.B. Mann, S. Chandrasekar and W.D. Compton, Low-Cost Ultra-Fine Grained Materials from Aerospace Machining Chips, Proceedings *SAE Aerotech Congress & Exhibition*, *Dallas, TX,* in press.

Related Grants

- Ford Motor Company University Relations program
- State of Indiana 21st Century Research and Technology fund
- USAF Phase 2 SBIR sub-contract from Nanodynamics