Wear Resistant $AlMgB_{14}$ Composites

Crosscutting Applications of a New Class of Ultra-Hard Materials
#1789

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EERE ITP Program Review
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AMES LABORATORY

IOWA STATE UNIVERSITY

Organization

- Project Summary
- Brief Background
  - (the importance of microstructure)
- Erosion & Abrasion testing
  - (recent milestones)
- Modeling
- Commercialization
- Future Plans

Crosscutting Applications for a New Class of Ultra-Hard Borides (#1789)

Goal: increase U.S. industrial energy efficiency through development of advanced, wear-resistant materials

Challenge: monolithic ceramics wear by brittle fracture; metals wear by plastic deformation. Neither possesses extreme resistance to tribological wear across a broad range of conditions; wear resistance has been limited by available materials and microstructures.

Benefits: extended lifetime of critical pump, valve, and material transport components; increased efficiency in mining and metal removal operations. energy savings of 5.6 trillion Btu/yr by 2020; 26 trillion Btu/yr by 2030.

Potential End-User Applications: abrasive water-jet cutting nozzles, material conveying system liners (particularly bends and elbows), valves, seals, and bearings; cutting and milling inserts, extrusion and drawing dies; low-friction coatings for MEMS and other rotating/sliding components

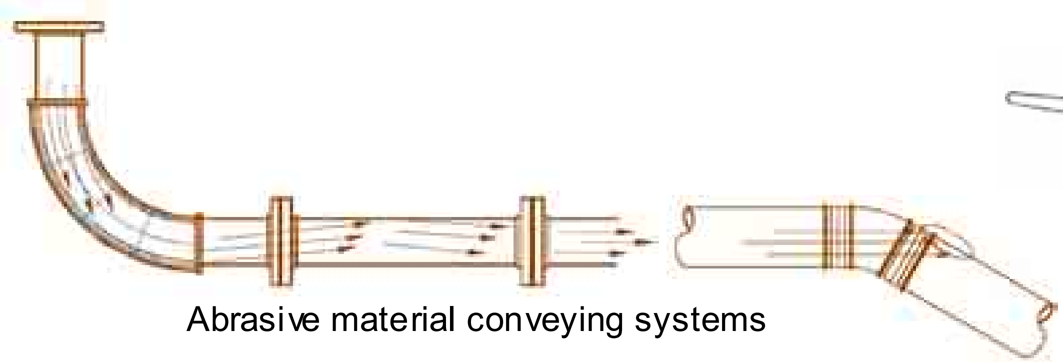
FY06 Activities: optimize processing technology and process scale-up, continue microstructural refinement, advance theoretical modeling efforts to enable accurate prediction of best composition and microstructure for each application, establish industry-wide consensus on performance of superior wear resistant compositions; re-tool commercialization infrastructure

Participants: (primary) Kennametal Advanced Solutions Group, IMI Vision, Inc., University of Missouri-Rolla High Pressure Waterjet Laboratory

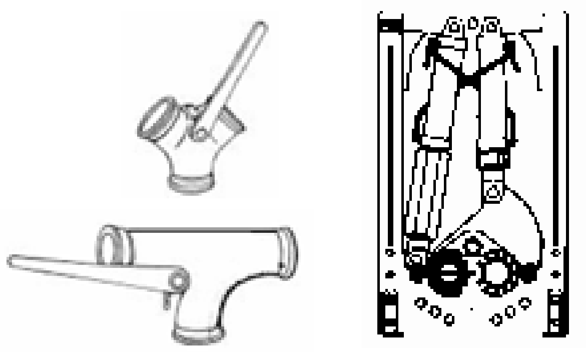
(secondary) Praxair Surface Technology, Inc, Norton St. Gobain, Ceracon, CeramTec, Concurrent Technologies, Inc. Harris Corp., Sunnen Corp., Engis Corp.

Crosscutting Applications for a New Class of Ultra-Hard Borides (#1789)

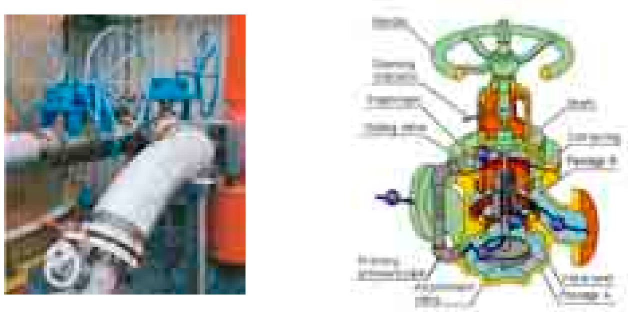
Erosion-resistance Applications:



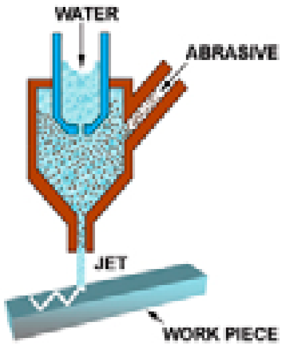
Abrasive material conveying systems



Diversion valve systems



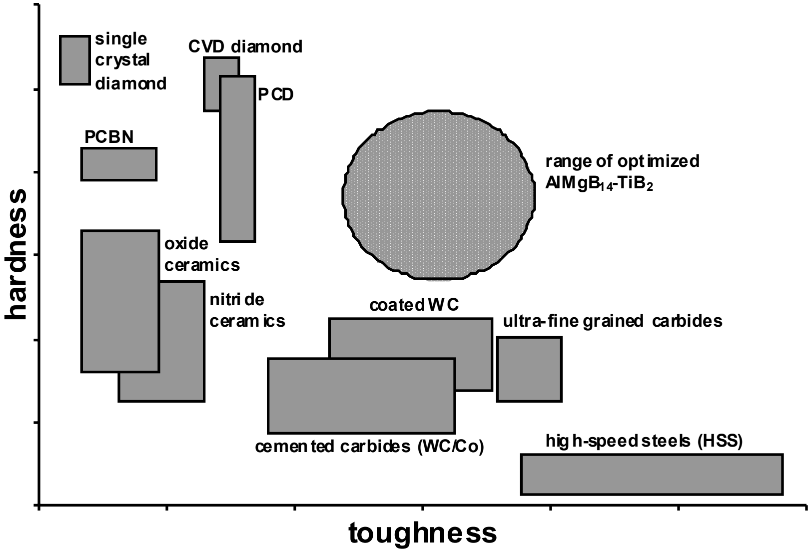
Pump components (shaft seals, impellers, linings)



Abrasive water jet nozzles

Background:

- **Exploratory work began in 2001 on high-hardness borides**
  - Discovered relationship between unit cell occupancy and Fermi energy in single phase $AlMgB_{14}$
- **Observed microhardness values of 35 GPa in $AlMgB_{14}$+Si polycrystalline samples**
  - Higher hardness in $AlMgB_{14}+TiB_2$ composites (45+ GPa)
- **Examined role of dopants vs. second phase additives**
- **Performed preliminary lathe cutting tests using hot pressed inserts**
- **Wear resistance unknown until 2004**



N.B., hardness alone does not guarantee good wear resistance…

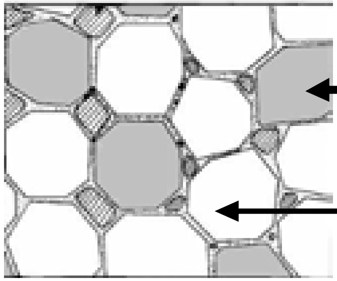
"BAM" ≠ $AlMgB_{14}$

- Maximum hardness of (single phase) $AlMgB_{14}$ ~ 30-35 GPa

Higher hardness requires ***microstructural engineering***:

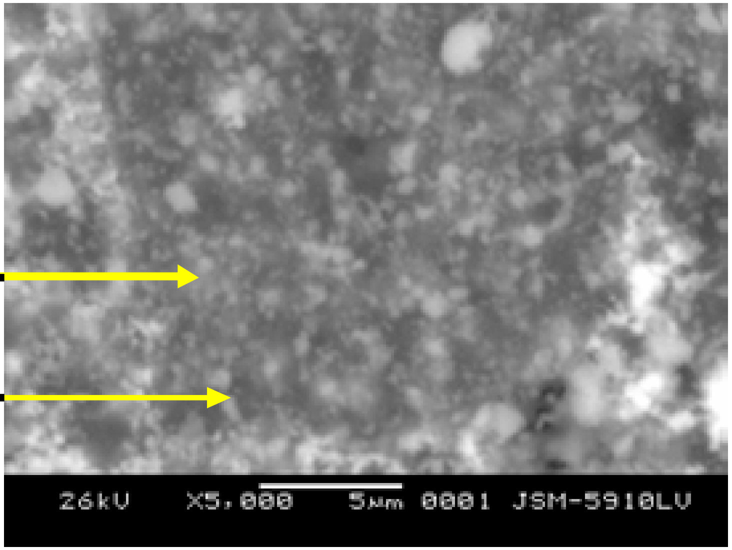
Composition	Microhardness, $HV_{0.1}$, GPa	Wear Coefficient ($m^2/N \times 10^{-15}$)
$AlMgB_{14}$	32	1.6
Al_2O_3-TiC	22	2.9
Al_2O_3-SiC_w-TiC	19	2.7
WC/Co	23	1.8

Bhat, Deepak G.; Bedekar, Vikram A.; Batzer, Stephen A, "A Preliminary Study of Chemical Solubility of Ultra-Hard Ceramic AlMgB14 in Titanium: Reconciliation of Model with Experiment," Machining Science and Technology 8(3) (2004), 341-355.



TiB_2 -rich region

$AlMgB_{14}$ -rich region



Wear resistance:

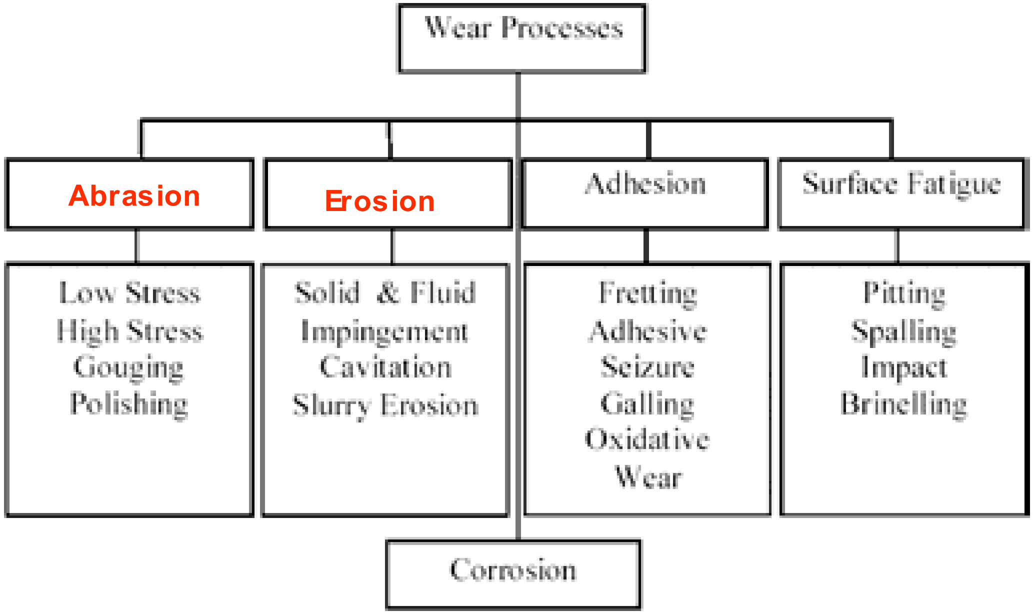
Objective: Combat energy losses due to material degradation by development of new, wear-resistant materials

“wear” encompasses a number of distinct physical mechanisms

- Abrasive and erosive wear are functions of hardness **AND** fracture toughness:

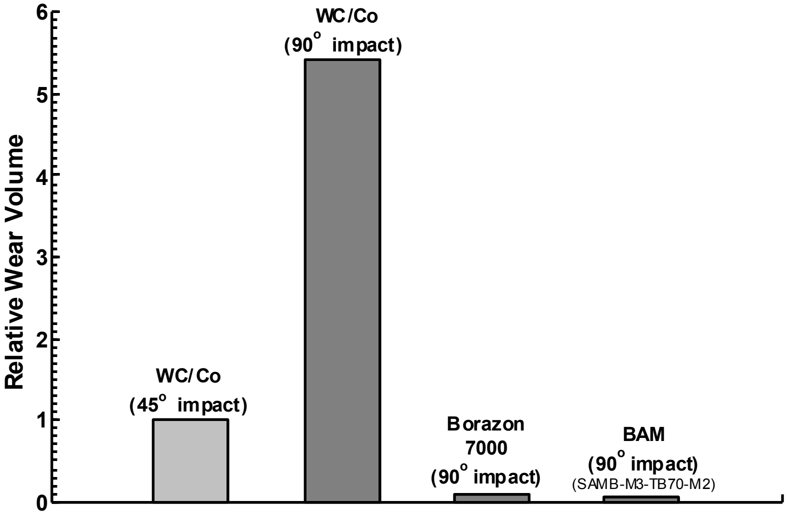
$$V = cK_{1C}^{m}H_{v}^{n}$$

V = volume removed/impact
K_{1c} = fracture toughness
H_v = hardness
m ≅ - 1.3
n ≅ - 0.5
C = proportionality constant

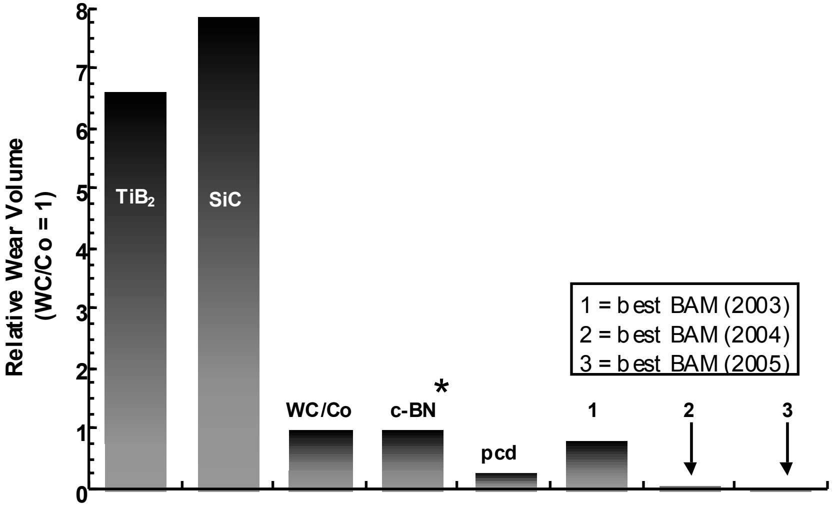


Erosion resistance: relative wear volume

60 sec erosion with 100 m/s Al_2O_3



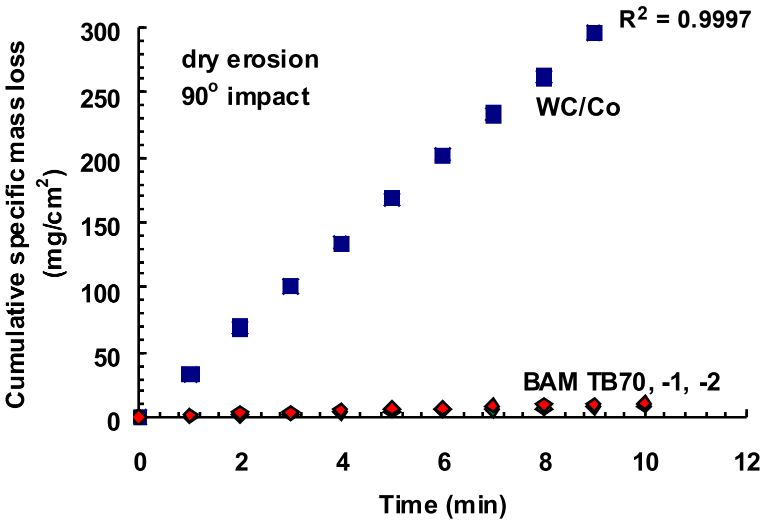
Effect of impingement angle (WC/Co)

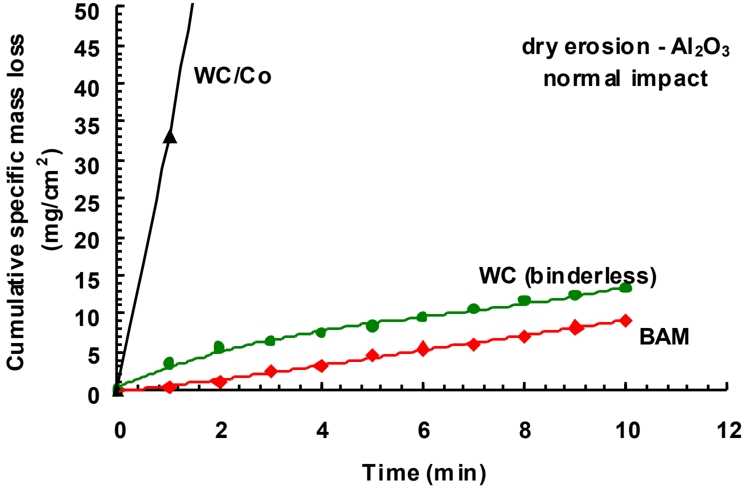


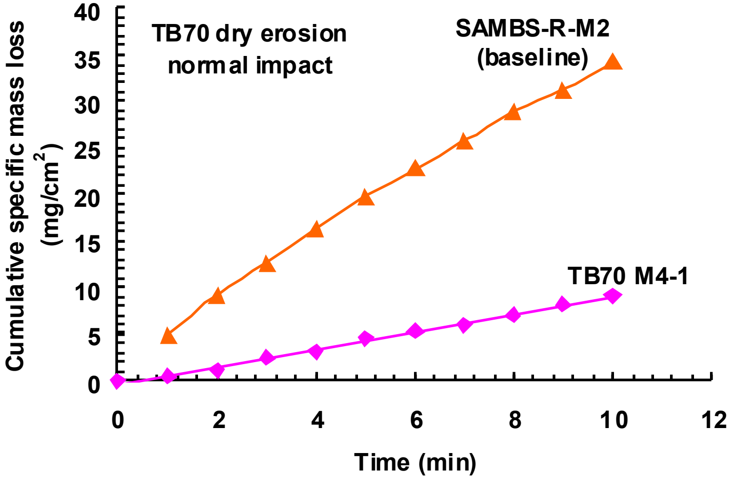
Comparison with other materials

* "standard grade" CBN

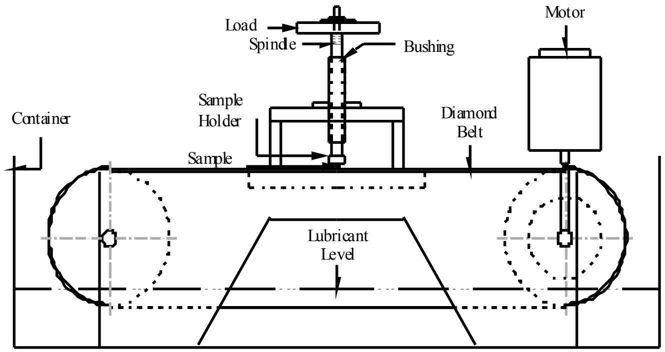
Steady state erosion:

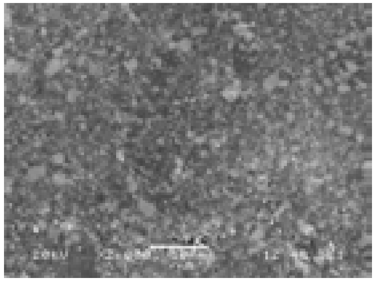




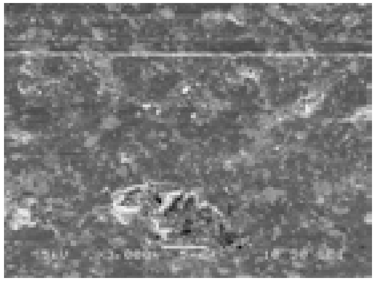


Diamond belt abrasion test:



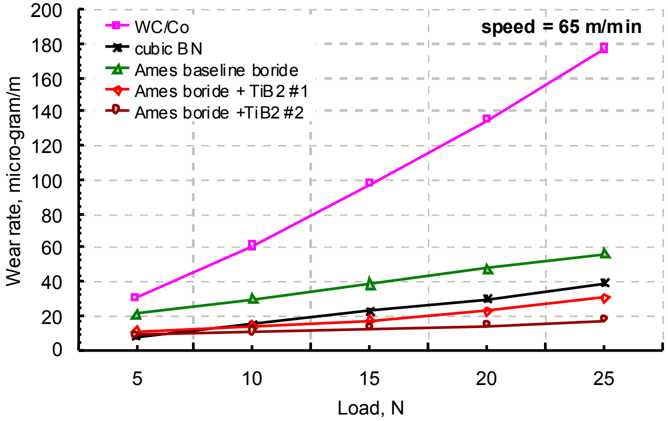


5 N load + 0.42 m/s belt speed

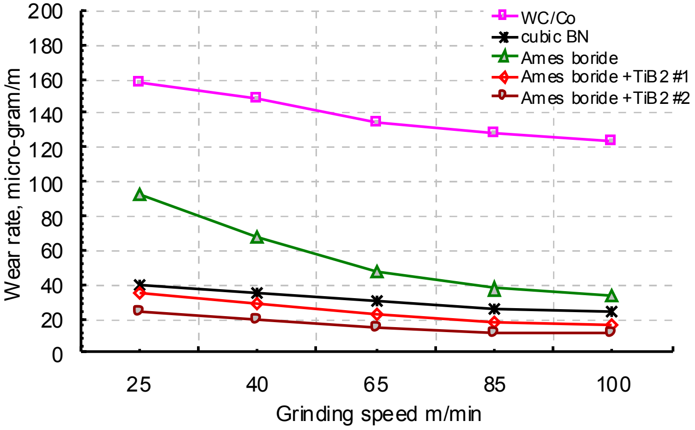


20 N load + 1.67 m/s belt speed

Variation in abrasive wear rate with applied loading at a constant speed of 65 m/min

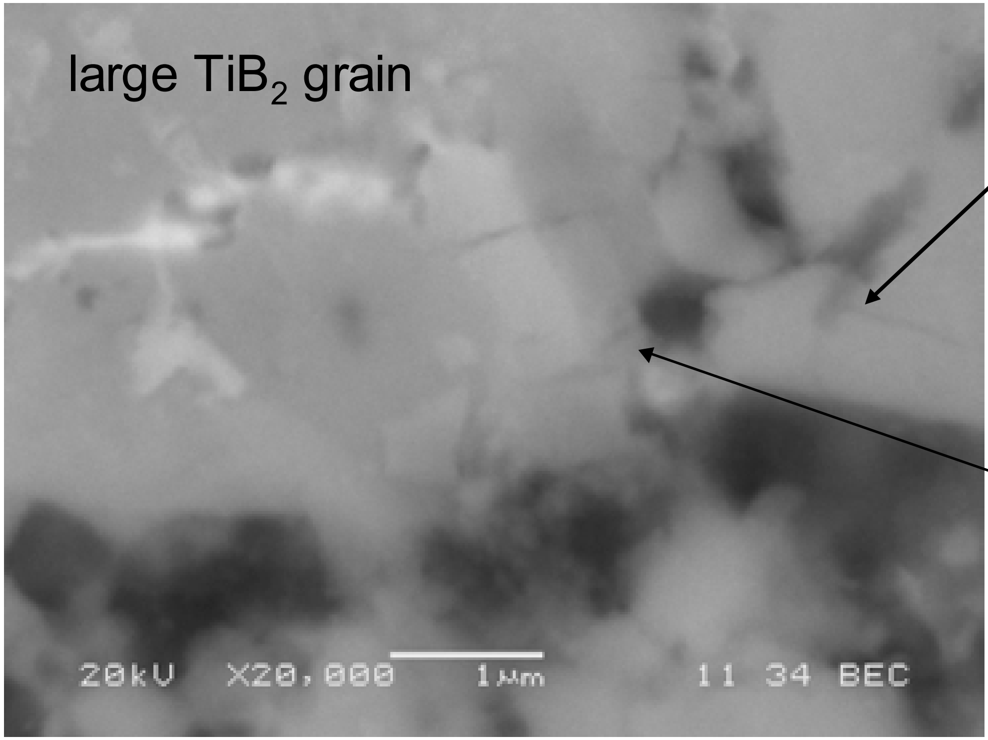


Variation in wear rate with speed at a constant 20N loading



Proposed damage mechanism:

TB70-M3 5 seconds at 45° impact angle



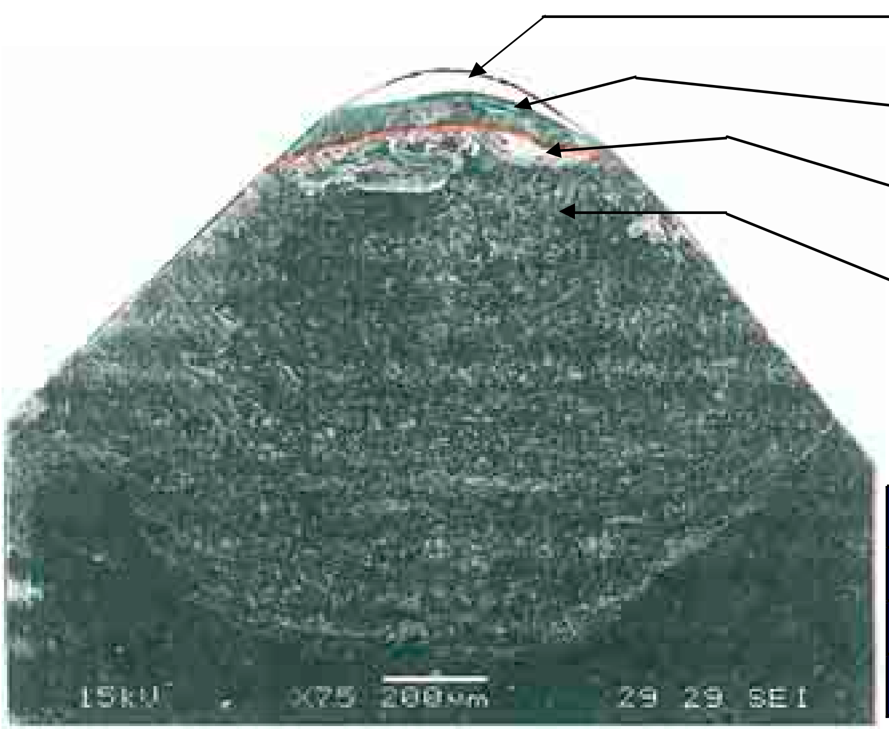
Transgranular cracks in TiB_2

Crack arrest at TiB_2 - $AlMgB_{14}$ interface

Lathe cutting tests:

Workpiece material: Ti-6Al-4V

Comparison of nose wear in different tool materials after machining tests



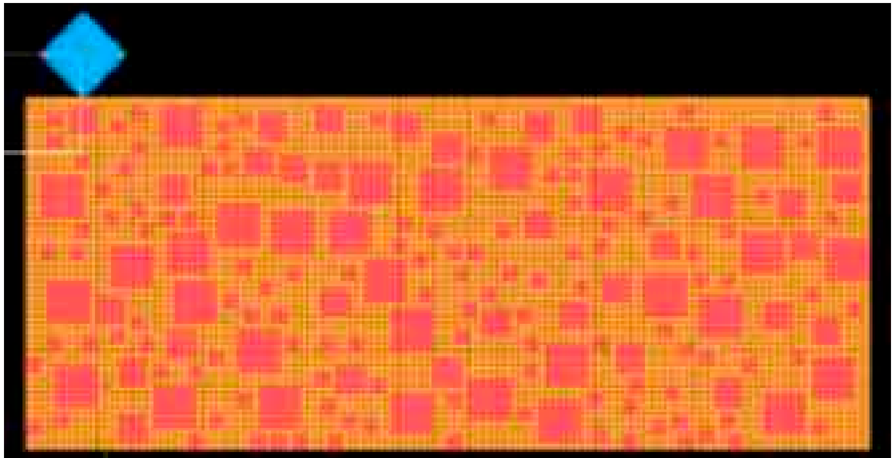
Material	Volume loss, mm^3
WC-Co	6.85
β–BN	8.95
$AlMgB_{14}$ – 70 wt. % TiB_2	1.44

Theoretical modeling:

Wear of composites was simulated by a "micro-scale dynamic model:"

Objective: to understand the relationship between microstructure and wear:

- Volume fraction of reinforcement
- Size and size distribution of reinforcement
- Abrasive grit size



rectangular or square reinforcing particles are randomly distributed throughout the matrix

"*micro-scale dynamic model:*"

- Discretize composite and erodent
- Define small volume of material
- Connect volume elements by springs
  (incorporate Young's modulus, yield strength, tensile strength; estimate interfacial bonding strength)
- Calculate force coefficients for each volume element
  (Newton's 2nd law)
- Calculate total force on each site
- Calculate new velocity of each site
- Calculate deformation of bonds
- If deformation > critical value (i.e., fracture strain of material), then bond is broken and fracture is nucleated

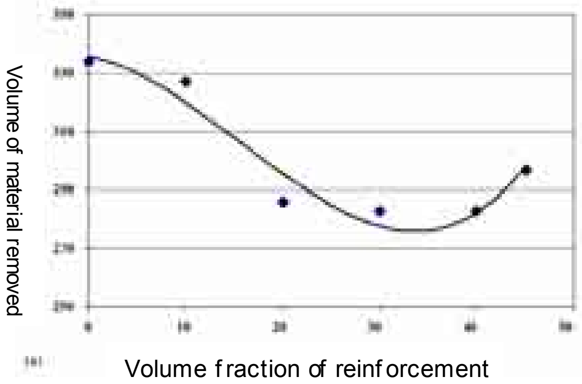
J. Hu, D.Y. Li, and R. Llewellyn
"Computational investigation of microstructural effects on abrasive wear of composite materials
Wear (2005) (in press)

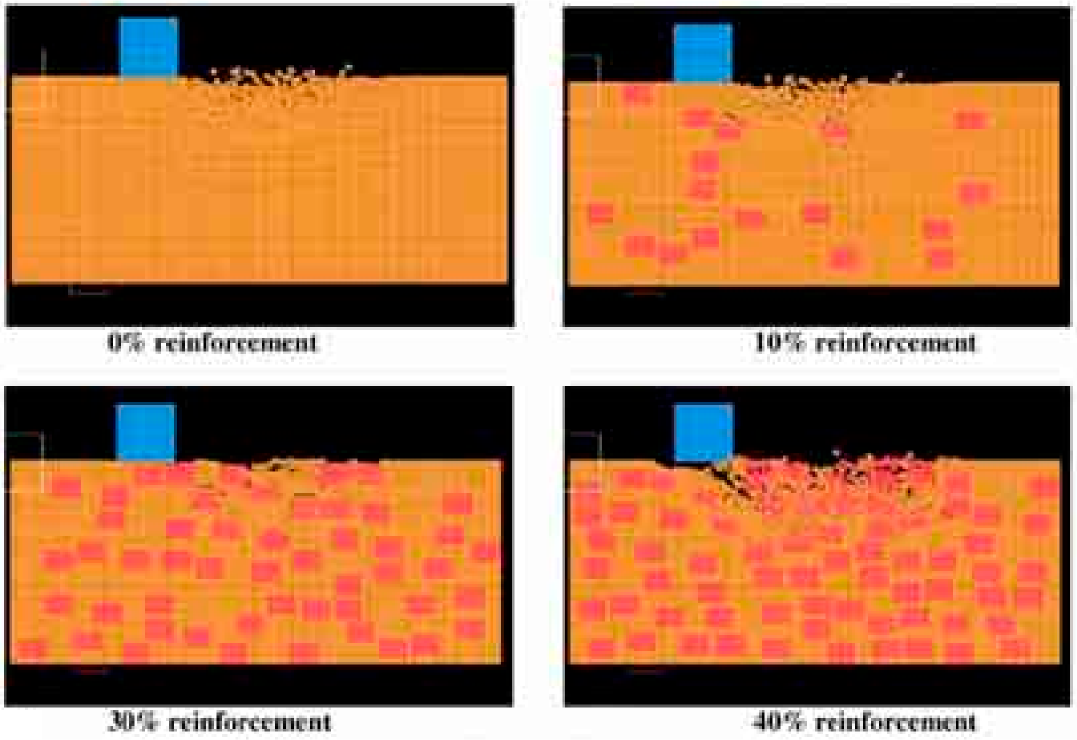
Predictions of model:

Effect of reinforcement volume fraction

Model predicts existence of a critical volume fraction of reinforcement

- varies with material
- increases with increased bond strength between reinforcement and matrix

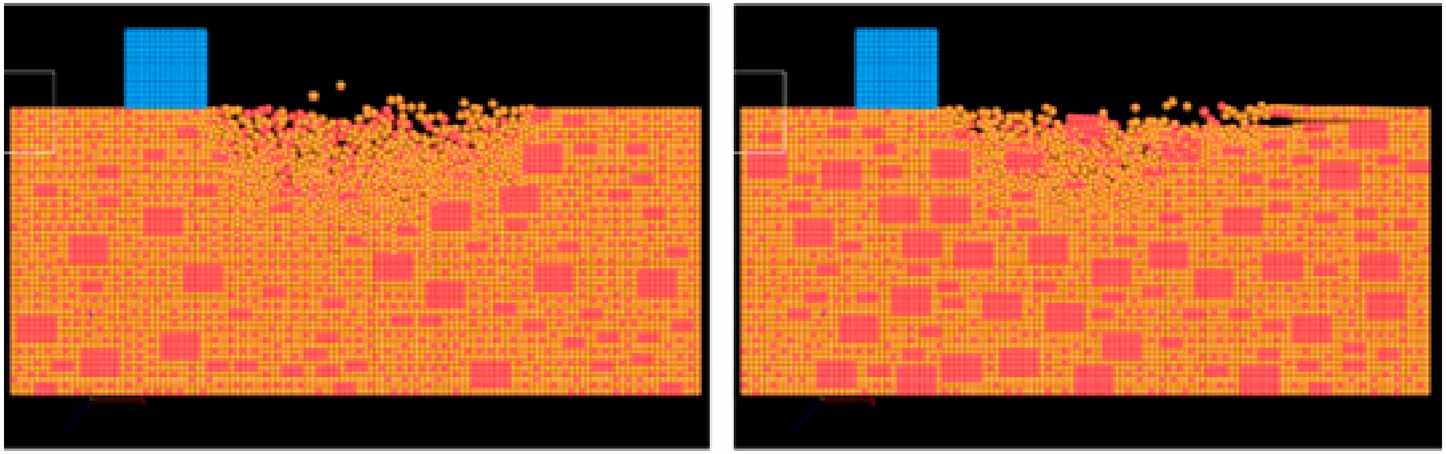



0% reinforcement 10% reinforcement

30% reinforcement 40% reinforcement

Predictions of model:

Effect of reinforcement distribution



8% large+8% middle+19% small

19% large+8% middle+8% small

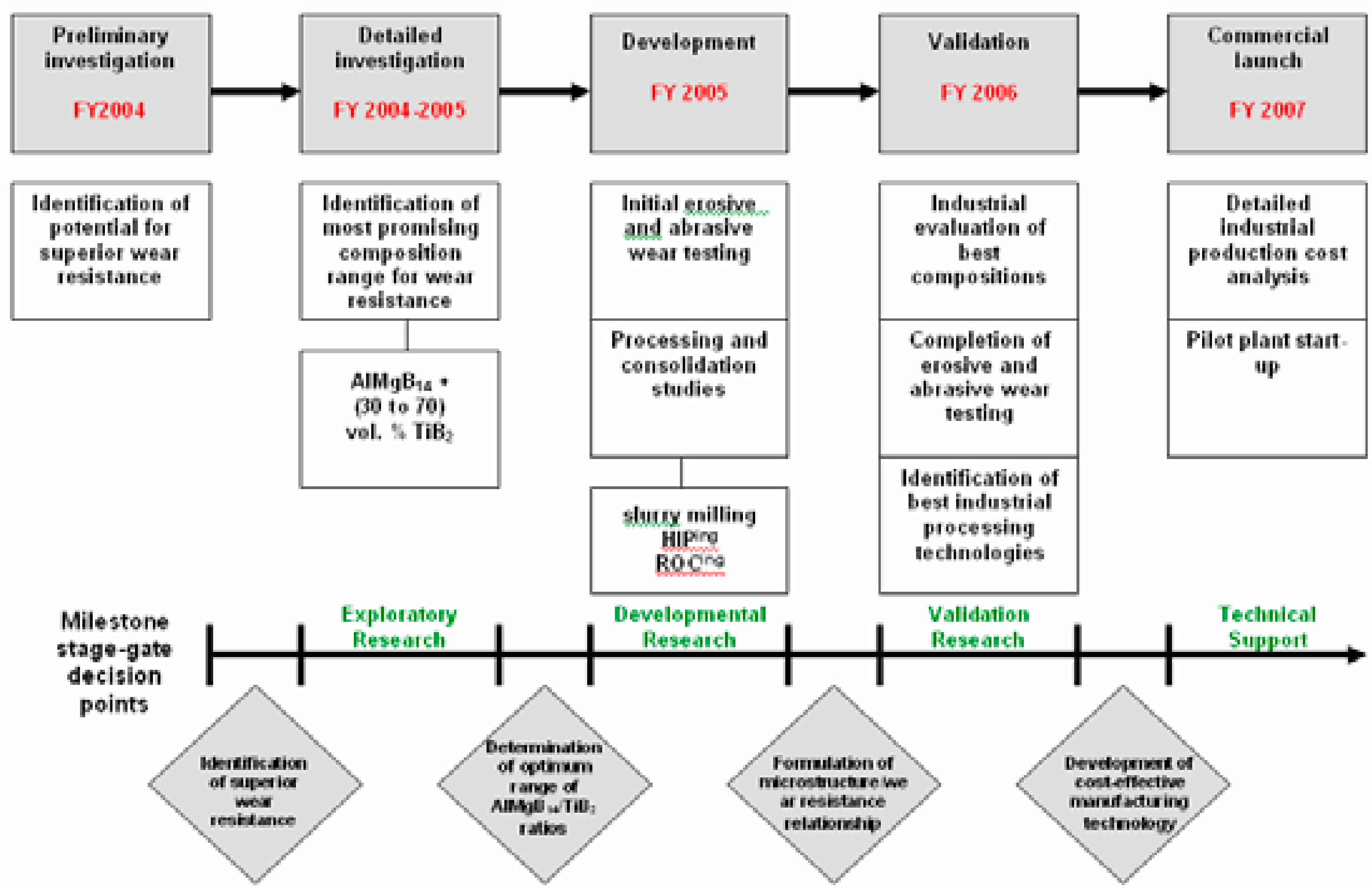
Volume fraction held constant at 35%

Model predicts

- Distribution of sizes leads to higher wear resistance than monosized reinforcement

Ultra-Hard Boride Technology Maturation via Stage-Gate

Project milestones and stage-gates



Preliminary investigation
FY2004

Identification of potential for superior wear resistance

Detailed investigation
FY 2004-2005

Identification of most promising composition range for wear resistance

$AlMgB_{14}$ + (30 to 70) vol. % TiB_2

Development
FY 2005

Initial erosive and abrasive wear testing

Processing and consolidation studies

slurry milling
HIPing
ROCing

Validation
FY 2006

Industrial evaluation of best compositions

Completion of erosive and abrasive wear testing

Identification of best industrial processing technologies

Commercial launch
FY 2007

Detailed industrial production cost analysis

Pilot plant start-up

Milestone stage-gate decision points

Exploratory Research

Developmental Research

Validation Research

Technical Support

Identification of superior wear resistance

Determination of optimum range of $AlMgB_{14}/TiB_2$ ratios

Formulation of microstructure/wear resistance relationship

Development of cost-effective manufacturing technology

Commercialization

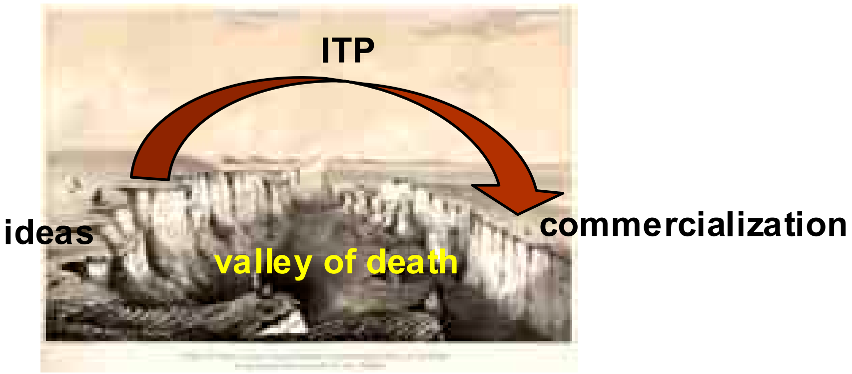
$$N_c = N_i \times f_w \times f_d \times f_p \times f_s \times f_l \times f_a \times f_v$$

N_c = # successfully commercialized laboratory technologies

N_i = # of "good" ideas

f_w = fraction that actually "work"
f_d = fraction of ideas that are disclosed
f_p = fraction that are patentable
f_s = fraction that are "scalable"
f_l = fraction that are licensed
f_a = fraction that attract attention from investors
f_v = fraction that survive the "valley of death"

w/o ITP	with ITP
$f_w \sim 0.50$	$f_w \sim 0.50$
$f_d \sim 0.75$	$f_d \sim 0.75$
$f_p \sim 0.75$	$f_p \sim 0.75$
$f_s \sim 0.50$	$f_s \sim 0.50$
$f_l \sim 0.40$	$f_l \sim 0.60$
$f_a \sim 0.40$	$f_a \sim 0.60$
$f_v \sim 0.05$	$f_v \sim 0.50$



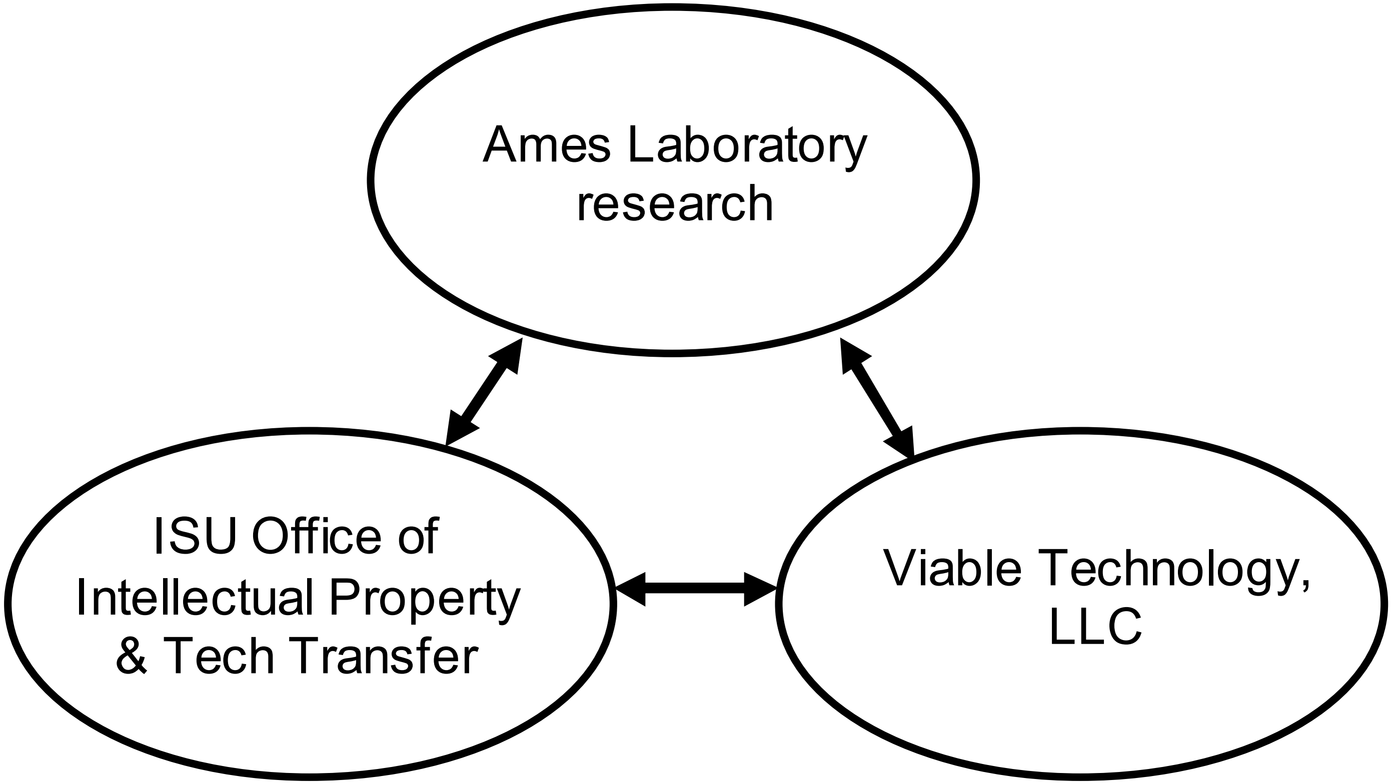
$N_c \approx 0.001 N_i$ w/o ITP

$N_c \approx 0.025 N_i$ with ITP

$N_c^{\text{w/ ITP}} \approx 25\ N_c^{\text{w/o ITP}}$

Commercialization...

Status as of 1 January, 2005



Ames Laboratory research

ISU Office of Intellectual Property & Tech Transfer

Viable Technology, LLC

Commercialization...

Status as of 1 May, 2005

General consensus that Viable Tech lacked sufficient…

- *Experience*
- *Facilities*
- *Capital*

License surrendered by Viable on March 31, 2005 to ISU's OIPTT

- OIPTT currently negotiating terms of *due diligence* from interested parties

- New licensing agreement expected to be in-place by Fall, 2005

Scale-up and commercialization of a complex materials-related technology imposes significant demands on the commercialization entity

must possess sufficient resources to bridge the Valley of Death

Commercialization…

Risks / Variables (and Lessons Learned):

Resources (and background) of licensee

Exclusive license vs. non-exclusive license

Communication of expectations between participants

Public sector support tends to be technology driven; private sector interest is market driven.

Assertiveness of the inventor/researcher in establishing / defining terms of licensing agreements

Licensing ≠ commercialization!!!!

Plans for FY’06…

- Research:
  - Processing technology / scale-up
  - Microstructural refinement
  - Thin film technology – start with PLD; advance to magnetron sputtering
  - Modeling - enable accurate prediction of best composition and microstructure for each application
- Industrial out-reach / collaboration:
  - establish industry-wide consensus on performance of superior wear resistant compositions
- Commercialization:
  - Patent protection on wear resistant compositions (and related materials)
  - Re-tool “commercialization infrastructure”
  - Have processing partner on-board and ready to manufacture “pilot-plant” scale quantities by end of FY’06

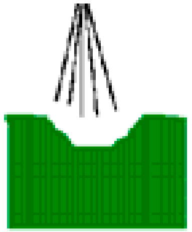
Additional slides...

Erosive wear:

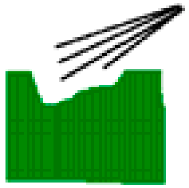
Erosive wear is the loss of material from a solid surface by impingement from abrasive particles contained within a fluid

Erosive wear depends on:

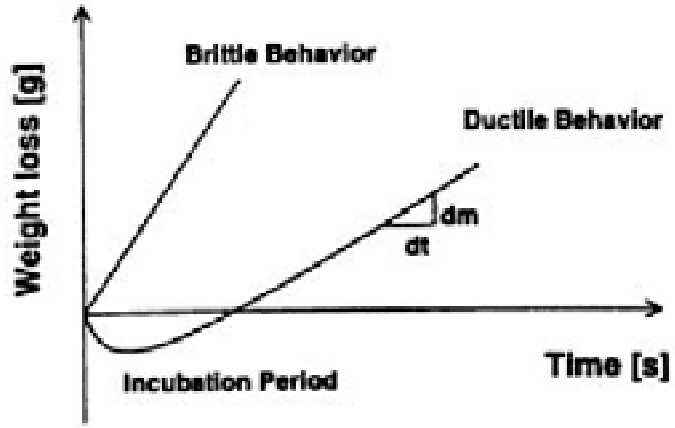
- **hardness of erodent**
- **morphology of erodent**
- **hardness of target**
- **fracture toughness (ductility) of target**
- **impact velocity**
- **impact angle**
- **temperature**
- **duration of exposure to erodent**

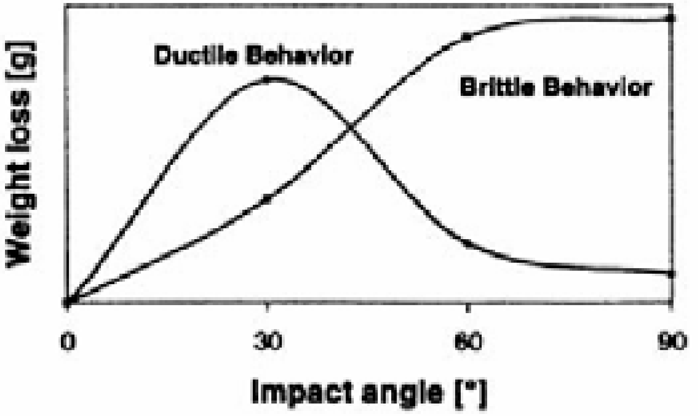


High angle erosion



Low angle erosion



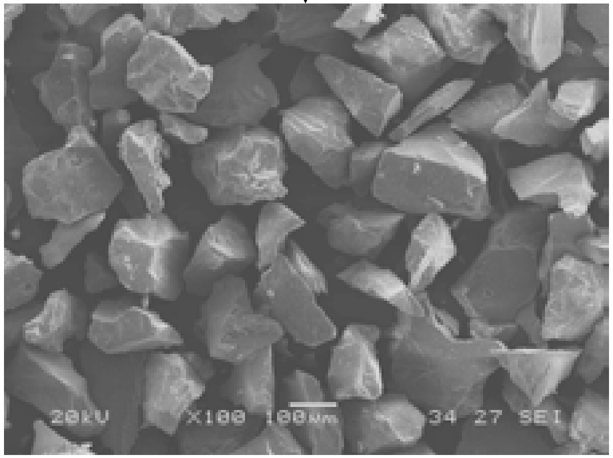


Ductile metals behave qualitatively different than brittle ceramics

Erosion resistance test parameters:

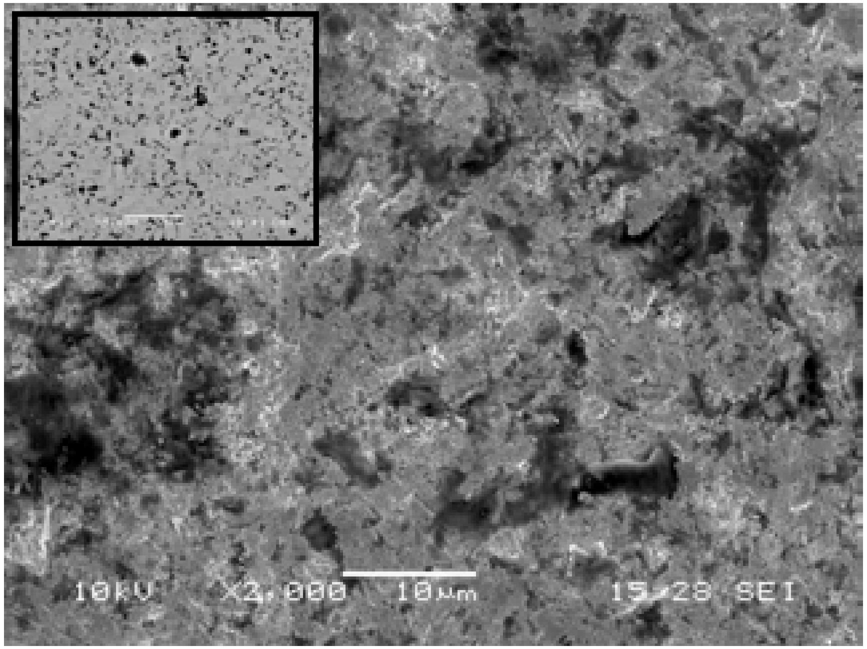
Erosive wear test parameters

Erodent	**Al_2O_3**
Ave. erodent particle size	**100 – 200 microns**
Hardness of erodent	**20 GPa**
Elastic modulus of erodent	**375 GPa**
Particulate velocity	**~ 100 m/s**
Nozzle diameter	**10.4 mm**
Nozzle length	**80.2 mm**
Stand-off distance	**1 inch (25.4 mm)**
Erosion angles	**45° and 90°**
Target	**Hot pressed "BAM" coupons**



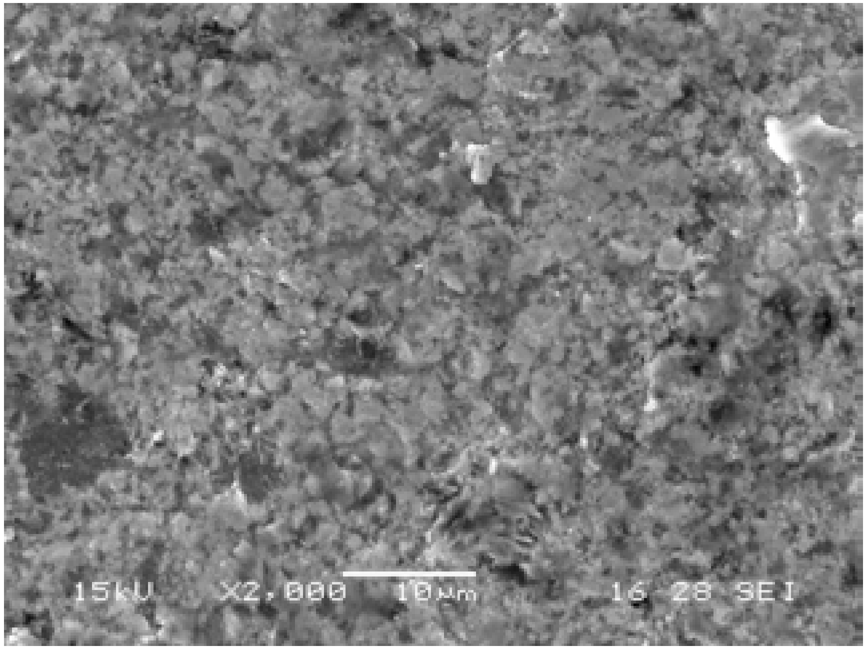
Microstructural damage (5 sec at 45°):

WC/Co



Removal of Co binder
Large-scale ejection of WC grains

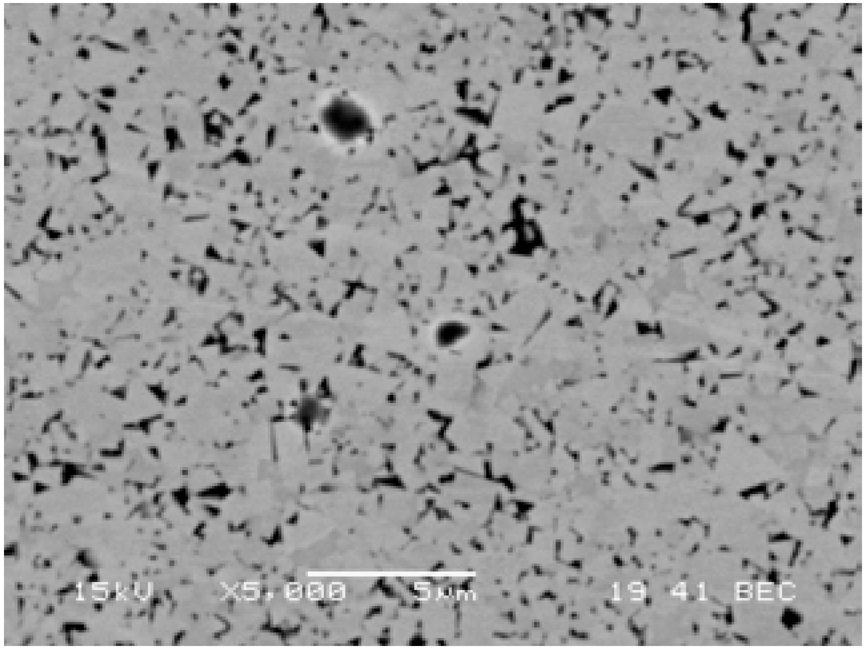
BAM (TB70-M2)



Minimal grain ejection
No evidence of transgranular fracture

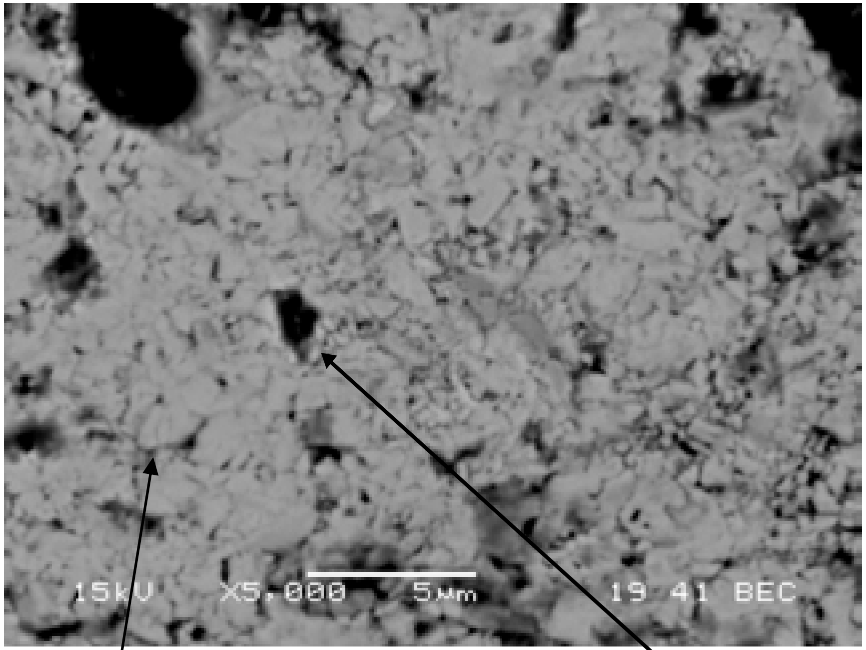
Microstructural damage (5 sec at 45°):

WC/Co - before erosion



2 - 5 micron WC grains

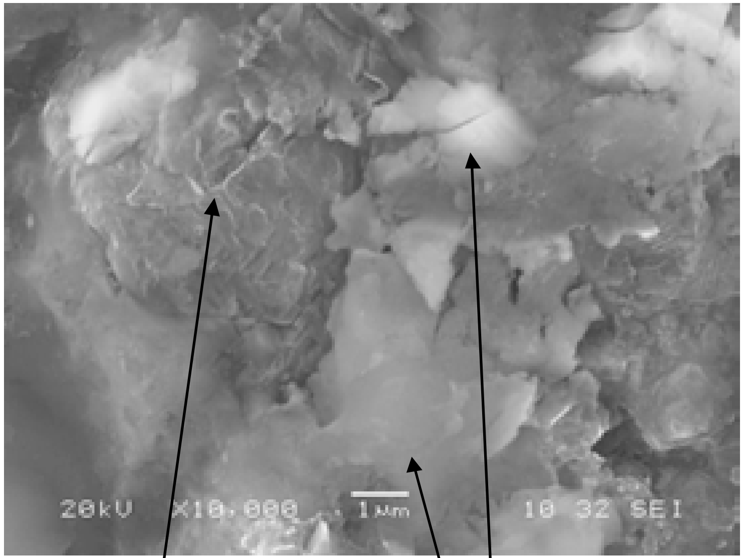
WC/Co - after erosion



Transgranular fracture,
grain ejection

Microstructural damage (2 minutes at 45°):

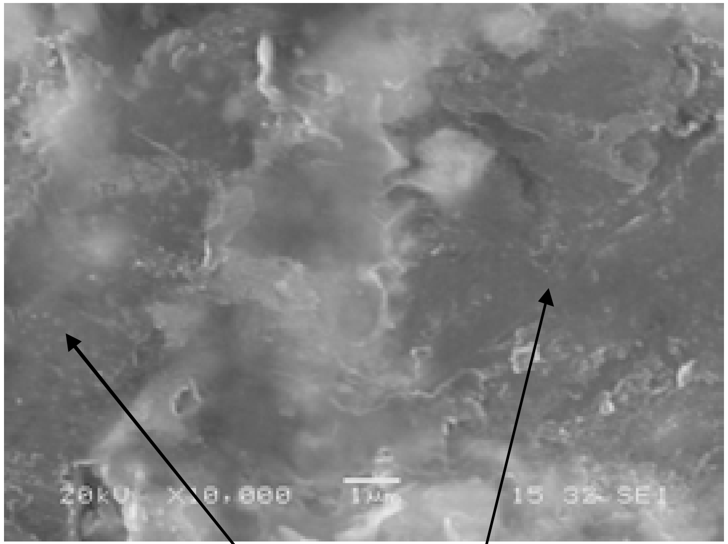
C-BN



Transgranular fracture

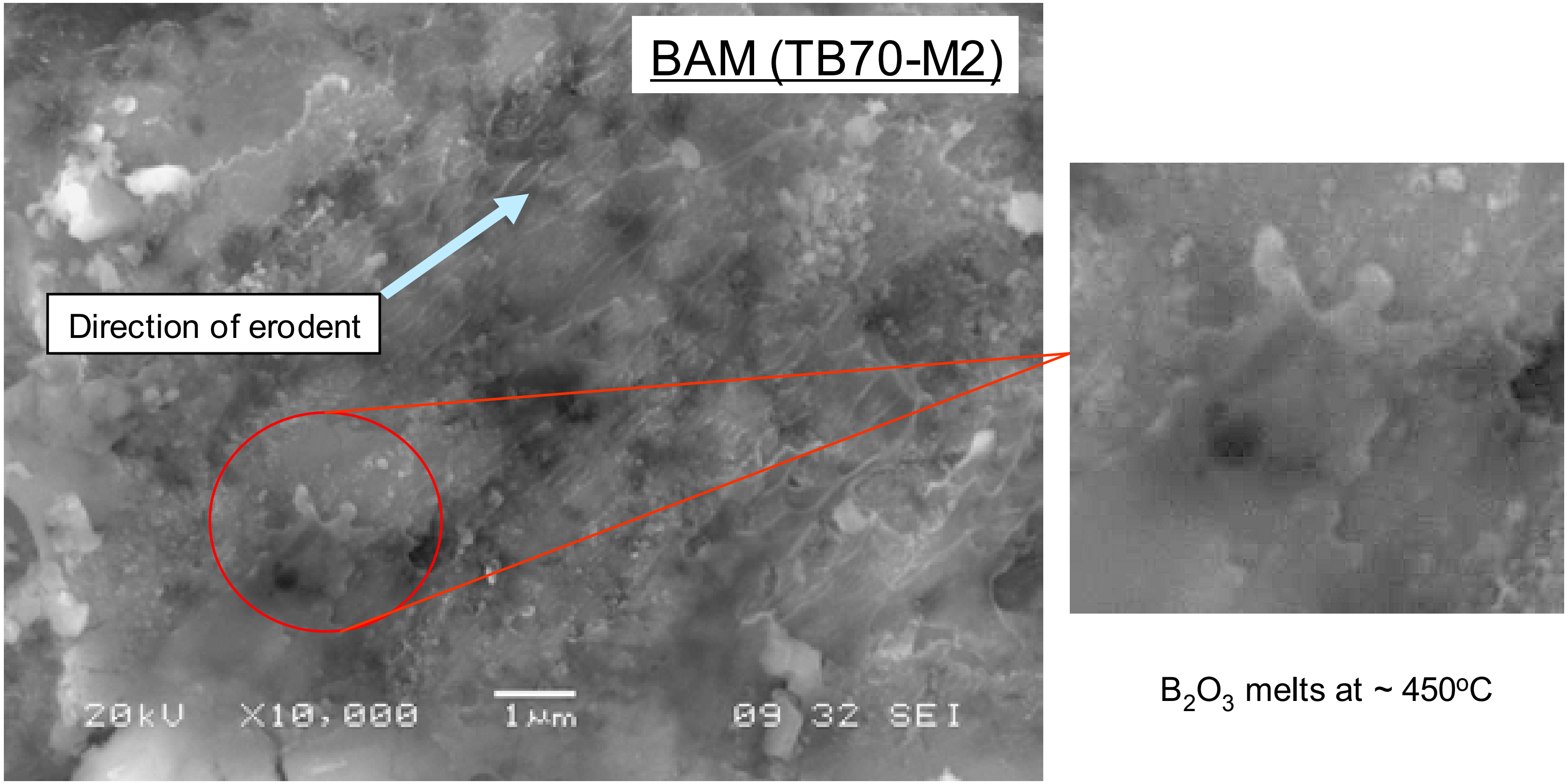
Al_2O_3

BAM (TB70-M2)



Few fractures; relatively smooth surface

Thermal effects (2 minutes at 45°):



B_2O_3 melts at ~ 450°C

Note presence of droplets - possible formation of molten B_2O_3

Thermal effects:

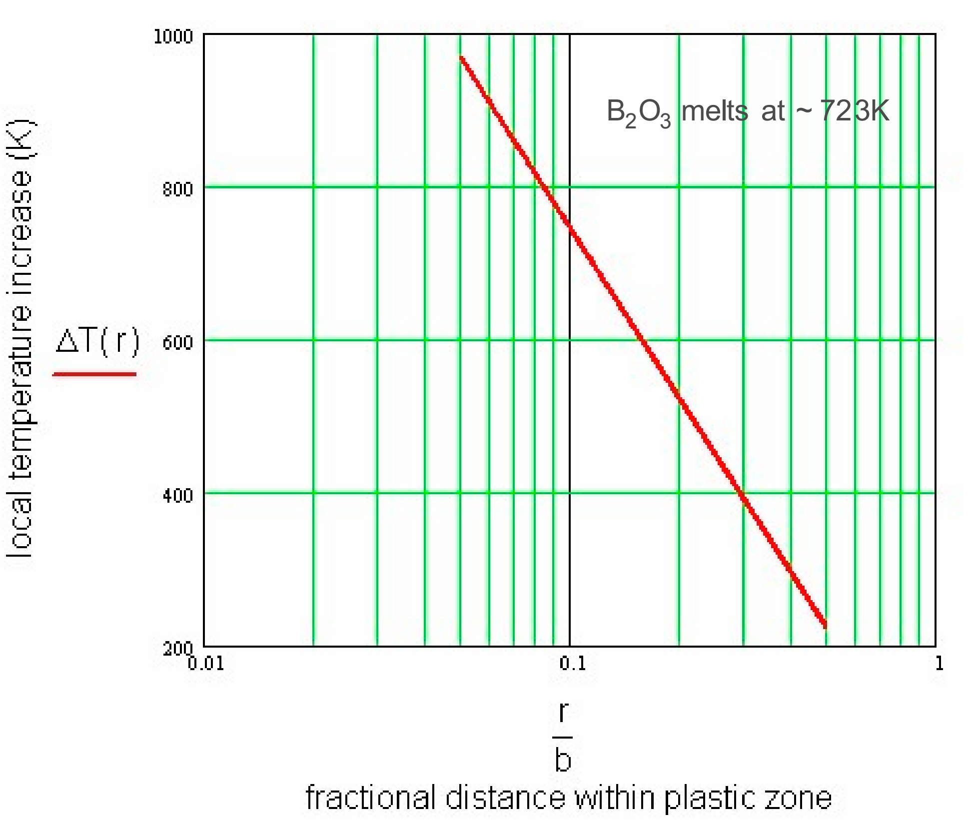
Model impact of erodent as an adiabatic indentation

Work performed in plastic deformation = localized heat absorbed by material

$$fP\delta z = mc\Delta T$$

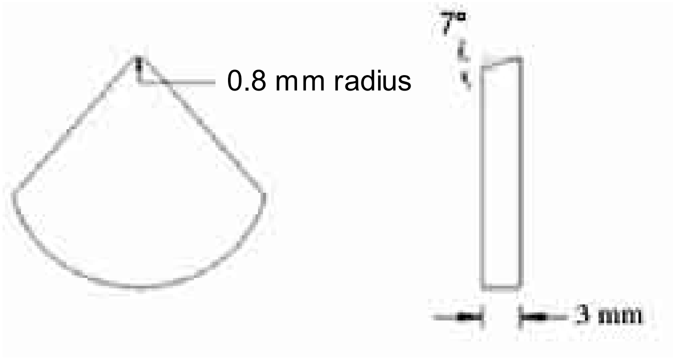
where
P = impact load
δz = penetration depth
m = mass
c = heat capacity
ΔT = local temperature increase
f = fraction of indentation energy dissipated within plastic zone

<u>Values used in model</u>:
hardness = 40 GPa
pyramid angle = 74°
density = 3.3 g/cm^3
heat capacity = 1026 J/kg-K (boron)
f = 0.4 (40% energy dissipated as heat)
b (plastic zone radius) = 20 microns



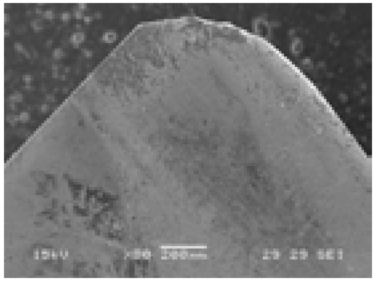
Lathe cutting tests:

Workpiece: Ti-6Al-4V

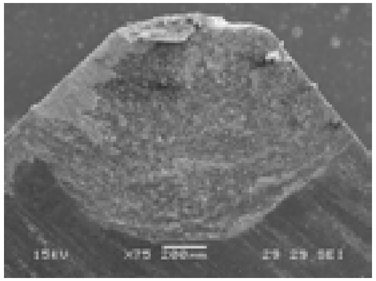


- relief angle = 7°
- no chip breaker
- insert was glued and clamped to a tool holder
- depth of cut = 0.5 mm
- feed rate = 0.25 mm/rev
- cutting speed = 25 m/min
- turning performed on 38 mm diameter rod
- unlubricated (dry) cutting
- ISO 3685-1977 (International Standard for ‘Tool-life Testing for Single Point Turning Tools)
- tool wear for one pass over a length of 200 mm measured to a resolution of 1µm using a Toolmaker’s microscope

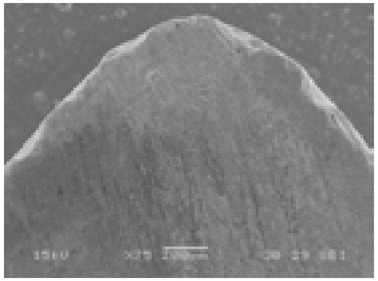
WC/Co



CBN



TB-70



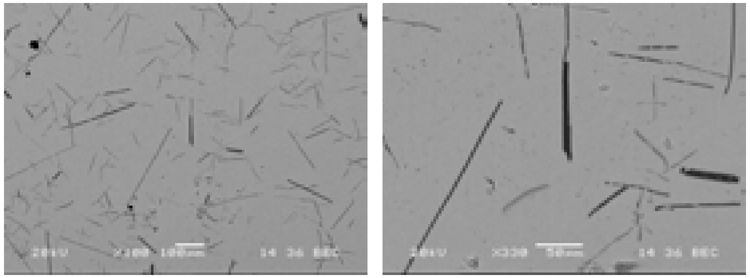
Commercialization

"…the new generation of clean energy entrepreneurial firms is finding it very difficult to make the leap from public sector funding for their innovations. Without new capital, many of our nation's most promising energy entrepreneurs will fail."

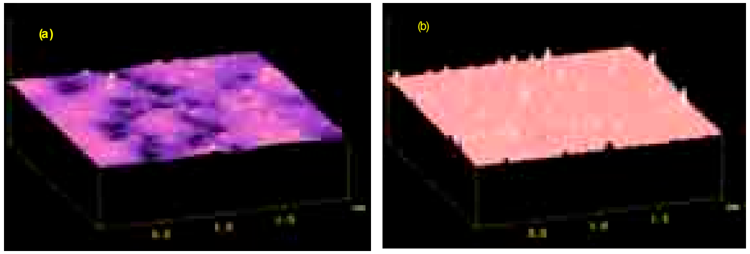
L. Murphy and P. Edwards, "Bridging the Valley of Death," TechComm – The National Journal of Technology Commercialization, June-July (2004) p. 25.

Other promising technologies and applications...

In-situ Aluminum reinforcement



Ultra-low friction coatings for MEMS



Friction-stir welding tips