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San Diego, CA

Spallation Resistant HVOF Coatings

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Standard Form 298 (Rev. 8-98)
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- Ongoing Work
  - University of Utah-Nano Composite Carbides
  - HVOF tough wear resistant coatings

Project Goal

- The goal of this project was to find a High Velocity Oxygen Fuel (HVOF) applied coating system that would enable a thick (up to 0.030 inch) coating to replace current Chrome over Nickel repair applications.
- The coating would be used across all US Air Force weapon system platforms on both high strength and low strength steels.
- Previous tests of HVOF applied WC/Co and WC/Co/Cr resulted in spallation under high stress. Spallation was a function of coating thickness and application parameters.
- Coating thickness limit at the time of the USAF Chrome replacement project start was .015", has now been reduced to .010".

- In Phase I, 14 different HVOF powder chemistries where sprayed and tested using the Praxair JP8000 spray system. Criteria for selection was that the powder must be commercially available from multiple vendors. Desired chemistries included Nickel, Cobalt and Iron based “bond coat” materials.

- Of these 14 chemistries, 4 where found to be top performers to be used in Phase II testing.

- A Design Of Experiment (DOE) was performed on the 4 selected chemistries to optimize coating characteristics given identified controllable input parameters.

- A statistical model was developed that can predict coating hardness, bond strength, ductility, and porosity given the input parameters resulting in an optimized coating for the “Duplex” application.

Spallation Resistant HVOF Coatings
DOE Prediction Tool

Amdry 9954 Low

Almen		Bend		Microhardness		Bond		Porosity	
Term	*Coeff*	*Term*	*Coeff*	*Term*	*Coeff*	*Term*	*Coeff*	*Term*	*Coeff*
Constant	-5.780E-02	Constant	-3.349E+01	Constant	-2.354E+02	Constant	-8.876E+03	Constant	-1.572E+00
O_2	1.899E-05	O_2	5.231E-02	O_2	7.177E-01	O_2		O_2	5.230E-03
F	4.841E-03	F		F	1.837E+02	F	-6.004E+03	F	-1.377E+00
P	4.448E-03	P	-1.472E+00	P	-3.243E+01	P		P	-8.200E-02
D	1.861E-03	D	-1.991E+00	D	-6.846E+01	D	4.622E+03	D	5.418E-02
O_2^2		O_2^2	-1.441E-05	O_2^2	-1.677E-04	O_2^2		O_2^2	
F^2		F^2		F^2	-2.708E+01	F^2		F^2	1.099E+00
P^2	-2.007E-04	P^2		P^2		P^2	0.000E+00	P^2	
D^2		D^2	7.807E-02	D^2		D^2	-2.842E+02	D^2	
O_2*F		O_2*F		O_2*F		O_2*F		O_2*F	-3.059E-03
O_2*P		O_2*P	8.333E-04	O_2*P		O_2*P		O_2*P	
O_2*D	-1.172E-06	O_2*D		O_2*D		O_2*D		O_2*D	7.891E-04
F*P	-3.802E-04	F*P		F*P		F*P		F*P	
F*D		F*D		F*D	8.668E+00	F*D	5.632E+02	F*D	-2.990E-01
P*D		P*D		P*D	2.435E+00	P*D		P*D	
R^2	85.98%	R^2	39.68%	R^2	91.79%	R^2	43.89%	R^2	77.94%
± 95%	0.0016	± 95%	1.6	± 95%	26.6	± 95%	4,525	± 95%	1.36
Prediction	0.0024	Prediction	2.3	Prediction	351.0	Prediction	4,956	Prediction	1.11
measured	0.002	measured	2	measured	347.4	measured	4557	measured	0.869

Prediction	
Input	*Value*
O_2	1800
F	4.2
P	8.7
D	16

Measured Results

Predicted Results

Input variables

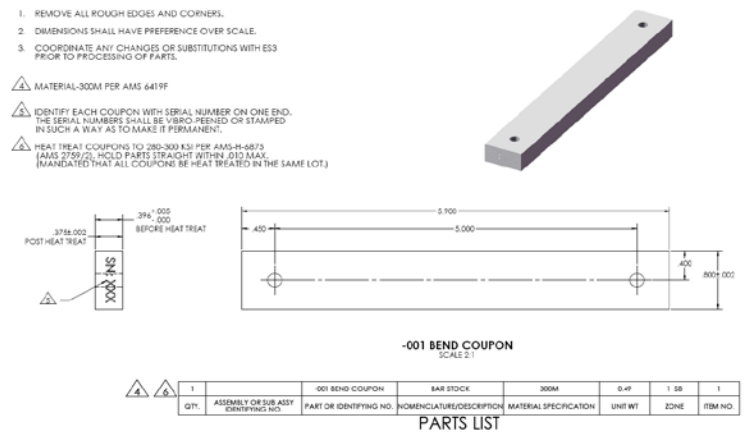
O_2: Oxygen Rate
F: Fuel Rate
P: Powder Rate
D: Standoff Distance

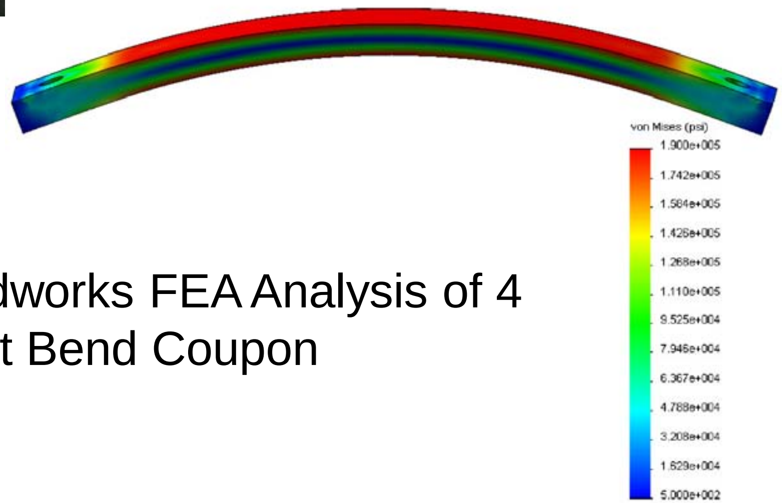
Phase II

- The 4 down-selected chemistries from Phase I where tested/evaluated as follows
  - Coating Integrity (300M Steel Flat Bar)
  - Corrosion (B-117 Salt Fog)
  - Cost Analysis

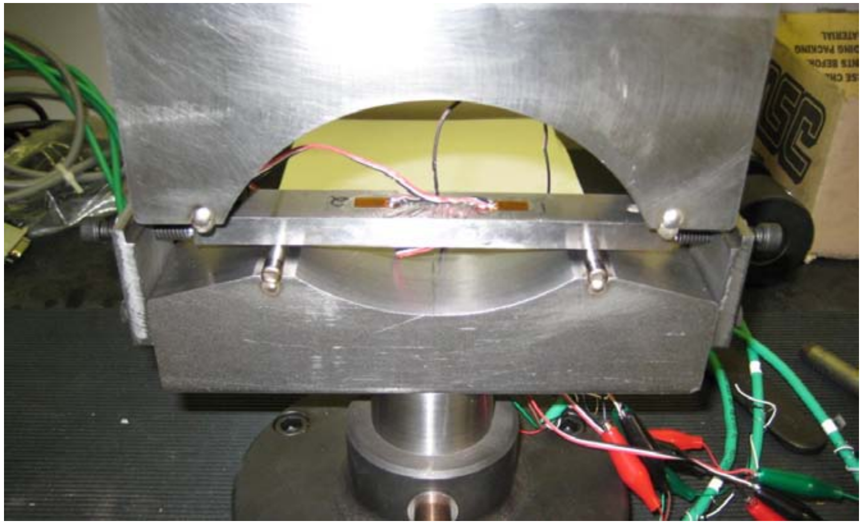
Powder	Chemistry
Praxair 1262 F	80%Ni 20%Cr
Praxair 1275H	Ni-Bal 14.5%Cr 4.5%Fe 4.5%Si 3.2%B
Amdry 9954	Co-Bal 32%Ni 21%Cr 8%Al 0.5%Y
Liquid Metal M	Fe-Bal 44%Cr 6%B 2%Si

Spallation Resistant HVOF Coating
Phase II Coating Integrity

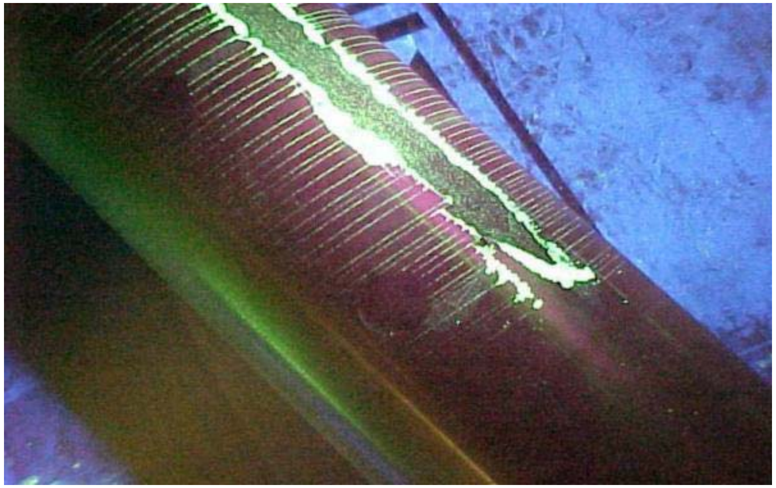




Solidworks FEA Analysis of 4 Point Bend Coupon



Spallation Resistant HVOF Coating
Phase II Coating Integrity



To test a coating's integrity, a 4 point bend test was developed that creates a constant stress state across the gage section where the coating is applied.

- Each Chemistry was tested for 5 cycles at each of the following stress levels (or until spallation occurred)
  - 190ksi
  - 210ksi
  - 230ksi
- Initial testing with WC-Co top coat on all of the duplex chemistries did not perform as expected (spallation at 190ksi)
- A "Triplex" coating was then optimized and tested where a thin layer of WC-Co was applied to substrate before applying the "Duplex" build coat.
- Only one of the 4 chemistries spalled with the Triplex Configuration.

Spallation Resistant HVOF Coating
Phase II Coating Integrity

Duplex Chemistry	Coupon Serial #	Total Coating Thickness	190ksi	210ksi	230ksi	Notes
1262 F	4	0.020	Pass	Pass	Pass	no spallation
1262 F	5	0.020	Pass	Pass	Pass	no spallation
1262 F	11	0.030	Pass	Pass	Pass	no spallation
9954	79	0.020	Pass	Pass	Pass	no spallation
9954	33	0.020	Pass	Pass	Pass	no spallation
9954	24	0.030	Pass	Pass	Pass	no spallation
LMC-M	27	0.020	Pass	Pass	Pass	no spallation
LMC-M	55	0.030	Pass	Pass	Pass	no spallation
LMC-M	47	0.020	Pass	Pass	Pass	no spallation
1275H	72	0.020	Pass	Pass	Pass	no spallation
1275H	14	0.020	Pass	Pass	Pass	no spallation
1275H	20	0.030	Spalled			blade style spallation at 190 ksi

All data shown is for coupons coated with the triplex coating system as follows:

- 0.017 and 0.027 inch total coating thickness of duplex coating bond coat layer
- Additional 0.003 inch after grind WC-Co top coat for a total coating thickness of 0.020 or 0.030 inch respectively.

Spallation Resistant HVOF Coating
“Triplex” Coating

ASTM B-117 Corrosion Testing

Cr over Ni | Cr only | 1262 F with WC-Co Cap | WC-Co only

- Corrosion testing was performed per ASTM B-117 for a total of 1000 hours
- ASTM B-117 was used to duplicate the original HCAT accelerated corrosion testing.
- The specimens where evaluated at 250 hour increments.
- Base line samples of Chrome only, Chrome over Nickel, and WC-Co only where tested for comparisons to the Duplex/Triplex chemistries.
- A double blind evaluation study was performed to ensure accuracy of results.

Spallation Resistant HVOF Coating
Phase II Summary

Chemistry	Corrosion	Integrity	Total Points
1262F	4	4	8
LMC-M	2	4	6
9954	2	4	6
1275H	4	1	5

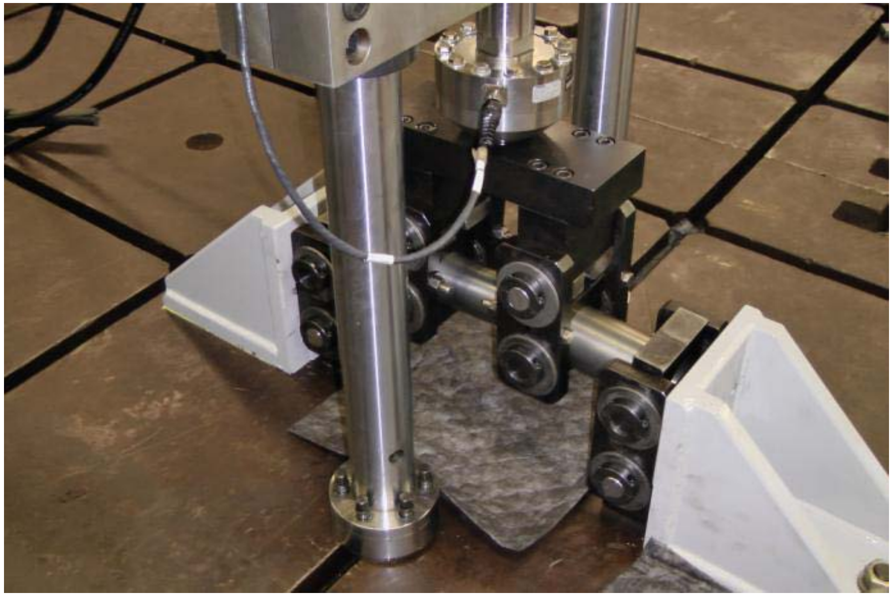
- Each Chemistry was ranked from best to worst in each category. 4 points where then given to the best with 1 given to the worst.

- The total application cost for all 4 chemistries is half the price of applying the same amount of WC-Co and does not factor in to the end result.

- Praxair 1262F (Ni-Cr) performed the best of the 4 chemistries and was moved to final coating integrity/fatigue testing in Phase III.

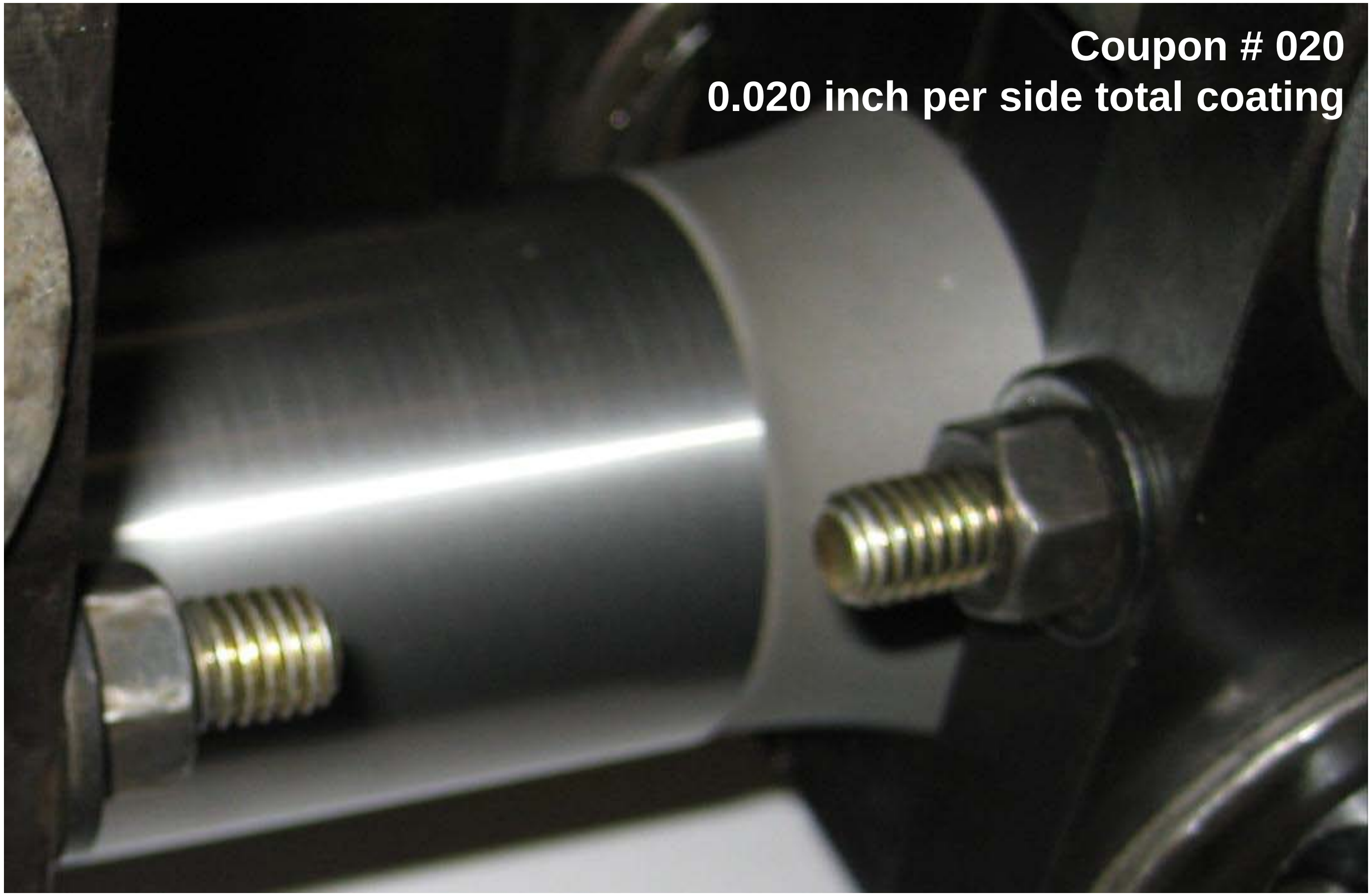
Spallation Resistant HVOF Coating
Phase III Large Bar Integrity Testing

- Phase III Testing- Metcut
  - Large Bar Coating Integrity/Fatigue per USAF 200925098
    - 190ksi: 100 cycles @ R=-1
    - 210ksi: 100 cycles @ R=-1
    - 230ksi: Till Coupon Failure @ R=-1



Bare   As Sprayed   Finish Ground

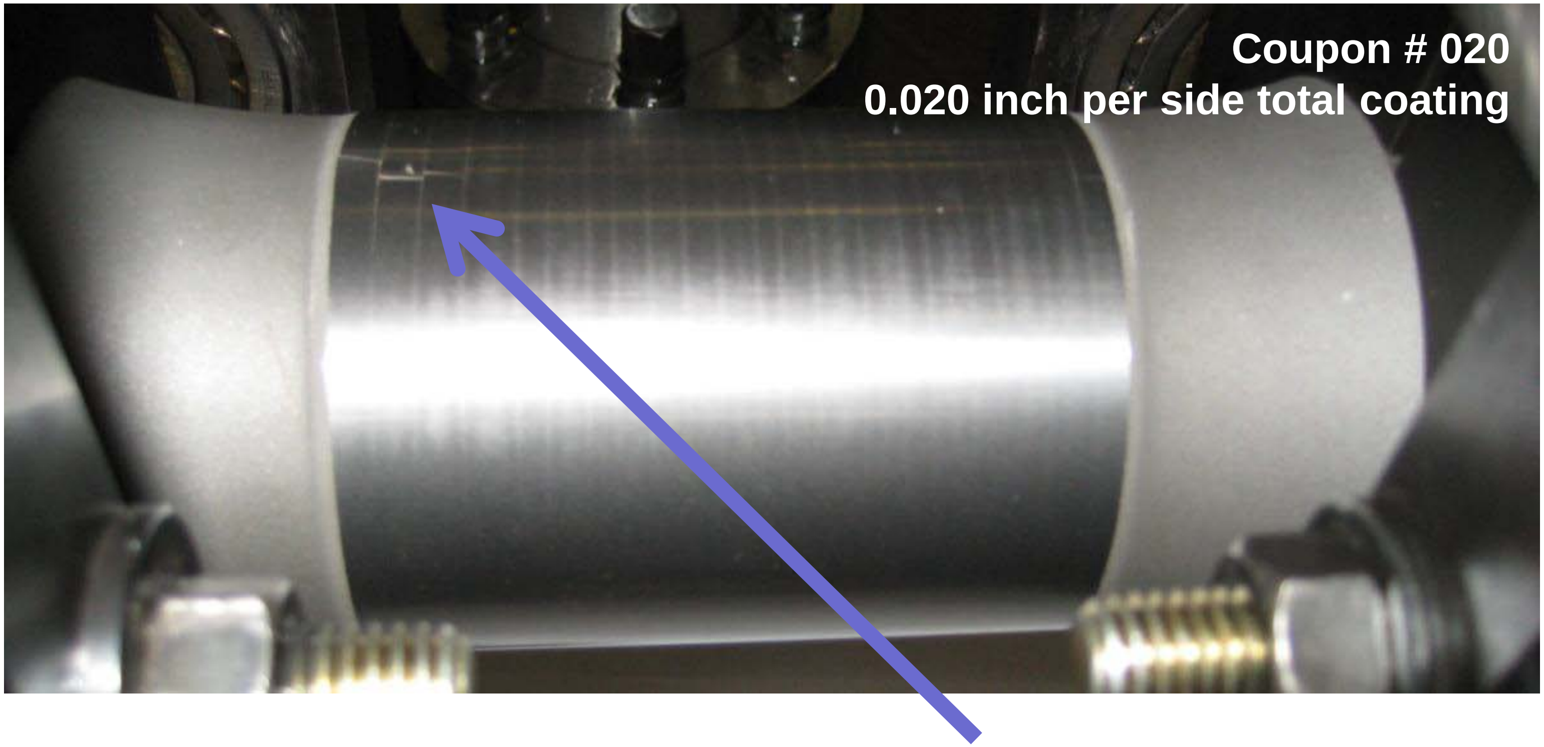
Spallation Resistant HVOF Coating
Phase III Large Bar Integrity Testing



After 100 cycles at 190ksi and 100 cycles at 210 ksi.

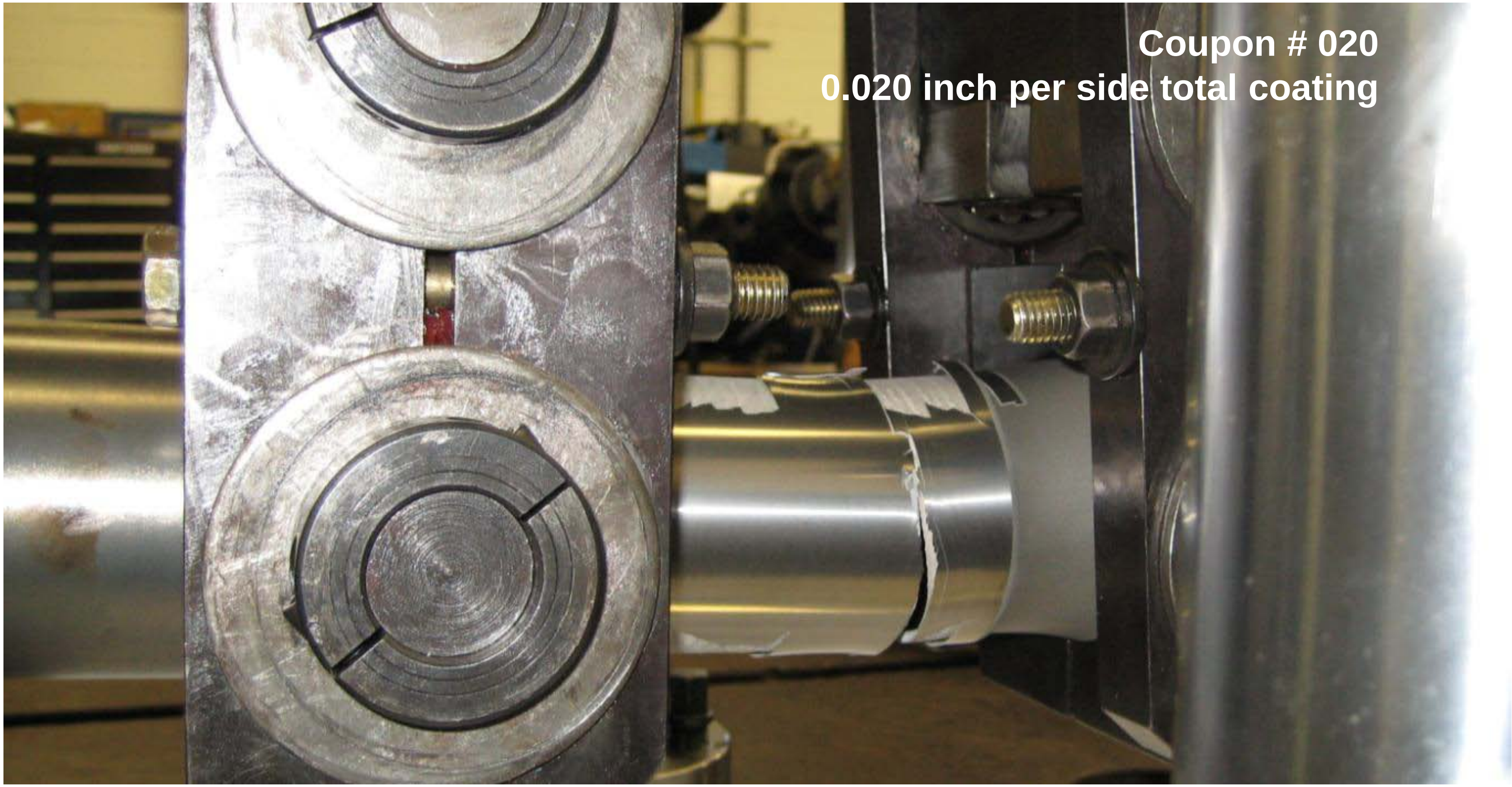
<u>No Spallation!</u>

Spallation Resistant HVOF Coating
Phase III Large Bar Integrity Testing



Coating Delamination at 200 cycles into the 230ksi stress level

Spallation Resistant HVOF Coating
Phase III Large Bar Integrity Testing



Cycles until Substrate coupon failure: 1538 @ 230ksi

Spallation Resistant HVOF Coating
Phase III Large Bar Integrity Testing

Serial #	Coating Type and Total Coating Per Side Thickness	Cycles Until Coating Failure			cycles @ 230ksi until coupon failure
		190 ksi	210 ksi	230 ksi	
032	bare	n/a	n/a	n/a	1563
049	bare	n/a	n/a	n/a	1168
053	bare	n/a	n/a	n/a	1263
WC-Co 8	WC-Co only 0.004 inch	100 Cycles[1]	100 Cycles[1]	970 Cycles[1]	970
WC-Co 20	WC-Co only 0.010 inch	36 Cycles[2]			1843
019	Duplex 0.020 inch	100 Cycles[1]	100 Cycles[1]	185 Cycles[2]	1793
020	Duplex 0.020 inch	100 Cycles[1]	100 Cycles[1]	200 Cycles[2]	1538
021	Duplex 0.020 inch	100 Cycles[1]	100 Cycles[1]	320 Cycles[2]	1942
WC-Co 15	WC-Co only 0.030 inch	4 Cycles[2]			N/A
022	Duplex 0.030 inch	100 Cycles[1]	20 Cycles[2]		1029
023	Duplex 0.030 inch	100 Cycles[1]	3 Cycles[2]		1612
024	Duplex 0.030 inch	85 Cycles[2]			1706

Notes
1: No spallation
2: Number of cycles at which Coating spallation occurred. Coupon was still tested for 100 cycles at each stress level.

WC-Co only data points are the best performing coupons from the original large bar testing performed in 2007 and 2008 and are included for comparison only.

Spallation Resistant HVOF Coating
Phase III Results

- Coating Integrity/Fatigue Testing Results
  - 0.020 inch total coating thickness- Passes stress, corrosion and application cost requirements and is recommended for implementation for Landing Gear applications.
  - More testing may be done to determine coating durability at lower stresses.

- What's Next?
  - Improvements to eliminate the multiple layered coating structure. Goal is to create an optimized single coating structure.
  - ES3 working with Dr. Zak Fang, University of Utah College of Metallurgical Engineering- CTO Heavystone Labs.
  - Develop coatings using proven concepts of composite hard materials

Spallation Resistant HVOF Coating
Ongoing Work

- Dr. Z. Zak Fang, Professor, Metallurgical Engineering, the University of Utah
- PhD 1991 (U of Alabama at Birmingham)
- MS and BS from Beijing University of Science and Technology
- 11 years industrial R&D and management experience (RTW Inc. and Smith International Inc.)
- Joined the University of Utah in 2002. Tenured full professor.
- ~ 40 US patents. Over 100 publications.
- Most downloaded articles of International Journal of Refractory Metals and Hard Materials
- The largest academic group in US on tungsten carbide
- Group well known in the world of hardmetals industry.

Functionally Designed Hard Materials

- Wear resistance of materials are affected by many factors:

  - Hardness and toughness of the material

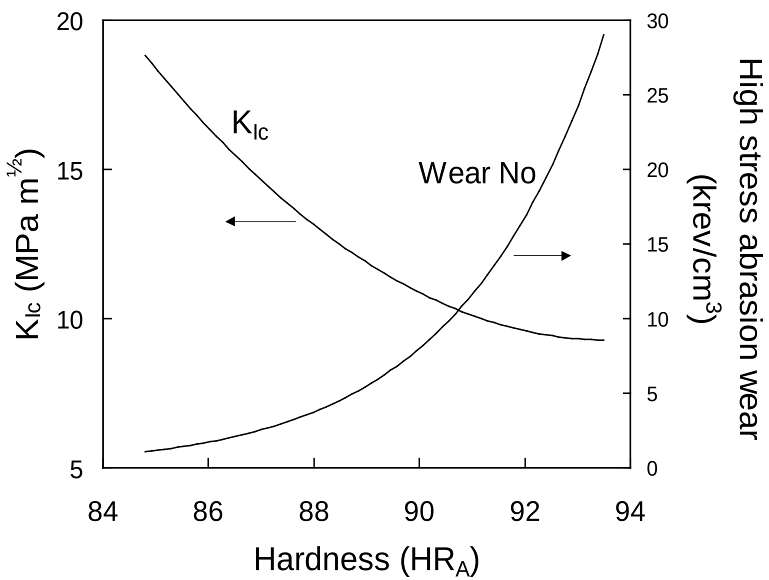
  - Microstructure of the material

  - Wear mechanisms / environment

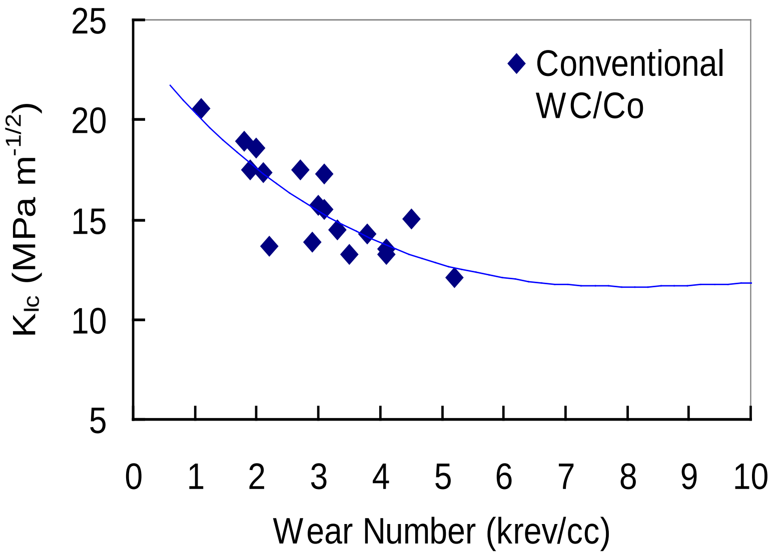
- A common practical issue:

  Trade-off between wear resistance and toughness

The Classic Trade-off



K_{Ic} and Wear number vs. HR_A

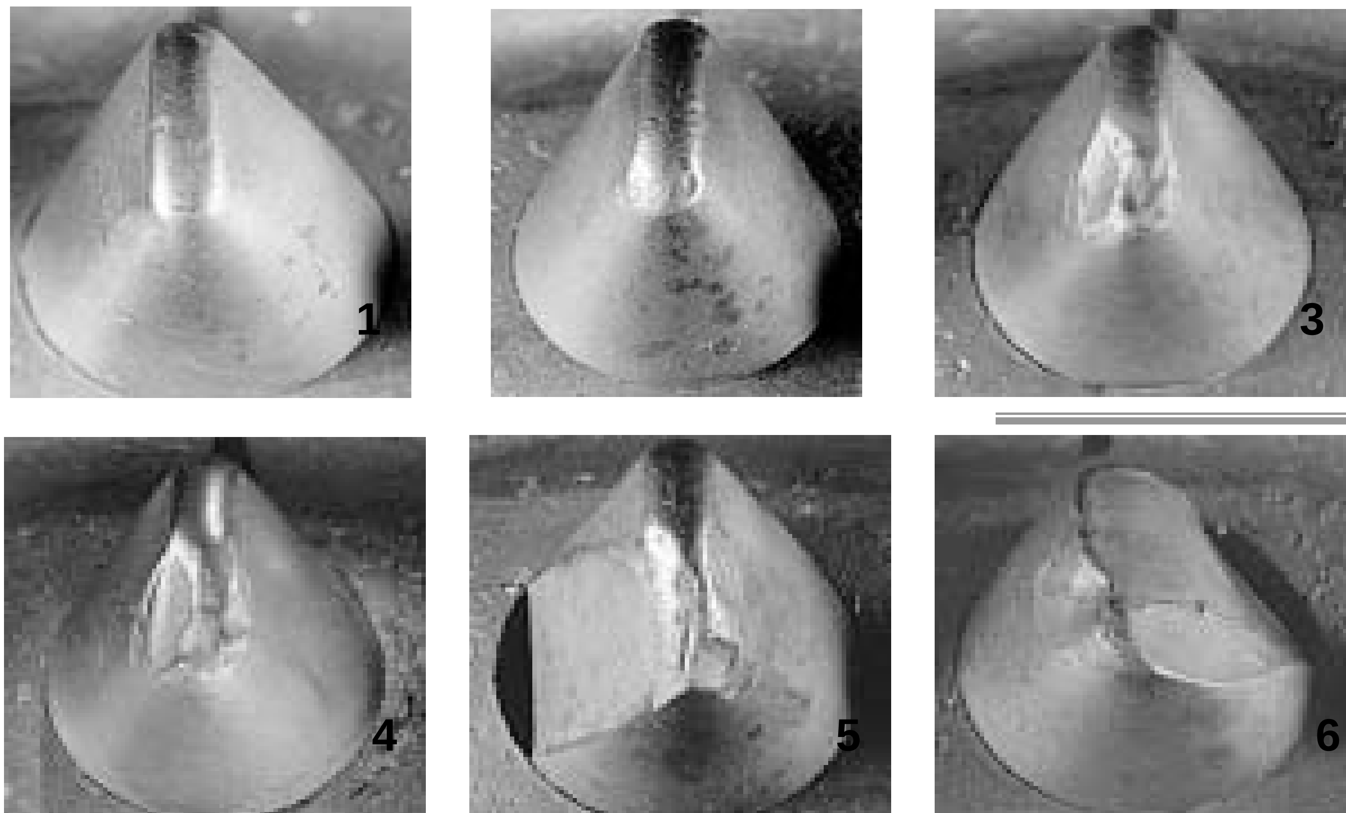


K_{Ic} vs. Wear number

Mechanical properties of conventional cemented tungsten carbide.

Ref. Z. Fang, "Wear Resistance of Powder Metallurgy Alloys", ASM Handbook, Volume 7, Powder Metallurgy, 1998, pp.965-977

An example - Cemented tungsten carbide compacts employed for rock drilling



From minor chipping to fracturing

Challenge

Improve toughness without sacrificing wear resistance

Question: How?

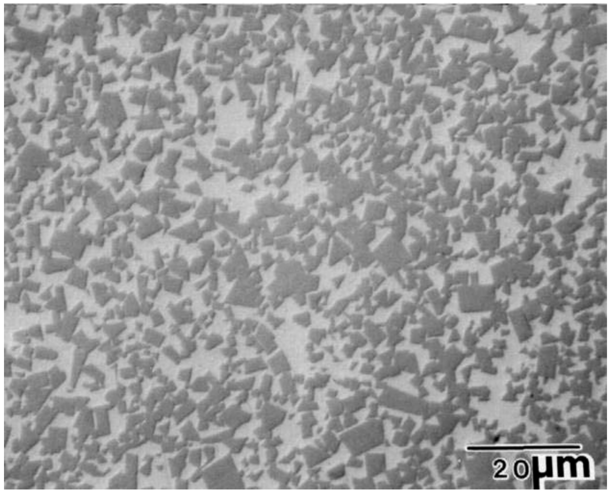
A microstructure engineering approach

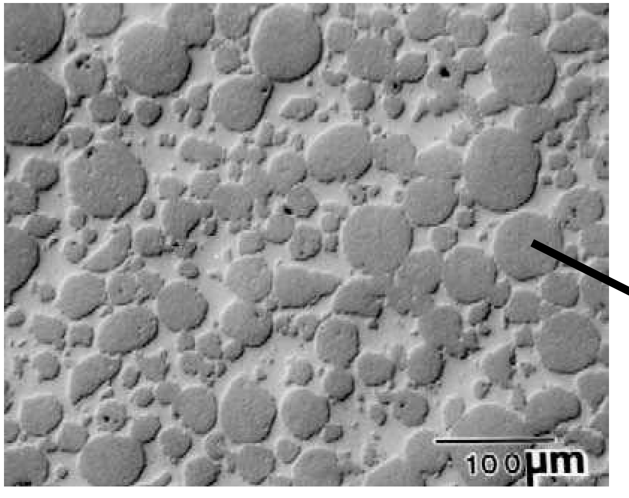
Functionally Designed Microstructure

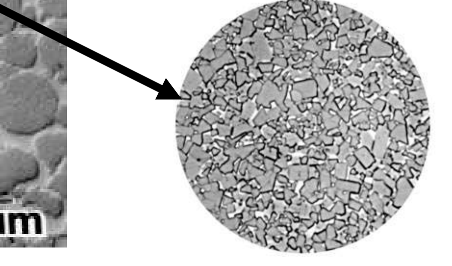
- Tailor properties solely by microstructure, no / little changes in composition
- Microstructure by design, not necessarily a product of phase transformation or thermal mechanical processing
- Improves functional properties of components, with or without concurrent boost of intrinsic mechanical properties

Functionally Designed Hard Materials

Example - Double Cemented Carbide







Conventional microstructure of WC-27%wt Co

DC carbide rearranged microstructure with same chemistry

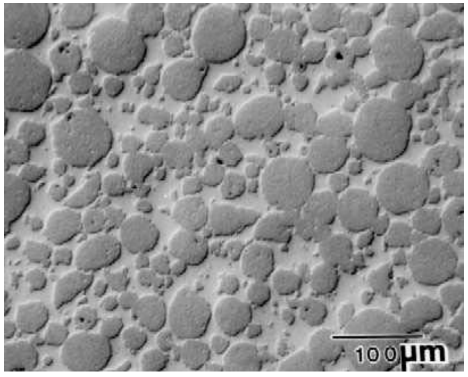
- Maximize Mean Free Path for high toughness,
- Using large granules of the hard phase for good wear resistance

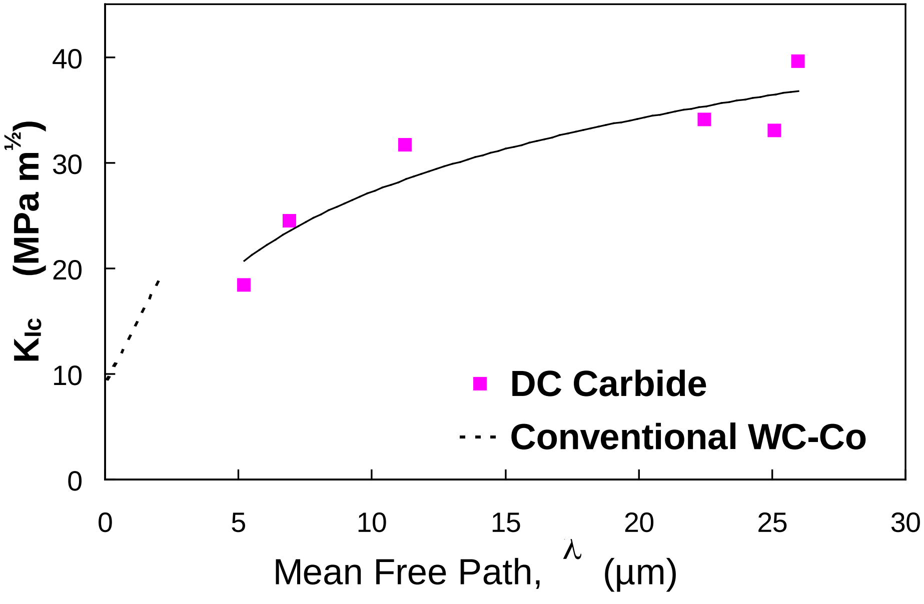
Ref. Z. Fang, G. Lockwood, and A. Griffo, "A Dual Composite of WC-Co", *Metall. Trans. A.*, 1999, *30A*, 3231-3238

Functionally Designed Hard Materials

Properties and Performance

DC Carbide

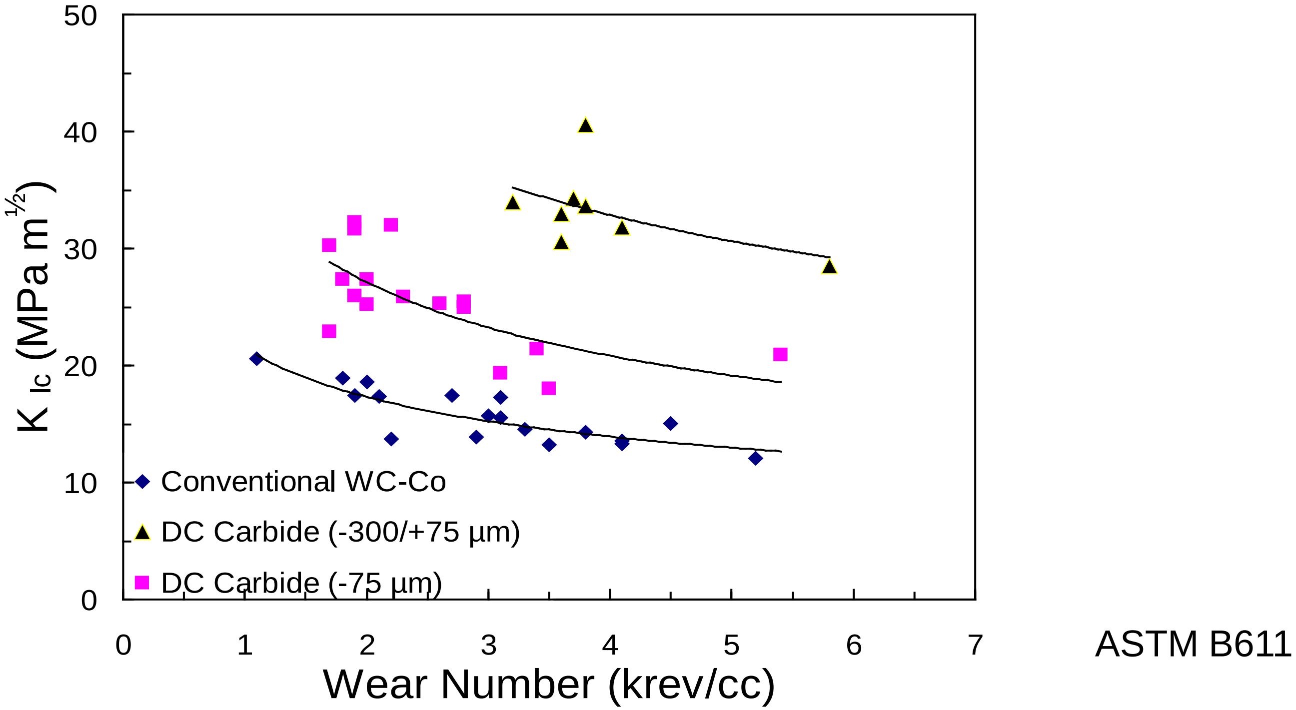




- DC carbide is dramatically tougher than conventional WC-Co
- DC carbide is equivalent in toughness when compared to tool steel

Ref. Z. Fang, G. Lockwood, and A. Griffo, "A Dual Composite of WC-Co", *Metall. Trans. A.*, 1999, *30A*, 3231-3238

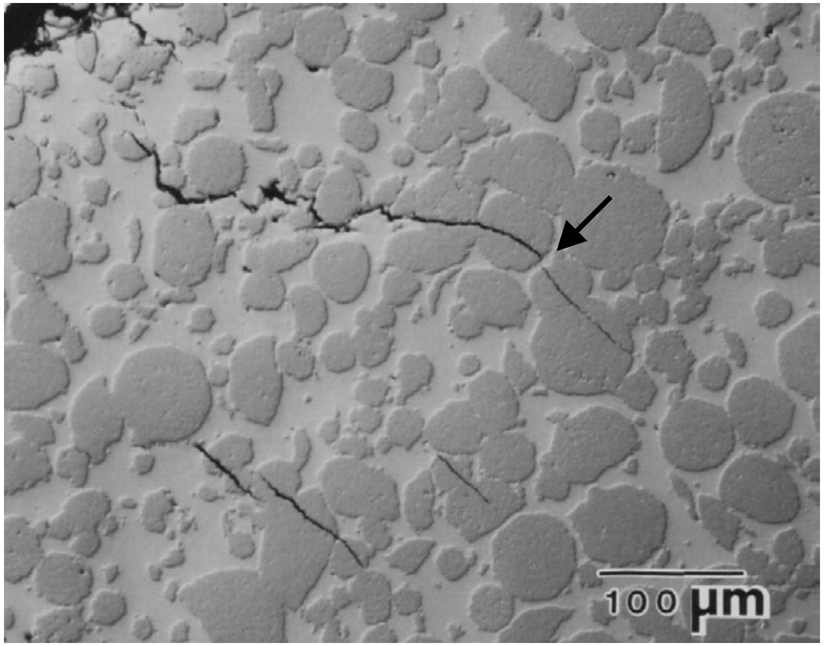
Fracture Toughness / Wear Combination



K_{Ic} vs. High Stress Abrasion Wear #, comparing DC carbide to conventional WC-Co

Ref. Xin Deng, B. R. Patterson, K. K. Chawla, M. C. Koopman, C. Mackin, Zhigang Zak Fang, G. Lockwood, A. Griffo, "Microstructure / Hardness Relationship in a Dual Composite", *J. Mater. Sci. Lett.*, 2002, *21*, 707-709

Fatigue Crack Resistance and Damage Tolerance



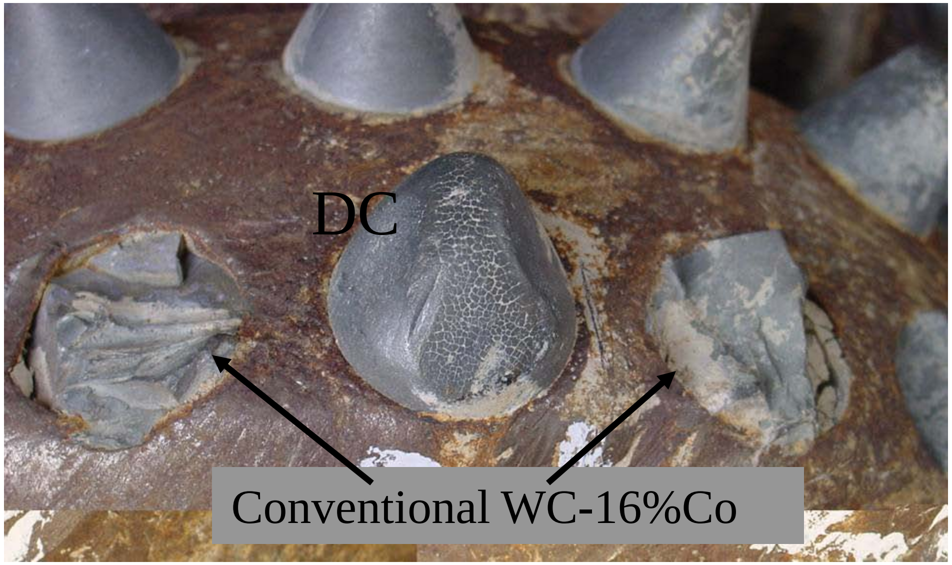
Lab fatigue test - Crack blunting - Crack bridging

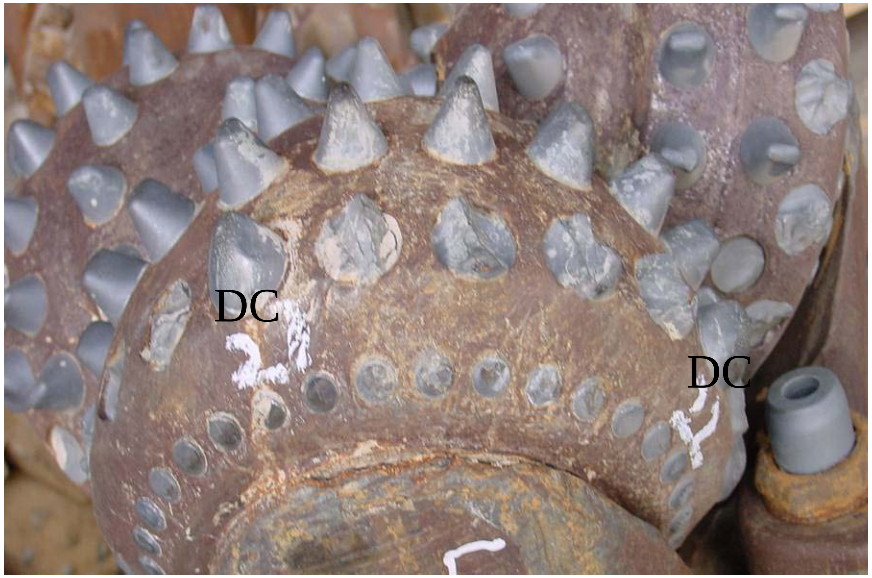
DC carbide rock drilling insert survived severe damage. No catastrophic breakage.

Ref. Zhigang Zak Fang, A. Griffo, B. White, D. Belnap, and G. Hilmas; "Fracture Resistant Super Hard Materials and Hardmetals Composite with Functionally Designed Microstructure"; *Int. J. Refract. Met. Hard Mater.*, 2001, *19*, 453-459

Field Test Performance

Damage tolerance of components is greatly improved as the result of higher K_{IC}





Ref. Zhigang Zak Fang, A. Griffo, B. White, D. Belnap, and G. Hilmas; "Fracture Resistant Super Hard Materials and Hardmetals Composite with Functionally Designed Microstructure"; *Int. J. Refract. Met. Hard Mater.*, 2001, *19*, 453-459

Conclusions

- Fracture toughness of Hard materials can be improved without sacrificing wear resistance.
- Functionally designed microstructure is a viable approach for improving properties of WC composites independent of chemical or phase compositions.