Final Report-Process Mapping

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Participants

- HCAT (NRL)
- Hill (GA)

- Praxair
- Stellite
- Sulzer-Metco

- Sauer Engineering
- NRC

Outlines

- Goals
- Project Description
- Phase 1
  - Operating Ranges
  - Bend Test Results
  - Coating Characterization
- Phase 2
  - Cyclic Loading Results
  - Fatigue Cracking
- What's Next

Goals

- The main goal or purpose of the program is to understand or “map” the varied HVOF processes that have been used in the HCAT program for numerous qualification protocols and final part spraying.
- Unfortunately, with the past HCAT evaluations performed at both different test facilities and spray shops, there has been some considerable spread in results concerning the coating performance thresholds for both cracking and spalling capabilities.

Goals

- With the information generated in one location in this study, the *robustness of the HVOF process* as related to HCAT usage can and was determined.
- A secondary outcome could also be another type of QC (quality control) tool that would allow further differentiation of varied spray processes other than the current metallography, Almen, hardness, and tensile analyses.
- In addition to the varied sets of parameters from the HCAT population, two additional sets of spray parameters were also evaluated:
  - Jet Kote parameters as supplied by David Lee of Stellite which would encompass work on the Messier Dowty installation for HCAT type landing gear work
  - Newer spray parameter sets from both Sulzer Metco and Praxair which were developed to address the spalling issue during fatigue and coating integrity evaluations

Project description

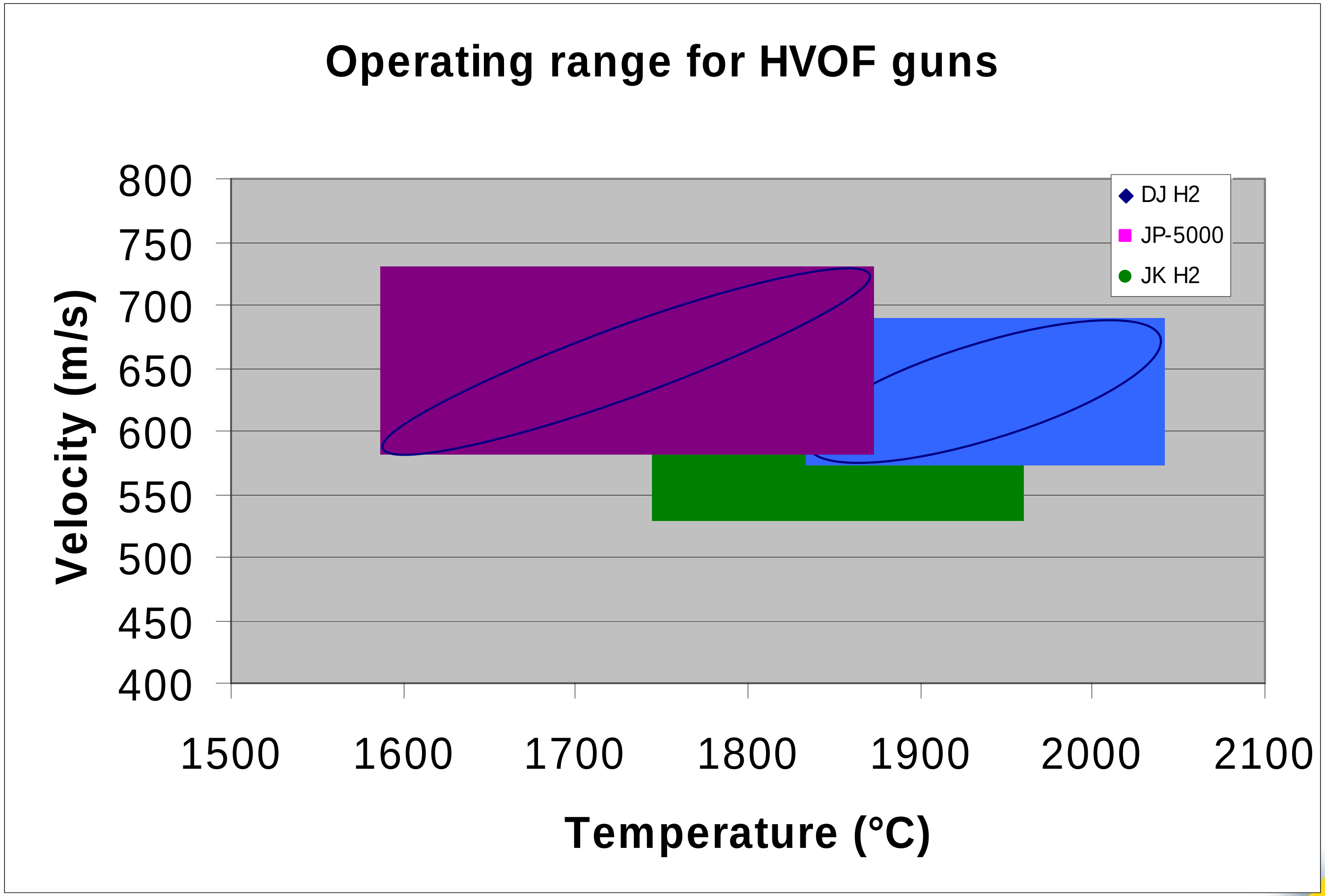
- **Phase 1** (directly related to the previous work carried out within the HCAT/C-HCAT projects)
  - Analysis of the current ‘HCAT related’ sets of parameters
    - DPV 2000 velocity/temp measurements
    - General QC tests (Almen, micro, hardness)
  - Bend tests of Almen strips using acoustic emission
  - Coating characterization via the SEM
  - Phase analysis with carbide degradation index (X-Ray)
  - Selection of spray parameters to be used in Phase 2
- **Phase 2** (cyclic loading)
  - Step Loading
  - Fatigue testing

Operating Ranges

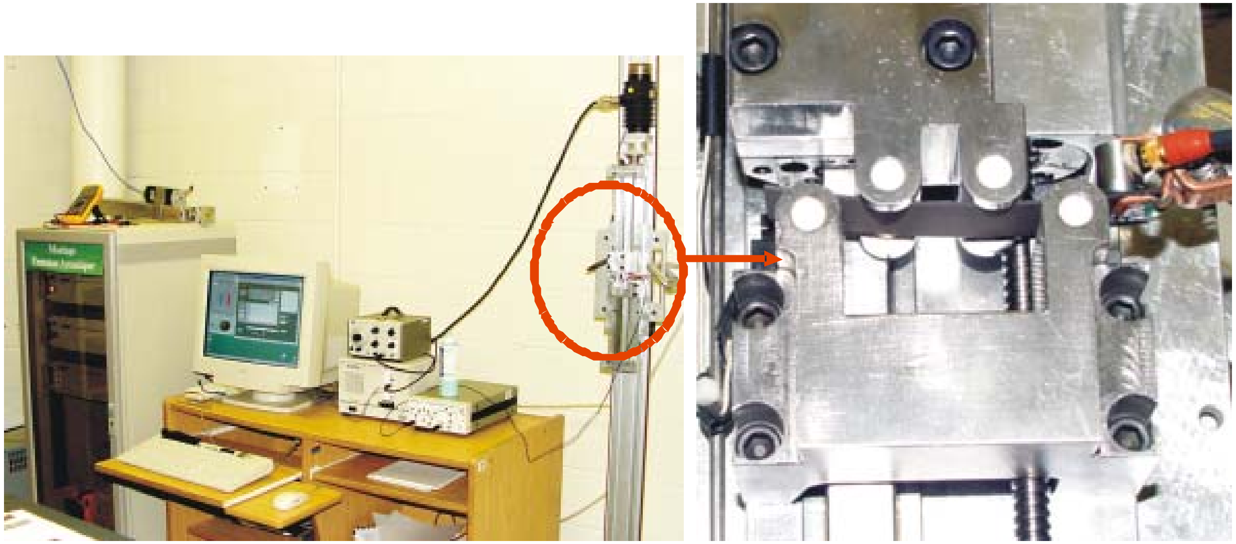
DJ H2: Diamond Jet using H2 as fuel (D-2005)

JP-5000 using Kerosene (1343 VM)

JK H2: Jet Kote III using H2 as Fuel (Stelcar JK 117)

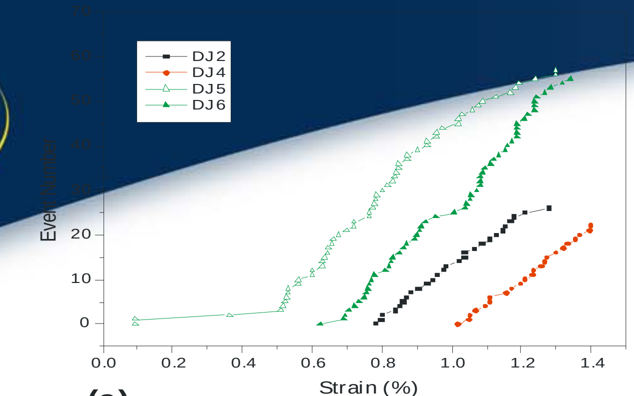


Bend Testing

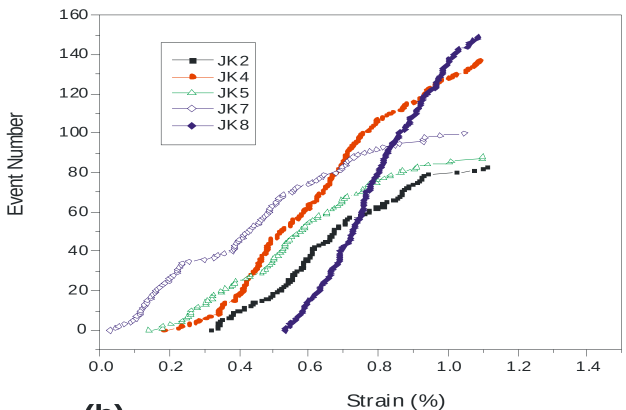


- Use Common Almen strips as specimens
- Use Acoustic Emission to record initial event and total energy
    - Onset of Cracking
    - Total Cumulative Energy generated by the cracking process

Typical AE Plots



(a)



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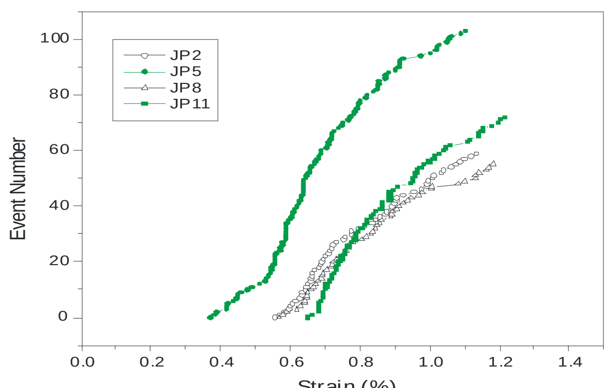

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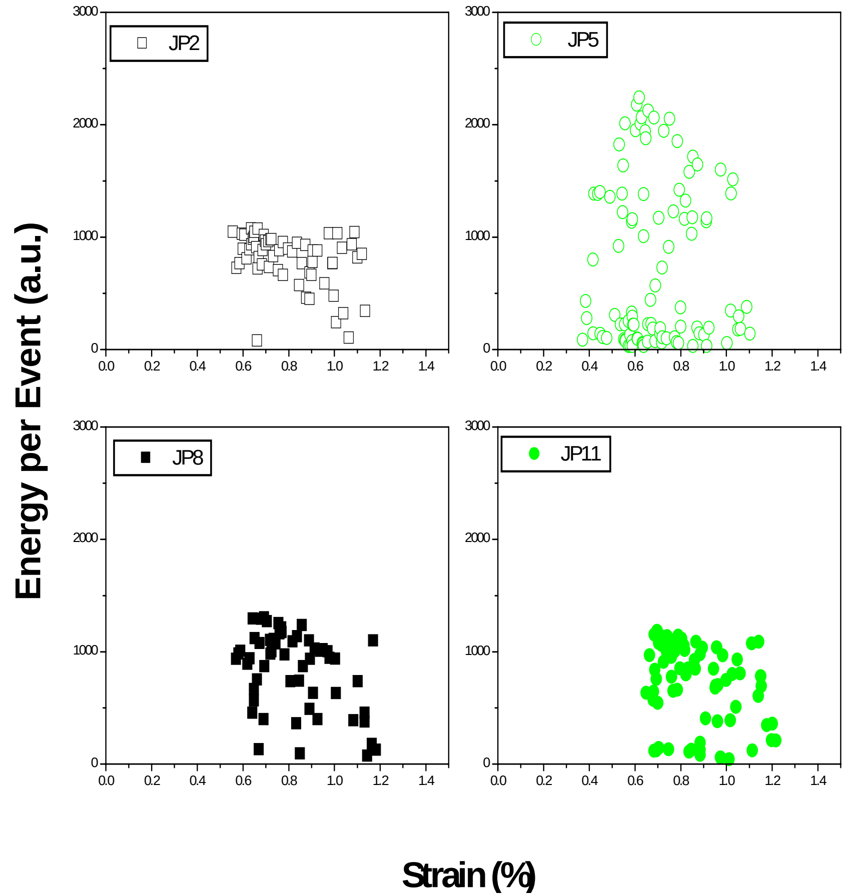
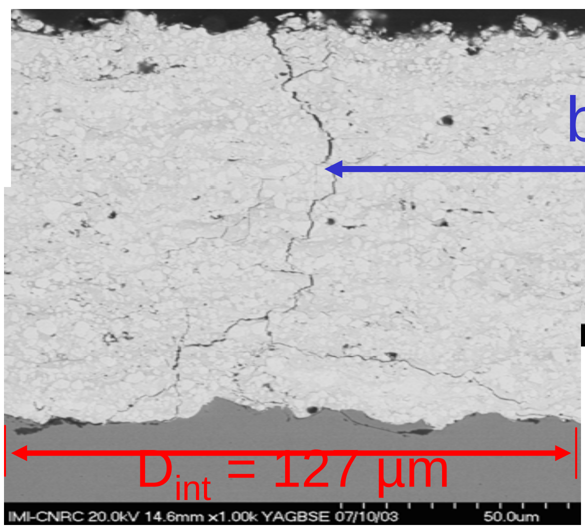


Characterization of Crack Propagation

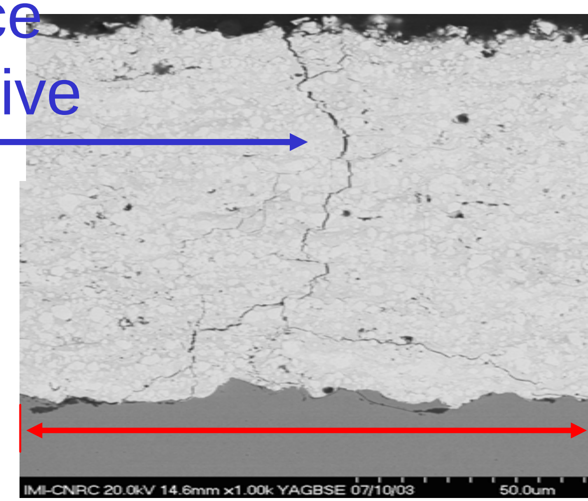
D Mean Distance Between Two Successive Cracks
Dint Distance of propagation at interface
Is Ratio of “Dint/D”, (cracks join together at ratio of 1 for spalling)



Average distance between successive cracks

D = 635 µm

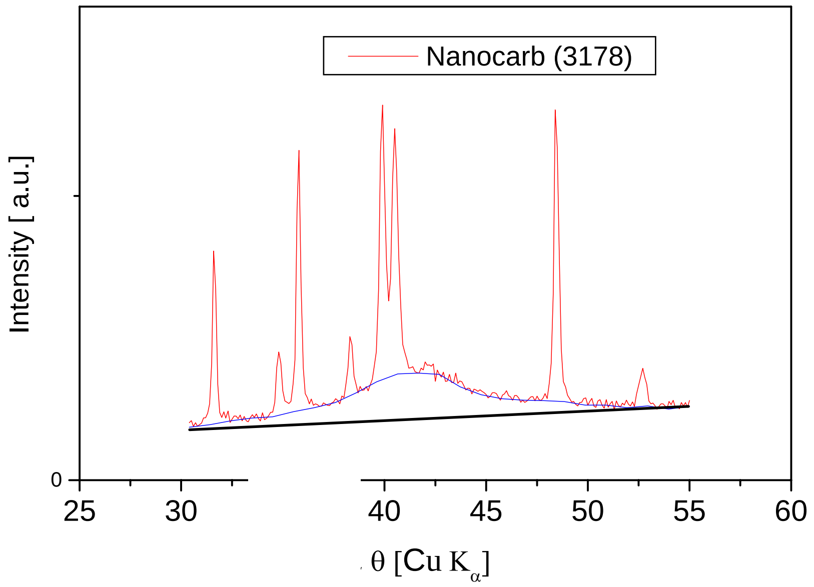
$Is = D_{int}/D = 0.2$



Characterization of Coatings

- X-Ray Diffraction
  - Phase Quantification
  - Amorphous Content (Ic)

    It is the ratio between the areas of the Bragg peaks (crystalline material red - blue) and the total areas of the spectrum for a specific range of 2θ



Cyclic Testing

- Crack initiation or threshold testing
- Coating integrity/spalling threshold testing
- Fatigue testing

			Step Loading			Fatigue Testing at 160/190 ksi				Fatigue Testing at 160/190 ksi				Fatigue Testing at 170/190 ksi			
Condition	S/N	Cracking Initiated	S/N	Highest Stress at Delamination or Spalling	Observed	S/N	Cycles	Cracks First Observed	Observations at Failure	S/N	Cycles	Cracks First Observed	Observations at Failure	S/N	Cycles	Cracks First Observed	Observations at Failure
JK																	
JK #2	41	180	42	240	fractured at 3895 cycles	43	23,079	150 cycles	Spalled adjacent to fracture plane at time of failure	44	27,514	60 cycles	Spalled adjacent to fracture plane at time of failure				
JK #4	48	180	49	240	delaminated, specimen fractured at 3210 cycles while being photographed	50	22,279	no cracks at 9900	Spalled adjacent to fracture plane at time of failure	51	51,919	40 cycles	Spalled adjacent to fracture plane at time of failure				
JK #5	54	180	53	240	delaminated, spalled away from fractur plane at 3673 cycles	55	49,976	100 cycles	Spalled adjacent to fracture plane at time of failure	58	59,430	50 cycles	Spalled adjacent to fracture plane at time of failure				
JK #4 Thick	59 & 60	170		--	not reloaded-no delam testing	61	40,472	54 cycles	Spalled outside fracture plane at time of failure	62 (1)	158,252	790 cycles	Vertical crack and delamination at cycles shown caused test to be stopped. NO FRACTURE				
DJ																	
Boeing	13	200-210	4	240	delaminated, but did not separate/spall	17	12,274	500 cycles	Spalled adjacent to fracture plane at time of failure	65	12,728	3400 cycles	Spalled adjacent to fracture plane at time of failure				
Sulzer	27	230	15	240	fractured at 1909 with significant spalling at time of fracture	36	127,069	2300 cycles	Spalled outside fracture plane at time of failure	80	130,735	1900 cycles	Coating spalled in transition area - test stopped				
Hitemco	14	190-200	12	240	spalling at lower 'blend' region	66	14,146	70 cycles	Spalled outside fracture plane at time of failure	69	11,248	125 cycles	Spalled in one transition area and outside fracture plane at time of failure	90 (2)	18,397	3000 cycles	three cracks in coating observed at 8200 cycles - coating spalled adjacent to fracture plane at time of failure
Hitemco thick	22 & 75	190		--	both specimens spalled - one worse than the other - neither reloaded	87 (1)	4,644	no cracks at 1900	Spalling of coating prompted operator to stop test NO FRACTURE	92 (1)	10,520	--	At 1300 cycles, no cracks were observed - testing was stopped due to major spallation observed when operator returned from lunch NO FRACTURE				
JP																	
Jerry	79	180	2	240	delamination noticed at ~200 cycles - test was continued to failure at 3810 cycles with 'banding' of coating at time of failure	94	60,106	40 cycles	Evidence of delamination at time of failure NO FRACTURE	96	29,950	35 cycles	Evidence of delamination caused test to be stopped NO FRACTURE				
Praxair	81	190	70	230	coating cracked at 35 cycles into 190 ksi run - specimen reloaded - coating spalled 42 cycles into 230 ksi run stopping test	86	12,205	60 cycles	Spalled outside fracture plane at time of failure	88	12,791	50 cycles	Coating spalled at time of fracture				
Hill	37	180	21	230	delamination at 230 ksi - test stopped by PE even though coating appears to be cracked only	82	21,289	1300 cycles	Spalled adjacent to fracture plane at time of failure	85	24,800	48 cycles	Coating spalled at time of fracture	93 (2)	35,144	1300 cycles	multiple cracks observed - coating spalled adjacent to fracture plane at time of failure
Hill Thick	8 & 28	160		--	spalling is imminent on one of two specimens tested - neither reloaded	72 (1)	4,133	45 cycles	Multiple cracks in various directions implying delamination - test stopped NO FRACTURE	76 (1)	38	38 cycles	Major coating spall occurred at cycles indicated - test stopped NO FRACTURE				

(1) This test was run at 160 ksi
(2) This test was run at 170 ksi

What questions do we hope to answer with the Process Mapping Data?

- Within the parameter sets used in the HCAT community, is there process robustness and consistency?
- Does the characterization of property techniques produce data which shows significant differences between the varied HVOF parameter sets?
- Is there a relationship between coating characterization data and coating performance?
- Can we logistically and economically use the characterization procedures as QC /process qualification tools for future HCAT and HVOF work?

Robust Process
Within HCAT?

Robust Process?
HCAT Only Data

	Step Loading					Fatigue Testing at 190 ksi				Fatigue Testing at 190 ksi				Bend test			IC		Hardness
Condition	S/N	Cracking Initiated-test stopped	S/N	After cracking observed, Highest Stress at Delamination or Spalling	Observed	S/N	Cycles	Cracks First Observed	Observations at Failure	S/N	Cycles	Cracks First Observed	Observations at Failure	Onset of cracking of cracking (5 mil coating)	distance between cracks (μm)	AE cumul energy		Almen A (mils)	HV (300g)
JK																			
JK #2	41	180	42	240	fractured at 3895 cycles	43	23,079	150 cycles	Spalled adjacent to fracture plane at time of failure	44	27,514	60 cycles	Spalled adjacent to fracture plane at time of failure	0.291	0.585	60047	0.565	6.4	1167
JK #4	**48**	180	49	240	delaminated, specimen fractured at 3210 cycles while being photographed	50	22,279	no cracks at 9900	Spalled adjacent to fracture plane at time of failure	51	51,919	40 cycles	Spalled adjacent to fracture plane at time of failure	0.268	0.532	34082	0.484	13	1252
JK #5	54	180	53	240	delaminated, spalled away from fracture plane at 3673 cycles	55	49,976	100 cycles	Spalled adjacent to fracture plane at time of failure	58	59,430	50 cycles	Spalled adjacent to fracture plane at time of failure	0.18	0.619	58419	0.517	7.2	1222
DJ																			
Boeing	**13**	200-210	4	240	delaminated, but did not separate/spall	17	12,274	500 cycles	Spalled adjacent to fracture plane at time of failure	65	12,728	3400 cycles	Spalled adjacent to fracture plane at time of failure	1.036	0.911	16911	0.418	18.7	1159
Hitemco	14	190-200	12	240	spalling at lower 'blend' region	66	14,146	70 cycles	Spalled outside fracture plane at time of failure	69	11,248	125 cycles	Spalled in one transition area and outside fracture plane at time of failure	0.38	1.573	61986	0.509	10.2	1169
JP																			
Jerry	79	180	2	240	delamination noticed at ~200 cycles - test was continued to failure at 3810 cycles with 'banding' of coating at time of failure	94	60,106	40 cycles	Evidence of delamination at time of failure NO FRACTURE	96	29,950	35 cycles	Evidence of delamination caused test to be stopped NO FRACTURE	0.371	0.7	65933	0.695	11.32	
Hill	**37**	180	21	230	delamination at 230 ksi - test stopped by PE even though coating appears to be cracked only	82	21,289	1300 cycles	Spalled adjacent to fracture plane at time of failure	85	24,800	48 cycles	Coating spalled at time of fracture	0.5345	0.695	38321	0.689	13.9	
					Not similar to other trends														

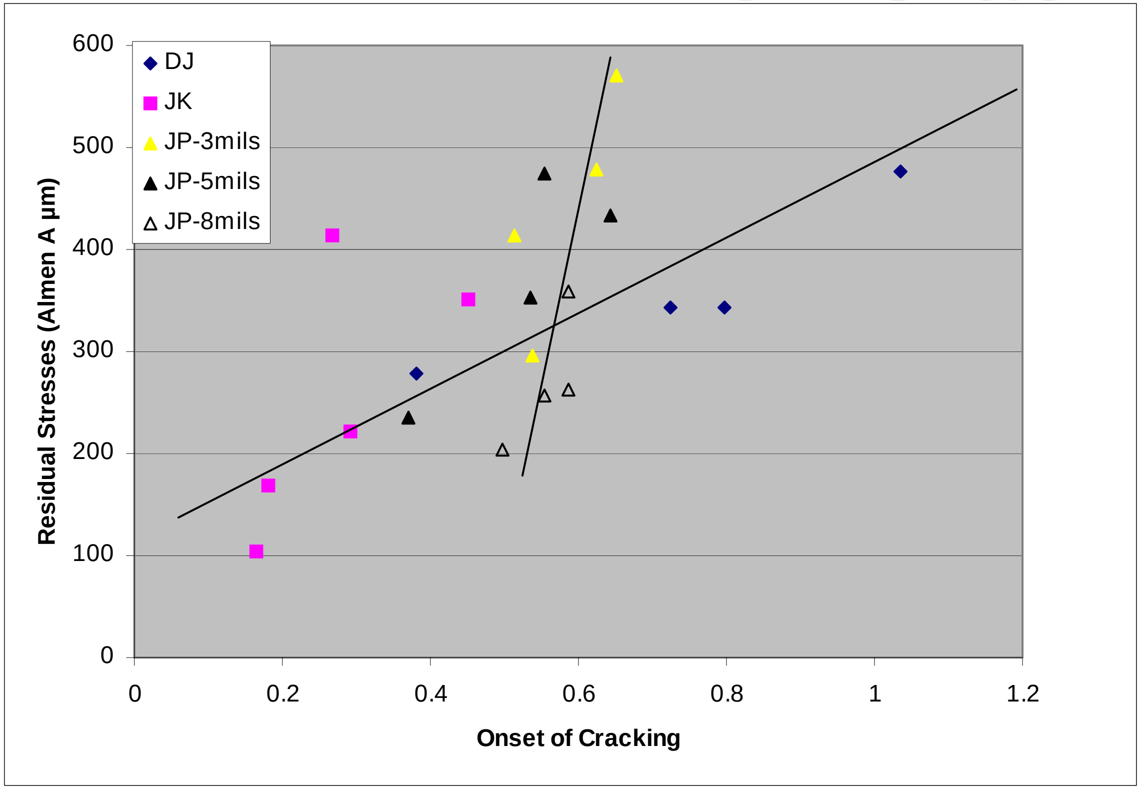
Is the HVOF Process within HCAT Robust?

- All HCAT parameter sets included in study
- Cracking threshold 180-190 ksi
- Spalling did not occur until 240 ksi
- No significant variation between DJ, JP, and JK systems

HVOF Process Robust with in the HCAT Program

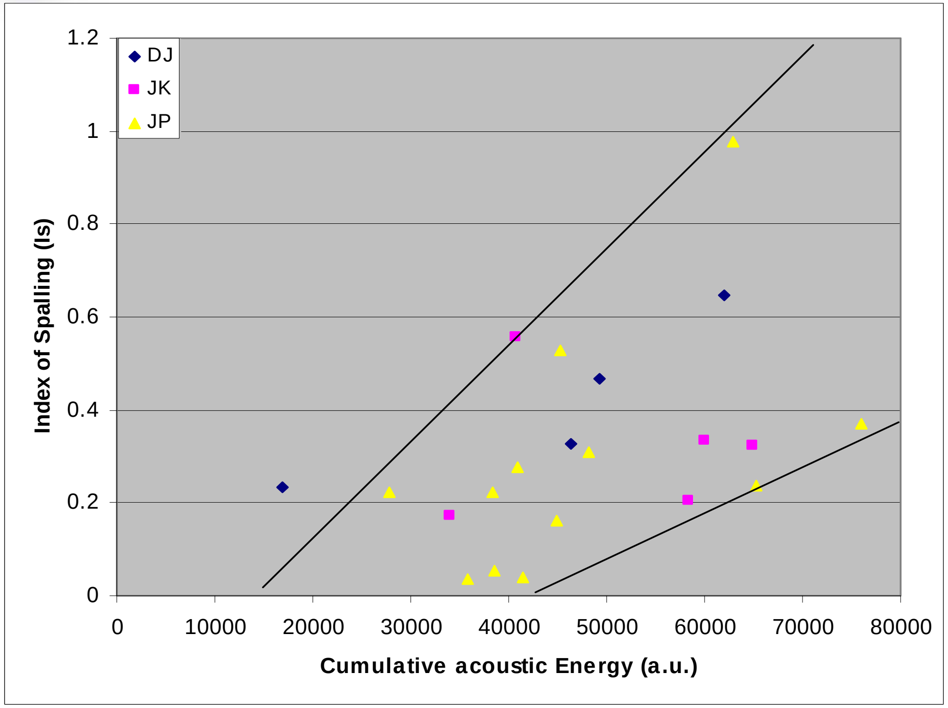
Does Coating Characterization show Differences between the Processes?

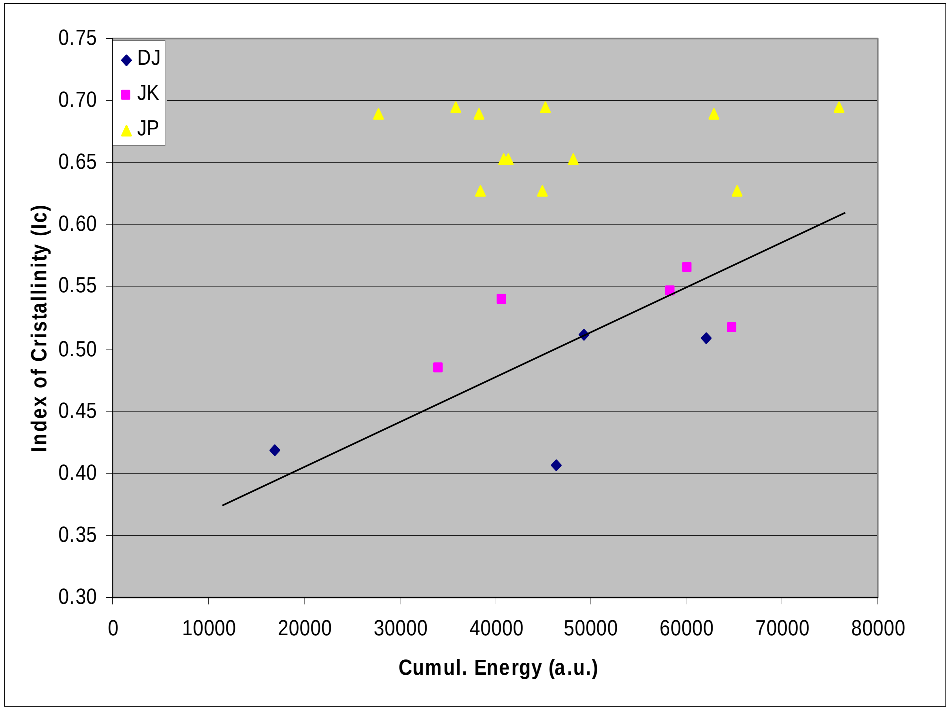
Effect of Residual Stresses on Crack Initiation



- Increasing compressive residual stress delay the onset of cracking.
- The effect is gun or powder dependant

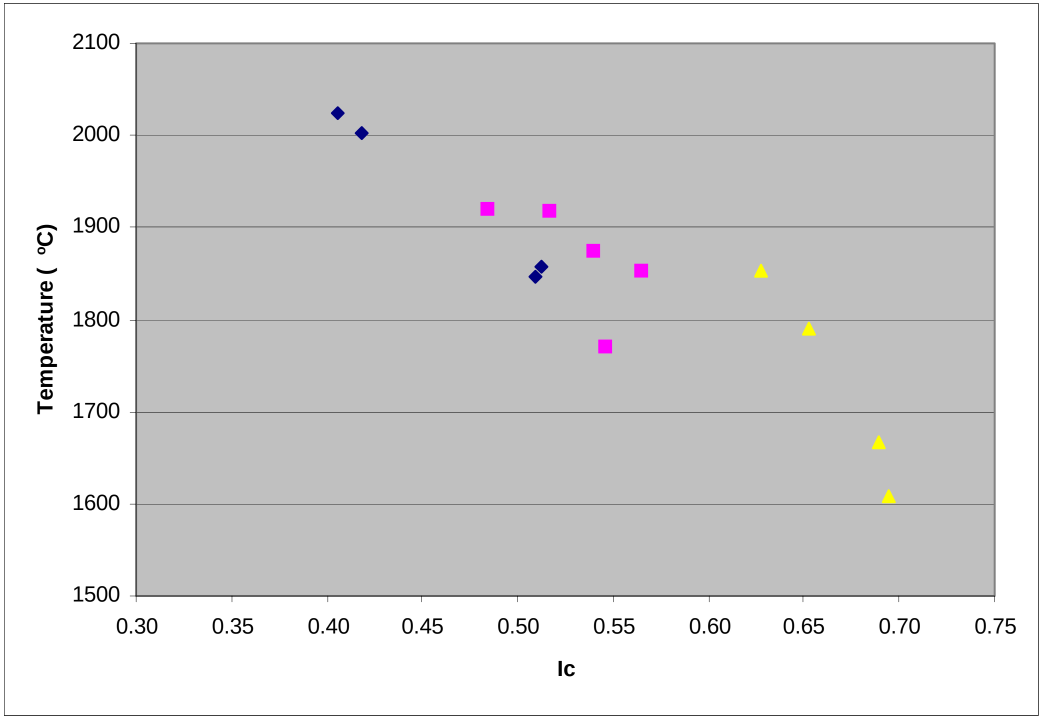
Acoustic Emission
(Relationship to Microstructure)





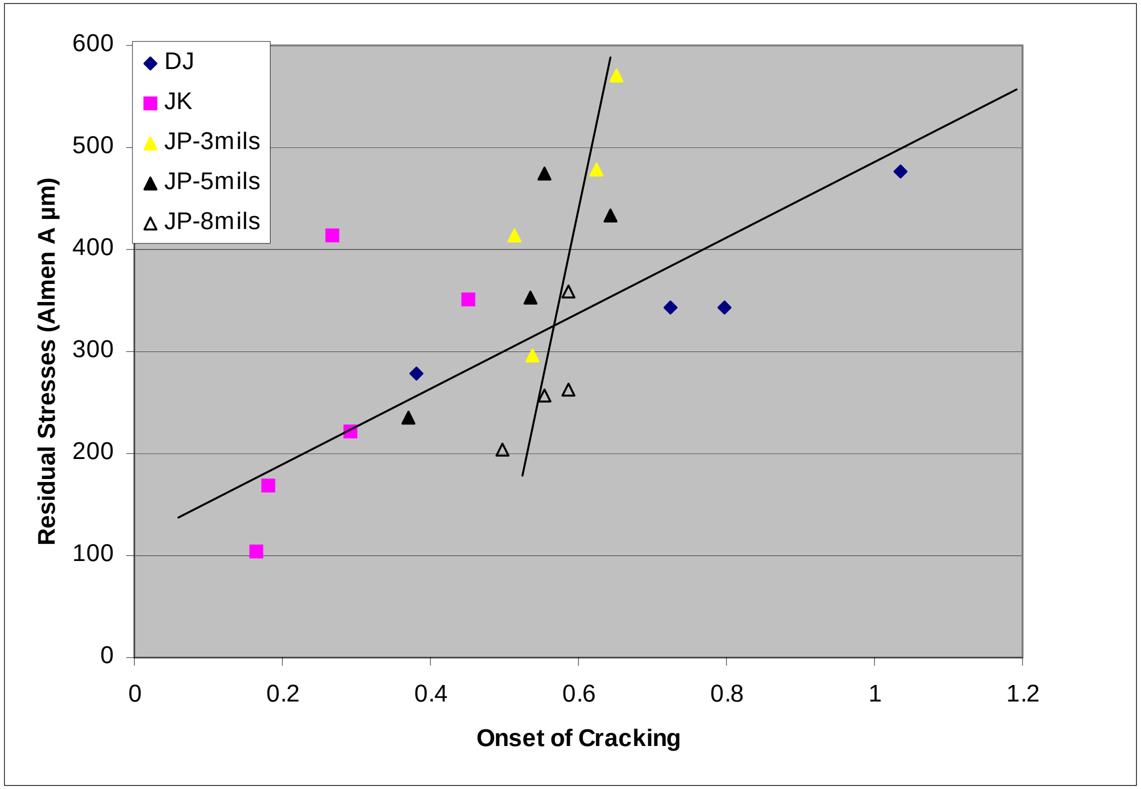
- A coating more prone to spalling produces longer cracks and thus releases more energy
  - **Is** Ratio of "Dint/D", (cracks join together at ratio of 1 for spalling)
- More crystalline coatings tend to have a higher propensity to spall and thus again releases more energy.

Effect of Particle Temperature on Structure



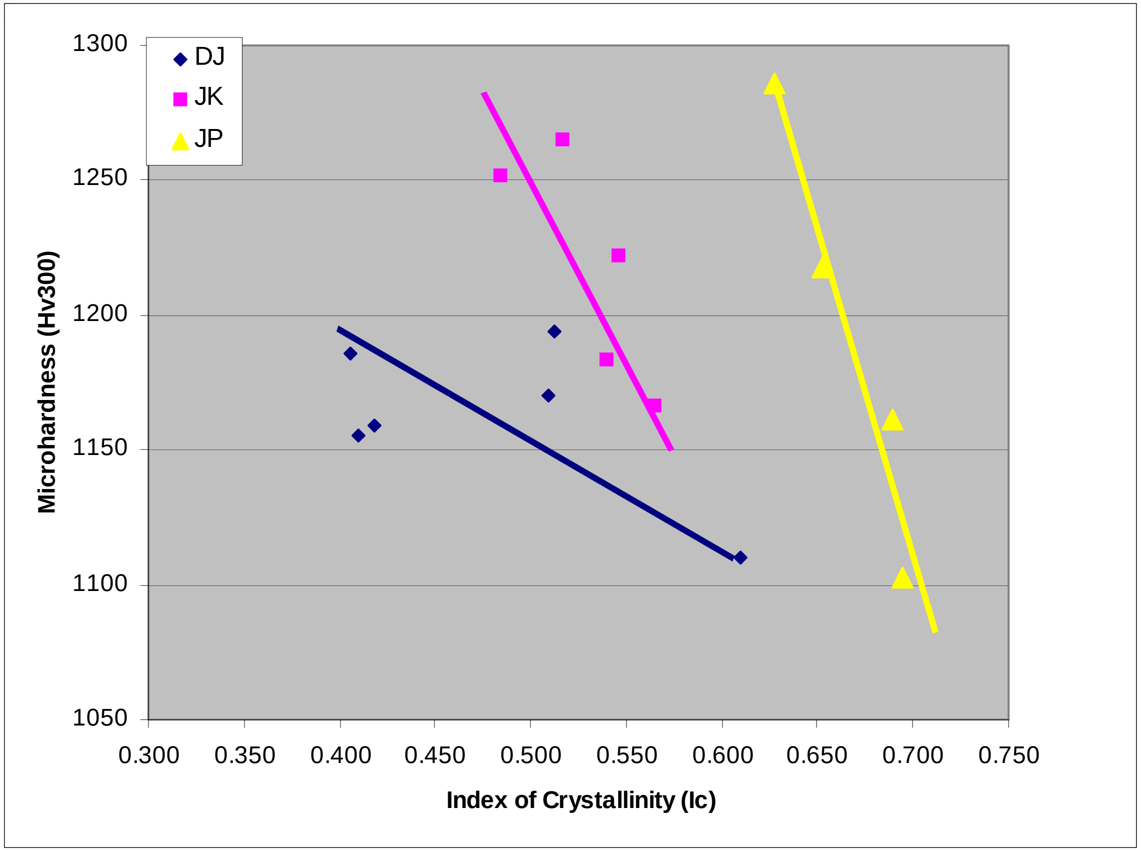
- There is a strong correlation between in flight temperature and coating phase content or Amorphous Content (Ic)

Effect of Residual Stresses on Crack Initiation



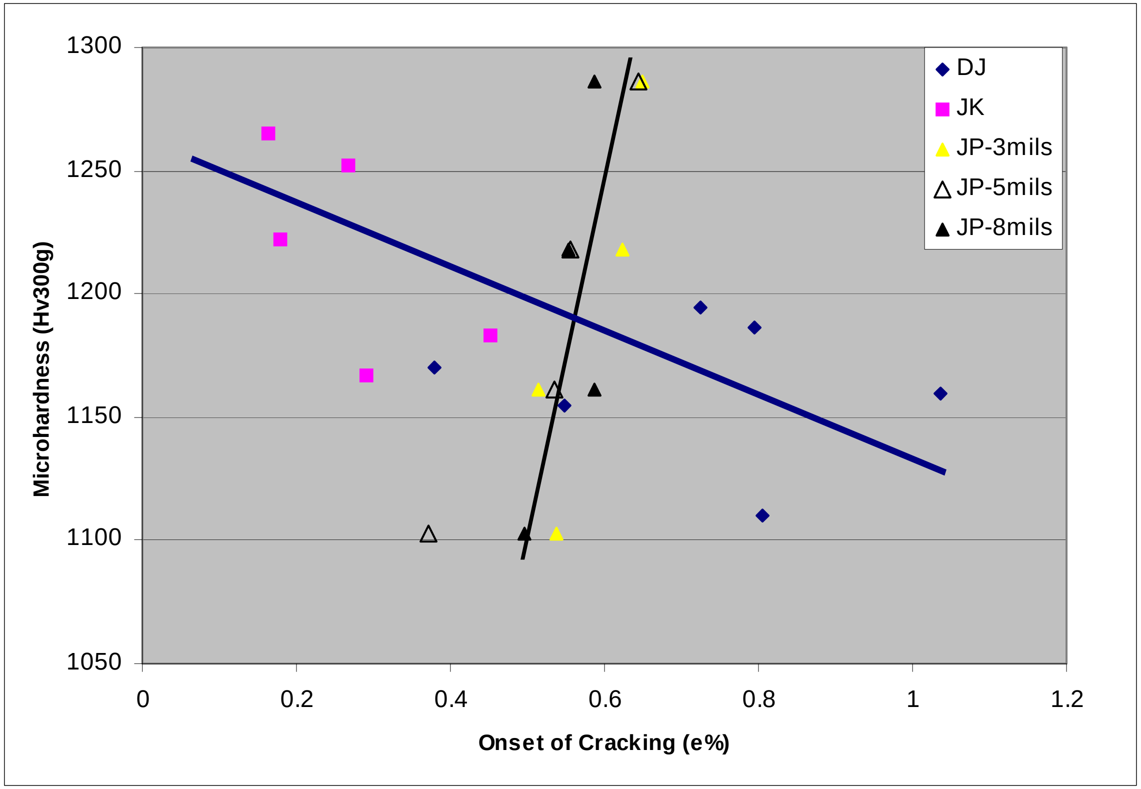
- Increasing compressive residual stress delay the onset of cracking.
- The effect is gun or powder dependant

Effect of Structure on Microhardness



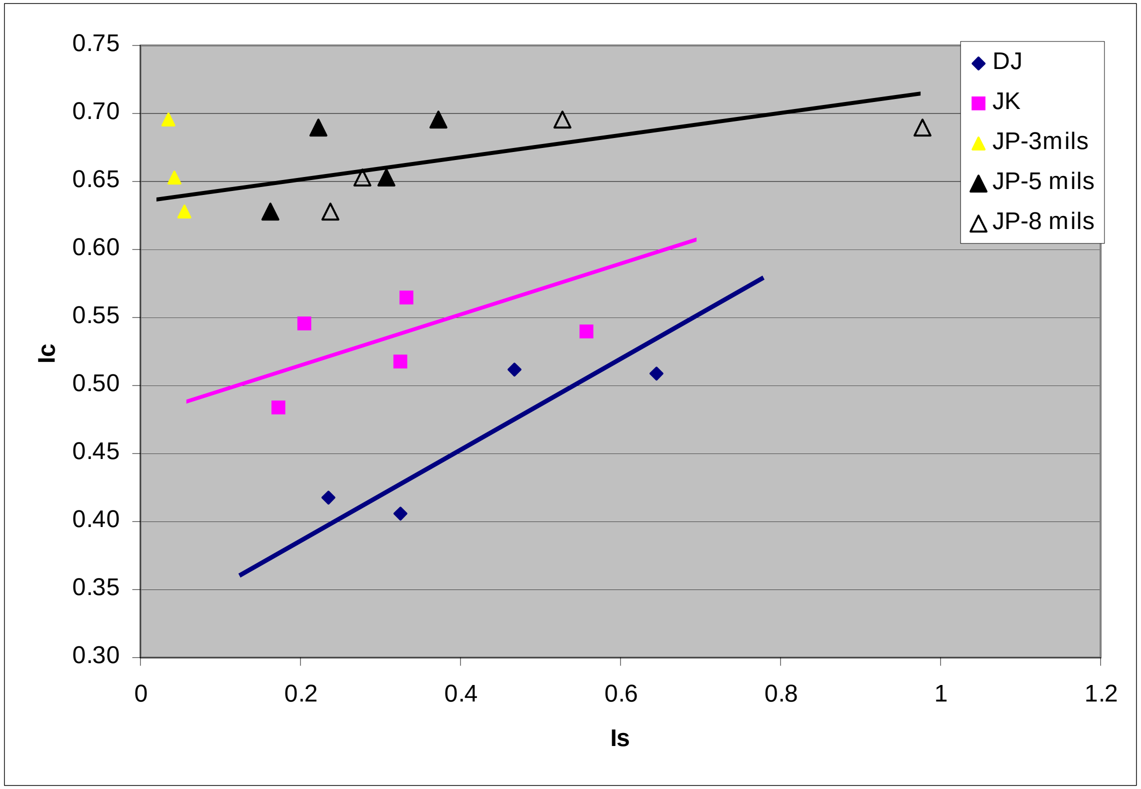
- Coating hardness is strongly related to the phase content.
- The effect is strongly material or gun dependant

Effect of Hardness on Crack Initiation



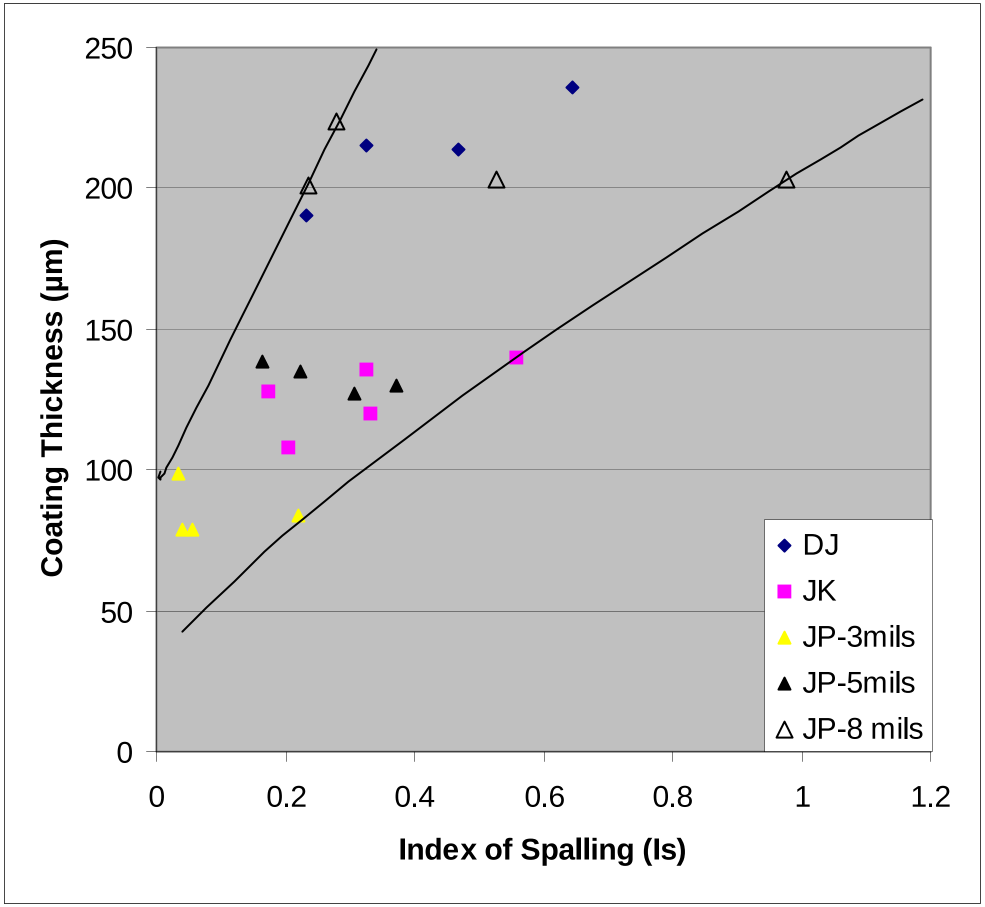
- For the DJ and the JK softer coatings tend to delay crack initiation
- An inverse relation is observed for the JP-5000 indicating a strong gun or material dependence

Effect of Coating Structure on Spalling



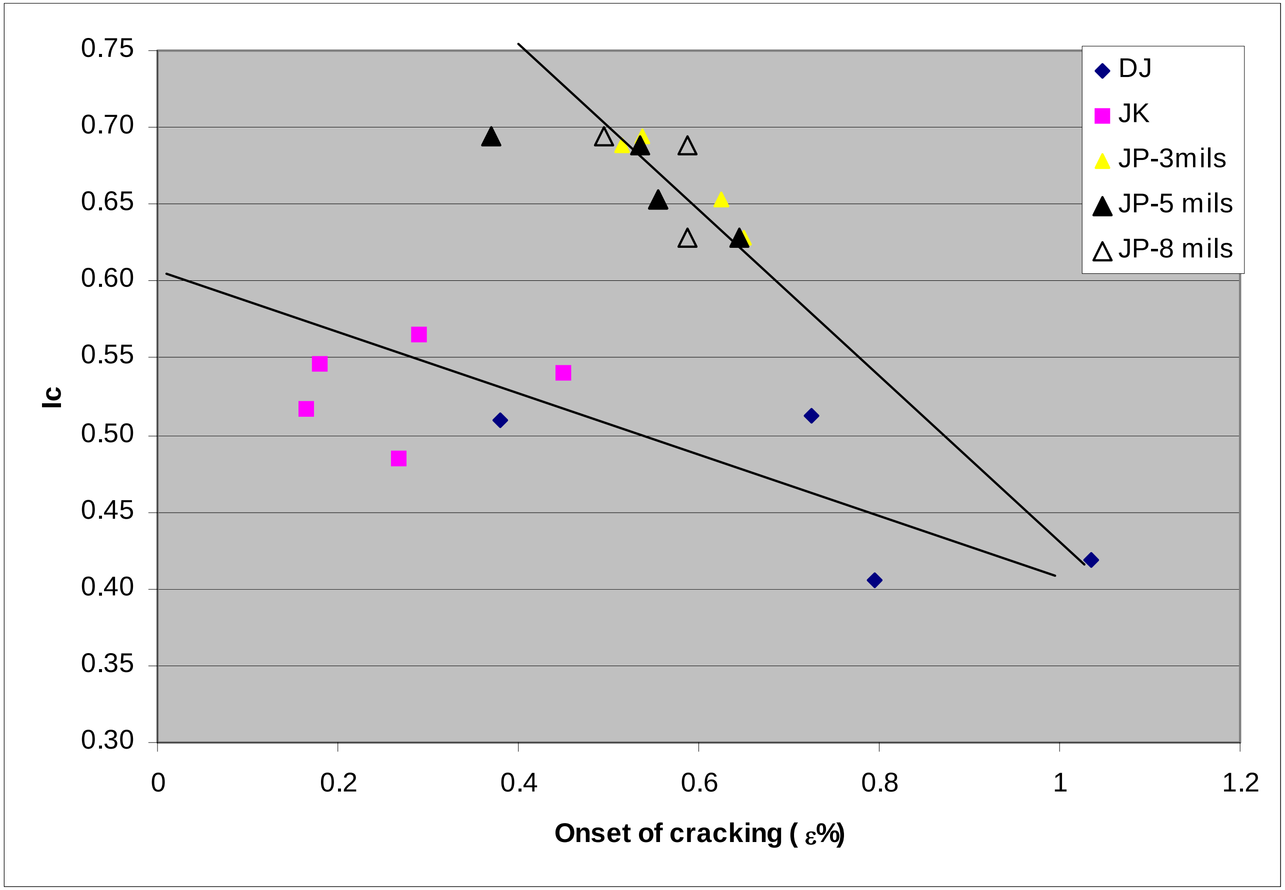
- Higher crystallinity tend to increase spalling for all gun.
- A strong material or gun effect is also observed.

Effect of Coating Thickness on Spalling



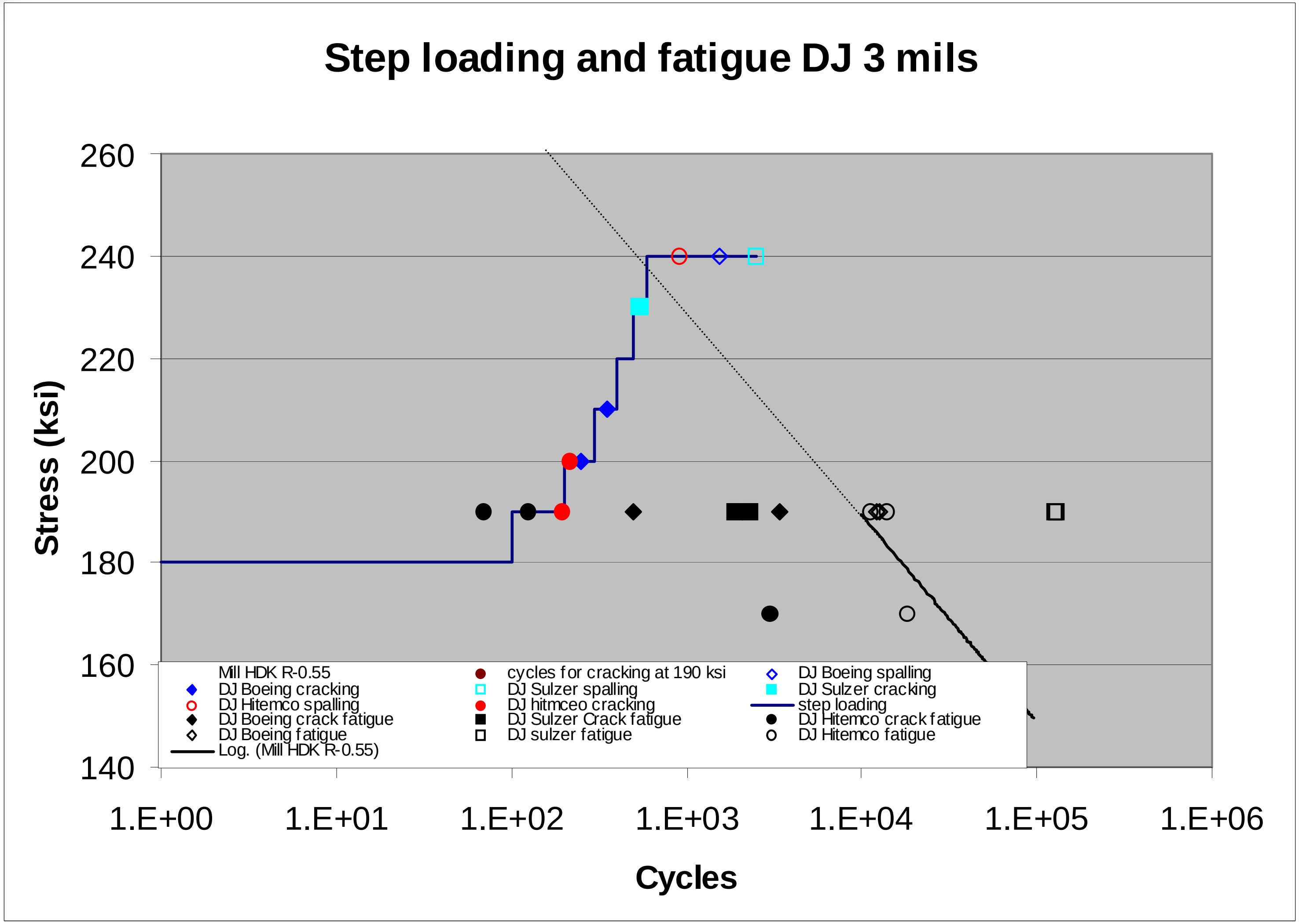
Thicker Coating are more prone to spall

Effect of Structure on Crack Initiation

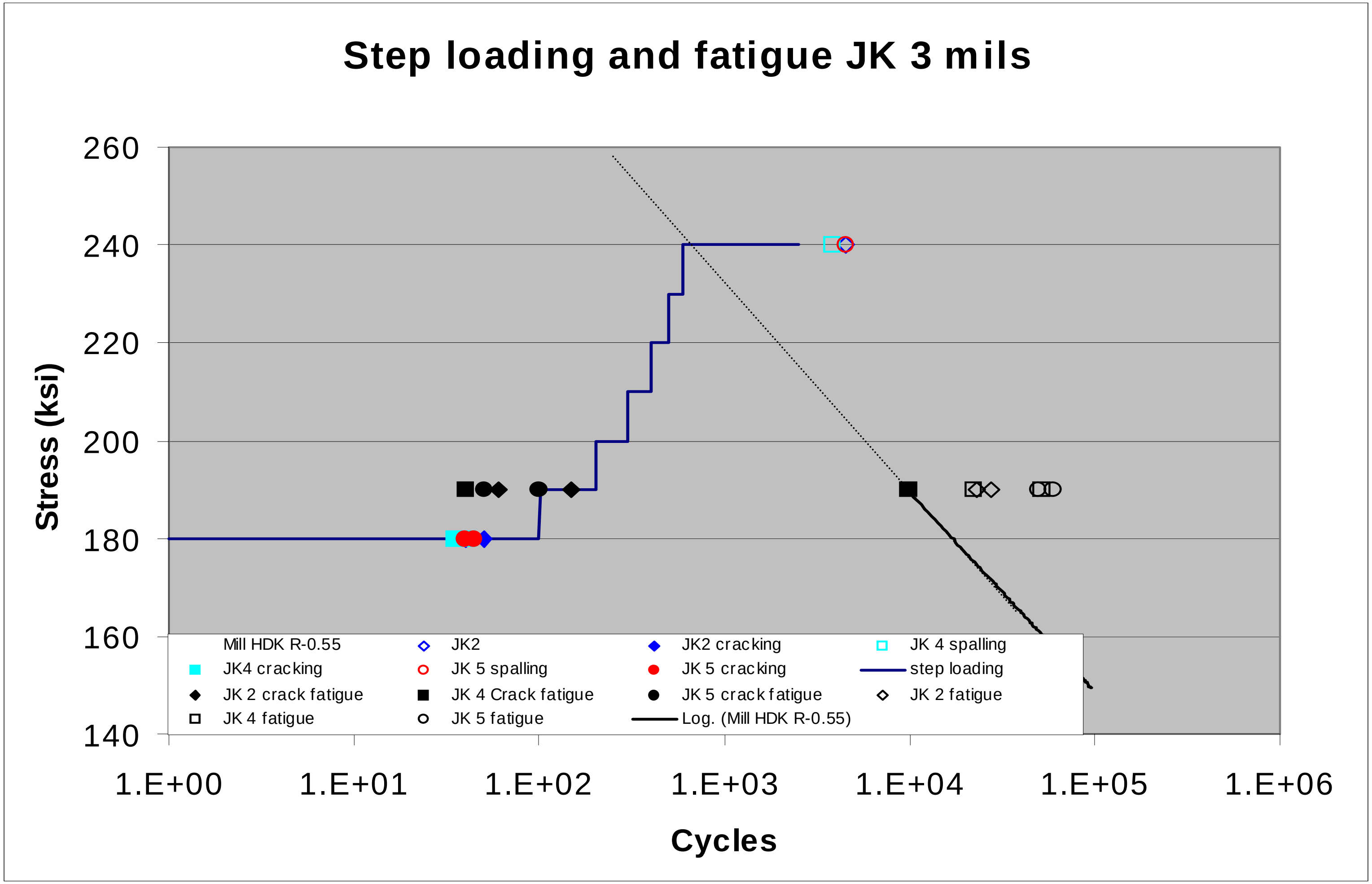


- More crystallinity for coatings tend to favour crack initiation.
- Again there is a strong component related to material or gun

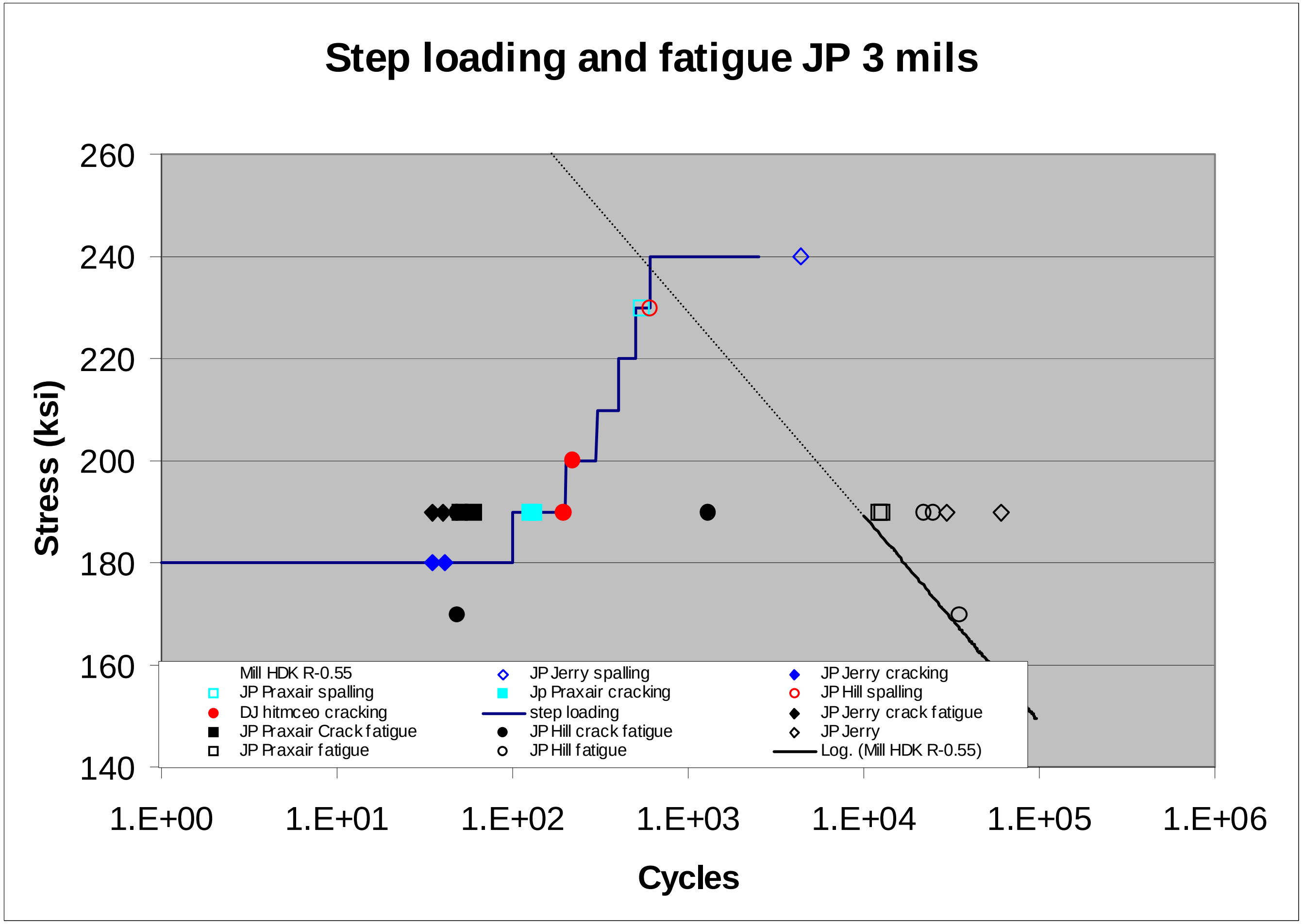
Cyclic Loading DJ



Cyclic Loading Jet Kote



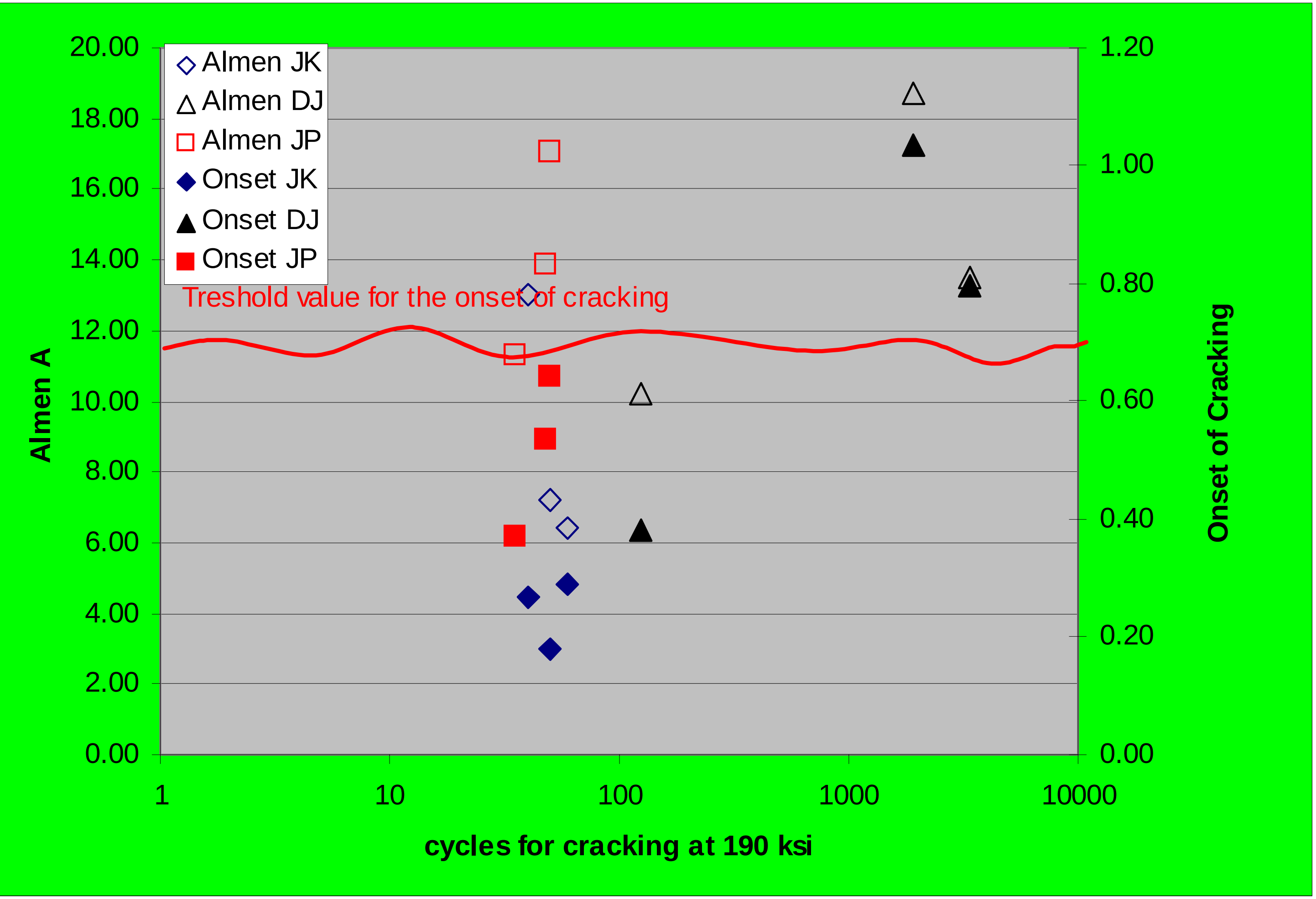
Cyclic Loading JP-5000



General Comments on Cyclic Loading

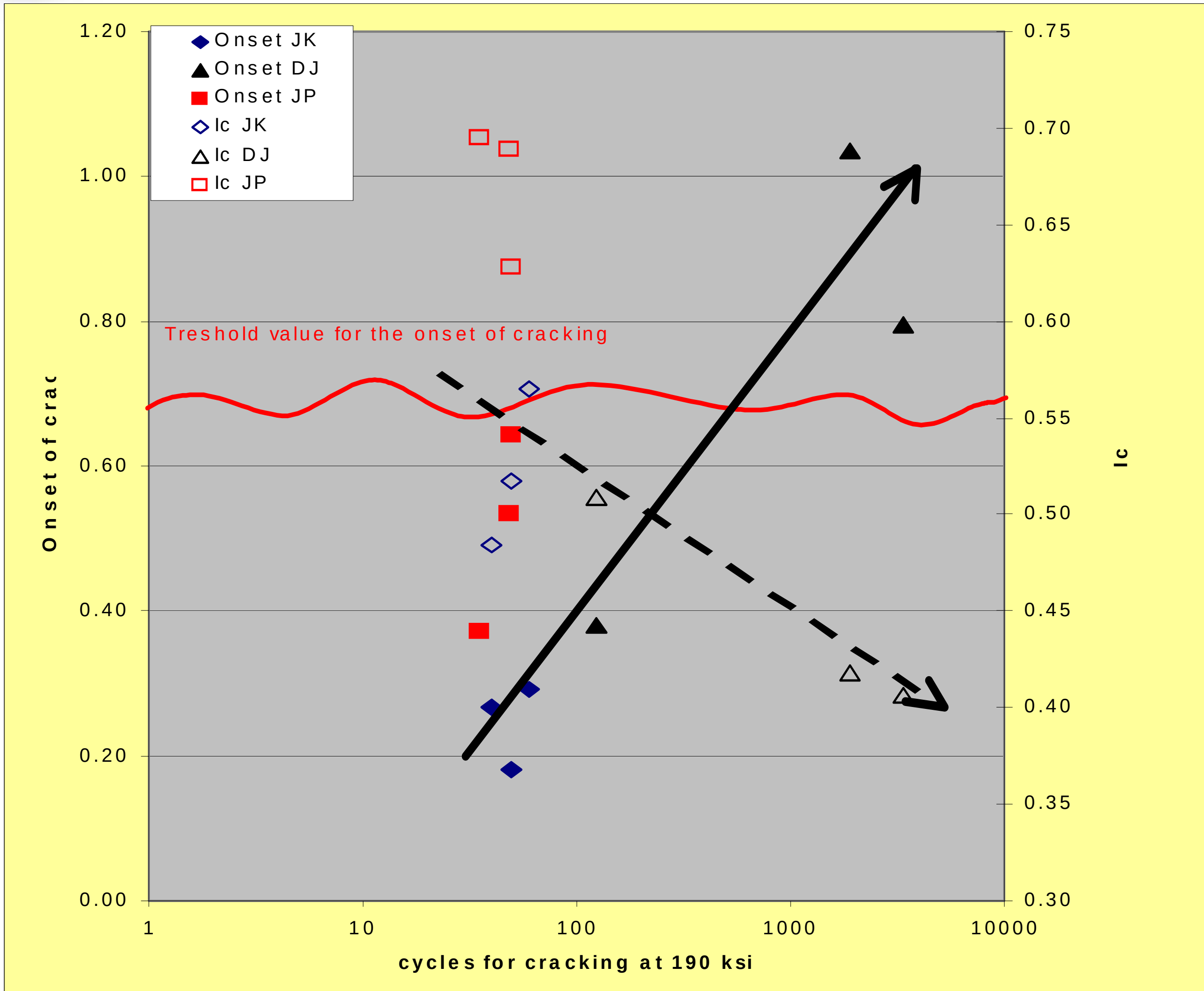
- Coating failure occur in or over the expected fatigue life for all the tested coatings.
- There is no direct relation between the cracking and the spalling of coatings.

Crack initiation in relation to static properties



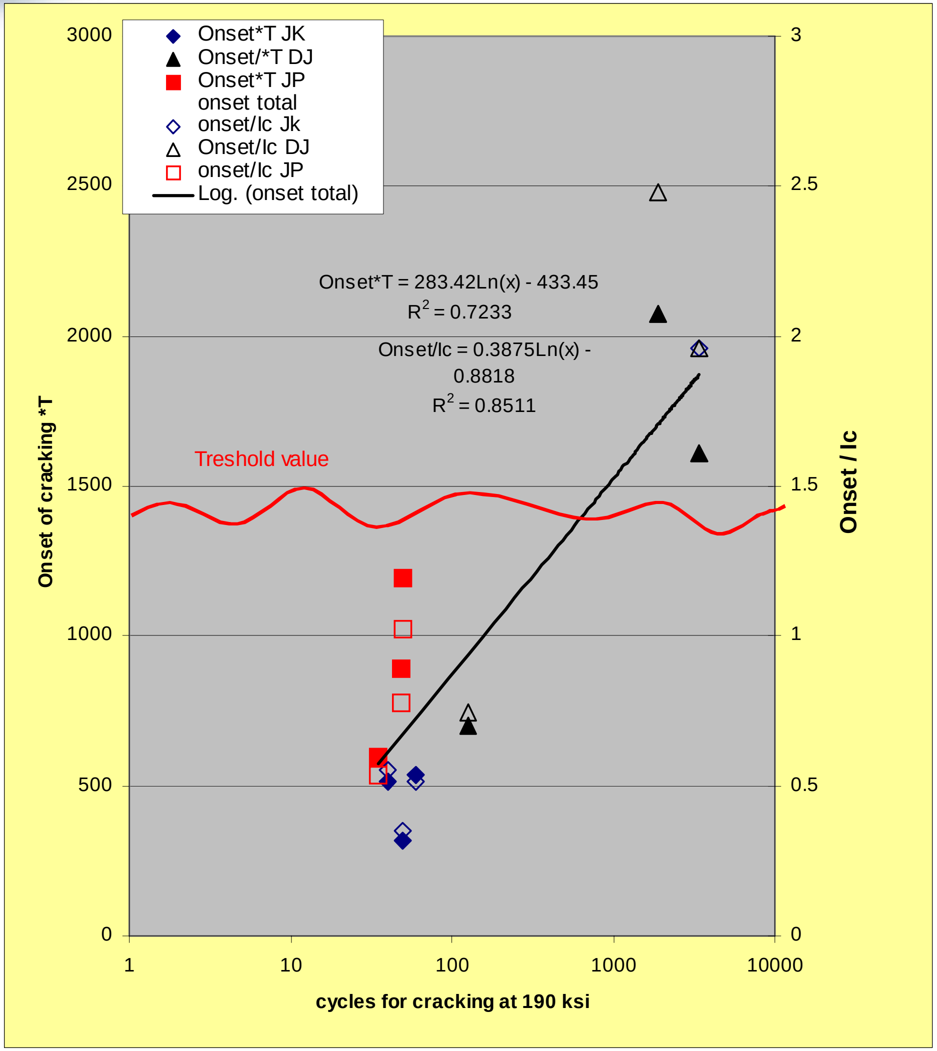
- No relationship between crack initiation and residual stresses
- The onset of cracking seems to have a strong influence on the crack initiation.

Crack initiation in relation to coating structure



- The index of crystallinity and the static onset of cracking seems to have inverse effect on the cyclic crack initiation.

Proposed indicator of cyclic performance



- Combining static bend test results (onset) and coating structure seams to correlate well with the cyclic behaviour of coatings.
- More work is needed to confirm the validity of the indicator.

Can we use Characterization Tools in Qualification and QC Testing?

Qualification and Quality Control

<u>**Test**</u>	<u>***QC***</u>	<u>***Qual***</u>
Temperature and velocity profiles	***Yes***	***Yes***
X-ray diffraction	***No?***	***Yes??***
Bend Test (onset determination only)	***Yes***	***Yes***
Bend testing of Almen strips for crack quantification via acoustic emission	***No??***	***Yes***

What's Next ?

The Process Mapping Program was very thorough but as in all investigative arenas, good research answers many questions but also identifies other areas for more work:

- ***Powder dependency*** The coatings were sprayed with powders as recommended by the manufacturers. What is the dependence on spalling behavior for different powder sizes and manufacturing methods??
- ***Spraying at higher operating temperatures -XRD Dependence*** What will happen to cracking behavior when temperature ($\mathbf{I_C}$)rises.
- ***Fatigue testing at other loads 200-210-220*** What will happen to coating results if fatigue testing is performed at higher loads?
- ***Review cracked specimens*** Some of the fatigue specimens were run to cracking but not spalling. Will investigation of the crack pattern formation help in understanding final spalling resistance??
- **Follow up ongoing experiments** to validate the QC control criteria