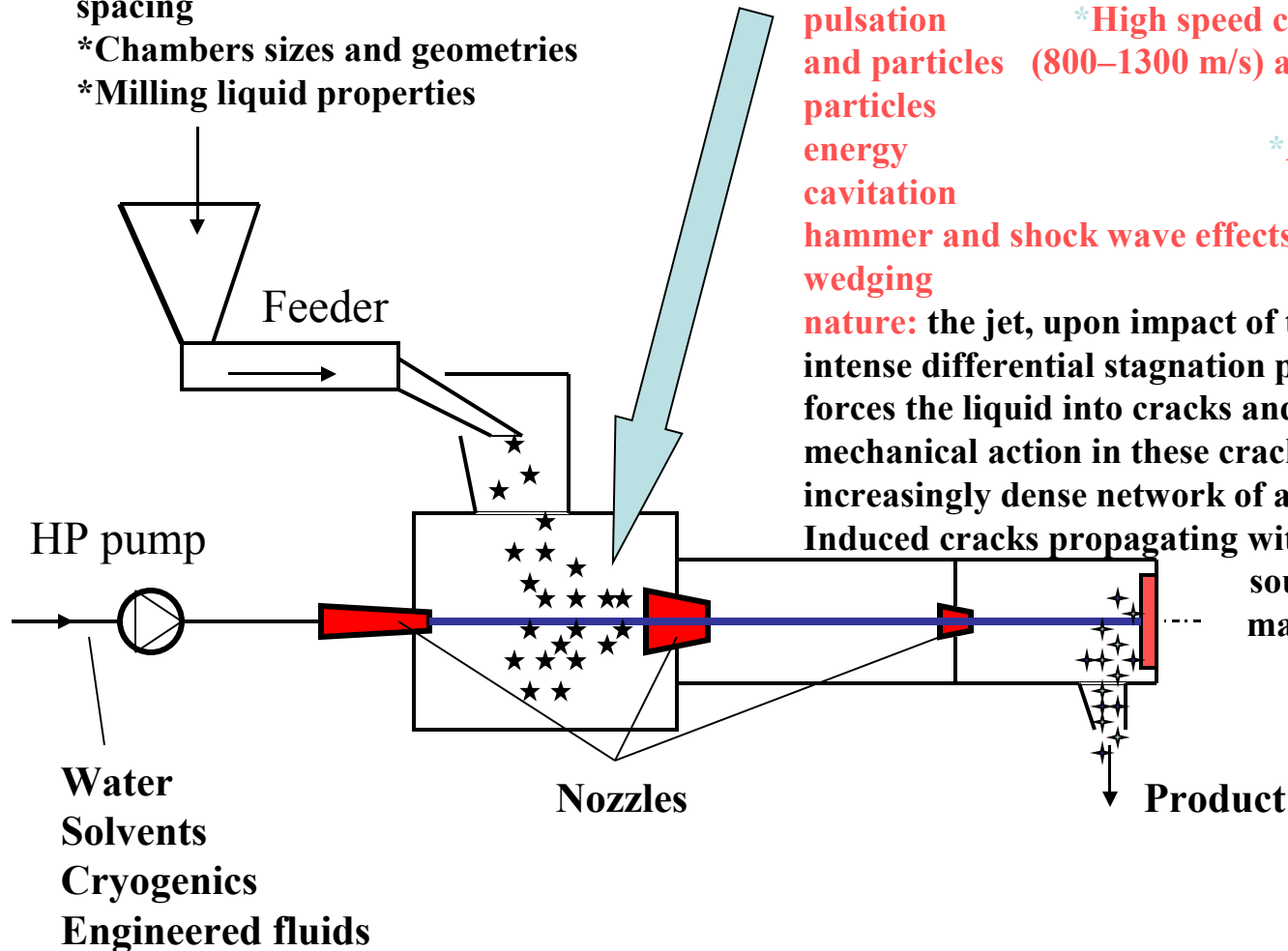


Aktywacja surowców farmaceutycznych i
kosmetycznych w mikro i nanoskali
procesów mechanochemicznych

Process Parameters:

- *Jet power (pressure and volume)
- *Feed rate
- *Nozzles geometry, size and spacing
- *Chambers sizes and geometries
- *Milling liquid properties



Process Physics:

*Tremendous energy concentration and stress application where there are particles

*High turbulence and intensive abrasion

*Ultra-high frequency pressure

*High speed collisions between jet and particles (800–1300 m/s) and between particles

*High shearing energy cavitation

*Aggressive

*Water-hammer and shock wave effects

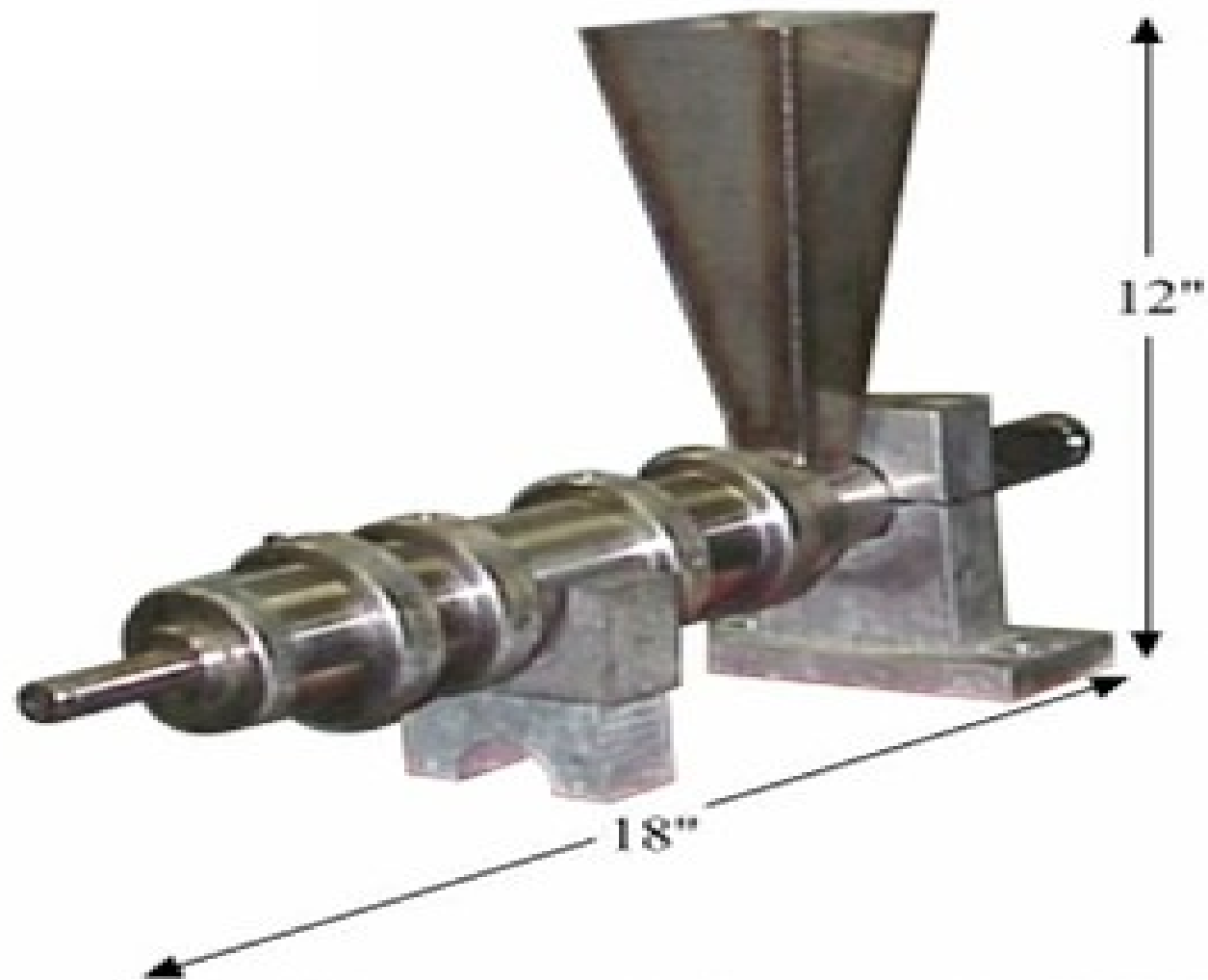
*Hydro-

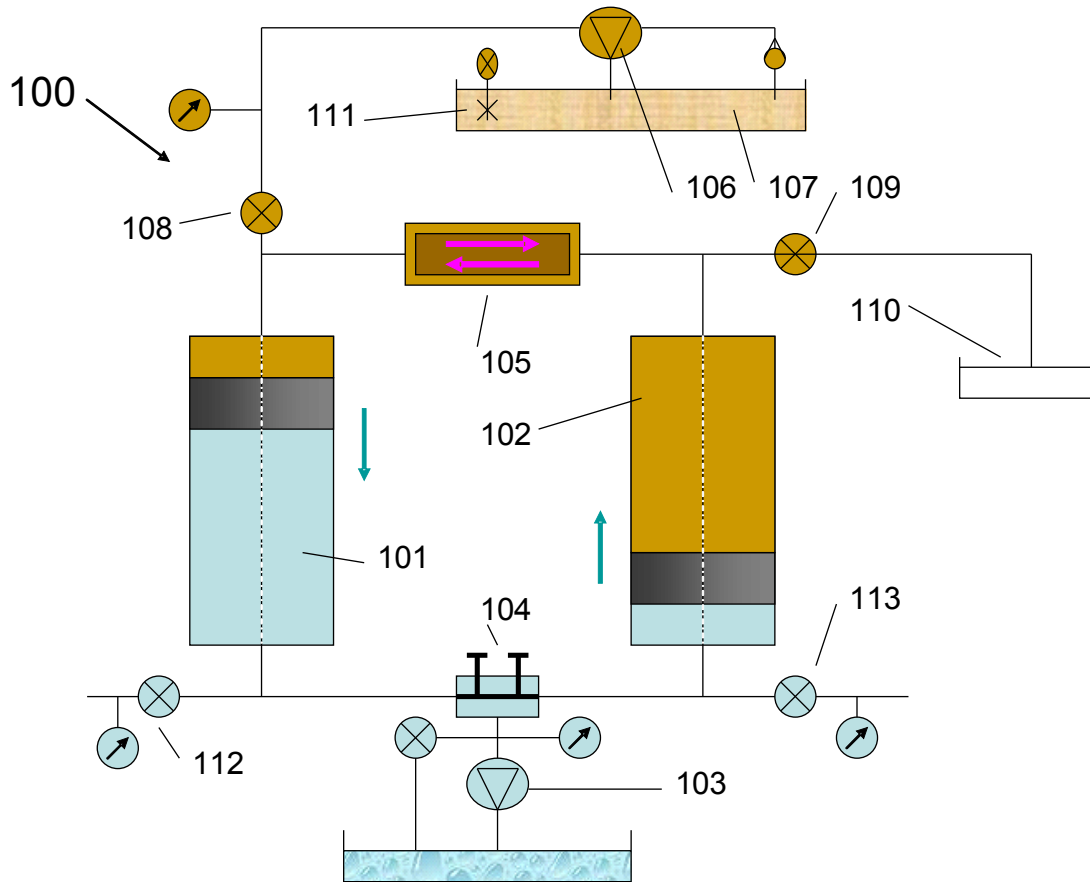
wedging

*Jet's

nature: the jet, upon impact of the target generates intense differential stagnation pressure which forces the liquid into cracks and creates a hydro-mechanical action in these cracks, to form an increasingly dense network of a new cracks.

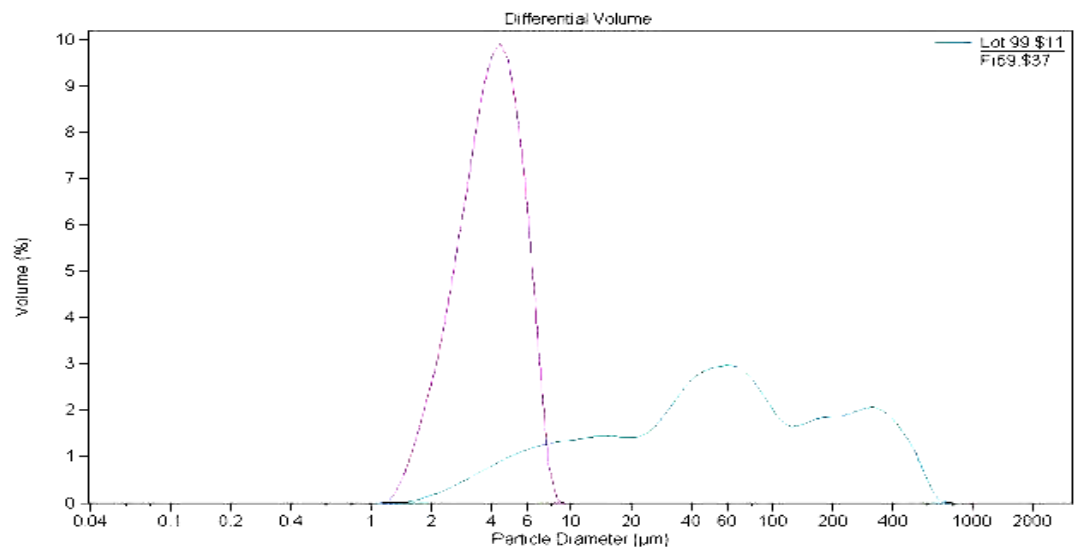
Induced cracks propagating with the speed of sound in particular material.



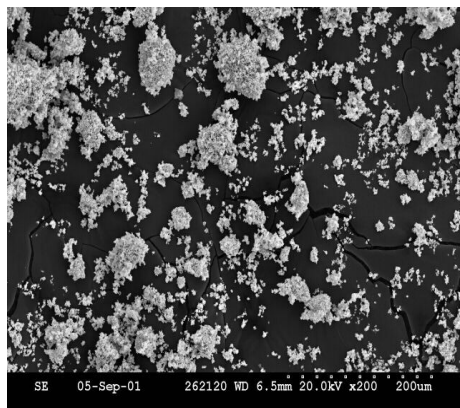


The modified milling system

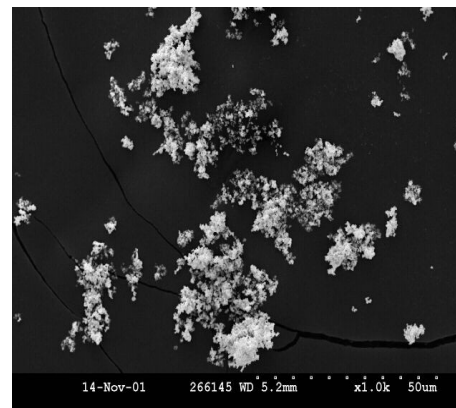
100 – recirculation system, 101 i 102 high pressure cylinders, 103 – high pressure water pump,
 104 – flow control valve , 105 –flow through mill, 106 – slurry supply pump, 107 – slurry tank,
 108 i 109 – cut-off valves, 110 – product tank, 111 – mixer, 112 i 113 - water supply cut-off valves



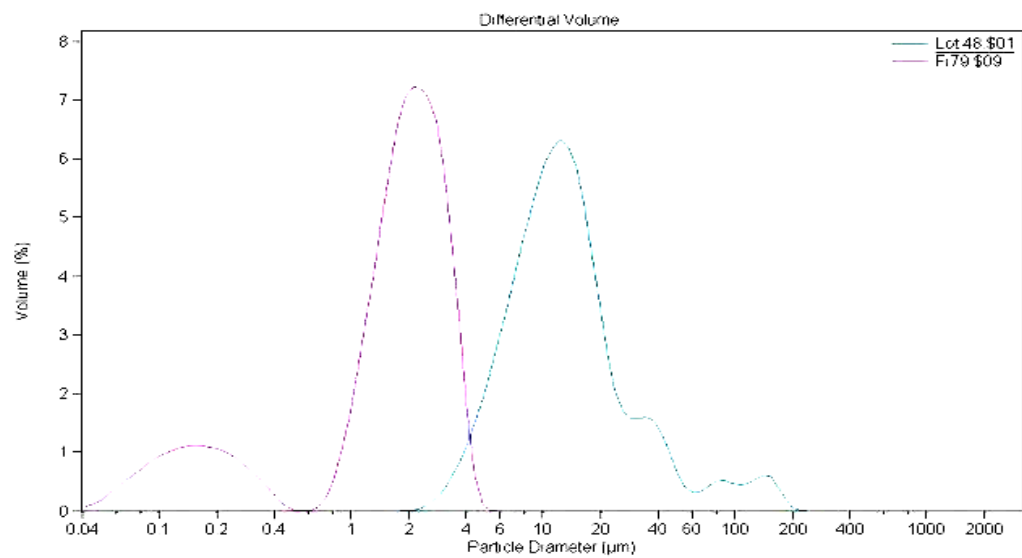
Single Pass Densification of Silver



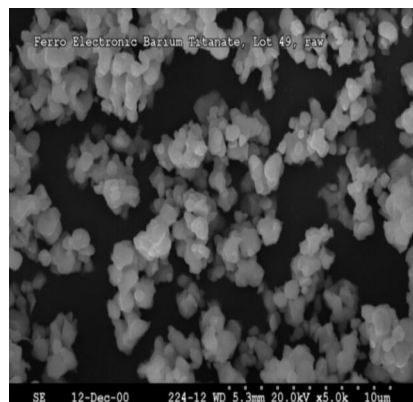
Raw Silver 200x



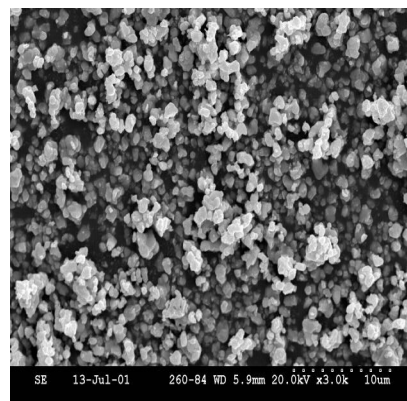
Milled Silver 1k



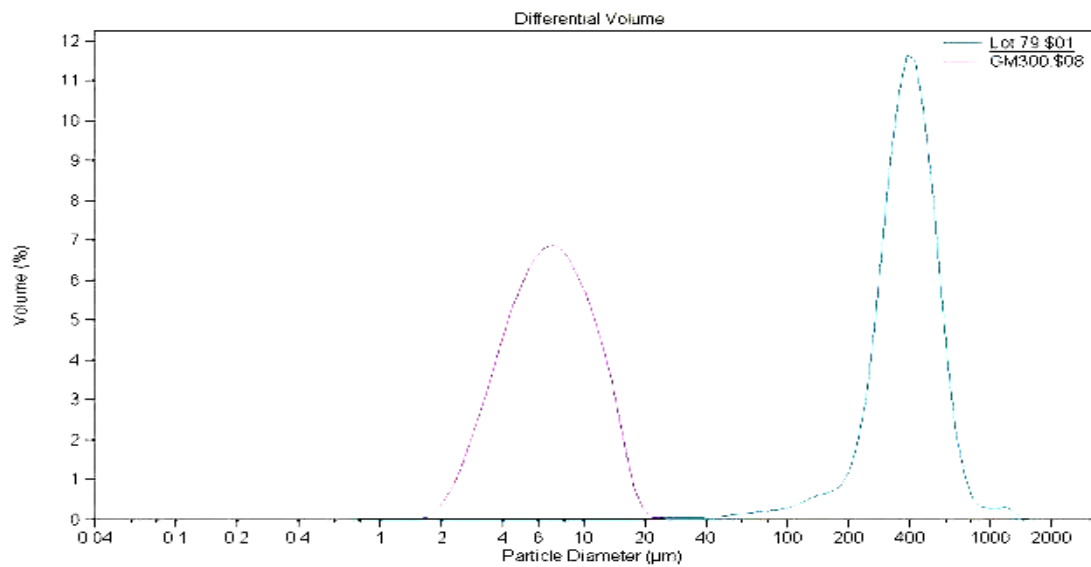
Single Pass Barium Titanate



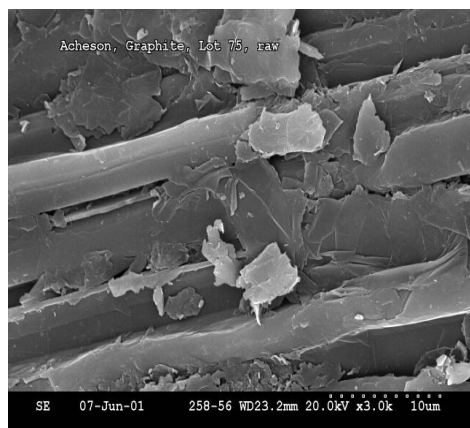
Raw Barium Titanate 5k



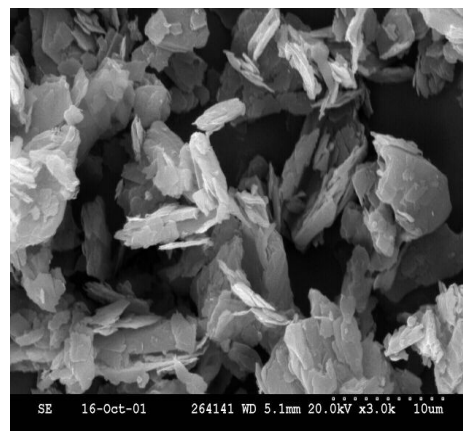
Milled Barium Titanate 3k



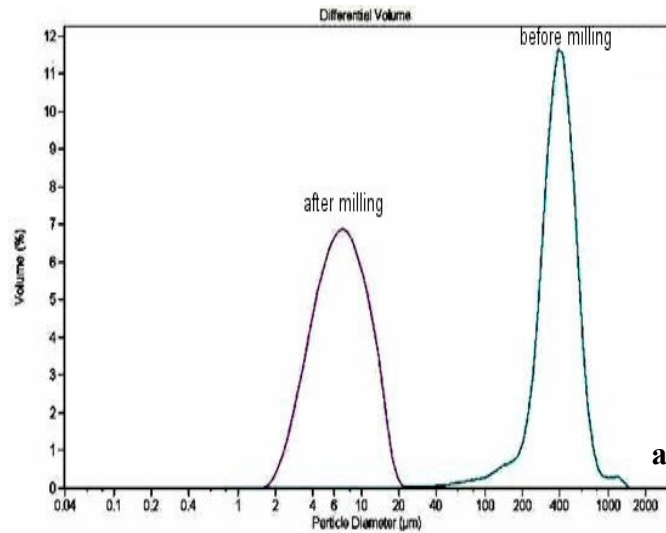
Multi Pass Crystalline Graphite



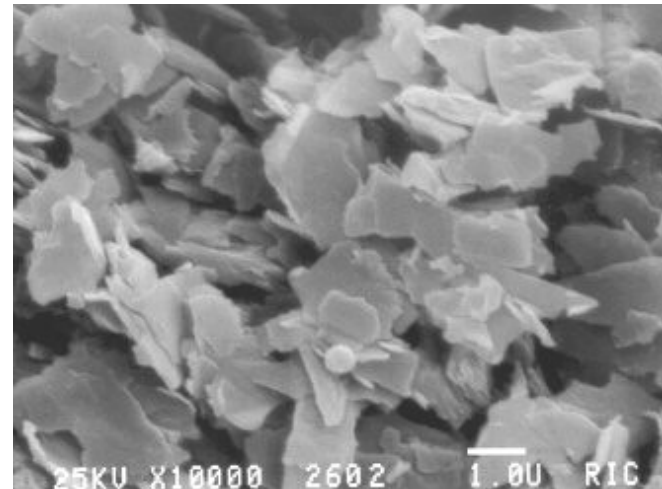
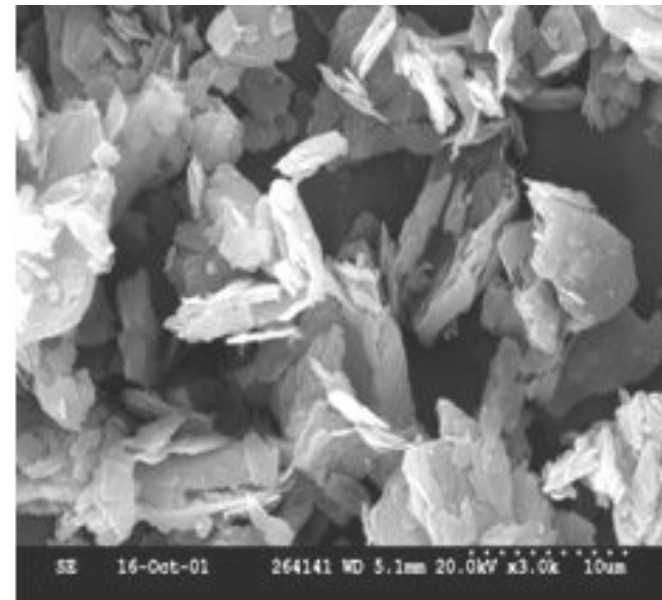
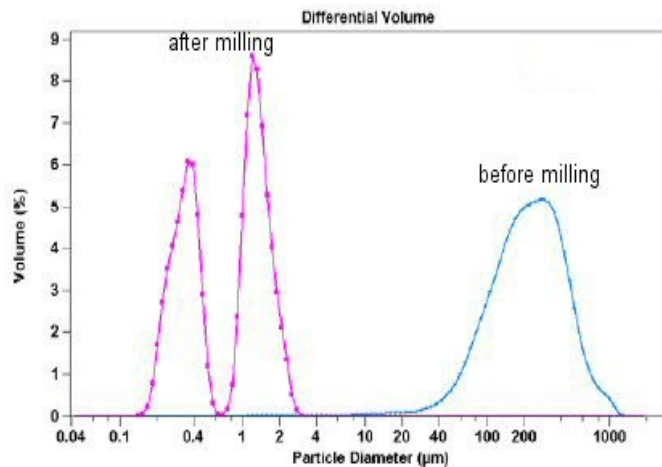
Raw Graphite 3k



Multi Pass Graphite 3k



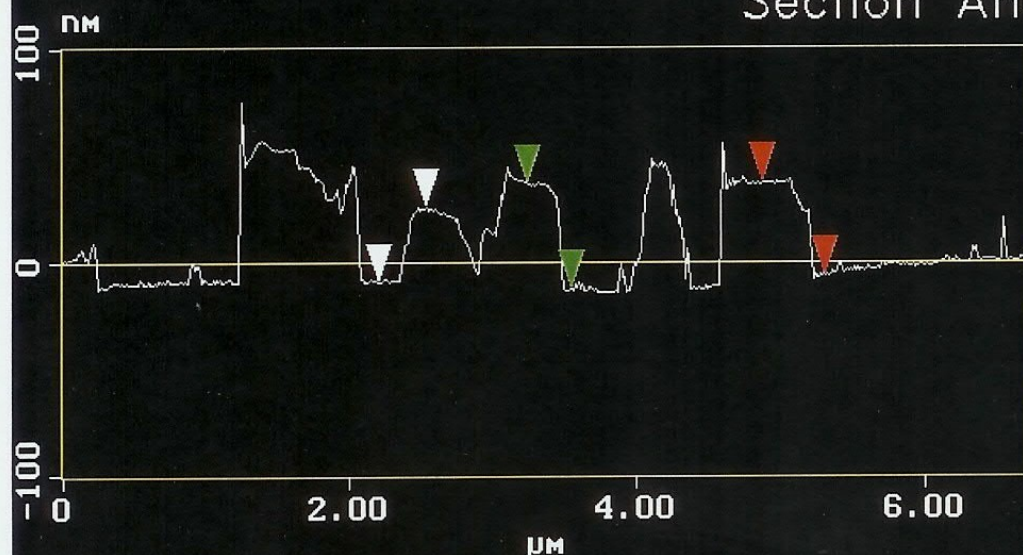
c



Examination of the multi-pass milled synthetic graphite with laser diffraction (LD) and scanning electron microscopy (SEM): (a) high-pressure mill (LD), (b) high-pressure mill (SEM), (c) modified high-pressure mill (LD), (d) modified high-pressure mill (SEM)

Cursor Marker Spectrum Zoom Center Line Offset Clear

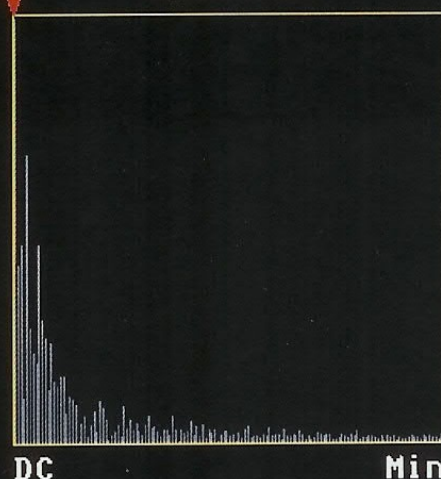
Section Analysis



L	328.13 nm
RMS	14.842 nm
lc	DC
Ra(lc)	4.334 nm
Rmax	20.148 nm
Rz	10.924 nm
Rz Cnt	6
Radius	341.24 nm
Sigma	11.729 nm



Spectrum

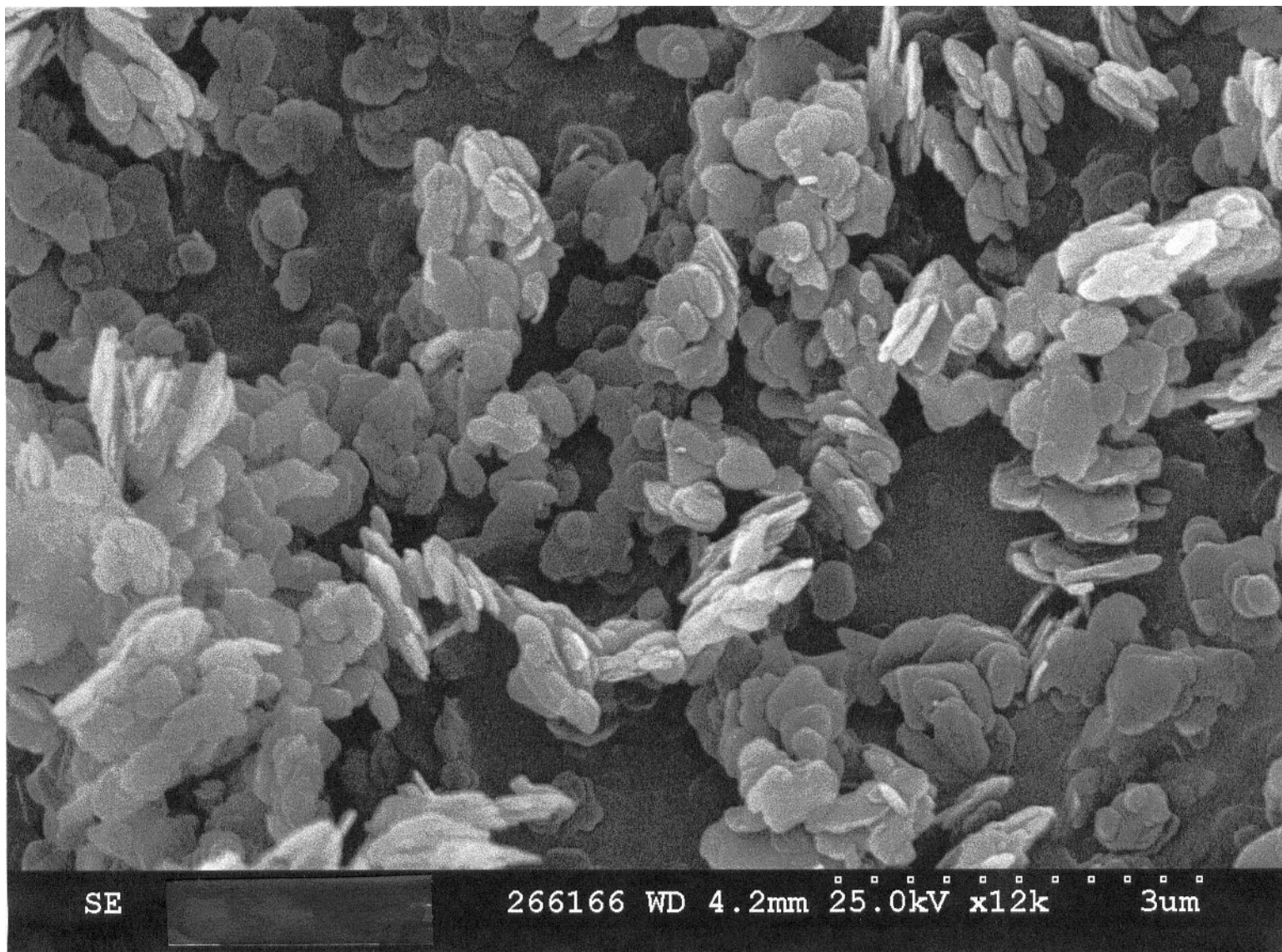


Surface distance	448.67 nm
Horiz distance(L)	433.59 nm
Vert distance	43.715 nm
Angle	5.757 deg
Surface distance	323.26 nm
Horiz distance	304.69 nm
Vert distance	49.818 nm
Angle	9.286 deg
Surface distance	336.78 nm
Horiz distance	328.13 nm
Vert distance	34.529 nm
Angle	6.007 deg
Spectral period	DC
Spectral freq	0 Hz
Spectral RMS amp	0.016 nm

ct4.012

Cursor: fixed Zoom: 2:1

Cen line: Off Offset: Off

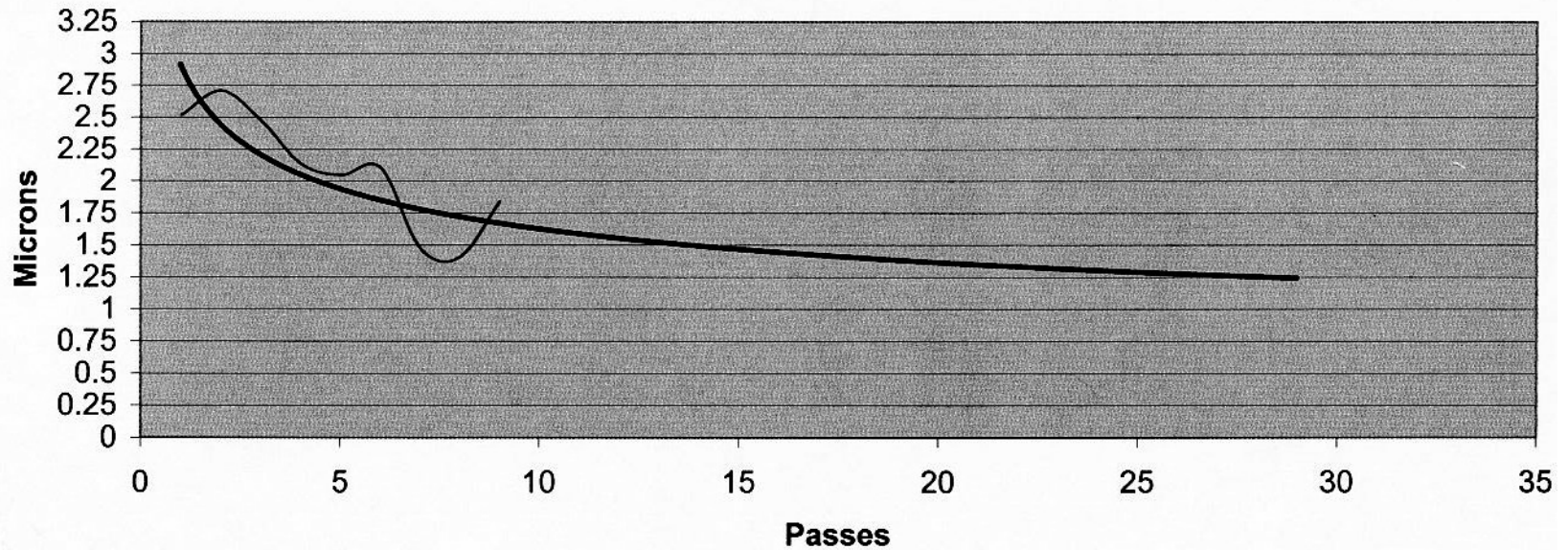


Opposite Jet Milling Nozzles .014" -> -> -> -> .011" -> ->

Analysis and Trend of d10's For Recirculation of Boron Nitride

	Raw	1st Pass (recirc)	2nd Pass (recirc)	3rd Pass (recirc)	4th Pass (recirc)	5th Pass (recirc)	6th Pass (recirc)	7th Pass (recirc)	8th Pass (recirc)
Pass Number	1	2	3	4	5	6	7	8	9
Test Numbers	Lot 109	GM551-552	GM553-556	GM557-560	GM561-564	GM565-568	GM569-572	GM573-576	GM577-580
	2.515	2.568	2.424	2.057	2.111	2.168	1.47	1.509	1.798
		2.847	2.385	2.171	2.015	2.054	1.599	1.32	1.953
			2.427	1.981	1.919	2.069	1.611	1.33	1.749
			2.656	2.325	2.147	2.128	1.209	1.464	1.872
Averages of d10's	2.515	2.7075	2.473	2.1335	2.048	2.10475	1.47225	1.40575	1.843
Standard Deviation		0.19728279	0.10694625	0.129602276	0.088741197	0.045811434	0.1617272	0.08237832	0.07713948

Trend of d10 vs Number of Passes

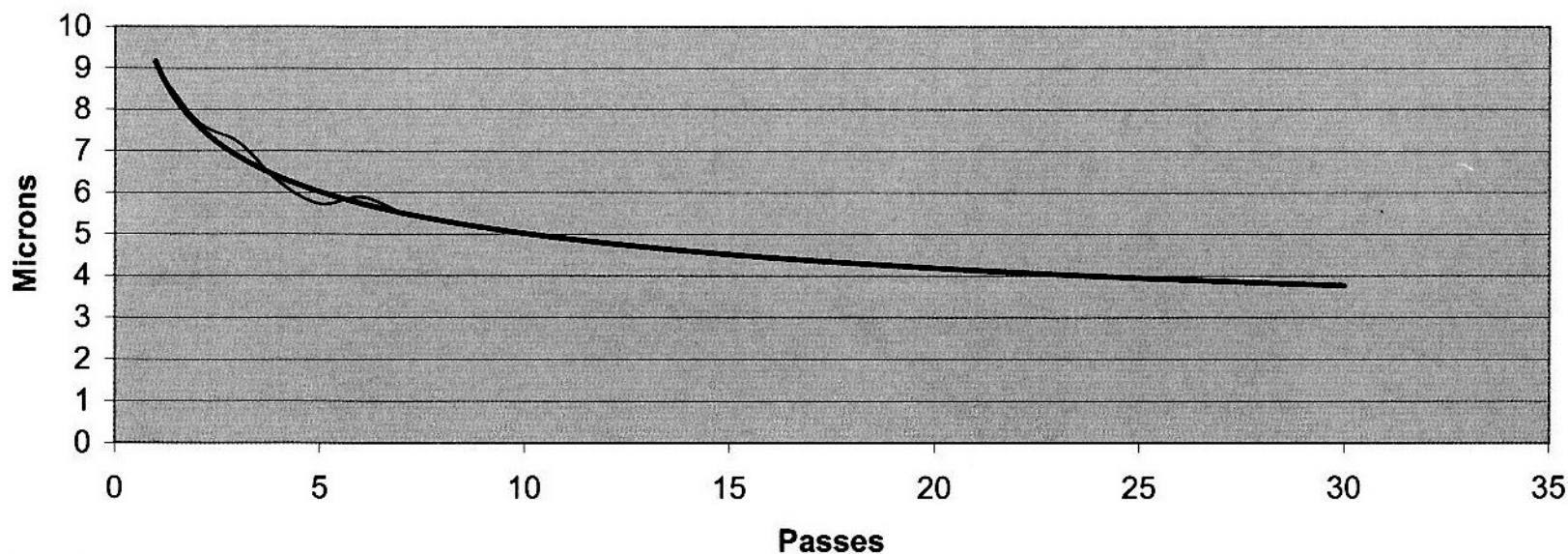


Opposite Jet Milling Nozzles .014" -> -> -> -> .011" -> ->

Analysis and Trend of d50's For Recirculation of Boron Nitride

	Raw	1st Pass (recirc)	2nd Pass (recirc)	3rd Pass (recirc)	4th Pass (recirc)	5th Pass (recirc)	6th Pass (recirc)	7th Pass (recirc)	8th Pass (recirc)
Pass Number	1	2	3	4	5	6	7	8	9
Test Numbers	Lot 109	GM551-552	GM553-556	GM557-560	GM561-564	GM565-568	GM569-572	GM573-576	GM577-580
	9.022	7.272	7.066	6.039	6.12	5.792	5.359	5.632	4.954
		8.151	6.927	6.234	5.513	5.731	5.718	5.053	5.218
			7.006	5.941	5.43	5.927	6.136	5.213	5.152
			7.823	6.76	5.836	6.111	4.885	5.429	5.192
Averages of d50's	9.022	7.7115	7.2055	6.2435	5.72475	5.89025	5.5245	5.33175	5.129
Standard Deviation		0.62154686	0.35990589	0.316302466	0.2740049	0.14585502	0.46035991	0.21875257	0.10373524

Trend of d50 vs Number of Passes

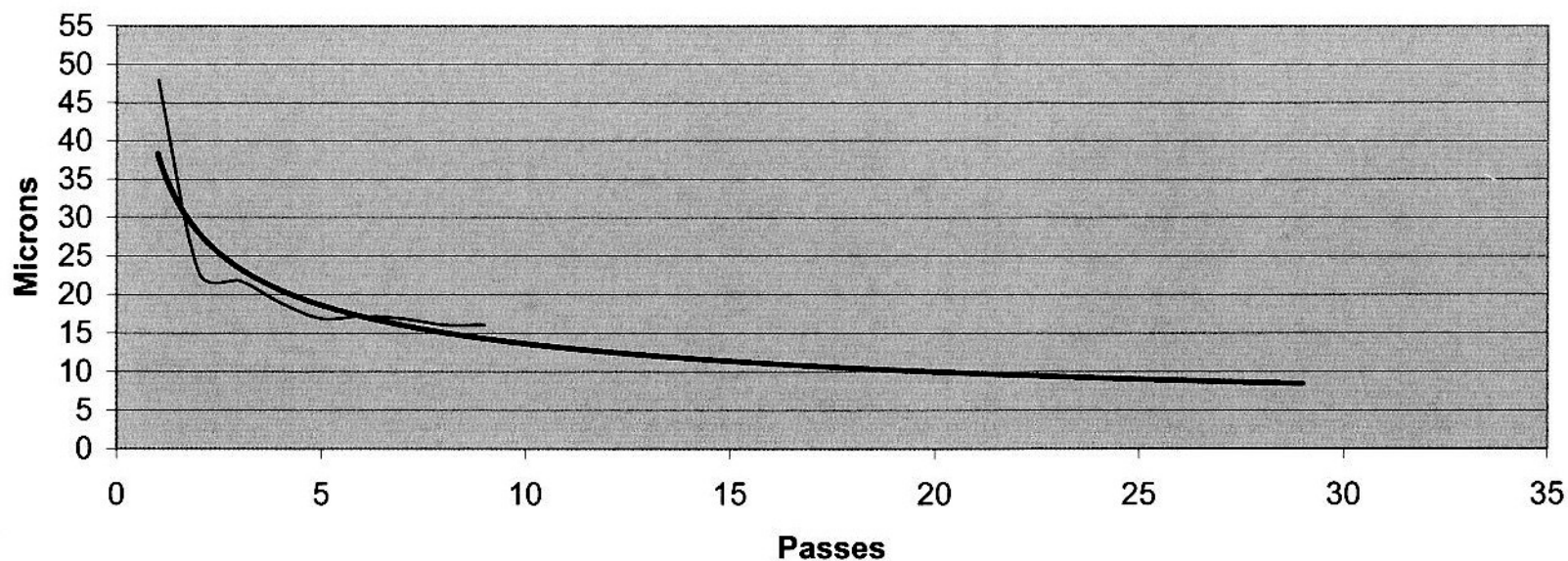


Opposite Jet Milling Nozzles .014" -> -> -> -> .011" -> ->

Analysis and Trend of d100's For Recirculation of Boron Nitride

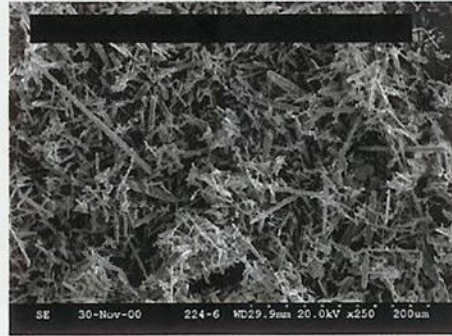
	Raw	1st Pass (recirc)	2nd Pass (recirc)	3rd Pass (recirc)	4th Pass (recirc)	5th Pass (recirc)	6th Pass (recirc)	7th Pass (recirc)	8th Pass (recirc)
Pass Number	1	2	3	4	5	6	7	8	9
Test Numbers	Lot 109	GM551-552	GM553-556	GM557-560	GM561-564	GM565-568	GM569-572	GM573-576	GM577-580
	47.94	20.71	20.71	18.86	17.18	17.18	15.65	17.18	17.18
		24.95	20.71	18.86	15.65	17.18	17.18	15.65	15.65
			20.71	18.86	15.65	17.18	18.86	15.65	15.65
			24.95	18.86	18.86	17.18	15.65	15.65	15.65
Averages of d100's	47.94	22.83	21.77	18.86	16.835	17.18	16.835	16.0325	16.0325
Standard Deviation		2.99813275	2.12	0	1.53058812	0	1.53058812	0.765	0.765

Trend of d100 vs Number of Passes

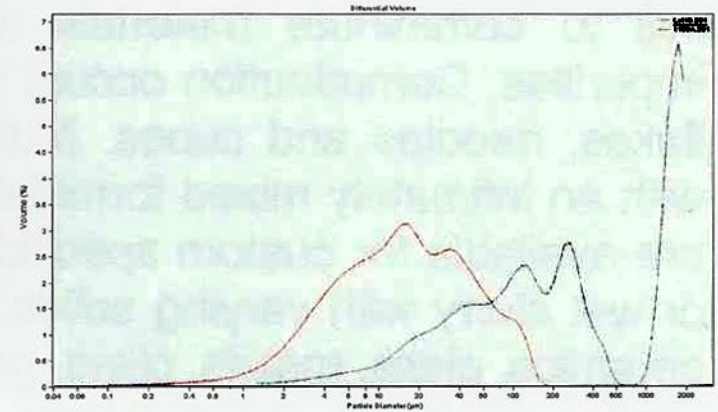




FE-SEM micrograph at 250x magnification.



FE-SEM micrograph of typical product at 250x magnification. Note the very high aspect ratio needles created. Further comminution to produce lower aspect ratios can be achieved.



Raw Feedstock $d_{50} = 188 \mu\text{m}$, $d_{100} = 2000 \mu\text{m}$
Milled material $d_{50} = 15 \mu\text{m}$, $d_{100} = 194 \mu\text{m}$

Material Name	Raw Material [μm]				Product [μm]			
	d10	d50	d90	d100	d10	d50	d90	d100
Barium Titanate	2,383	4,429	8,026	22,73	0,362	2,44	4,208	7,421
Boron Nitride	0,993	2,962	5,111	8,943	1,704	5,759	9,991	17,18
Boron Nitride	2,515	9,022	24,68	47,94	2,496	7,816	13,48	22,73
Boron Nitride	1,872	7,159	17,48	33,01	8,906	21,65	34,59	47,94
Calcium Carbonate	0,175	1,648	8,036	20,71	0,185	1,774	6,41	18,86
Cordierite	6,615	40,57	168,6	309,6	2,178	11,86	33,19	121,8
Dental Glass	0,45	2,5	40,68	111	0,39	2,496	4,021	5,61
Dental Glass	0,506	89,26	213,8	373,1	0,239	2,713	6,214	24,95
Dental Glass	2,642	7,898	22,01	63,41	2,106	5,262	16,56	52,62
Electronic Glass Frit	-	-	-	<2mm	10,13	59,28	137,4	786,9
Graphite	49,02	399	907,6	1 822	2,806	7,195	22,25	52,62
Graphite	310,2	477,7	774,3	1 660	9,273	19,29	32,02	43,67
Graphite	368,1	638,6	991,5	1 660	6,57	19,95	63,61	121,8
Graphite	87,17	343,9	782,8	1 041	8,372	20,8	46,15	92,09
Graphite	387,9	590,6	891,8	1 660	2,903	7,222	15,8	39,78
Graphite	-	-	-	>2mm	9,394	19,31	35,11	52,62
Graphite	302	556,7	942,1	1 377	7,849	17,41	29,57	39,78
Amorphous Graphite	-	-	-	>2mm	3,285	8,177	20,63	63,41
High Purity Alumina	-	-	-	~2mm	1,51	2,825	5,539	24,95
Pin Milled Graphite	248,8	441,3	756	1 660	9,085	18,71	32,12	47,94
Pyrex B	6,69	23,3	61,59	161,2	1,499	6,914	21,25	57,77
Silica	90,22	665,4	1 636	2 000	4,638	24,45	73,11	194,2
Silica	508,6	777,2	1 172	1 660	0,522	28,33	126,3	786,9
Silver	7,091	56,57	327,8	786,9	2,579	4,634	7,501	17,18
Titanium Dioxide	1,168	2,73	4,9	8,943	1,52	2,737	4,455	6,76
Zirconium Oxide	486,3	759,9	1 093	1 660	8,727	107,9	334,1	1 143
Zirconium Titanate	16,27	30,94	52,98	111	3,196	15,39	29,86	57,77

Characterization (LD) of particles of selected materials milled by single-pass through the high-pressure mill