WB4101 Water Based Binder Compared to Competitive Binders

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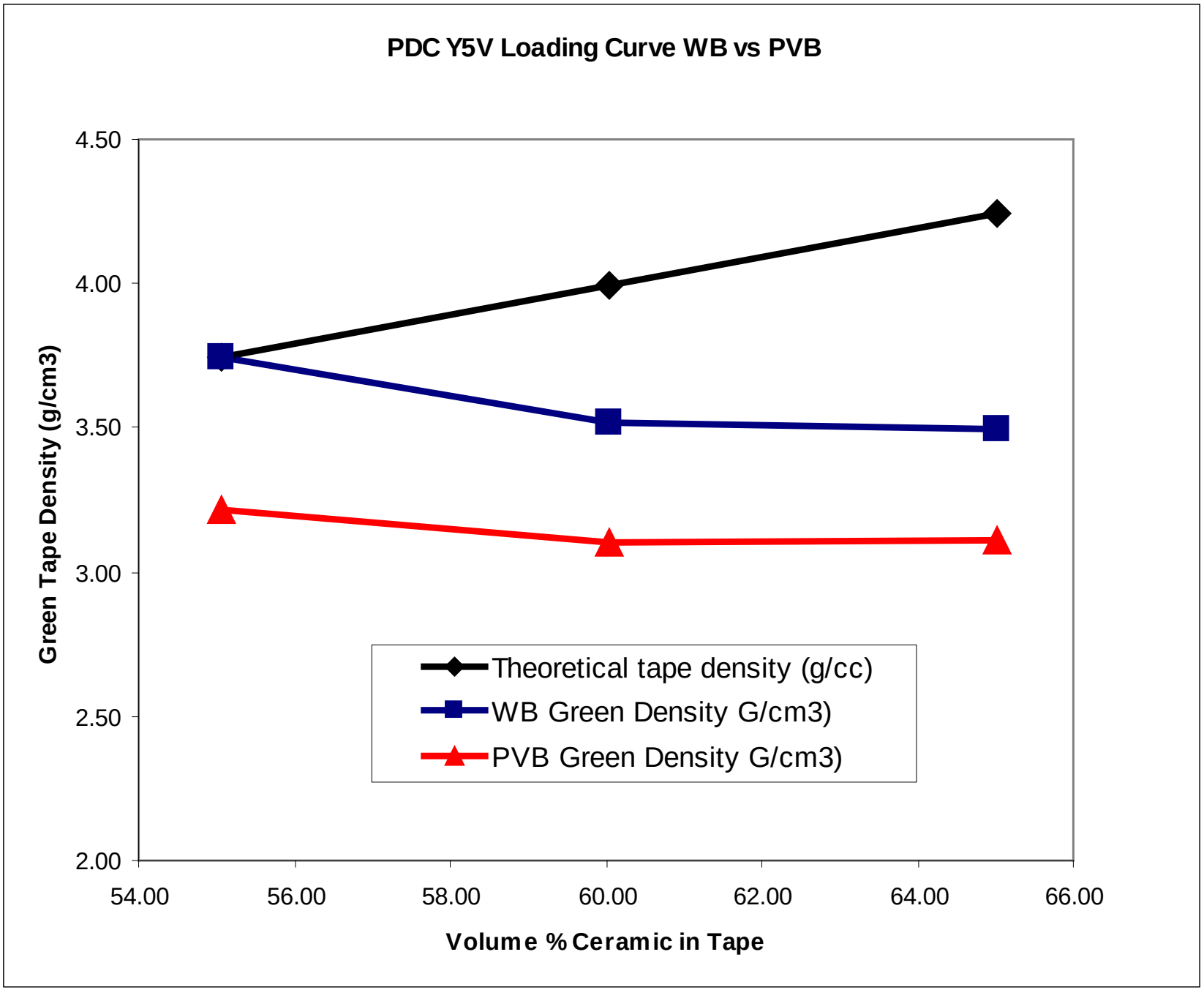
Introduction

- Standard Two Stage Ball Milling Methods Used
- Choose Some Standard BME Powder
- Choose Some Nano Powder
- For PVB Use Standard Ferro 2 Stage Type (Toluene-Ethanol Solvent Mix).

WB4101 vs. PVB
w/ Prosperity BME Y5V

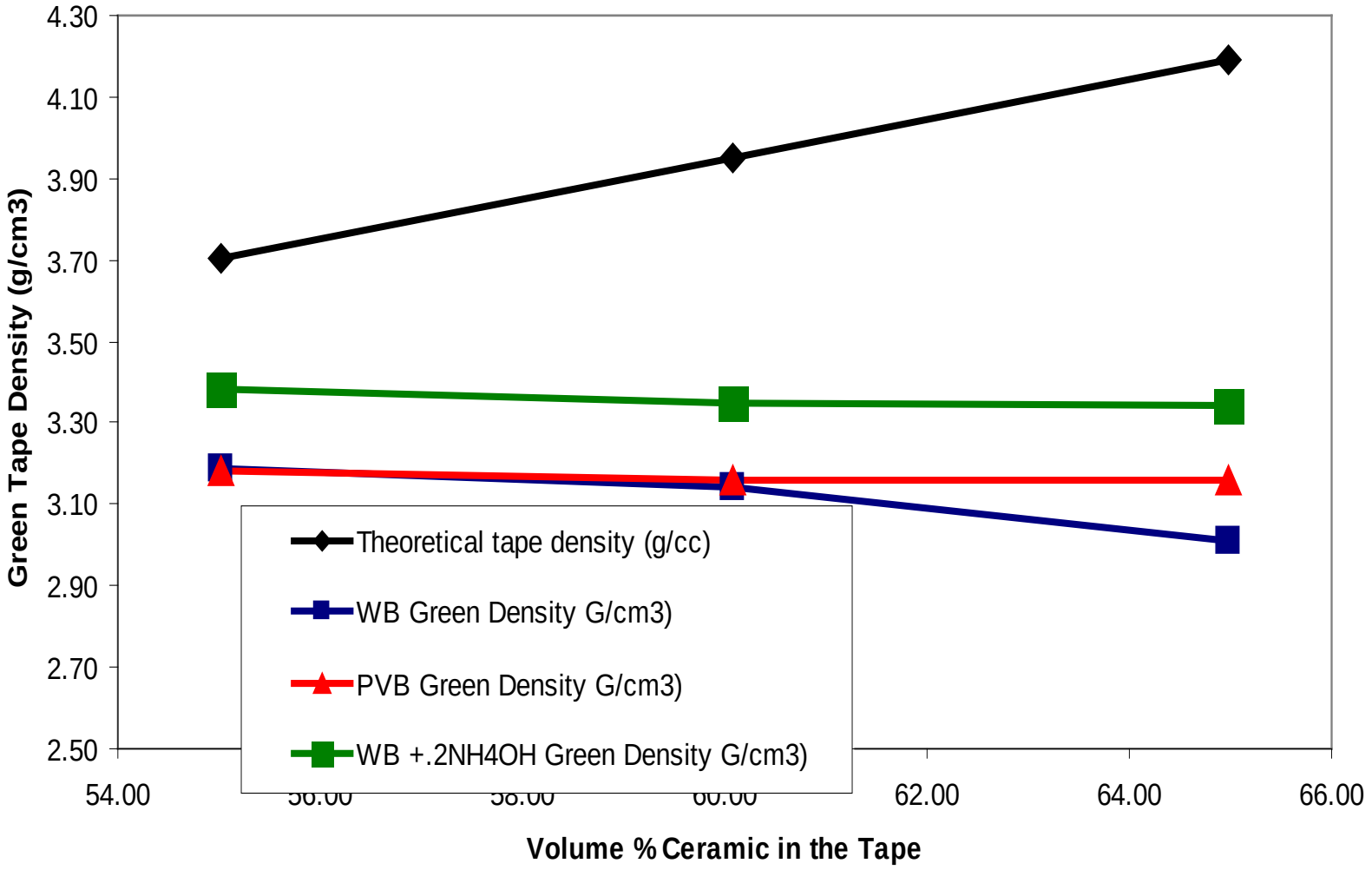
Sample Number	WB55	WB60	WB65	PVB55	PVB60	PVB65
PDC BME-Y5V153 (wt% slip)	60.60	63.90	67.00	60.00	63.00	66.00
Volume Ceramic Loading in Tape	55.07	60.04	65.03	54.87	59.57	64.60
weight % ceramic	88.01	90.00	91.76	87.93	89.82	91.62
Green Density (g/cc)	3.74	3.52	3.49	3.22	3.10	3.11
Approximate % porosity	0.2	12.0	17.7	13.9	21.9	26.4
Puncture strength(grams/mil)	297	125	126	75	58	47
Flexibility (crease test)	0	0	0	50	0	0
Lam. 160F 330 psi 15 sec.	150+	150+	55	33	24	8


PDC Y5V Loading Curve WB vs PVB
Green Tape Density (g/cm3)
4.50
4.00
3.50
3.00
2.50
2.00
54.00
56.00
58.00
60.00
62.00
64.00
66.00
Volume % Ceramic in Tape
Theoretical tape density (g/cc)
WB Green Density G/cm3)
PVB Green Density G/cm3)

WB4101 vs. PVB
w/ Prosperity BME Y5V

Sample Number	WB55	WB60	WB65	PVB55	PVB60	PVB65
PDC BME-X7R342 (wt% slip)	58.90	62.20	65.20	59.00	62.00	65.00
Volume Ceramic Loading in Ta	55.01	60.07	64.98	55.50	60.31	65.46
weight % ceramic	87.84	89.89	91.64	88.05	89.98	91.80
Green Density (g/cc)	3.19	3.14	3.01	3.18	3.16	3.16
Approximate % porosity	14	21	28	15	20	25
Puncture strength(grams/mil)	173	107	84	78	58	39
Flexibility (crease test)	10	0	0	100	30	0
Lam. 160F 330 psi 15 sec.	150+	150+	150+	150+	34	19
WB4101 was about .2g/cm3 denser and stronger with .2% added NH4OH (Data not shown in table)						

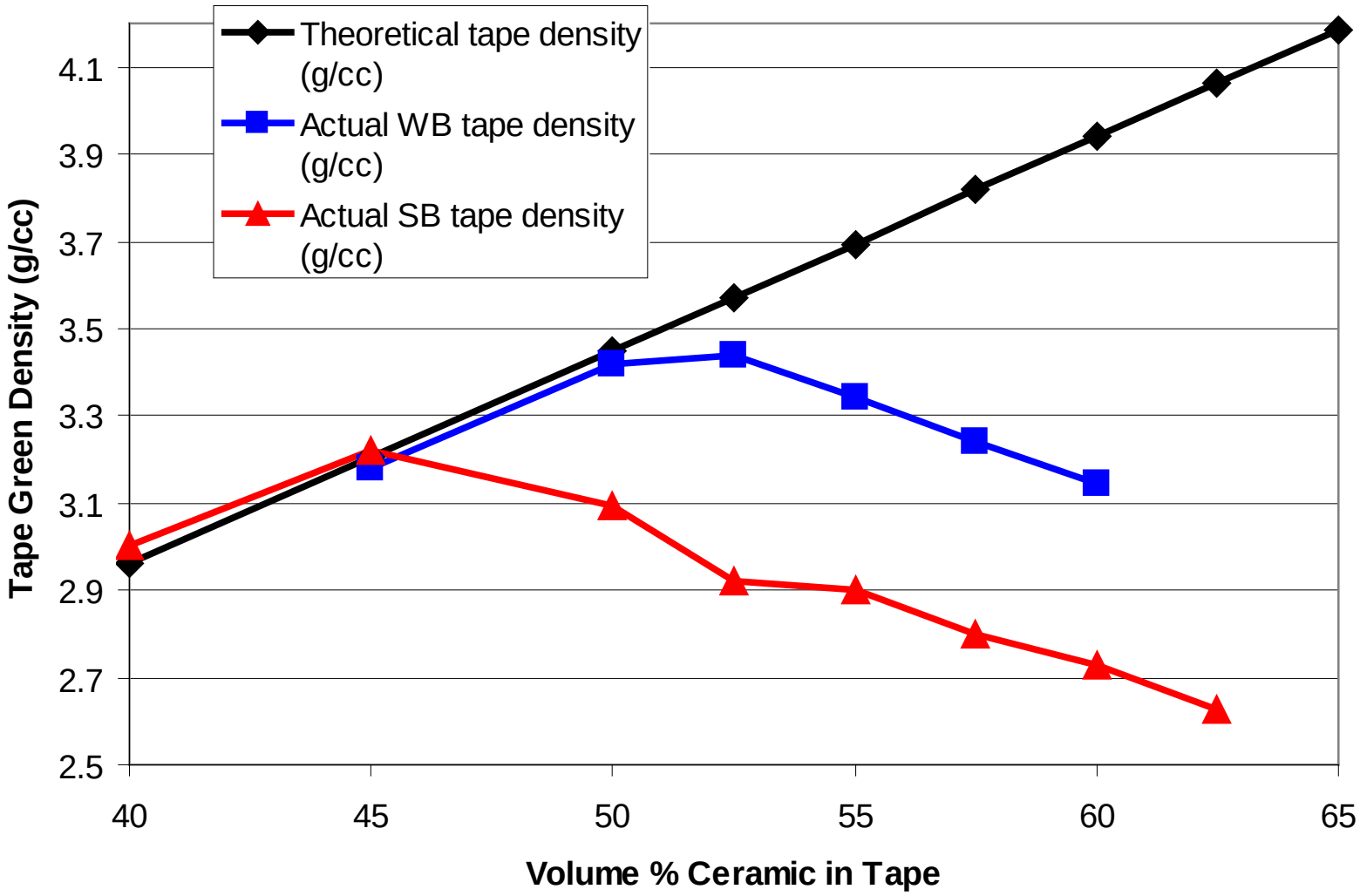
Loading Curve PDC BME X7R WB vs PVB



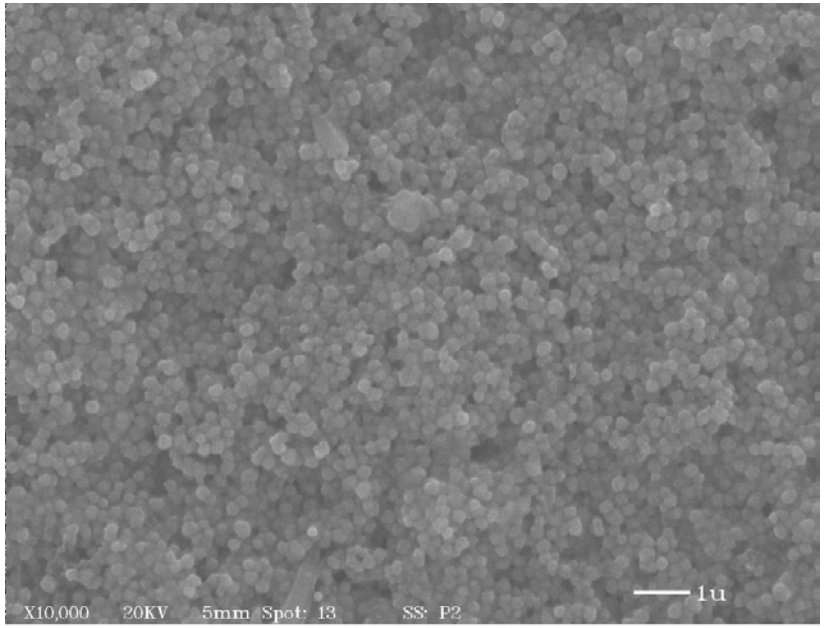
WB4101 vs. Solvent Based Acrylic Binder w/ Ferro ULF101 Ceramic

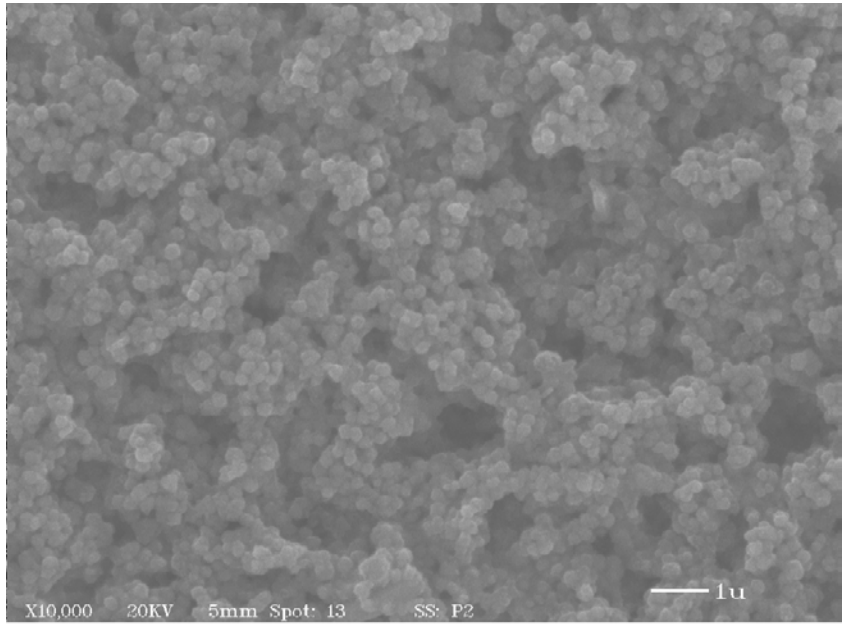
Volume Ceramic Loading in Tape	**45.0**	**50.0**	**52.5**	**55.0**	**57.5**	**60.0**	**62.5**
Theoretical Tape Density (g/cc)	**3.21**	**3.45**	**3.57**	**3.70**	**3.82**	**3.94**	**4.06**
WATER BASED WB4101 BINDER							
Measured tape density (g/cc)	**3.18**	**3.42**	**3.44**	**3.34**	**3.24**	**3.14**	**3.11**
Puncture strength(grams/mil)	**431**	**391**	**266**	**256**	**223**	**212**	**175**
Approximate % porosity	**0.0**	**0.0**	**3.7**	**9.5**	**15.2**	**20.2**	**23.4**
SOLVENT BASED ACRYLIC BINDER							
Measured tape density (g/cc)	**3.22**	**3.09**	**2.92**	**2.90**	**2.80**	**2.73**	**2.63**
Puncture strength(grams/mil)	**230**	**96**	**76**	**68**	**48**	**35**	**28**
Approximate % porosity	**0.0**	**10.4**	**18.3**	**21.5**	**26.6**	**30.7**	**35.3**

Ceramic Tape Loading Curves for ULF101 Dielectric - WB vs SB Acrylic Binders



WB4101 Compared to Emulsion w/ Cabot Nano Powder (Top Surface of Foil)

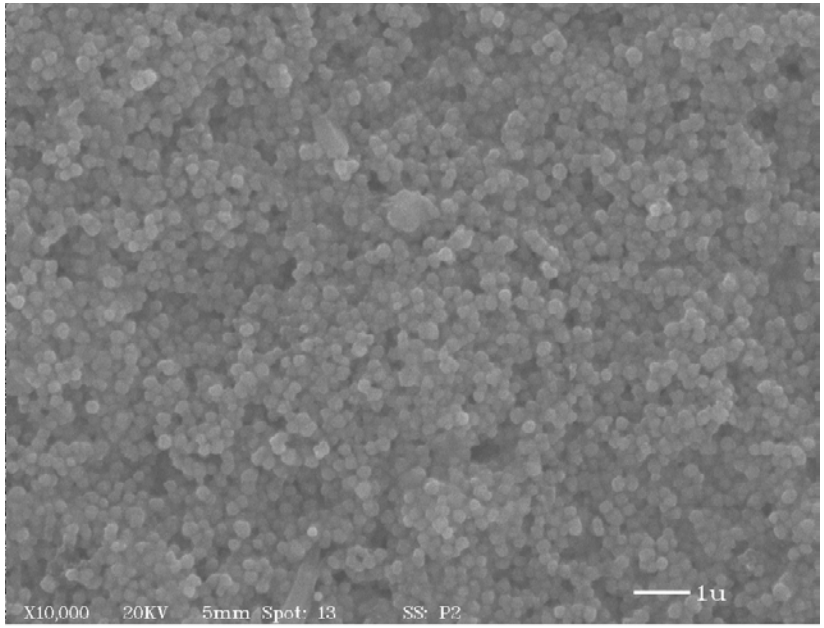




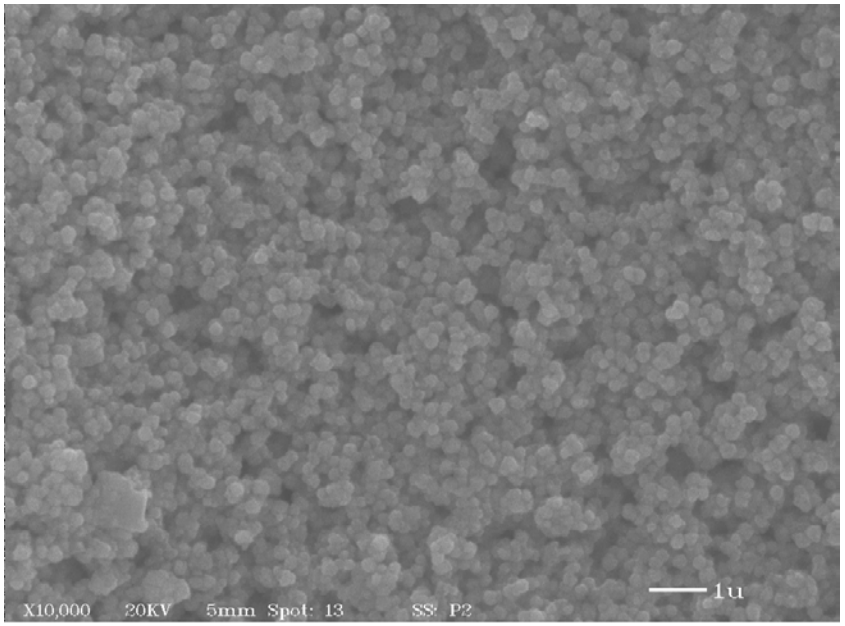
WB4101 type **Emulsion type**

(From Cabot 2000 ACERS Presentation)

WB4101 Compared to Water Soluble Type w/ Cabot Nano Powder (Top Surface of Foil)



WB4101 type

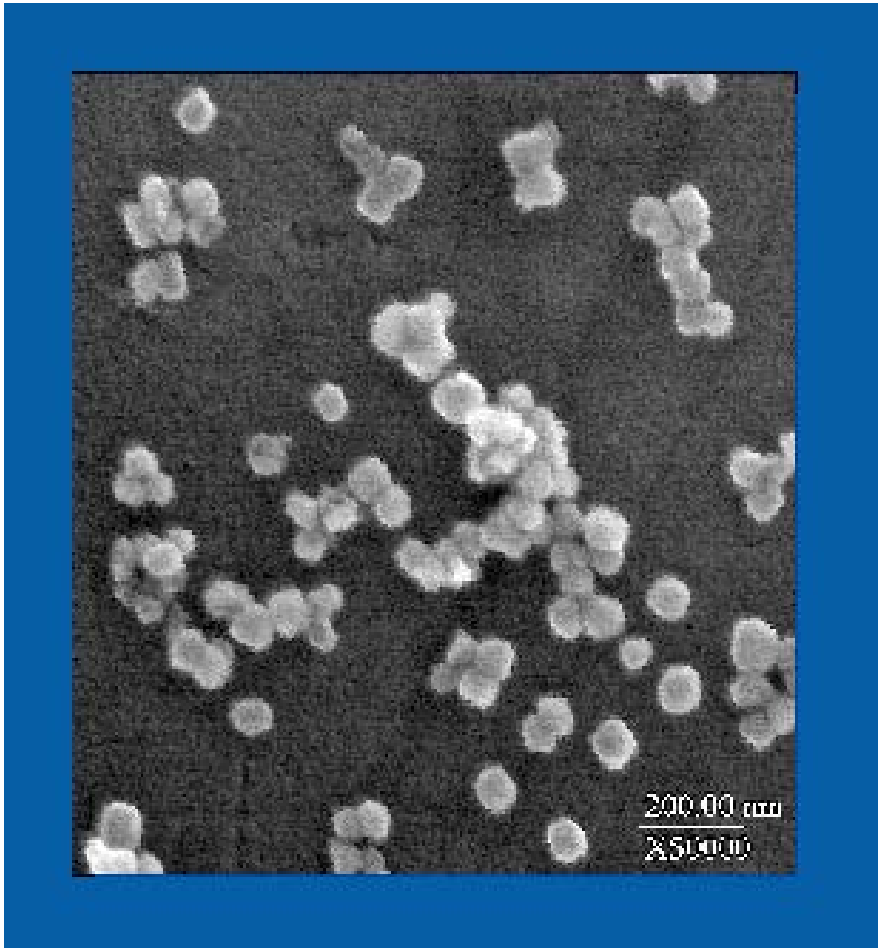


Water soluble type

(From Cabot 2000 ACERS Presentation)

TPL Nano BT Powder

- TPL Nano BT
- Dry Powder
- Some Dispersion Challenges for Regular Binders - Agglomerates
- WB4101 is Strong Dispersant and Binder

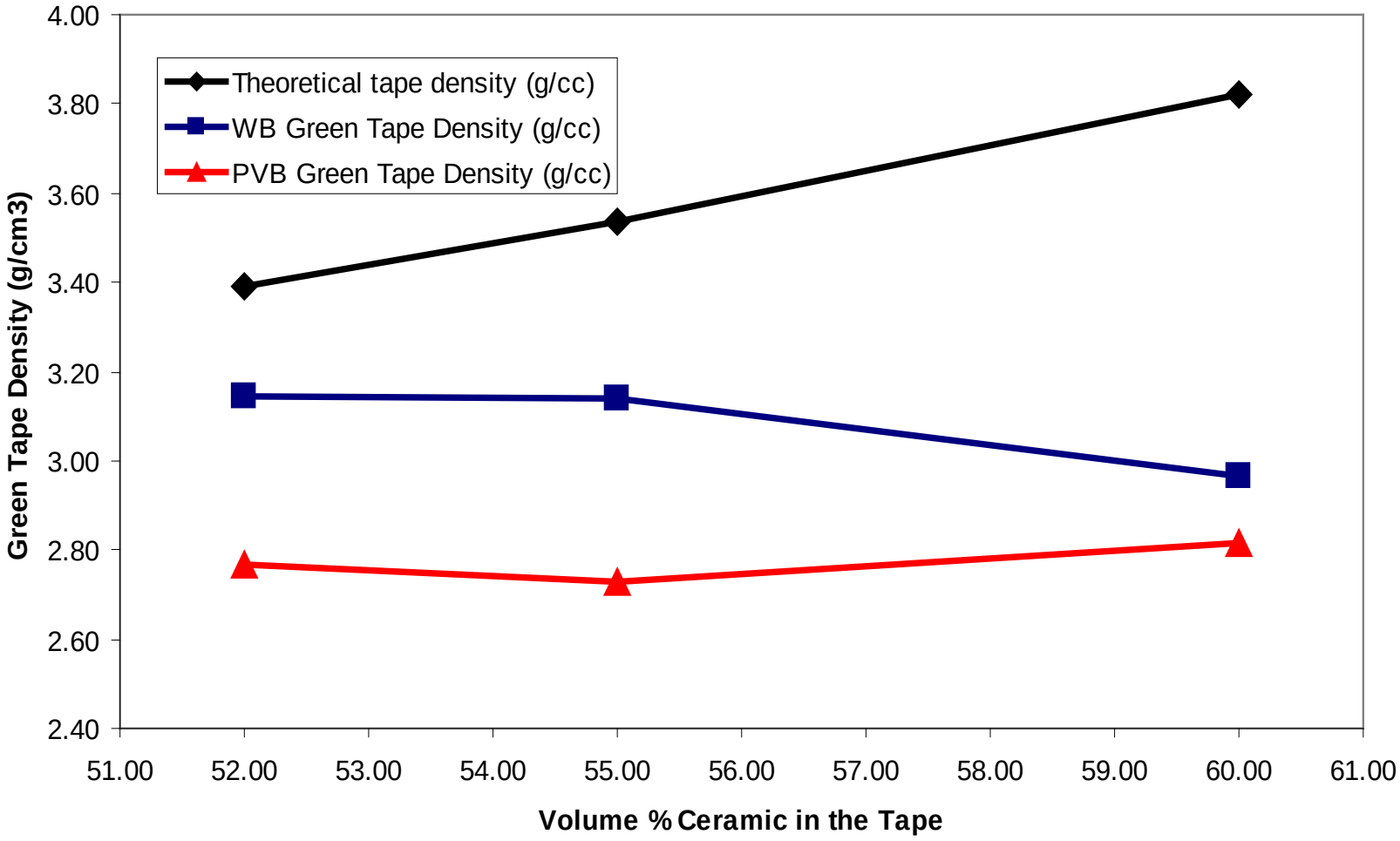


TPL 100 nm BT WB4101 vs. PVB

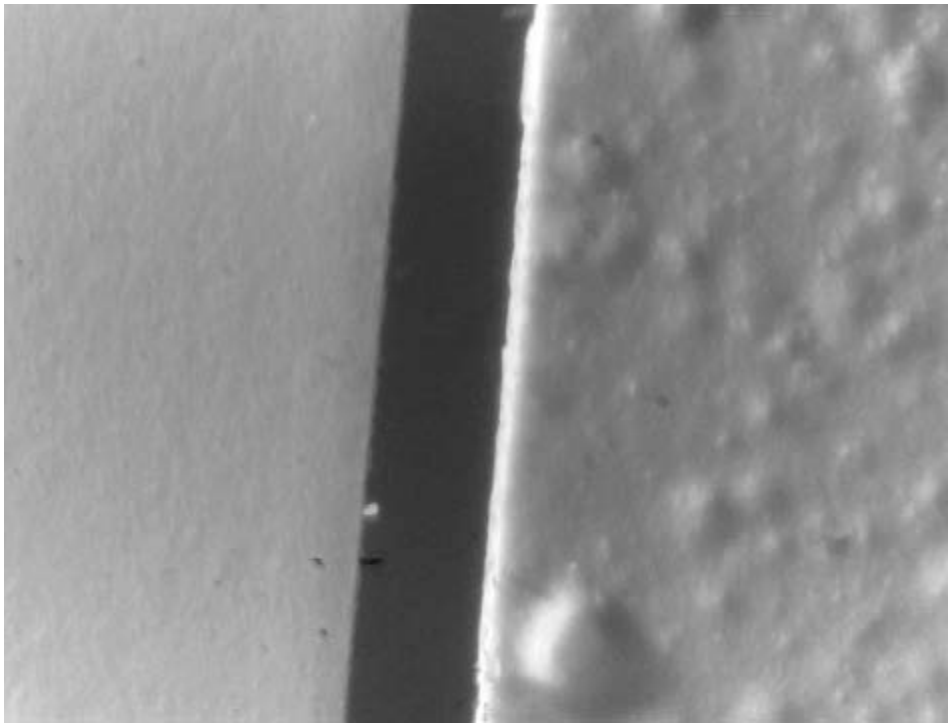
Sample Number	52WB	55WB	60WB	52 PVB	55 PVB	60 PVB
TPL - HPB1000 (wt% in slip)	52.00	54.80	58.00	56.00	58.00	61.00
Volume Ceramic Loading in Tape	50.87	53.97	60.01	51.90	54.86	59.54
weight % ceramic in tape	85.51	86.98	89.53	86.01	87.38	89.35
Theoretical tape density (g/cc)	3.39	3.54	3.82	3.44	3.58	3.80
Green Density (g/cc)	3.14	3.14	2.96	2.77	2.73	2.81
Approximate % porosity	7	11	22	20	24	26
Average Puncture (grams/mil)	141	166	123	124	101	91
Flexibility (crease test)	60	100	10			
Lam. 160F 330 psi 15 sec.3/10/05	120	145	26	40	27	10
Dispersion (agglomerates)	excellent	excellent	excellent	very poor	very poor	very poor

PVB tape agglomerates are so bad tape is not usable and data is not representative

Loading Curve TPL Nano Powder WB vs PVB



Dispersion Photo TPL 100 nm BT WB4101 vs. PVB



Both are 60 v/v% Powder and 2 stage ball milled
PVB tape is unusable due to large agglomerates (45X)

Conclusions

- Compared to PVB, solvent based acrylic, emulsion and water soluble binders, WB4101 typically results in higher packed density (green tape density) and easier lamination characteristics
- WB4101 is superior for nano powders and can easily achieve well dispersed tapes with typical ceramic loadings. PVB did not.
- WB4101 is capable of achieving high packing densities for thin (<6 micron) layers when other water based types can not.

Nano Refinement

- Work in part 1 achieved workable tape with ~100 nm BT powder with densities of about 2.9 g/cm2 at 60 vol% ceramic loading in the tape.
- However, SEM showed the 100 nm particles were still agglomerated in groups of 3 or more particles.
- This work now focuses on trying to further improve WB4101 ability to disperse Nano BT.

Procedures

- Initial work was using TPL Nano BT with regular milling (3/8” cylindrical milling media).
- This work uses highly basic modifiers to increase the dispersing power of the ammonium polyacrylate binder.
- Smaller 2 mm YSZ media and longer milling times were used.
- Even greater dispersion results are achieved as seen in green tape density, loading and finally SEM (broken down to primary particle size).

Below is the Formula and Process Used to Further Increase the Dispersion

		MILL CHARGES CLEAN							MILL CHARGES DIRTY						
Proposed Formula	**Formula**	Formulas in Grams for **Clean** Mill Of Given Size							Formulas in Grams for Dirty (7% retain) Mill Of Given Size						
Mill Size (gallons)	TP9-2	52	27	6.6	4.6	2.2	1.5	**0.3**	52	27	6.6	4.6	2.2	1.5	0.3
Dielectric															
STAGE 1															
TPL - HPB1000 (barium titanate)	51.60	50122	26025	6362	4434	2120.5	1445.8	**289.2**	46613	24203	5916	4123	1972.1	1344.6	268.9
WB4101	5.50	5342	2774	678	473	226.0	154.1	**30.8**	4968	2580	631	440	210.2	143.3	28.7
DF002	0.20	194	101	25	17	8.2	5.6	**1.1**	181	94	23	16	7.6	5.2	1.0
DS005	1.00	971	504.4	123.3	85.9	41.1	28.0	**5.6**	903	469.1	114.7	79.9	38.2	26.1	5.2
PL005	0.50	486	252.2	61.6	43.0	20.5	14.0	**2.8**	452	234.5	57.3	40.0	19.1	13.0	2.6
		0	0	0	0	0.0	0.0	**0.0**	0	0	0	0	0.0	0.0	0.0
NH4OH	0.50	486	252	62	43	20.5	14.0	**2.8**	452	235	57	40	19.1	13.0	2.6
DI Water	35.00	33997	17652	4315	3007	1438.3	980.7	**196.1**	31617	16417	4013	2797	1337.7	912.0	182.4
STAGE 2		0	0	0	0	0.0	0.0	**0.0**	0	0	0	0	0.0	0.0	0.0
WB4101	5.50	5342	2774	678	473	226.0	154.1	**30.8**	4968	2580	631	440	210.2	143.3	28.7
DF002	0.20	194	101	25	17	8.2	5.6	**1.1**	181	94	23	16	7.6	5.2	1.0
WT007		0	0	0.0	0.0	0.0	0.0	0.0	0	0	0.0	0.0	0.0	0.0	0.0
		0	0.0	0.0	0.0	0.0	0.0	0.0	0	0	0.0	0.0	0.0	0.0	0.0
Total	100	97135	50436	12329	8593	4110	2802	560	90336	46905	11466	7991	3822	2606	521
Milling time first (hours)								72							
Mixing time second (hours)								24							
Recommended mill RPM		33-36	35-38	**	**	**	56	**							
Aim pounds of media for mill		745.7	387.2	94.6	66.0	31.5	21.5	4.3							
Aim first stage slurry volume (gal)		13.00	6.75	1.65	1.15	0.55	0.38	0.08							
Aim first stage slurry weight (grams)		91792.7	47662	11651	8120.1	3883.5	2647.9	529.57							
Aim slurry final weight (grams)		97135.2	50436	12329	8592.7	4109.6	2802	560.4							
Calculated organic solids	5.250	ratio of slurry vol to mill vol below													
Calculated ceramic in tape	90.77	0.25	0.25	0.25	0.25	0.25	0.25	0.25							
Ceramic density	5.7														
Calculate ceramic volume %	63.29														
Theoretical green tape density	3.97														
Estimated first stage density	1.87														
Estimated final slip density (g/cc)	1.78														

Per TPL SEM 1/25/06 this formula has outstanding dispersion. Small lab ball mill used 2 mm YTZ media. Larger mills will use much shorter milling times.

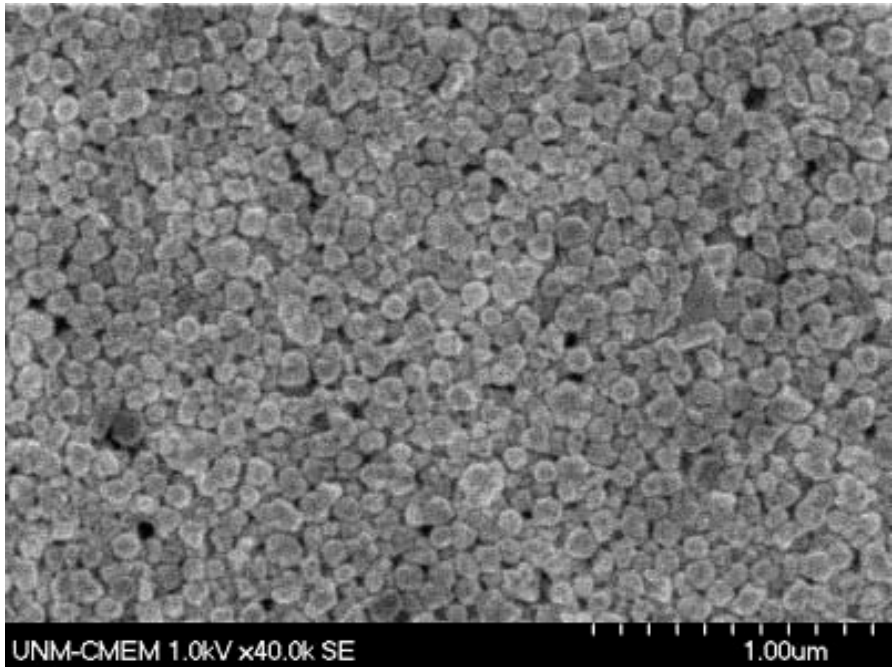
** For mill RPM use between about 60-65% of the critical calculated rpm. Critical rpm is calculated by taking 54.2 divided by the square root of the mill inside radius in feet.

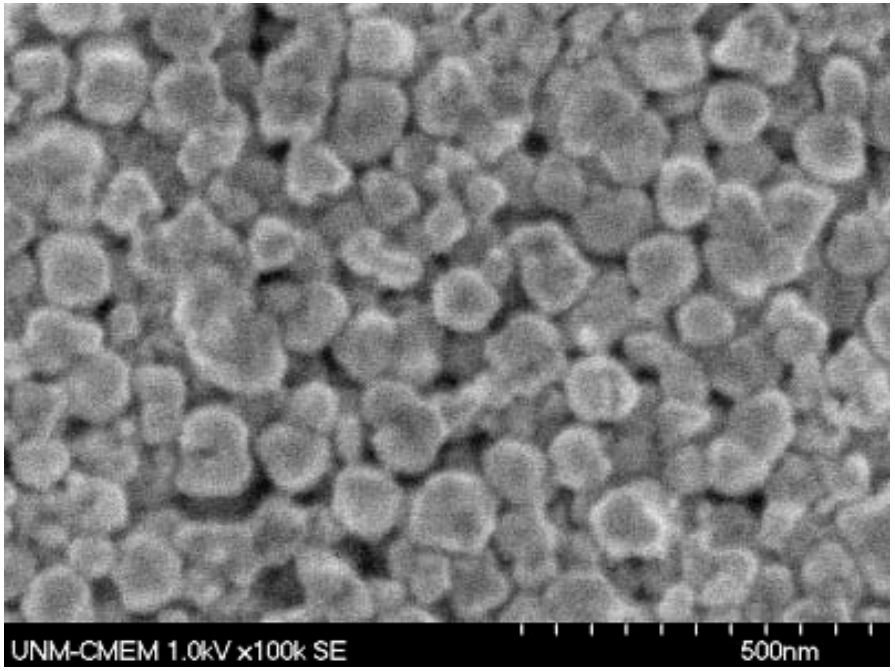
Note when changing formulas for new mill size use ~25% of slurry volume ratio to mill volume for 2 stage milling. All the numbers are calculated from line 3 (mill size) and aim first stage slurry volume (use 25% of total mill capacity).

Results

- The use of stronger additives and processing further increased the dispersing ability of WB4101.
- Good quality tape with 100 nm BT was produced at an astonishing 63 volume % BT in the green tape.
- Green tape density of 3.5 g/cm3 was obtained despite the high loading and Nano particle size.
- Please see attached SEM

SEM of Green Tape





- As can be seen, the primary particle size is about 100 nm

Conclusions

- WB4101 is superior for Nano powders and can easily achieve well dispersed tapes with typical ceramic loadings. PVB did not.
- In this part 2 the use of PL005 and DS005 additives were compatible with the WB4101 and further increased the dispersing power of the WB Binder System.
- For finer powders finer milling media provided faster results.
- WB4101 is capable of achieving high packing densities for thin (<6 micron) layers when other water based types can not.