Water Based Tape Casting Demonstration Kit

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Polymer Innovations, Inc. (PII)

Founded 1996

WB4101 Type Binder Advantages

- Unique and highest performing WB binder available.
- Environmentally friendly - eliminate dangerous and expensive solvents. Non flammable.
- Equivalent performance to solvent based but with superior packing and dispersion performance - especially with Nano-size powders.
- Long history of successful use in production tape casting facilities since 1998.
- Water based but acts similar to solvent based.
- Reversible water solubility allows tape water resistance yet ability to rework old tape with pH adjustment.
- Cleans easily with water containing couple % ammonium hydroxide (window cleaner with ammonia also effective).

Demonstration Kit

- Due to various misconceptions regarding water based binders as well as differences in processing at various facilities many customers have had difficulties trying to institute water based tape casting.
- In addition not every ceramic responds to the exact same mixture of binder ingredients.
- PII has found the supply of a complete Demonstration Kit provides a working example with a known powder and can greatly accelerate implementation.
- This Kit provides all the ingredients and formula to produce tape from 0.5 micron alumina powder. Customer just to supply ball mill and grinding media. The small ball mill and media shown in this report can be supplied by PII for a nominal charge if desired.
- Tape should be cast per the formula and method outlined in this report and can be compared to example tape supplied by PII.

The Sample Kit Contains All Ingredients to Reproduce PII Results and Includes a Few Extra Helpful Ingredients for Other Ceramics

Water Based Tape Casting Demonstration Kit		
WB4101	2 liters	Most Popular PII WB Binder
DF002	100g	All Organic Defoamer
PL002	100g	Most effective plasticizer if increased lamination or cutting performance is required
PL005	50g	Very high pH plasticizer useful for reactive ceramics and to enhance dispersants (glass bottle packed inside vermiculite powder)
DS001	100g	Dispersant compatible with WB4101
DS005	100g	Dispersant compatible with WB4101
TK004	8oz	Thick Casting Additives that work together to prevent cracking and ceramic settling by stabilizing thick sections of cast slurry during drying. ***Please refer to ThickTapeCastingDemo.pdf for proper usage guidelines***
TK005	8oz	
A16SG Alumina	400g	Almatis Alumina powder used in example formula
KE2-6	.3 meter	Example Tape cast at PII
SiPET Casting Film	6 meters	Example of suitable silicone treated PET casting film (release coating on only one side)
CD	1	CD containing SDS, Certs, Pictures of Process and other useful technical articles and resources.

The Volume of the Empty Ball Mill was Calculated from the Dimensions. The Amount of Media and Other Ingredients is Calculated from the Mill Volume.

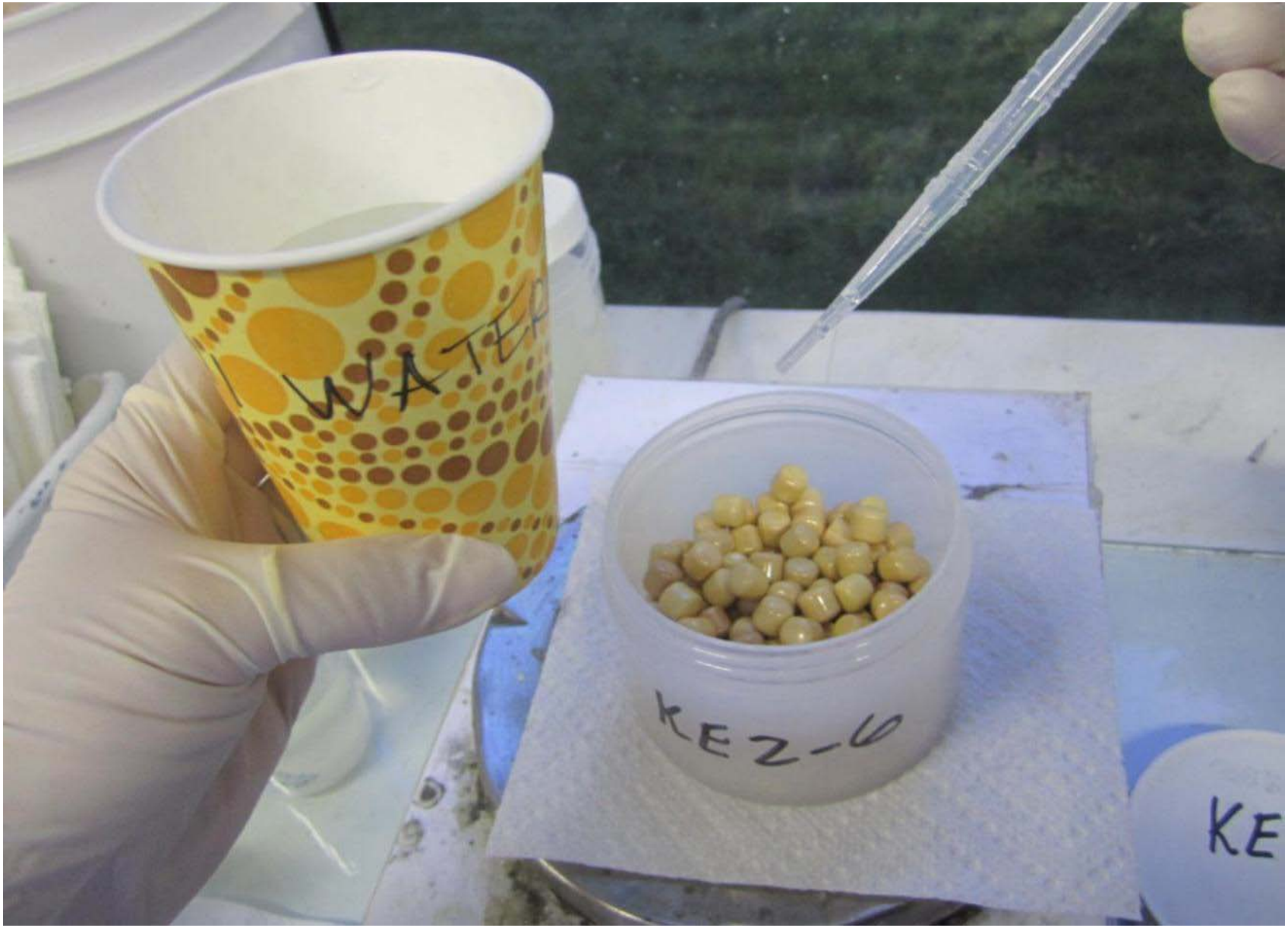
PII Provides Batching Mill Sheet. Insert Mill Volume in Liters and Spreadsheet Calculates the Formula and Proper Amount of Milling Media to add .The actual Active Excel Spreadsheet with Formulas can be Found on the Supplied CD.

			MILL CHARGES CLEAN Ball Mill						
KE2-6	Formula		Formulas in Grams for **Clean** Mill Of Given Size						
Mill size (liters)			100	75	50	15	10	5	0.307
Mill Size (gallons)			26.4201	19.8151	13.21	3.96301	2.64201	1.321	0.08111
Dielectric									
STAGE 1									
Almatis A16SG Alumina	56.00		29715	22286	14858	4457	2971.5	1485.8	91.23
WB4101	11.00		5837	4378	2918	876	583.7	291.8	17.92
DF002 (defoamer)	0.10		53	39.8	26.5	8.0	5.3	2.7	0.16
DI Water	21.80		11568	8676	5784	1735	1156.8	578.4	35.51
STAGE 2									
WB4101	11.00		5837	4377.7	2918.4	875.5	583.7	291.8	17.92
DF002 (defoamer)	0.10		53	39.8	26.5	8.0	5.3	2.7	0.16
Total	100		53063	39797	26531	7959	5306	2653	163
Milling time first (hours)			6 to 16	6 to 16	6 to 16	6 to 16	6 to 16	6 to 16	6 to 16
Mixing time second (hours)			4	4	4	4	4	4	4
Recommended mill RPM			33-36	35-38	**	**	**	56	**
Aim pounds of YTZ media for mill			378.9	284.1	189.4	56.8	37.9	18.943	1.163
Aim first stage slurry volume (gal)			6.61	4.95	3.30	0.99	0.66	0.33	0.02
Aim first stage slurry weight (grams)			47172.6	35379.4	23586.3	7075.89	4717.26	2358.63	144.82
Aim slurry final weight (grams)			53062.5	39796.9	26531.3	7959.38	5306.25	2653.13	162.902
Calculated organic solids	7.900		ratio of slurry vol to mill vol below						
Calculated ceramic in tape	87.64		0.25	0.25	0.25	0.25	0.25	0.25	0.25
Ceramic density	3.7								
Calculate ceramic volume %	65.70								
Theoretical green tape density	2.77								
Estimated first stage density	1.89								
Estimated final slip density (g/cc)	1.73								

** 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).

The Mill Should be Filled Half Full With Media for Proper Milling. Formula Calculates at Rate of 1.7 kg Zirconia Media or 1.1 kg Alumina media per 1 Liter Total Mill Volume.

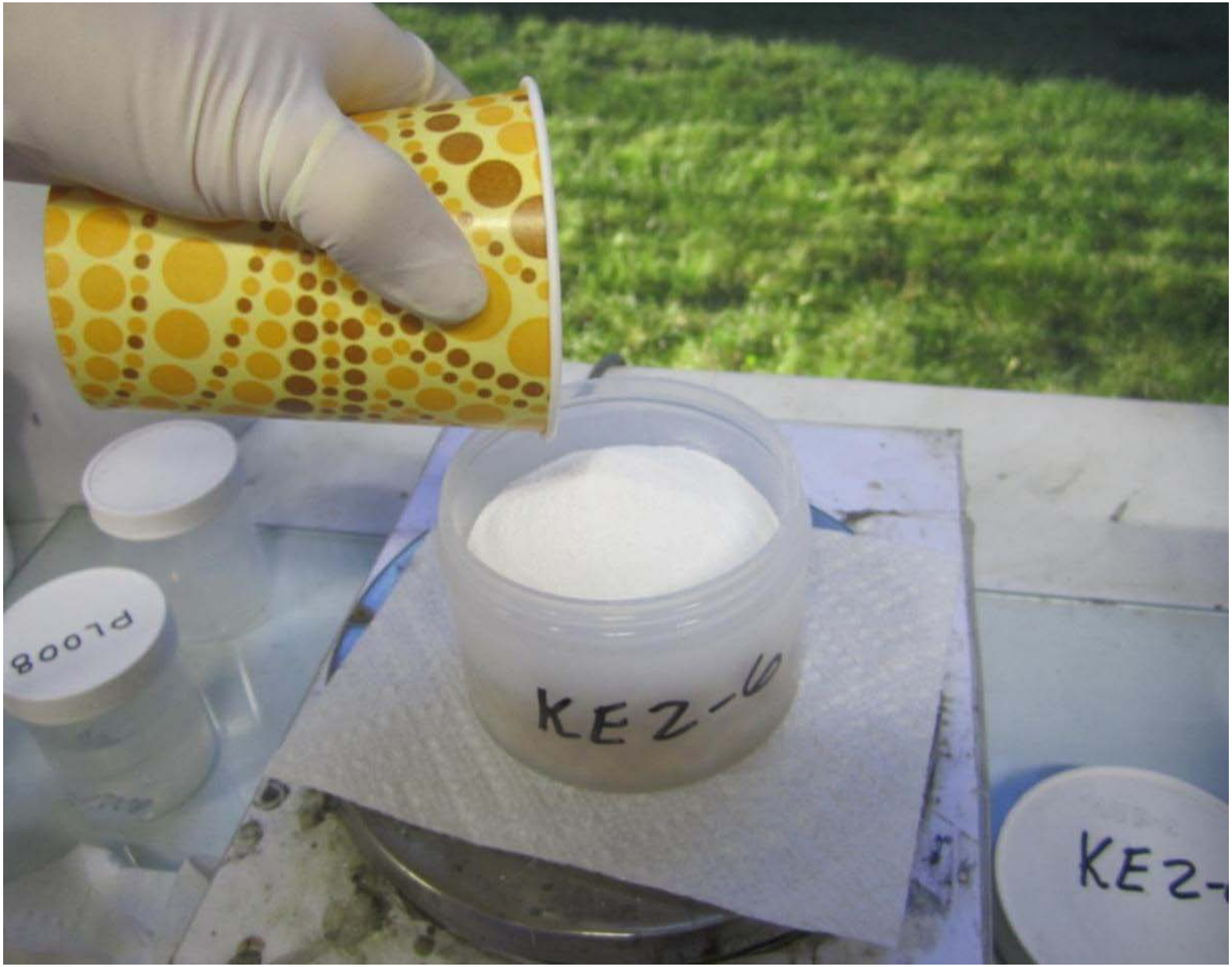
Deionized Water (Distilled Water also Acceptable) is Added to the Ball Mill



The WB4101 Water Based Binder is Added to the Ball Mill

The DF002 Defoamer is Added to the Ball Mill

The Almatis SG16 Fine Alumina Powder is Added to the Ball Mill



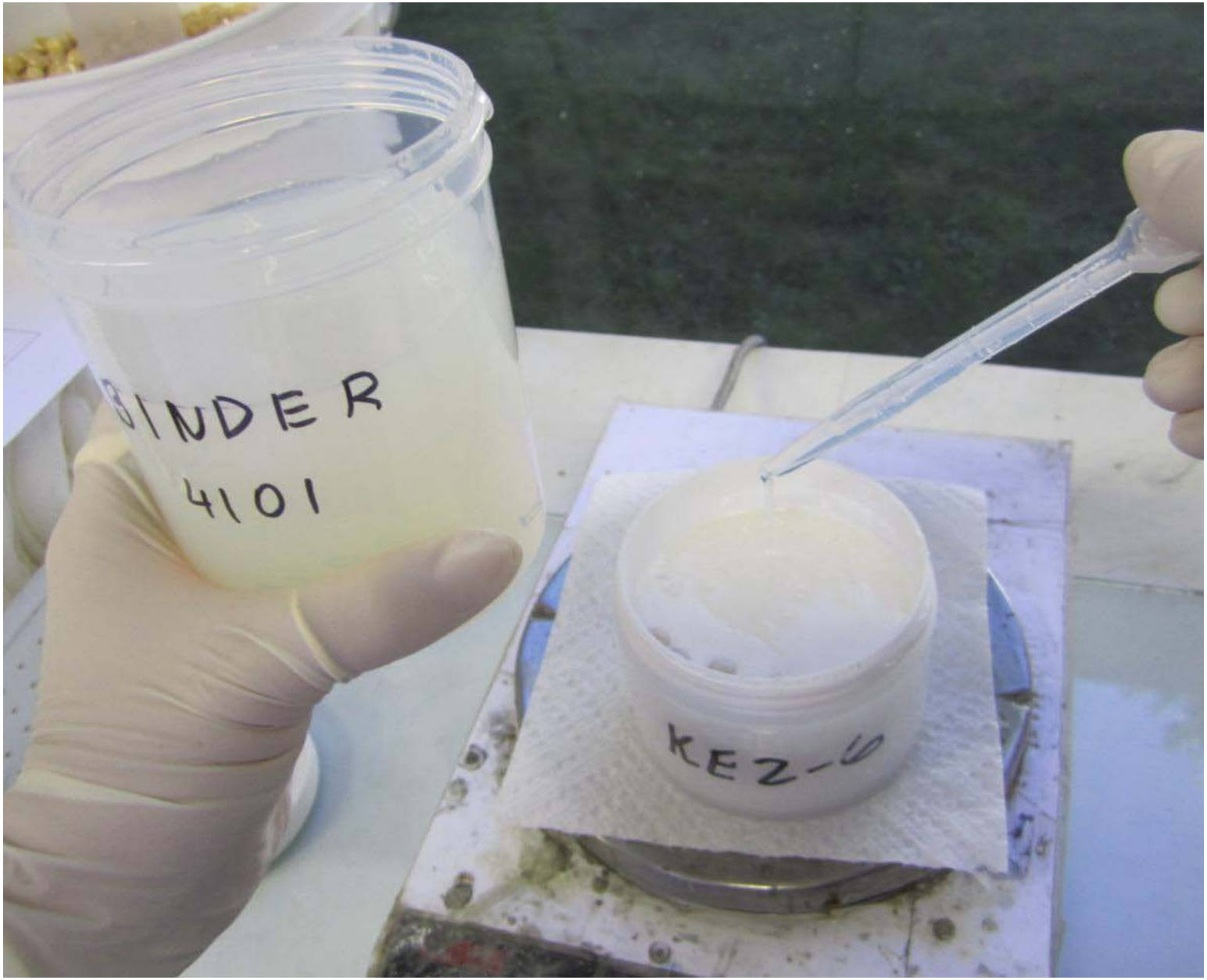
The Ball Mill was Placed on a Roll Rack for 16 Hours at 92 rpm. Electrical Tape was Used to Keep the Lid from Coming Loose. The Correct rpm for the Mill is Calculated from the Inside Diameter of the Mill Jar (Formula on supplied CD).

The Mill was Opened After 10 minutes just to Show the Bubbles and Viscosity. PII Sometimes also uses Teflon Pipe Tape on the Jar Threads if Leaking is a Problem.

The Mill Jar Was Opened after 16 Hours of Milling to Show Typical and Acceptable Foam Level and Lower Viscosity

The DF002 for the Second Stage of Milling was added

The WB4101 for the Second Stage of Milling was Added

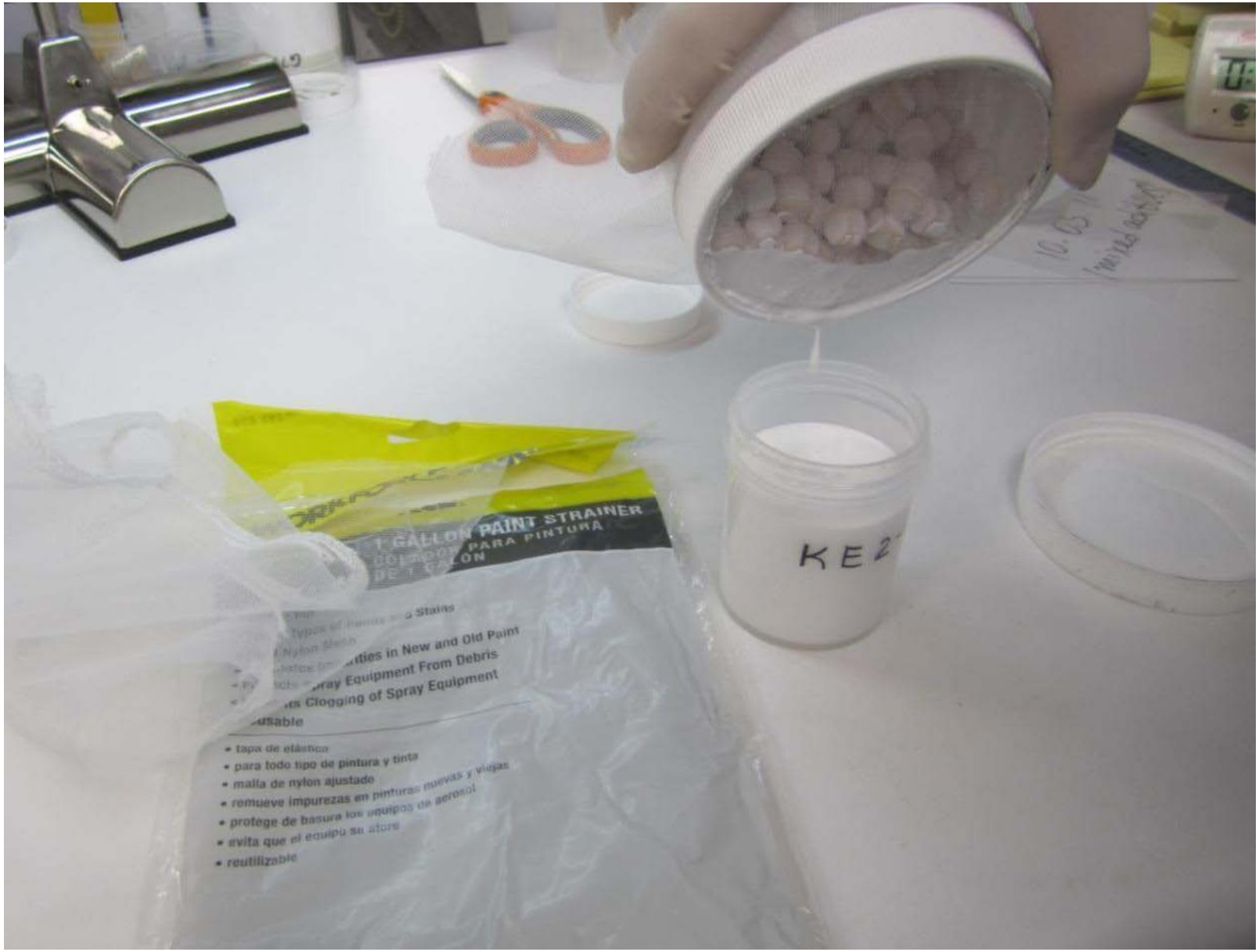


The View of Unmixed Second Stage Ingredients Before Further Milling

The Mill was Placed Back on the Roll Rack for 4 Hours for Second Stage Milling

For Ease of Draining a Mill Paint Strainer and Cut Out Lid Are Used

The Finished Slurry (or Slip) is Drained from the Ball Mill. Mill Can be Cleaned with Water Containing Couple % NH4OH or Can be Re-batched



Close Up of Minor Foam on Freshly Drained Slurry. Easiest Way to Deair is to Allow Slurry to Sit Without Stirring for at Least Several Hours. For Other Powders if Slurry Viscosity is High or Needs to be Cast Immediately use Vacuum Deairing

The pH of the Slurry was Measured at 7.0. A Normal Range for this Powder is About 6.5 to 8.0 but for Other Ceramics the pH is Normally Higher. If the pH is less than about 6.5 the Binder Can be Unstable and Needs Adjustment

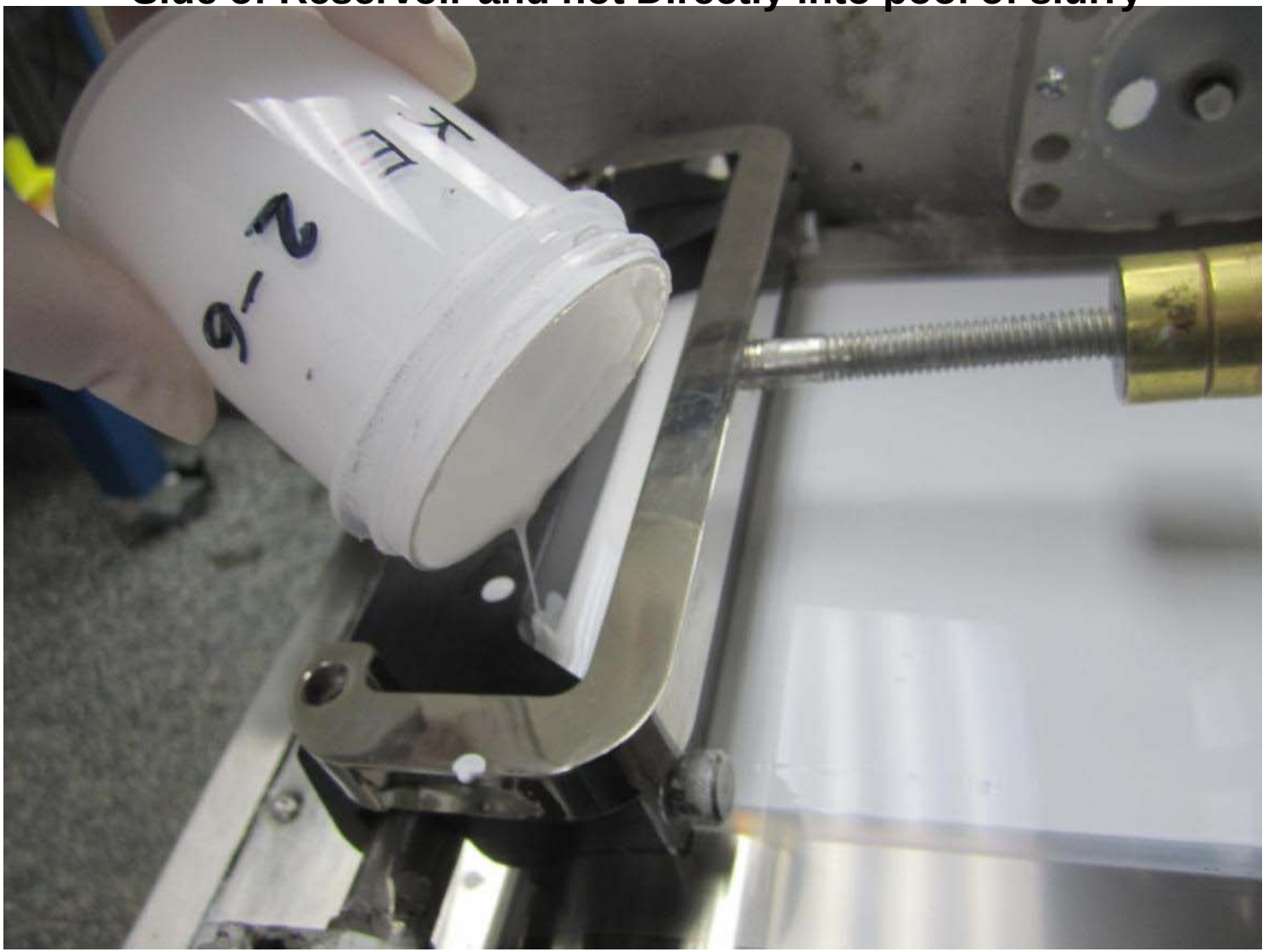
The Viscometer Used was a Brookfield HBT With Small Sample Adaptor

Close Up of the Small Sample Adapter SC4 21/13.

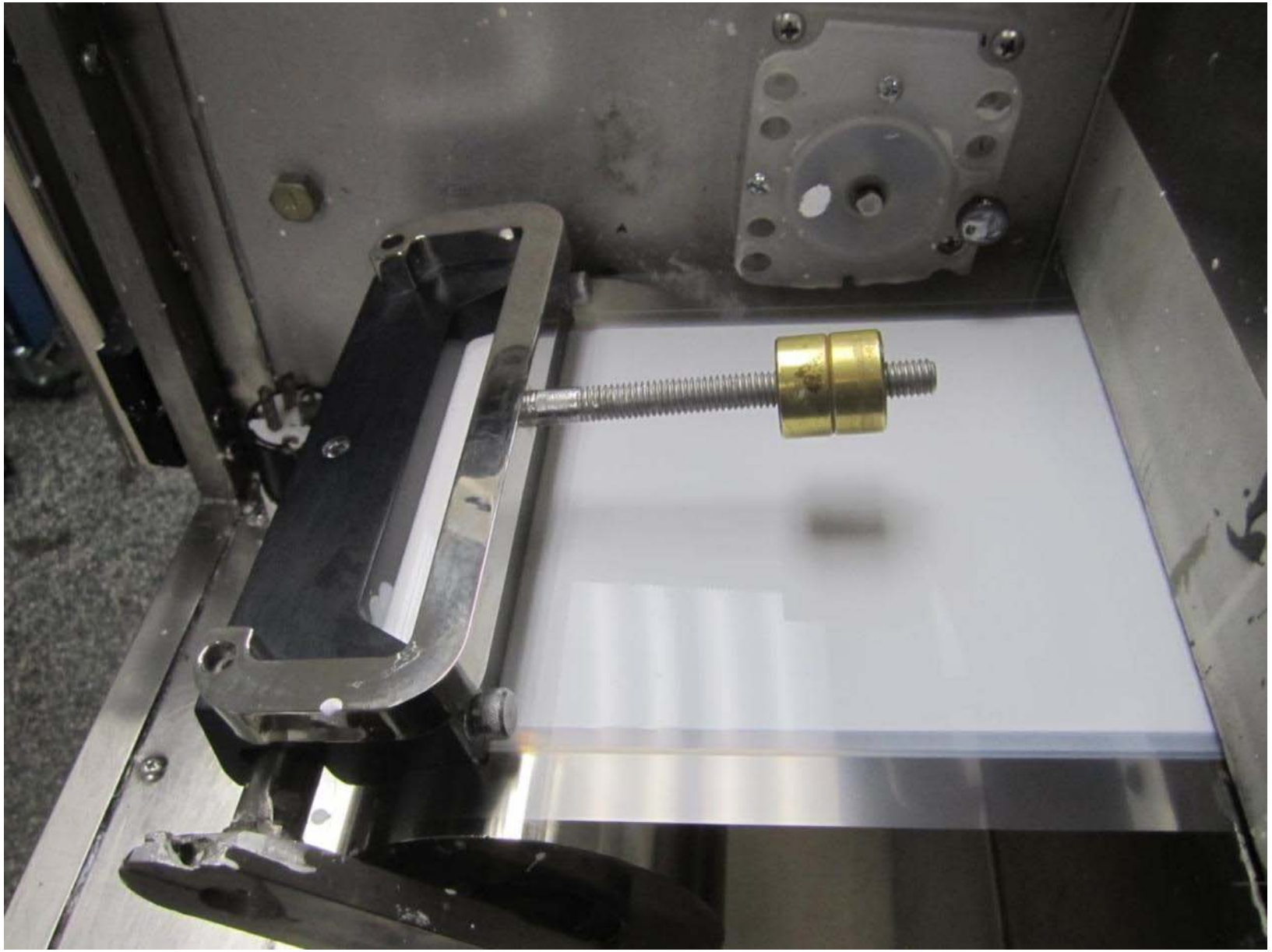
Slurry Viscosity Measured ~140 cps at 20 rpm (18 1/sec)

If Slurry Will Not be Cast Soon it is Stored on a Slow Roller. Roll Speed is about 1-5 rpm to Keep Slurry Homogenous. PII Uses a Cell Culture Roller for this Purpose

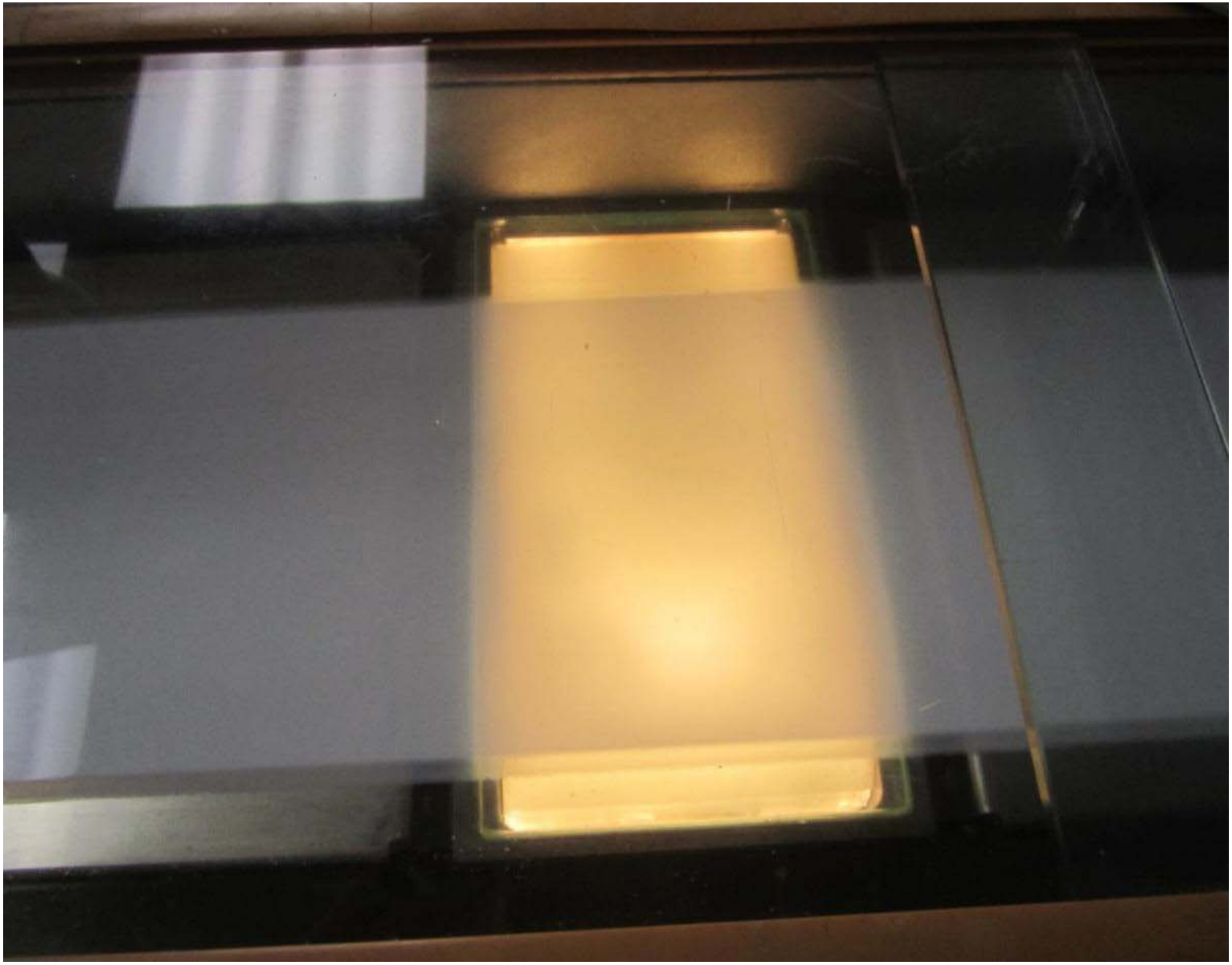
Photo of Slurry Being Cast using Dreitek Tape Caster with .0035 Inch Gap Doctor Blade. For Small Slurry Amounts Hand Pouring is Acceptable but Can Create Bubbles and therefore Pin Holes. For Less Bubbles it is Best to Pour Slurry on Side of Reservoir and not Directly into pool of slurry



Casting in Process



The Tape Passing over Caster Back Light for Observation

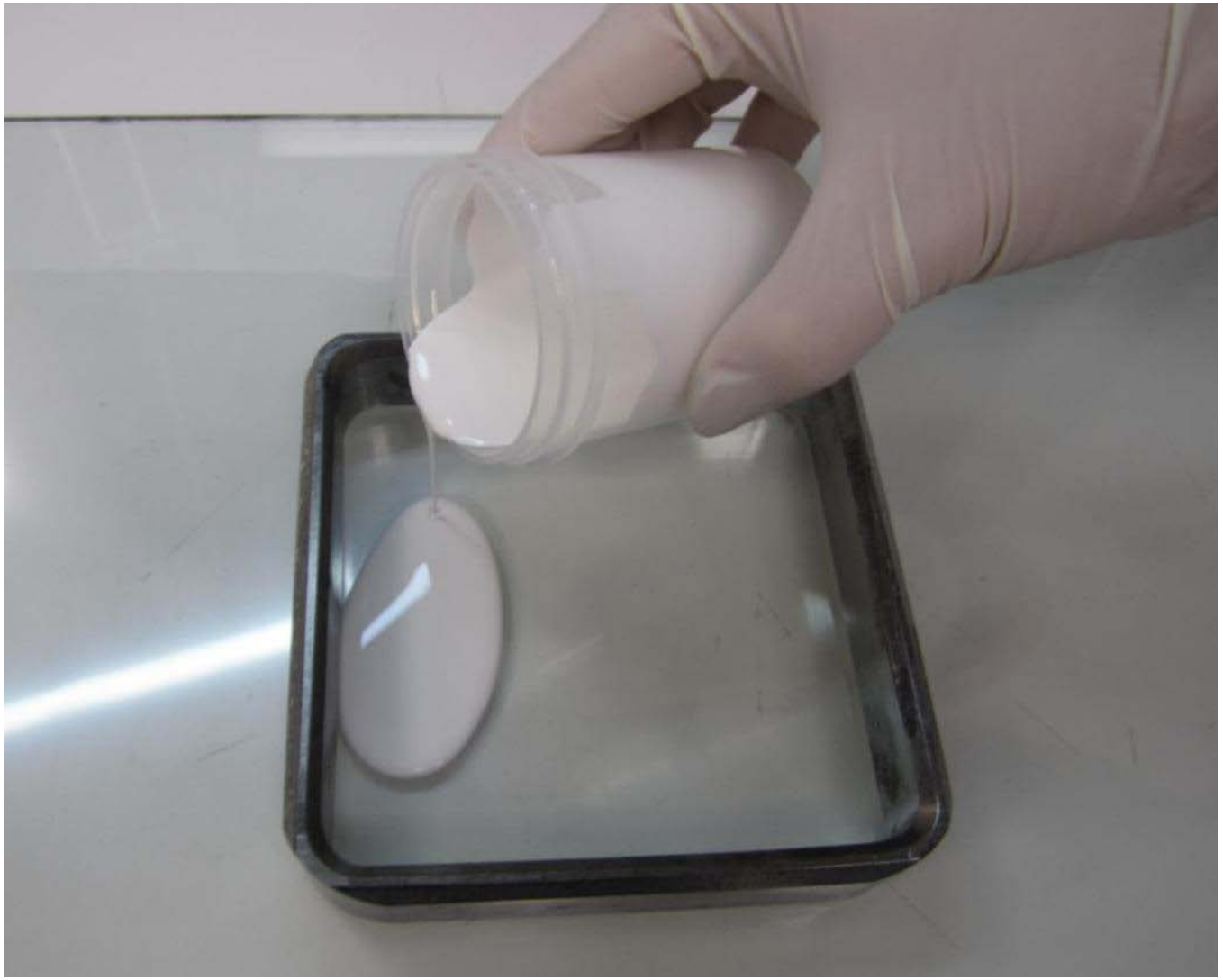


Zone Temperatures Depend On Tape Thickness, Caster Type and Desired Casting Speed (Typical Lab Settings Provided). Adequate Air Flow Depends on Caster.

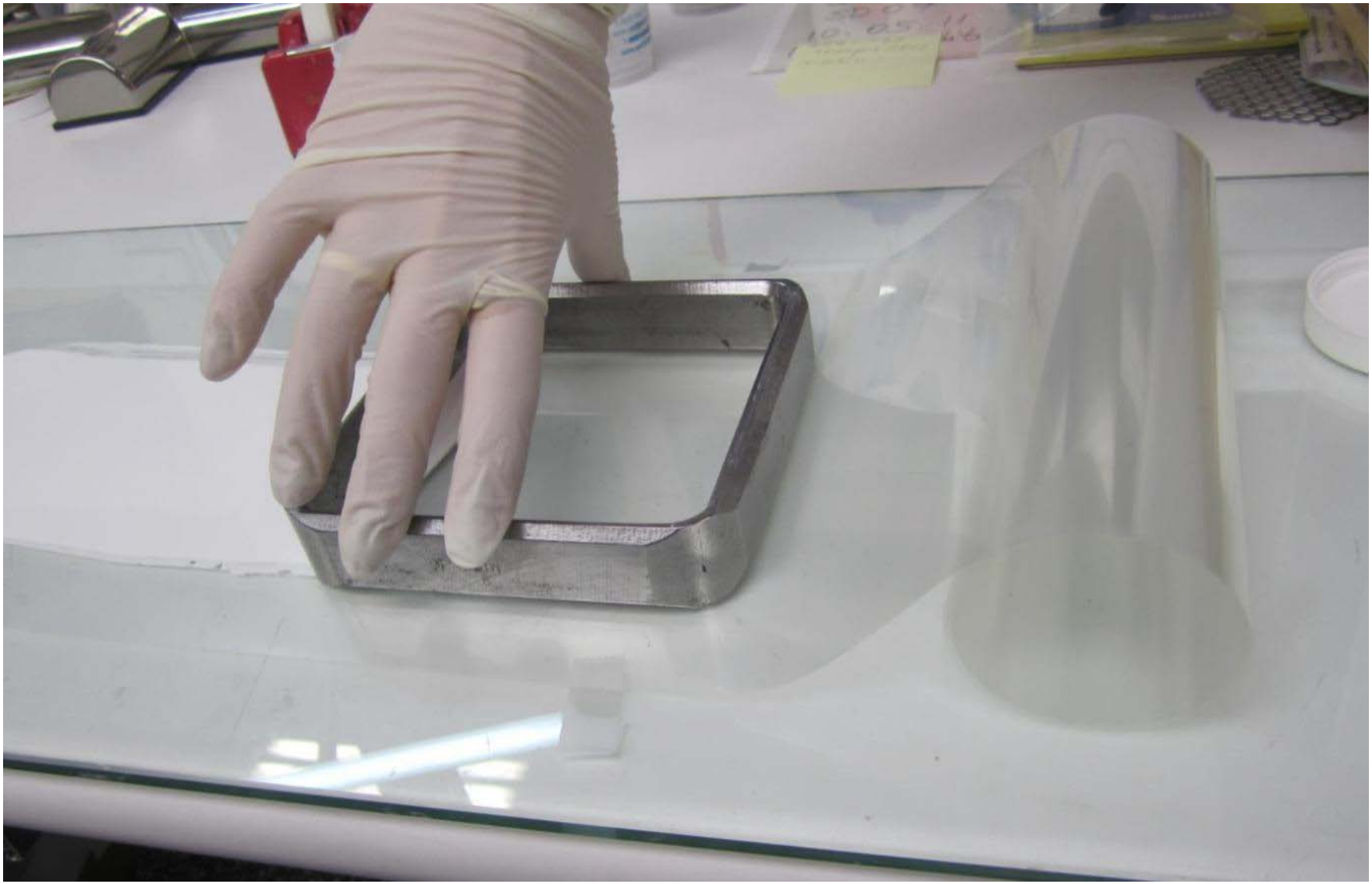
Typical Dreitek Settings	
Heater Zone #	Temperature (°C)
1	55
2	65
3	70
4	75
5	85

For Small Volume Experimental Purposes a Relatively Inexpensive Hand Doctor Blade Can be Used. Shown is an Example of 8 Side Doctor Blade with Gaps from 1 to 8 mils (25 to 200 Microns)

SiPET Rolled Out on Flat Glass Surface and Slurry Poured Into Hand Doctor Blade. This method will create thin spots as a result of air trapped under the siPET and glass but otherwise acceptable results. Tape can be left flat to air dry or hung from a vertical surface.



Casting Slurry with Hand Held Doctor Blade on to Release Side of siPET

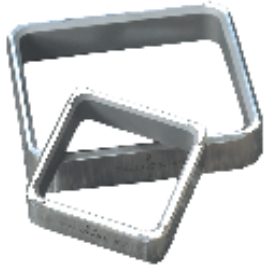


APPLICATION

8-PATH WET FILM APPLICATORS

For laying down wet films of materials in predetermined thicknesses. These precision wet film applicators are used in conjunction with paper charts, hiding power charts, plate glass or any other smooth, flat surfaces. They are simple, accurate experimental or quality control devices that require no adjustments.

GENERAL INSTRUCTIONS



A small quantity (depending on length and thickness of film required) of the material to be checked is placed on the inside of the channel near the correct opening for the thickness desired. The applicator is then drawn down over the chart or other surface and a uniform thickness of film is produced for observation and test. A path with a depth or a clearance of one mil will lay down a film approximately 1/2 mil thick. A latex film will shrink considerably more, so paths of greater depths are required in order to obtain a dry film thick enough to check flexibility and toughness.

Film thicknesses deposited may vary from 40% to 80% of the actual clearance.

- Adhesives
- Board
- Ceramic Colors
- Cosmetics
- Driers
- Duplicating Inks
- Resins
- Lacquers
- Cellulose
- Oils
- Pigments
- Plastics
- Printing Inks
- Waxes & Hot Melts
- Metallic Powders
- Floor Polishes
- Varnishes
- Fillers
- Emulsions

The 8-Path applicators are available in three widths

CAT. NO.	Overall Width	Path Width	Actual Path Depth, Mils	Approx. Wet Film Thickness	Stainless Steel
1	3"	2"		0.5, 1, 1.5, 2,	$476.00
14	4"	3"	1, 2, 3, 4, 5, 6,	2.5, 3, 3.5,	521.00
15	5"	4"	7, 8 mils	4 mils	581.00
2	3"	2"		2.5, 5, 7.5	$493.00
24	4"	3"	5, 10, 15, 20,	10, 12.5, 15,	535.00
25	5"	4"	25, 30, 40, 50	20, 25	598.00
3	3"	2"	0.5, 1, 1.5,	0.25, 0.5, 0.75,	$502.00
34	4"	3"	2, 3, 4, 5, 6	1, 1.5, 2, 2.5, 3	597.00
35	5"	4"			470.00

ORDER INFORMATION: To order please use the following example.

AP - (CAT. NO.) SS

Example of Hand Casting Doctor Blades from Paul N. Gardner Company. These casting heads are square and have 8 different casting gaps.

http://www.gardco.com/app.cfm

Example Data for Alumina Formula to Demonstrate Use of WB4101 Type Water Based Binder and Additives		
Sample Number	KE2-6	
ACTUAL FORMULA (grams):	%	Grams
First Stage:		
Alumina A16-SG (Almatis)	56.00	67.20
WB40B-53		
WB4101	11.00	13.20
DS009		
DF002	0.10	0.12
PL002		
Ammonium hydroxide		
DI water	21.80	26.16
Second Stage:		
WB40B-53		
WB4101	11.00	13.20
DF002	0.10	0.12
Total	100.00	120.00
Total organic solids	7.90	9.48
Ceramic density	3.70	3.70
Volume Ceramic Loading in Tape	65.70	65.70
Grams of media (3\8" cylinders)	528g in 300 ml jar	
First stage mill time (hours)	**16 hr @ 92rpm**	
Second Stage mill time (hours)	**4 hr @92rpm**	
25 C Viscosity HBT SC4 21/13R	reading	CPS
Stage 1 visco at 100 rpm	0.8	32
Stage 2 Slip Properties:		
25 C Viscosity HBT SC4 21/13R	reading	CPS
2.5 rpm	0.1	160
5 rpm	0.2	160
10 rpm	0.3	120
20 rpm	**0.7**	**140**
50 rpm	1.4	112
100 rpm	2.4	96
Foam stage 1	0.8	
Foam stage 2	0.8	
pH	7.05	
Cast method	Dreitek	
Number of layers in the stack	6	
Stack Thickness corner 1 (inches)	0.0047	
Stack Thickness corner 2 (inches)	0.0047	
Stack Thickness corner 3 (inches)	0.0046	
Stack Thickness corner 4 (inches)	0.0046	
Stack Thickness center (inches)	0.0047	
Average Stack Thickness (mils)	4.64	0.00
Average tape thickness (mils)	0.77	#DIV/0!
Weight (g)	0.7394	
Theoretical tape density (g/cc)	2.77	2.77
Green Density (g/cc)	2.47	#DIV/0!
Approximate % porosity	10.8	#DIV/0!
Puncture Test (grams) spl1	135	
Puncture Test (grams) spl2	125	
Puncture Test (grams) spl3	140	
Puncture Test (grams) spl4	115	
Average Puncture Test (grams)	129	0
Average Puncture (grams/mil)	**166**	**#DIV/0!**
Crease Strength (~ % of crease intact)	20	

Comments and Precautions

- If processing has been performed as per the instructions the resulting tape should be similar to the cast tape example supplied by PII.
- It is helpful to use a piece of adhesive “Scotch” tape to adhere to a corner or edge of the cast tape to help to release it from the siPET casting substrate.
- If there are problems please contact PII for assistance.
- If WB slurry comes in contact with bare aluminum metal it will create ceramic agglomerates and casting problems. So ensure there is no bare aluminum contact. Alternately if the aluminum metal is painted, covered with adhesive tape or anodized it will prevent the reaction.
- WB4101 binder is a strong dispersant which is why typically half to a third of the total amount of binder is in the first stage. The use of common water based dispersants typically causes a 70% loss in strength. PII supplied dispersants do not cause this problem. Further adding strong base to the first stage increases the dispersing nature of the binder or compatible dispersants.

Customer Ceramic / Scale Up

- The best concept for tape formulation is to load the tape to a similar volume level since ceramic densities vary considerably. This is covered in more detail in another article on the included CD.
- As a first attempt with a new powder, calculate the volume of the alumina in this provided example tape formula (alumina weight/actual density (3.8)) and replace it with an equal volume of the new ceramic. Due to chemical and surface area differences the level of water or additives will probably need to be adjusted for acceptable results with different ceramics.
- The milling times given in this presentation (16 hour first stage and 4 hours second stage) are appropriate for small lab scale ball mills less than a liter but larger ball mills provide more energy and first stage milling times might be more like 2-8 hours and second stage 2-4 hours. The second stage milling time and mixing is important since the WB4101 binder is surface active and needs time and mixing to equilibrate with the ceramic surface.
- PII has formulated WB tapes with hundreds of different ceramics, glasses and metals and can provide some suggestions of appropriate additives or for a reasonable fee develop a formula with customer supplied powder.
- PII formula development is the fastest route to scale up, all formulas and test data are provided to the customer and the work can be performed confidentially.

Order of Addition Comments

- Order of addition of ingredients can become more critical in large production ball mills. For small mills they can easily be shaken by hand and are more forgiving.
- Media can be in the ball mill first or can be added after ingredients.
- It is usually best to put the liquid additives in the mill first.
- Some binder additives when mixed with WB4101 in concentrated form can cause the formation of temporary regions of high viscosity or even polymer precipitation. So it is often more convenient to add the water to the mill first followed by binder and additives. The mill can be mixed briefly in-between additions if desired.
- Some ceramic powders can be very fluffy and will not fit in the ball mill in just one portion. In this case the amount of powder can be divided into two or three portions, the ball mill run for a few minutes in-between each addition to help wet the powder out and reduce its volume.
- Addition of thick casting ingredients TK004 and TK005 require special attention and is covered in specific thick casting literature.

Partial Product List* and Some Formulating Guidelines

Product	Solids (%)	Typical level (wt.% in slip)	Comments
Typical Water Based Binder Systems:			
WB4101	35%	10 to 30	Water based binder with basic defoamer, wetting agents and other additives. Binder also acts as ceramic dispersant and therefore typically 25-50% of the total binder should be included in the grinding stage. (28% polymer and 7% plasticizer)
WB40B-53	26%	10 to 30	Similar to above (same polymer type) but without any of the plasticizer used in the WB4101. It is useful for making more stiff and higher thickness (100 Micron+) tapes. Less plasticizer seems to make the thick tapes less sensitive to drying induced cracks. Also see TK additives for thick casting.
Binder Additives:			
Defoamers			
DF001	100	.05-.25	Modified silicone copolymer especially effective when combined with DF002 but with some powders can cause crater or fish eye defects. Must be careful to not over dose as it can cause casting defects such as fish eyes.
DF002	100	.05-1.0	Most popular and useful defoamer. Non-silicone mild defoamer most compatible so over dosing is not critical.
Plasticizers			
PL002	100	.2-1.5	High pH plasticizer, similar to PL001 but more reactive with binder. Also increases binder dispersion power if included in stage 1. Due to high pH can make dried tape more susceptible to water attack.
PL005	100	.2-1.0	Very high pH plasticizer, similar to PL002 but even more reactive with binder. Strong reactivity can help manage certain difficult powders. If very reactive ceramics are causing problems such as precipitation of binder or adverse tape reaction it is often helpful to include PL005 in the formula. It also increases the dispersion power of the WB4101 or other used dispersants if it is included in the grinding stage allowing effective dispersion of nano powders. Due to high pH can make dried tape more susceptible to water attack.
PL008	~100	.1-1.0	Semi-volatile plasticizer can be used as a low odor replacement for ammonium hyrdroxide, slurry clean up and help stabilize reactive ceramics.
Tape Release agents			
BR008	100	.5-1.5	Internal belt release additive. Added to first or second stage will help tapes release from steel belt casting surfaces. Should avoid premixing with the binder as it can cause temporary binder precipitation. Since use of internal belt release compromises tape properties to achieve steel belt release an external belt release such as BR021 is usually the best option.
BR010	100	.01-.2	Modified silicone helpful in preventing pinholes, craters and mud cracking. It is particularly useful if it is noticed thicker tape is weaker than thinner tape and it is helpful if casting when mixing solvent with a WB system (hybrid water/solvent systems). BR010 is also useful to reduce the adhesion of cast tape to silicone treated PET film.
BR021	100	NA	Best used as an external belt release additive for pre-coating on the steel belt to allow easy release of the cast tape from the belt. Only a very thin film is required. Typically a dilute solution of about 5-10% in hydrocarbon such as naptha or toluene is made and a very thin film applied to the steel belt by means of a felt wick type applicator or roller far enough in front of the casting head so the solvents are dry. The BR021 can also be high shear mixed into a water emulsion for application if hydrocarbons should be avoided.
Additives to allow thicker casting (Useful when casting greater than ~ 100 microns depending on the ceramic).			
TK004	30	.3-.6	Typically used about .4% TK004 and TK005 together. These additives work to stabilize thick sections of tape during drying to allow thicker casts. They help prevent cracking and ceramic settling. They can also stabilize small bubbles so require more rigorous defoaming or defoaming additives. Order of addition of these additives is critical. Please refer to the specific literature on thick casting.
TK005	100	.3-.6	See TK004.
Dispersing Agents			
Note: Normally the best dispersant for WB4101 is actually WB4101 since it is similar in nature to popular ammonium polyacrylate dispersants. The use of the popular ammonium polyacrylate dispersants will result in dramatically lower tape strength. Below are compatible dispersants for specific applications.			
DS001	40	.5-2.0	Strong polymeric dispersant useful for most ceramics and compatible with the WB binders. Usefull with or without any other binder in dispersing stage.
DS005	50	.3-1.5	first stage.
DS009	65	.3-1.5	Strong polymeric dispersant with different properties than DS005 which makes it especially useful for zirconia ceramics and can be tried if DS005 does not work well.
Wetting Agents			
BR010	100	.01-.2	Modified silicone helpful in preventing pinholes, craters and mud cracking. It is particularly useful if it is noticed thicker tape is weaker than thinner tape and it is helpful if casting when mixing solvent with a WB system (hybrid water/solvent systems). BR010 is also useful to reduce the adhesion of cast tape to silicone treated PET film.
WT007	100	.01-.15	Modified silicone helpful in preventing pinholes, craters and mud cracking. May increase foam and reduce ability to laminate. It is particularly useful if it is noticed thicker tape is weaker than thinner tape (sign of mudcracking). Also it is helpful if casting when mixing solvent with a WB system.
WT012	50	.05-.5	A modified silicone with main effect of increasing the wet ability of the slurry on silicone treated PET film. Rarely needed if proper siPET film used and slurry viscosity and drying controlled.
Other PII Product Lines: PII also produces compatible organic paste binders as well as custom screen printing pastes.			
*Partial listing. Contact PII for other products and details.			