Butvar®

Polyvinyl Butyral Resin

Ceramic Binder Applications Technical Bulletin

Butvar®

SOLUTIA® Solutions for a better life.®

Introduction

Butvar® polyvinyl butyral resins are recognized as the fugitive binder of choice in the processing of ceramic tape cast materials. The resin imparts excellent green strength and flexibility to the ceramic tape. It is compatible with many common solvents and plasticizers and burns out cleanly during sintering.

Butvar resins also are used as binder media in thick film processing. Butvar resins are formulated in the solvent vehicle used to deposit the circuit pattern on the ceramic surface. The primary advantages of using Butvar resins are their solubility in a wide range of solvents and uniform adhesion to conductive metals.

Typical Properties: Physical Characteristics (white, free-flowing powder)

Physical Characteristics:	Units	Method	B-76	B-79	B-90	B-98
*Volatiles, max.	%		5.0	5.0	5.0	5.0
Molecular weight (weight average in thousands)	-	(1)	90-120	50-80	70-100	40-70
Solution viscosity 15% by weight	cp.	(2)	500-1,000	100-400	600-1,200	200-400
Solution viscosity 10% by weight	cp	(2)	200-450	75-200	200-400	75-200
*Oswald solution viscosity	cp.	(3)	18.0-28.0	9.0-16.0	13.0-17.0	6.0-9.0
*Hydroxyl content expressed as % polyvinyl alcohol	-	ASTM D1396-58	11.5-13.5	11.0-13.5	18.5-20.5	18.0-20.0
Acetate content expressed as % polyvinyl acetate	-	ASTM D1396-58	0-2.5	0-2.5	0-1.5	0-2.5
Butyral content expressed as % polyvinyl butyral, approx.	-	-	88	88	80	80

1. Molecular weight was determined via size exclusion chromatography with low-angle laser light scattering (SEC/LALLS) method of Cotts and Ouano in P. Dublin, ed., Microdomains in Polymer Solutions (New York: Plenum Press, 1985), pp. 101-119.

2. Solution viscosity was determined in 15% by weight solutions in 60:40 toluene ethanol at 25ºC, using a Brookfield Viscometer. Also in 10% solution in 95% ethanol @ 25ºC using an Ostwald Viscometer.

3. Specification viscosity for each product type measured with an Ostwald-Cannon-Fenske Viscometer. The solvents and solids levels used are as follows:

Product	Percent Solids	Solvent	Temperature (ºC)
B-76,B-79	5.0	SD 29 E Ethyl Alcohol	25
B-90,B-98	6.0	Anhydrous Methanol	20

*Specification properties

Chemical Solubility*

Solvent	Butvar* B-76, B-79	Butvar* B-90, B-98
Acetic Acid (Glacial)	S	S
Acetone	S	SW
Butyl Acetate	S	PS
N-Butyl Alcohol	S	S
Butyl Cellosolve™	S	S
Cyclohexanone	S	S
Diacetone Alcohol	S	S
Diisobutyl Ketone	SW	I
N, N-Dimethylacetamide	S	S
N, Dimethylformamide	S	S
Dimethyl Ester	S	PS[1]
Dimethylsulfoxide	S	S
Ethyl Acetate, 99%	S	PS
Ethyl Acetate, 85%	S	S
Ethyl Alcohol, 95% or Anhydrous	S	S
Ethylene Dichloride	S	SW
Ethylene Glycol	I	I
Isophorone	S	S
Isopropyl Alcohol, 95% or Anhydrous	S	S
Isopropyl Acetate	S	I
Methyl Acetate	S	PS
Methyl Alcohol	SW	S
Methyl Ethyl Ketone	S	I
Methylene Chloride	S	S
Methyl Isobutyl Ketone	S	I
Naphtha (Light Solvent)	SW	I
n-Methyl-2-Pyrrolidone	S	S
Propylene Dichloride	S	SW
Santosol™ DME-1	S	PS[1]
Tetrachloroethylene	SW	SW
Tetrahydrofuran	S	S
Toluene	PS	SW
Toluene: Ethyl Alcohol, 95% (60:40 by weight)	S	S
1, 1, 1-Trichloroethane	S	SW
Xylene	PS	SW

Key: **S-** soluble **PS-**partially soluble **I-**insoluble **SW-**swells

*10% solids solution agitated for 24 hours at room temperature.
[1] clear solution at 80°C.

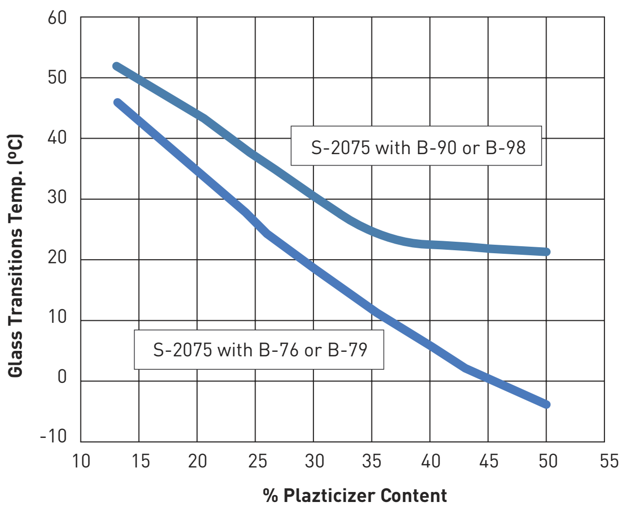
SOLUTIA

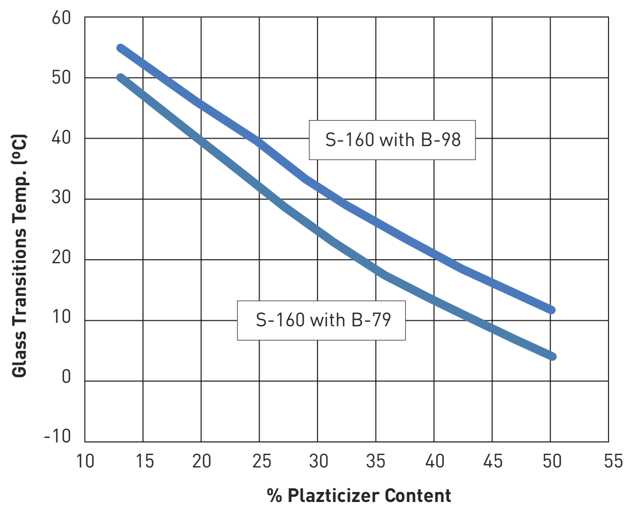
Plasticizer Compatibility

Plasticizer	Maximum Plasticizer : Butvar® Compatibility Level
Solutia S-2075 Plasticizer (Triethylene Glycol Di-2-Ethylhexanoate)	1:1 (B-76, B-79) 1:2 (B-90, B-98)
Santicizer® 160 (Butyl Benzyl Phthalate)	1:1
Alkyl Phthalate	1:1
Dibutyl Phthalate	1:1
Dioctyl Phthalate	1:4
Dioctyl Adipate	1:4
Santicizer® 141 (2-Ethylhexyl Diphenyl Phosphate)	1:1
Tricresyl Phosphate	1:1
Triphenyl Phosphate	1:2

Effect of Plasticizer Content on Tg

(Butvar® with Solutia S-2075 or Santicizer® 160 Plasticizer)





The glass transition temperature (Tg) profiles were determined by Dynamic Mechanical Analysis (DMA) with premixed Butvar® and plasticizer samples pressed into sheets.

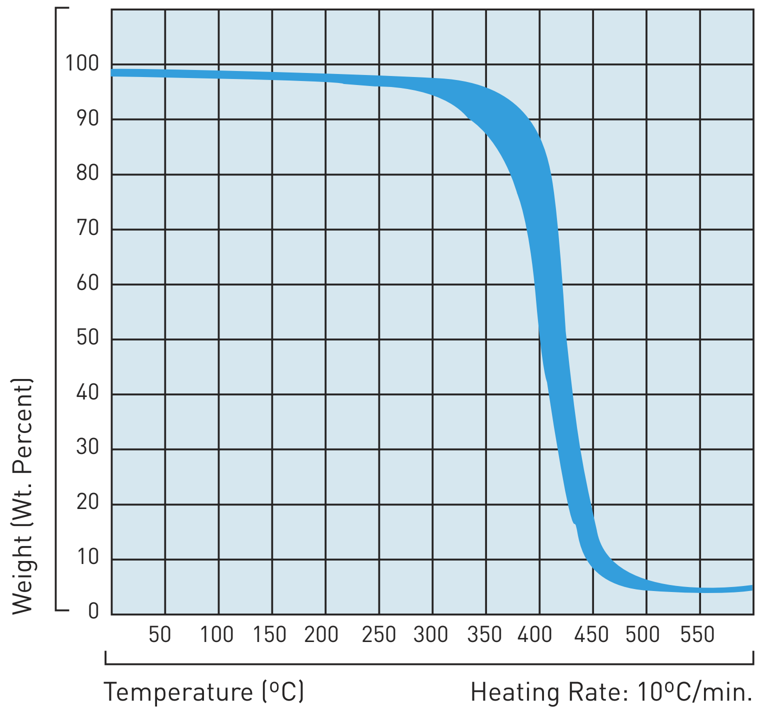
Thermal Properties

(Butvar® with Solutia S-2075 or Santicizer® 160 Plasticizer)

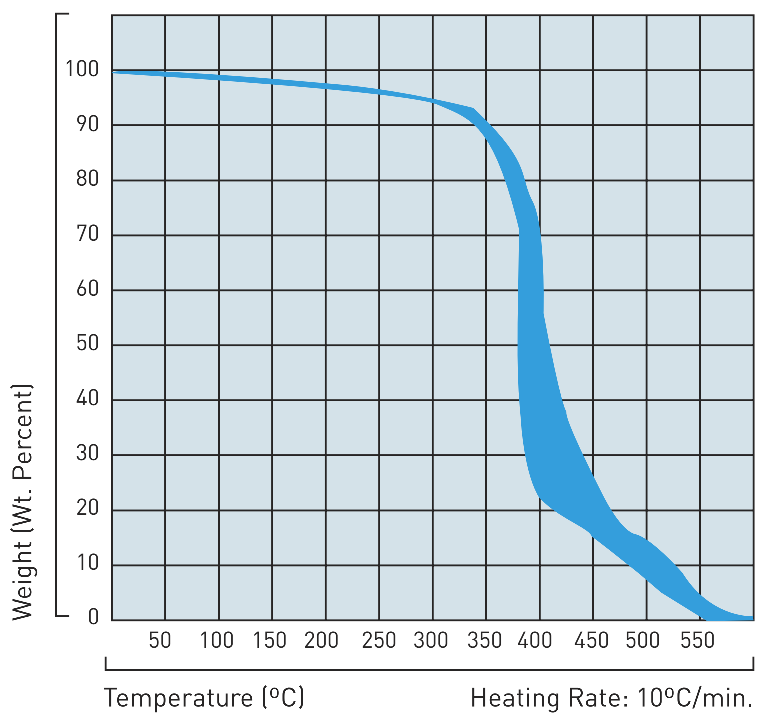
	Units	Test Method	Butvar® B-76, B-79	Butvar® B-90, B-98
Glass Transition Temperature, Tg	C	DSC	62-72	72-78
Ash Content at 550°C - In Nitrogen	%	TGA	<2.0	<3.0
Ash Content at 550°C - In Air	%	TGA	<0.75	<0.75

Glass transition temperature (Tg) was determined by Differential Scanning Calorimeter (DSC) over a range of 30°C to 100°C on dried granular resin.
Ash content of the Thermal Gravimetric Analysis (TGA) was determined as a weight loss versus temperature profile conducted at a heating rate of 10°C/min.

Thermal Gravimetric Analysis (In Nitrogen)



Thermal Gravimetric Analysis (In Air)



The Thermal Gravimetric Analysis (TGA) profiles of Butvar® weight loss versus temperature were determined at a heating rate of 10°C/min.

Applications

Thick Films

Butvar® resins can be used as a binder medium in vehicle formulations for thick film pastes. Our lowest molecular weight resins, Butvar B-79 and B-98, are recommended for either silk screen or steel screen processes. The advantages of using Butvar in thick films include:

- Butvar is an excellent binder and dispersant for the conductive metals used in thick films.

- Binder compatibility problems are minimized for co-firing systems when Butvar is used in both the thick film processing and as the binder in the ceramic tape casting process.

- Thick films with Butvar can be co-fired with the green tape in laminated ceramic substrates.

Tape Casting

Butvar® resins are regarded as the binder of choice for the ceramic tape casting process. Historically, the first referenced use for tape casting was in a U.S. Patent* filed in 1954 and issued in 1961. There are many reasons for this. The most important are as follows:

- Butvar resins provide excellent green strength to the unfired tape. This in turn allows the following:
  - Ease of handling during subsequent processing steps such as roll-to-roll metallization
  - Ease of lamination of multiple tapes using heat and pressure.
  - The use of low concentrations which yields higher unfired densities and as a result higher sintered densities.
- Butvar is soluble in many volatile, yet inexpensive, solvents. This in turn allows:
  - Flexibility in the selection of Butvar product types and concentrations to yield a wide range of binder solution viscosities and, therefore, ceramic slip viscosities.
  - Selection of a solvent system to meet governmental, environmental, safety and health restrictions.
- Butvar is compatible with many of the plasticizers used in ceramic tape systems. This allows:
  - Selection of a plasticizer or combination of plasticizers to yield the flexibility and plasticity desired in the unfired tape.
  - Selection from a list of phthalates, benzyls or phosphates. This includes a newly developed environmentally "friendly" plasticizer: S-2075 introduced by Solutia.
- Butvar burns out cleanly in an oxidizing atmosphere and relatively cleanly in nitrogen. This allows:
  - Uniform shrinkage during burn out with a minimum of warpage if the amount of organics utilized is optimized.
  - A minimum of surface defects due to low gel content in the dissolved binder system.
- Butvar has natural dispersing properties and is compatible with common dispersing agents, such as blown menhaden fish oils or phosphate esters.

The medium-to-low molecular weight resins, Butvar B-76, B-79, B-90 or B-98 are recommended for use in the tape casting process.

*J.L. Park, Jr., "Manufacture of Ceramics," U.S. Patent 2,966,719, January 3, 1961.

Typical Tape Casting Formulations and Procedures

A typical tape casting formulation based upon 100 parts by weight of solid ceramic powder is as follows:

Component	Parts by Weight
Aluminum Oxide	100.0
Butvar B-98	5.0
Santicizer® 160	2.5
Polyalkylene Glycol	2.5
Menhaden Fish Oil, Z-3	2.0
Xylenes	24.7
Ethyl Alcohol, 95% Denatured	24.7

Procedure: The procedure to be followed in batching a slurry for tape casting is almost as critical as the formulation itself since the final tape cast product depends upon following the process exactly. Following is a procedure which has been developed over many years and many tape casting batches:

- Dry the alumina powder at 90 to 100ºC for at least 24 hours.
- Weigh and dissolve the fish oil in the xylenes and add to a mill jar that will accommodate the alumina batch desired. The mill should be 1/3 filled with grinding media of the appropriate size for the mill; e.g. 1.27 cm cylindrical media for a one liter mill. The mill and media composition should be selected based upon the materials being processed: an alumina mill with alumina media would be selected for this formulation.
- Weigh and add the ethyl alcohol.
- Weigh and add the hot alumina powder.
- Dispersion mill by rolling at 58 rpm for 24 hours.
- Weigh and add the S-160 and the polyalkylene glycol plasticizers.
- Weigh and add the B-98 binder powder
- Mix and homogenize by rolling for an additional 24 hours at 58 rpm.
- Pour and de-air in a vacuum chamber with agitation at 635 mm of mercury for a minimum of 8 minutes (time depends upon batch size).
- An option at this point would be to filter the slip to remove agglomerates that may be left. Usually 50 to 100 micron filtration is sufficient.
- Check the viscosity of the slip for quality control.

At this point the slip is ready for tape casting. The carrier of choice is silicone-coated Mylar in a continuous tape casting machine. The blade gap, casting speed, etc. are selected to yield the tape thickness desired.

Following is a formulation for a 94% alumina tape utilizing Butvar as a binder and an environmentally friendly plasticizer:

Component	Parts by Weight
Alumina	92.0
EPK Kaolin Clay	2.0
Nytal 400 Talc	6.0
Butvar B-98	8.0
S-2075 Plasticizer	7.7
Polyalkylene Glycol	7.7
Menhaden Fish Oil, Z-3	2.0
Xylenes	21.4
Ethyl Alcohol, 95% Denatured	21.4

The procedure followed in the preparation of the tape casting slip is exactly as described in the previous example formulation.

The formulations reviewed here have been evaluated and tested to yield excellent tape cast products and the tapes have been sintered and compared to standard 99.9% alumina and 94% alumina substrate materials. The final sintered density, shrinkage during sintering, and properties of the sintered products have all been found to yield values which are equal to or better than standard control materials with S-2075 as the plasticizer in place of S-160. The Butvar concentration can be adjusted in the formulations to suit the use of the tape produced, e.g. the concentration can be raised to improve the lamination character of the tape produced.

Raw Material Sources

Product Designation	Supplier/Source
Butvar® Polyvinyl Butyral Resins, S-2075 Plasticizer	Solutia Inc.
Santicizer® Plasticizers (S-160, S-141)	Ferro Corp.
Polyalkyene Glycol, UCON50HB2000	DOW Chemical Co.
Blown Menhaden Fish Oil, Z-3 Viscosity	R.E. Mistler, Inc., Werner G. Smith, Inc.
EPK Kaolin	Hammil & Gillespie
Nytal® 400 Talc	R.T. Vanderbilt Co.

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