DMSO

Microelectronic Applications using DMSO (Dimethyl Sulfoxide)

Bulletin 165B

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Introduction

The manufacture of microelectronic devices uses a range of solvents in a variety of applications. Solvents are chosen based upon their properties to deposit coatings, remove photoresist and related polymers, and to carry out general cleaning activities. As the electronics industry continues to create new technologies and business models, serious challenges exist in their efforts to improve cleaning, comply with safety regulations, and to reduce cost. Dimethyl Sulfoxide (DMSO), a common solvent for many polymers, is a key solution to helping the industry meet their objectives. This bulletin provides pertinent information and guidelines in using DMSO in microelectronic applications.

Contents

Gaylord Chemical Company, L.L.C. (GCC) is th leading provider of Dimethyl Sulfoxide (DMSO) solutions. Beginning in the early 1960ôs, GCC has be to the development of new uses for DMSO. In order to meet customer-specific needs, GCC has pioneered the development of multiple grades of DMSO, including DMSO USP.

Gaylord Chem-icalôs based approach has contributed to the development and growth of industries including pharmaceuticals, hydrocarbons, electronics, polymers, coatings, agricultural chemicals, and industrial cleaners.

Gaylord Chemicalôs are located in Slidell, Louisiana with manufacturing, research, and development facilities in nearby Bogalusa, Louisiana. GCC remains the only producer of DMSO in the Western Hemisphere.

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Process Objectives

When designing and integrating a new process in microelectronic manufacturing, there are key objectives which must be achieved. Generally the process must meet minimum (a) performance and (b) selectivity criteria, while maintaining (c) stability for extended periods of time and (d) compatibility with a wide range of materials and construction commands pertinent in the tool. These are described in more detail below in Tabl

Table 1.
Microelectronic cleaning processes and DMSO benefits

Objective	Cleaning Process Requirements	DMSO Benefits
Performance	Ability to exhibit high solvency to a wide range of polymers and residues, rinse in water and other solvents, leaving the substrate clear and free of residue.	A wide variety of organic compounds including polymers are soluble in DMSO. DMSO is completely miscible with water and lower alcohols.
Selectivity	Ability to protect substrate, metal, and certain dissimilar polymers through-out the manufacturing process.	While acting as a strong solvent for polymers, DMSO is extremely mild on metals and oxides such as ceramics.
Stability	Ability for the sy[illegible] not change over an extended period of time. The longer this time period, the more robust the process and op-portunity for cost savings.	DMSO's [illegible] presence of acids and bases at ele-vated temperature produces cleaning formulations that are robust for manu-facturing processes.
Compatibility	Ability for the system to be easily inte-grated into existing tool platforms and have little or no effect on the materi-als of construction.	Teflon® fluoropolymer, polypropylene, polyethylene, and many metals and alloys are suitable for DMSO contact in the tool. Other materials should be evaluated for compatibility. Please ref-erence Appendix C for a list of com-patible materials.
Safety	Products deemed to be safe do not exhibit characteristics that present an acute or chronic hazard to workers or the equipment.	DMSO is safe to humans and biode-gradable in the environment. The ma-terial can be recycled using distillation technology. Additional Health, Safety and Environmental information in in-cluded in Appendix B.

DMSO Investigation

Dimethyl Sulfoxide exhibits properties which make it attractive in manufacturing. To better understand these benefits, Gaylord Chemical Company, L.L.C. has conducted an investigation of DMSO, and compared it to many other solvents for microelectronic applications. Common polymers known to be used in electronics were included in this investigation (See Table 2). The polymers were tested for their solubility in DMSO and other solvents.

Table 2.
Polymer Samples used for DMSO Benefits Investigation

	Product	Application	Polymer type
A	Wafer thinning-spin on adhesive	Wafer level packaging	TPU (EstaneÓ 5703,Noveon)
B	Neg PR (i-line)	WLP Bumping	Acrylic (Dianal Ó ACP-3, Dianal America)
C	Wafer thinning-spin on adhesive	Wafer level packaging	Rosin (Resinall Ó 833, Resinal Corp.)
D	Dielectrics	WLP Insulator	imide-based (TorlonÓ 4000T, Solvay)
E	Positive PR (DUV)	IC lithography	P-Host
F	Positive PR (i-line)	FPD, IC lithography	novolac

Over twenty (20) solvents were surveyed for solvency, blended together and mixed with acids and bases at different temperatures. These solvents were selected based on their common occurrence in photoresist stripping, casting, and rinse applications. They include amides, glycol ethers, lower alcohols, esters, ketones, and lactones. Although many of these solvents may exhibit good performance, very few provide the same benefits, in total, of DMSO. These benefits include: high flash point, high polarity / dielectric constant, water solubility, and low toxicity.

The acid and base classes tested in the study included alkyl ammonium hydroxides, alkanolamines, amines, and sulfonic acids. They were chosen based upon these being well known candidates in microelectronic formularies. These aggressive materials are needed to break up and hydrolyze long chain polymers complex with metals. Hard, baked polymers with impregnated metal are a common occurrence in many deposition or etch process.

Results

For ease of observation and in identifying key trends, dissolution rates of the noted polymers identified in Table 2 are assigned shades. The highest solubility is indicated by green, limited or no solubility is identified in orange, and partial solubility is light green.

1. General Polymer Solubility

DMSO exhibits excellent solubility for polymers used in microelectronic manufacturing as a compound to existing and common process of record (POR) solvents (see Table 3). Item #2 (DMSO), #1 (pyrrolidone), #5 (amide) and #19 (ketone) all exhibit excellent solubility at a tem-

Table 3
Dissolution of Polymers at 70ºC by Solvents Commonly Used in Microelectronics

	NMP	DMSO	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21
A	Soluble	Soluble	Soluble	Insoluble	Soluble	Insoluble	Insoluble	Soluble	Soluble	Insoluble	Insoluble	Soluble	Insoluble	Soluble	Insoluble	Insoluble	Soluble	Soluble	Soluble	Insoluble	Insoluble
B	Soluble	Insoluble	Insoluble	Insoluble	Soluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Soluble	Insoluble	Insoluble
C	Soluble	Soluble	Soluble	Soluble	Soluble	Insoluble	Insoluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Partially Soluble	Soluble	Soluble	Soluble	Soluble	Soluble
D	Soluble	Soluble	Insoluble	Insoluble	Soluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble
E	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Partially Soluble	Partially Soluble	Soluble	Soluble	Soluble	Soluble	Insoluble	Soluble	Soluble	Soluble	Soluble	Soluble
F	Soluble	Soluble	Soluble	Partially Soluble	Soluble	Soluble	Insoluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Insoluble	Insoluble	Soluble	Soluble	Soluble	Soluble	Partially Soluble

Soluble Partially Soluble Insoluble

2. DMSO Blends

The addition of DMSO as a cosolvent to an existing chemistry is expected to increase solvency for those polymers tested. The results in Table 4 (DMSO Blends) reflect an almost complete improvement from that observed in Table 3 (single solvent testing).

Table 4
Dissolution of Polymers by DMSO-Solvent Blends (1:1=50%DMSO) .

	NMP	DMSO	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21
A	Soluble	Soluble	Soluble	Partially Soluble	Soluble	Insoluble	Insoluble	Soluble	Soluble	Insoluble	Soluble	Soluble	Insoluble	Soluble	Partially Soluble	Soluble	Soluble	Soluble	Soluble	Insoluble	Partially Soluble
B	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble
C	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Insoluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble
D	Soluble	Soluble	Insoluble	Partially Soluble	Soluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Soluble	Insoluble	Insoluble	Soluble	Insoluble	Insoluble	Partially Soluble
E	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble
F	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble

Soluble Partially Soluble Insoluble

Note: Observe the increase in solvency, green shade=complete dissolution

3. Solvency with Additives

The solubility of these polymers tested is maintained in DMSO even after addition of alkalis. The observation of Table 5 and comparison with the baseline dissolution results for pure solvents in Table 3 suggest that many solvents result in a reduced solvency upon alkali addition.

Table 5
Dissolution of Polymers by Solvents with 20% Added Alkali Amine, at Room Temperature

	NMP	DMSO	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21
A	Partially Soluble	Partially Soluble	Insoluble	Insoluble	Partially Soluble	Insoluble	Insoluble	Soluble	Partially Soluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Partially Soluble	Insoluble	Insoluble	Insoluble	Partially Soluble	Insoluble	Partially Soluble
B	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble
C	Soluble	Soluble	Soluble	Soluble	Soluble	Insoluble	Soluble	Soluble	Insoluble	Insoluble	Insoluble	Soluble	Insoluble	Insoluble	Soluble	Insoluble	Insoluble	Soluble	Soluble	Soluble	Soluble
D	Soluble	Soluble	Insoluble	Insoluble	Soluble	Insoluble	Partially Soluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Soluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble
E	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Insoluble	Insoluble	Insoluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble	Soluble
F	Soluble	Soluble	Insoluble	Soluble	Soluble	Insoluble	Soluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Soluble	Insoluble	Insoluble	Insoluble	Soluble	Insoluble	Soluble

Soluble Partially Soluble Insoluble

Unlike most solvents, the results of Table 5 suggest that candidate solvents #1 (pyrrolidone), #2 (DMSO), and #5 (amide) will maintain their solvency for the noted polymers after addition of alkali. Many systems exhibit signi- #14=glycol ethers and reduct acetates).

4. Stability with Aggressive Additives

DMSO exhibits excellent stability as a process solvent in the presence of aggressive additives such as strong bases. Many common solvents react with bases such as alkylammonium hydroxides and amines.

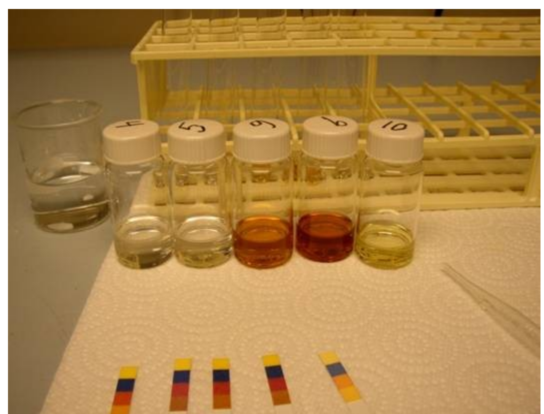


Figure 1.
Stability tests comparing basic DMSO formulas (Left vials=clear solutions) to other solvent blends.

Stability testing was performed by comparing pH changes in DMSO formulations with other solvent blends. Accelerated stability testing with DMSO / alkylammonium hydroxide formulations showed no change in pH after 24 hours at 80ºC. The two leftmost formulations in Figure 1 are DMSO-based products. It is worthwhile to mention that these formulations do not exhibit a color change during the test period.

Alkali formulations with amides and certain pyrrolidones appear to be especially sensitive to base exposure. Mixtures dropped at least 2 pH units suggesting a chemical reaction between the solvent system and the alkali.

5. DMSO in Microelectronic Applications

Test results suggest DMSO exhibits significant benefits in photoresist (PR) stripping from microelectronic devices. Information from the polymer dissolution work in this investigation indicates that DMSO is a good choice for common novolac and P-HOST PR stripping (see Tables 2-4). Stripping of a negative-tone PR[3] may require the addition of alkali to facilitate bond-breaking and hydrolysis. DMSO is a good choice as a high solvency medium with good stability when mixed with alkalis (compare Tables 3 & 5 and reference Figure 1). To this end, we have confirmed DMSO’s performance on example microe

Conclusions and Applications

DMSO has been investigated for its solvency and other performance-based properties that are key indicators of its ability to solve problems in the microelectronics industry. The following are general conclusions from these studies:

- DMSO is an excellent solvent for common polymers used in microelectronics.
- The addition of DMSO is expected to improve solvency of other solvents.
- Additions such as alkalis are not expected to harm the solvency character of DMSO.
- DMSO exhibits good stability when combined with aggressive materials such as strong bases.
- DMSO has been demonstrated to strip conventional novolac and negative tone acrylic PR from microelectronic devices.

TFT-LCD photoresist stripping applications. DMSO is a known formulation solvent for stripping novolac resist materials used in TFT-LCD flat panel displays. DMSO / amine and DMSO / alkylammonium hydroxide formulations have become industry standards.

High Dose Implanted (HDI) resists. The patterning conditions used to make sophisticated IC devices has a hardening influence on photoresist residues. Prior to stripping, a resist may be dosed with dopants, during an aggressive heated process. Strong solvents and aggressive stripping media are needed to safely remove carbonized resists from silicon and related substrates. The high polarity of DMSO makes it an attractive component of HDI resist residue cleaners.

Coating Solvent Applications. Wet photoresist formulations are polymer solutions, which are typically spun onto a substrate[5]. One or more baking steps are used to drive off the formulation solvent to produce a resist film appropriate for lithographic patterning. DMSO may be used as a cosolvent in resist formulations when formulation stability requires the use of polar additives.

Wafer Level Packaging applications. Lithographic processes using acrylic dry-film resist products[6] are being used increasingly in chip-scale packaging applications. DMSO is a useful cleaning solvent for stripping these materials after bump patterning. There are two dominant wafer-bumping processes in practice today, Flip-Chip and C4. Each process flow is shown in Figure 6. The different conditions experienced by resist materials in both processes has an impact on the formulation of cleaning / stripping products for these processes.

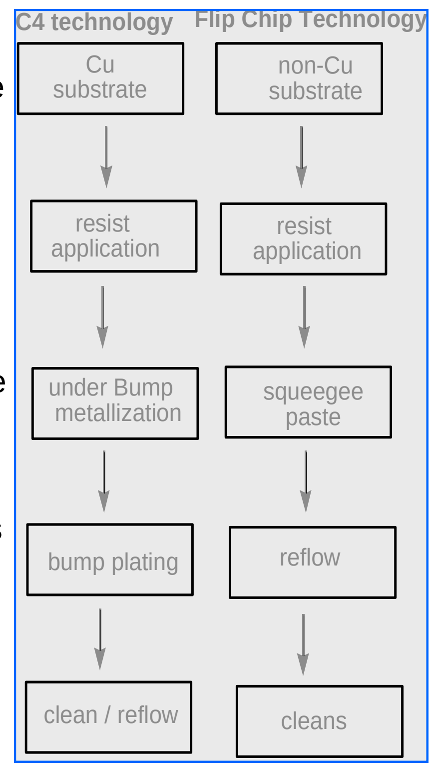


Figure 6. General process flow for bumping operations

Flip Chip processes often use acrylic dry film resist products, while the C4 process uses wet resists, with electroplated solder.

(novolac PR strip) and Figures 4 & 5 (negative tone acrylic PR strip).

Photoresist stripping - TFT-Array Patterning A single DMSO-based formula was demonstrated to work well in stripping novolac PR from specific areas on a TFT-LCD device (Figures 2 & 3).

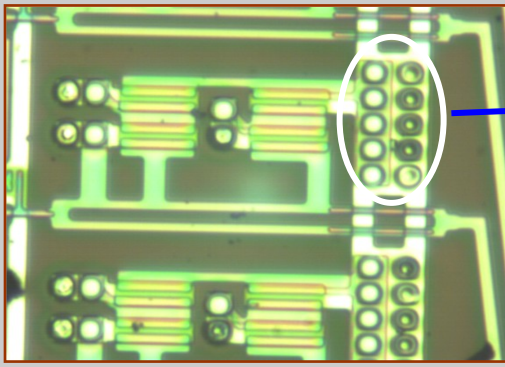


Figure 2. Area on device showing the presence of PR before cleaning.

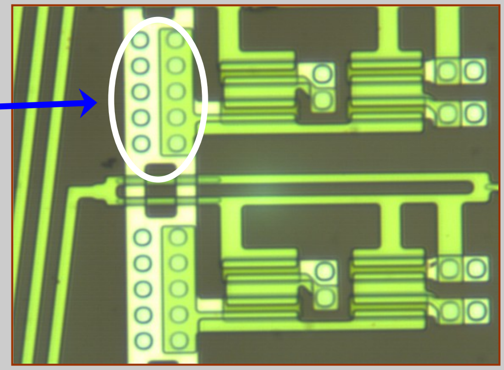


Figure 3. Area on device following PR stripping with a DMSO-based chemistry.

Photoresist Stripping - Solder Bump Patterning[4]. In another microelectronic application, a DMSO based formula was prepared with an alkali used to strip and dissolve negative-tone acrylic PR. This PR was used to pattern and define metallized areas that remained for solder bumping. The optical microscope photos in Figures 4 & 5 show the thick PR in a before condition and following exposure with the DMSO chemistry. (i.e. 80ºC, 30 min).

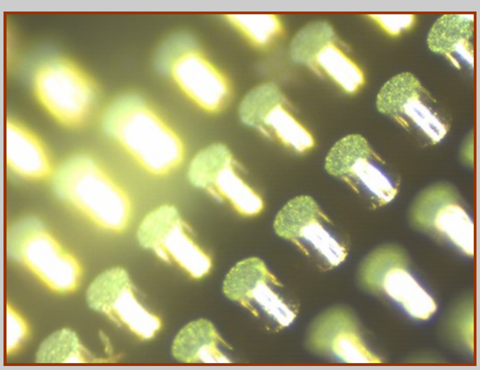


Figure 4. Area on the device showing the before condition of the top surface of the solder metal surrounded by cured thick PR. (*50x)*

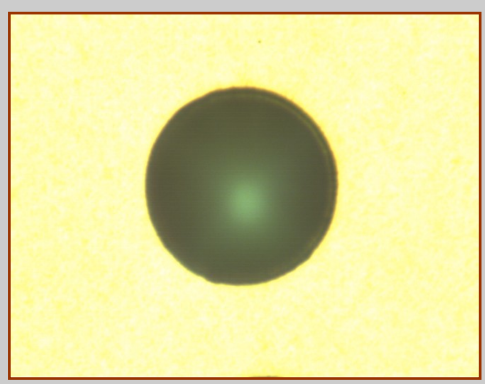


Figure 5. Area on the device following stripping of the thick PR with DMSO/alkali mixture. (*500x)*

Reflow is typically done in the resist pattern for Flip-Chip processing while the C4 process will remove the resist prior to reflow.

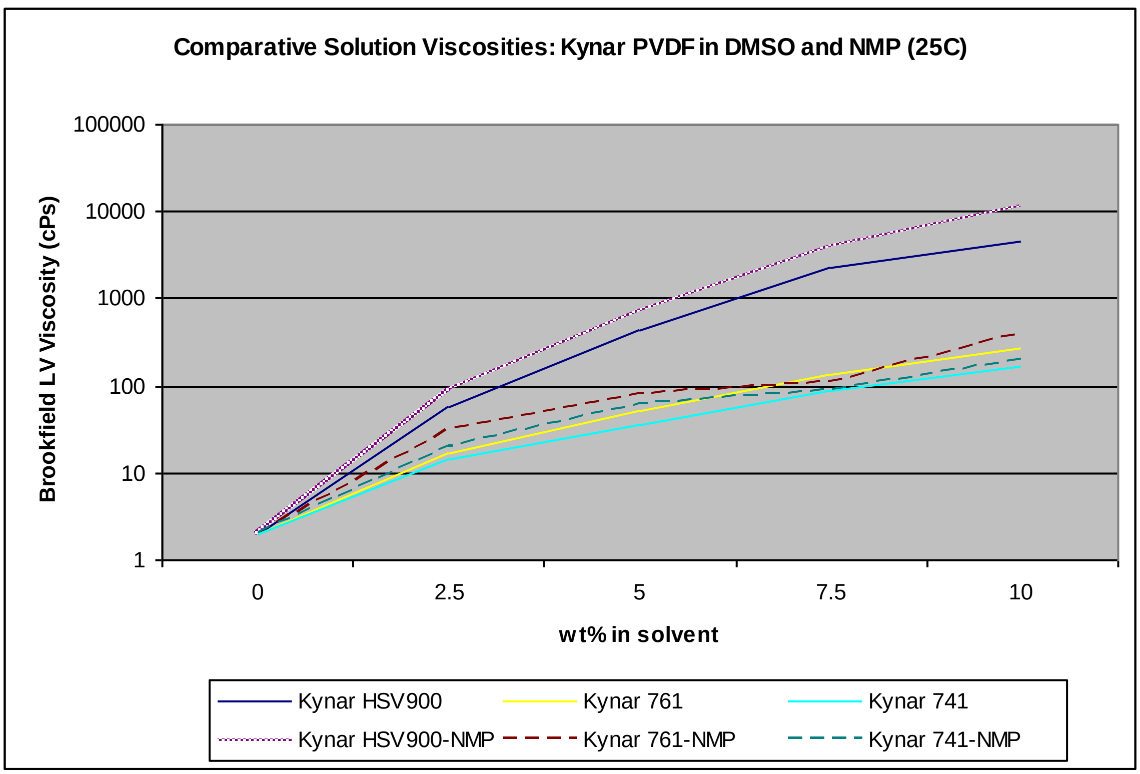
In the Flip-Chip process, the resist material is hardened requiring more aggressive DMSO-based formulations to remove photoresist and post-strip residue.

Rework Applications. DMSO has proven applications in solvent formulations to rework defective microelectronic components. These applications include removal of polyimide films used in TFT-LCD color filter applications and finished wafer passivation films. DMSO may also be useful in printed circuit rework applications.

Photoresist Stripping - Semiconductor Device Manufacturing. DMSO is a component of photoresist stripping products which are compatible with silicon substrates[7].

Wafer-Thinning Adhesives. The back lapping process[8] uses adhesive materials to hold GaAs wafers for grinding. DMSO is useful in removing adhesive materials when the process is complete.

Secondary Lithium Ion Battery Manufacturing. DMSO has been reported as a solvent for lithium salt electrolytes[9] and as a solvent for PVDF binder in batteries.[10] Below is data comparing the solubility of PVDF products used in the battery industry in DMSO and NMP. Solubilities of KynarÓ PVDF in both solvents are similar, with similar solution viscosities.



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10. Coustier, F. “ Method of Fabricating a Lamin
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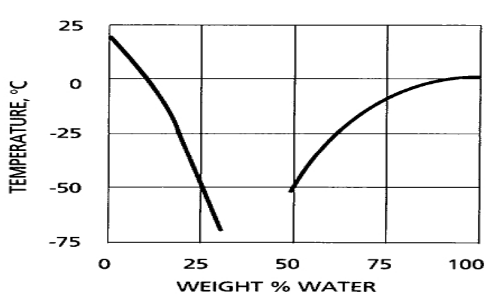
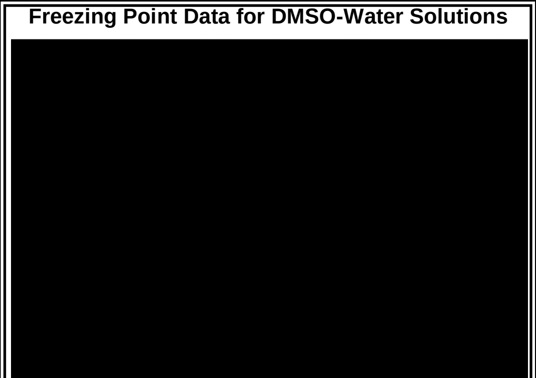
Appendix A
DMSO Properties

Parameter	Value
Miscible with water	
Density, at 25°C	1.0955 g / cm^3
Viscosity, cP, at 25°C	2.0
Surface tension at 20°C	43.53 dynes / cm
Refractive index ND@25°C	1.4768
Dielectric constant, 1 MHz, @ 20°C	48.9
Dielectric constant, 1 MHz, @ 40°C	45.5
Conductivity (Electrical), @ 20°C	3x10 (ohm^{-1} cm^{-1})
Conductivity (Electrical), @ 80°C	7x10 (ohm^{-1} cm^{-1})
pKa	35.1
pH (50% in water)	8.5
Boiling Point	189°C (372°F)
Freezing point	18.55°C (65.4°F)
Auto ignition temp, in air	300-302°C (572-575°F)
Flash point (open cup)	95°C (203°F)
Flash point (closed cup)	89°C (192°F)
Heat capacity (liq.), 25°C	0.47 cal / g / °C
Solubility parameters	
Hildebrand's	13.0 $(cal / cm^3)^{1/2}$
Hansen's	
- Dispersion	9.0 $(cal / cm^3)^{1/2}$
- Polar	8.0 $(cal / cm^3)^{1/2}$
- Hydrogen bonding	5.0 $(cal / cm^3)^{1/2}$

Freezing Point Data for DMSO-Water Solutions



Freezing Point Data for DMSO-Water Solutions



Weight % Water

Appendix B
DMSO
Health, Safety and Environment (HSE) Overview

Health (with comparison to other solvents)

Toxicological Indicator	**DMSO**	DMAc	NMP
Oral LD-50	14,500-28,300 mg / kg rat	4,300 mg / kg rat	3,914 mg/ kg rat
dermal LD-50	40,000 mg / kg rat	2,240 mg / kg rat	8,000 mg/ kg rabbit
Inhalation (rat)	none @ 2,900 mg / m^3	2,475 mg / kg @1 hr	NA
Reproductive toxin	no	yes	yes
Proposition 65	no	no	yes

Safety

Personal Protection-should include wearing butyl rubber or nitrile (NBR) rubber gloves and tight fitting safety goggles for eye protection. A recommended product is the TNT^{Ó} Disposable Nitrile Glove produced by Ansell Healthcare (VWR cat # 32889-888)

Use and Handling-Avoid contact with skin, eyes and clothing. Do not use near heat or open flame, distill with caution.

Storage conditions-Keep container closed, store away from heat and light

Temperature Parameters

Auto ignition temp, in air	300-302°C (572-575°F)
Flash point (open cup)	95°C (203°F)
Flash point (closed cup)	89°C (192°F)
Flammable Limits (% in air):	LEL: 3.0 - 3.5% by volume
Flammable Limits (% in air):	UEL: 42-63% by volume

Environmental

DMSO is biodegradable in biological systems, it is converted in part to methyl sulfone and to dimethyl sulfide.

Biological Oxygen Demand:

Theoretical Oxygen Demand at 10 ppm:	123 mg oxygen
Chemical Oxygen Demand at 10 ppm:	107 mg/1
Biological Oxygen Demand-5 at 10 ppm:	<1.0 mg/1

Note: For complete information please refer to the MSDS.

Appendix C

DMSO
Material Compatibility

	Solubility Grams/100 cc DMSO	Solubility Grams/100 cc DMSO
Material	**20-30°C**	**90-100°C**
Calcium methyl sulfonate	Soluble	
Diethanolamine	Miscible	
Ethyl alcohol	Miscible	
Methyl sulfonic acid	Miscible	
Phosphoric acid	Miscible	
Rosin	> 100	-
Triethanolamine laurylsulfate	Soluble	
Triethanolamine	Miscible	
Triethylamine	10	-
Polymers		
Teflon (DuPont)	Insoluble	Insoluble
Methacrylates Lucite 41, 45 (DuPont), Plexiglas (Rohm & Haas)	-	<1
Phenoplasts Modified Novalac R7522, R7550 (Ceca)	Soluble	-
Polycarbonates Lexan (General Electric)	-	>5
Silicones Dow Corning "Sylkyd 50", Z6018 (flake)	Miscible	
Neoprene	Insoluble	Insoluble
Polyetherether ketone (PEEK)	Insoluble	Insoluble
Polyethylene	Insoluble	Insoluble
Polypropylene	Insoluble	Insoluble

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