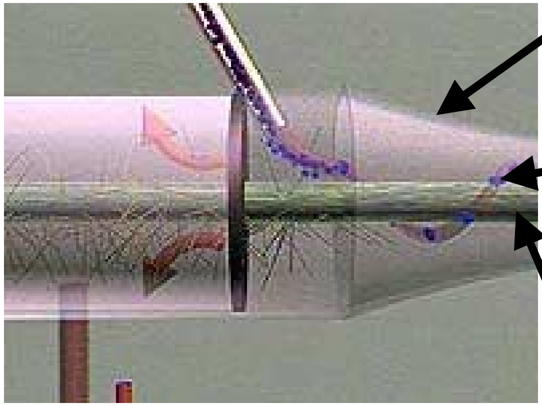
Polymer Additives

Textile Dyeing & Highly Reflective Material:

- SCF CO_2 infusion of dye to synthetic fibers
- SCF CO_2 infusion of Si containing additive to PEEK
- Rapid CO_2 depressurization leaves additives behind



Textile Sizing:

- Coating a smooth PVA film on fibers to add strength before weaving.
- Adjustment of conditions allows control of penetration.
- Fibers do not stick together after drawing through SCF.

http://scholar.lib.vt.edu/theses/available/etd-93097-173344/

Supercritical Fluid Drying

Silica aerogel :
permeability comparable to glass matrix, high porosity.

Useful as heat or acoustic insulation material

- Drying without vaporization
- No volume expansion from phase change
- No destruction of minute holes in silica gel

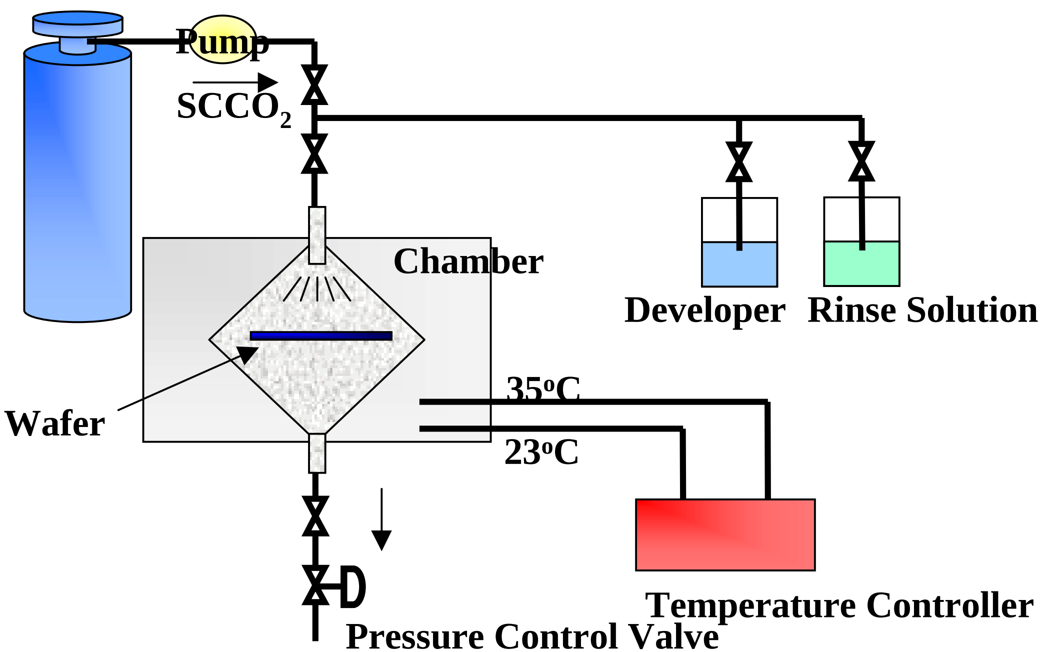
- Reactions in CO_2-swollen matrix to create composite materials
- Controlled depressurization renders dense composites
- Rapid pressure quenches yield expanded (microcellular foam) materials.

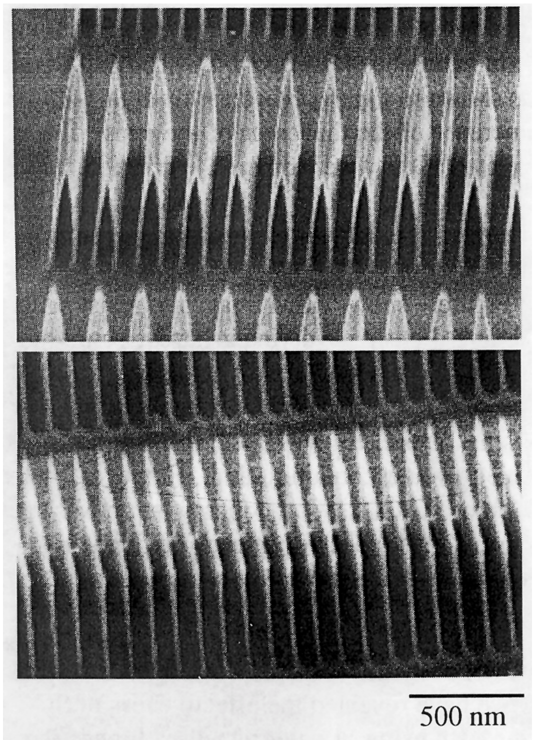
Thomas McCarthy http://www.pse.umass.edu/faculty/mccarthy.html

Supercritical Fluid Drying

- Use CO2 to replace water or polar solvents.
- Low surface tension avoids capillary forces/ pattern collapse

- N2 / CO2 combination used
- Very fine features created



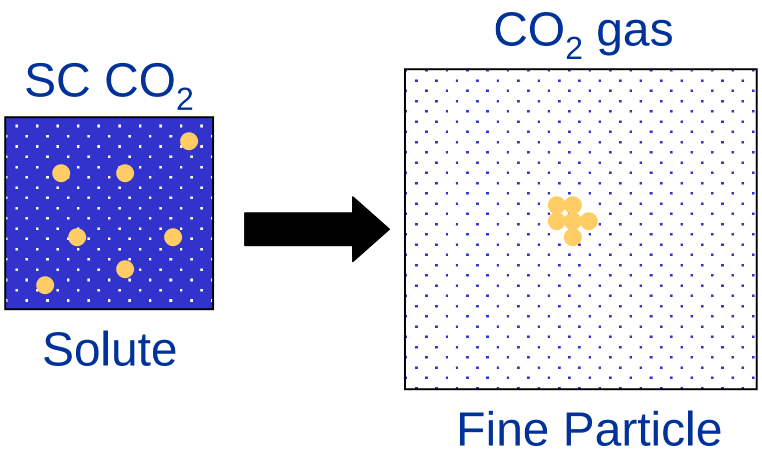


1999 NTT LSI Laboratories, Japan

Rapid Expansion of SCF Solutions : RESS

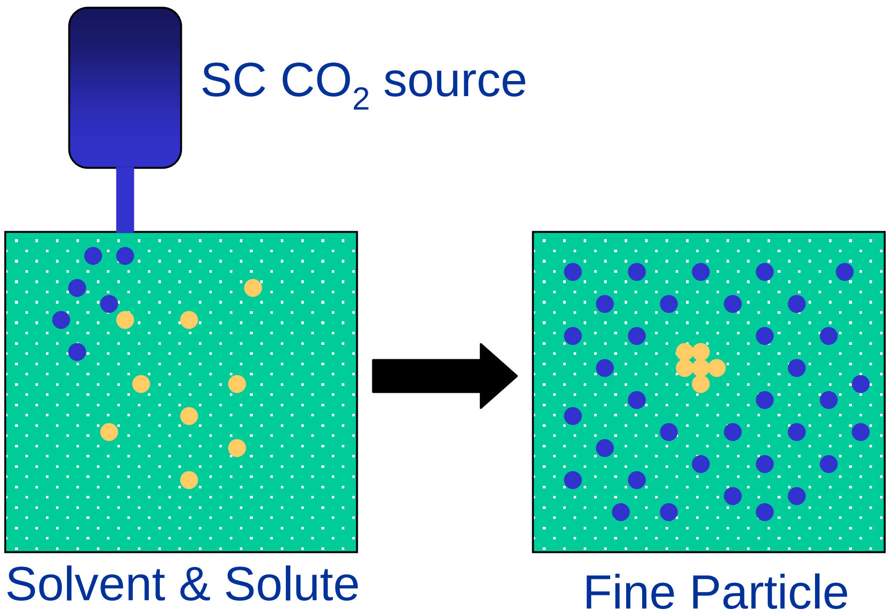
RESS: Rapid Expansion:

- Solute dissolved in SCF CO_2
- Expanded to ATM
- Solubility decreases by 10^6
- Supersaturation, Nucleation
- Highly uniform particles
- Controllable size



GAS: Gas anti-solvent Approach
SAS: Supercritical non-solvent

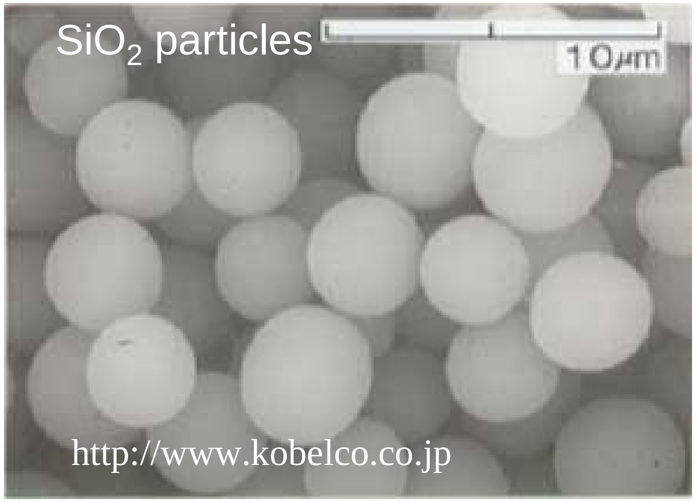
- Drug dissolves in carrier solvent, not CO_2
- Carrier solvent is partially miscible with CO_2
- Mixing carrier with SCF causes precipitation



RESS/ GAS Applications

Microencapuslation of Flavor and Fragrance: (volatile, themally labile, sensitive to oxidation, expensive)

- Release of flavors during microwaving
- Protection of aspartame during cooking
- Oxidation protection for oils and essences
- Protection during freeze/thaw cycles
- Mask potassium chloride taste
- Reduce flavor mixing: choline and vitamins
- Final product not contaminated with residual solvent
- Process does not degrade active agent as spray drying may



Pharmaceutical Application:

- Aerosol drugs require uniform particles and small size
- Must produce sterile powder
- CO_2 replaces aseptic conditions + use of EtO sterilizer

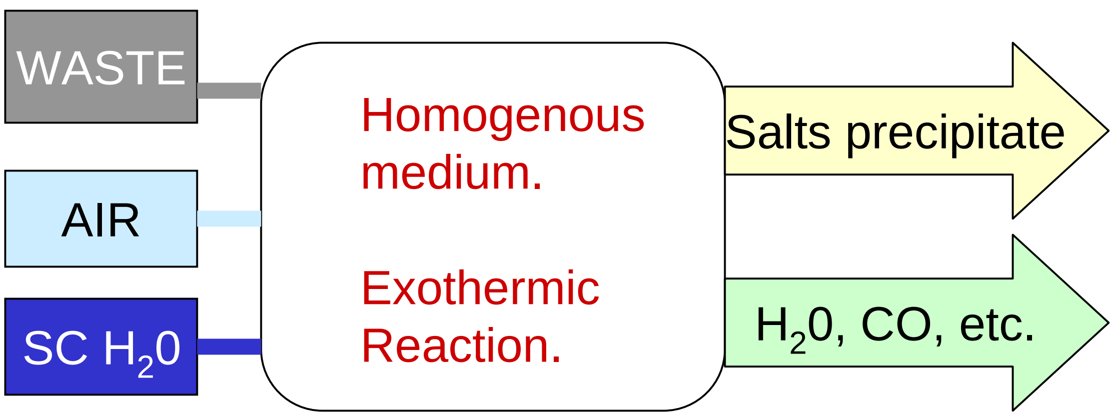
Supercritical Water Oxidation

<u>SCWO: The oxidative destruction of organic wastes in supercritical water.</u>

Above 550 °C toxic organic compounds are miscible, as are oxygen and peroxides in water. Inorganic salts are mostly insoluble.

Destruction of chemical warfare agents, toxic pharmaceuticals

Challenges include: separation of inorganic salts to prevent scaling, & corrosion arising from halogenated hydrocarbons.

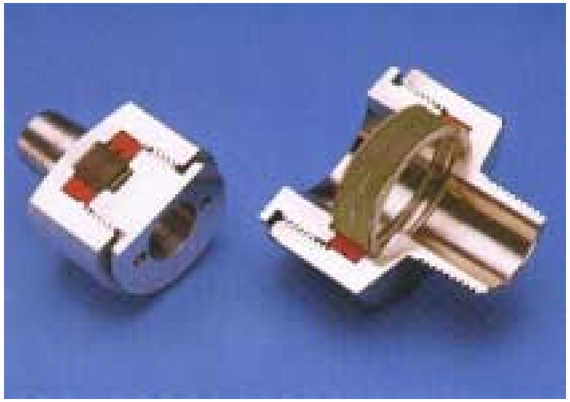


EcoWaste Technologies

Molecular-Level SCF Study

Requirements for Modeling Solubility:

•Properties of all solvents and solutes
•Equation of state: ideal gas, virial, VDW, cubic, Peng-Robinson, etc.
•Temperature dependence, attraction terms, component interaction parameters
•Short and long range (solvation and compresibility driven) effects for solutions



Determine solubility & dissolution behavior in CVD films; interferometery.

Modeling via regression:

•Based on quantitative structure-property relationships.
•Numerical descriptors may include electrostatic, geometric, and topological parameters, as well as F or other atomic content.

Challenges in SCF Technology

•Feasible designs for scale-up are necessary for commercialization

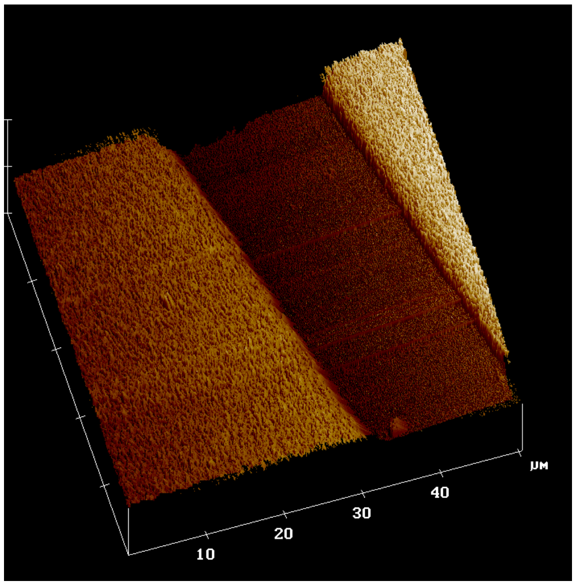
•Modeling for scale-up not always available/ accurate

•Absence of fundamental, molecular-based model of solutes in SCF

•Continuing instrumental & equipment improvements

•Continuing research & development for applications & new material design

Thrust D: Dry Lithography



- Continuing progress in CO_2 developing of CVD deposited fluoropoly(ethers)
- Environmentally benign alternative to aqueous base development
- Useful for patternable low k material
- Aid in resist design for 157 nm laser source or e-beam
- Study to identify structure/property relationships in SCF CO2
- Study of dissolution behavior in films

SRC / NSF

Karen Gleason,
Hilton Pryce Lewis
MIT

Conclusions

Supercritical Fluids:

•Have unique properties, including high diffusivity, low surface tension, low dielectric constant, and continuously variable density.
•Are non-flammable, non-toxic, inexpensive and do not require special disposal.

Provide alternatives for solvents in cleaning, extraction, and synthesis applications.
•Provide capability for manufacturing specially structured materials, which cannot be produced conventionally.
•Provide new capability for extraction, chromatography, and toxic waste elimination.