

# SOL-GEL PROCESS

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**Sol-Gel process** is a synthesis of an oxide ceramic in a liquid solution of an alkoxide based precursor in which submicron particles of a solid phase suspended in the liquid (**Sol**) are obtained and then condensed forming **Gel** - a 3-D network of polymerized macromolecules surrounded with the solution.

Applications of Sol-Gel process:

- ☐ Thermal insulation.
- ☐ Acoustic insulation.
- ☐ Protective optical coatings.
- ☐ Lightweight materials.
- ☐ Tough ceramics.
- ☐ Membranes and microfilters.
- ☐ Nuclear waste storage.
- ☐ Ultra fine powder abrasives.
- ☐ Encapsulation of biomolecules for controlled drug release.

The stages of the Sol-Gel process

- ☐ **Hydrolysis of precursor (sol formation)**
- ☐ **Polycondensation (gelation)**
- ☐ **Aging**
- ☐ **Drying**
- ☐ **Calcination**
- ☐ **Properties of some aerogels**

## Hydrolysis of precursor (sol formation)

The precursor is an aqueous solution of the metal **M** alkoxide: **M-OR**, where **R** is the alkyl group (e.g., C<sub>2</sub>H<sub>5</sub>).

The metal alkoxide reacts with the surrounding water and forms the colloidal suspension (sol) of the metal hydroxide **M-OH** according to the hydrolysis reaction:



to top

## Polycondensation (gelation)

Polycondensation of the hydrolyzed precursor occurs according to the reactions:

Reaction with the non-hydrolyzed metal alkoxide **M-OR** resulting in alcohol formation **R-OH**:  
**M-OH + M-OR = M-O-M + R-OH**

Reaction with the hydrolyzed metal alkoxide **M-OH** resulting in water formation **H<sub>2</sub>O**:  
**M-OH + M-OH = M-O-M + H<sub>2</sub>O**

Polycondensation stage results in a formation of the **Gel** - a rigid 3-D network built of polymeric molecules and surrounded with the solvent.

The gel structure obtained from the acidic solutions with PH<6 (acid catalyzed) is uniform and weakly cross-linked.

The gel structure obtained from the basic or neutral solutions with PH>6 (base catalyzed) consists of separate clusters .

## Aging

During the aging stage the polycondensation reactions continue completing the formation of the gel.

The gel structure is reinforced with additional cross-links, which cause contraction of the gel matrix and expulsion of the solution from the shrinking pores.

## Drying

The water and other liquids entrapped within the pores of the gel structure are removed during this stage.

Drying is performed at a temperature of about 400°F (~200°C).

After drying the gel converts into a monolith micro-porous structure called **Xerogel**.

Drying at super-critical conditions preventing collapsing of the gel network results in a formation of a macro-porous low density structure called **Aerogel**.

## Calcination

Calcination is performed at increased temperatures varying within the range 750-1470°F (400-800°C).

During the calcination stage the dry gel structure is dehydrated according to the reaction:



Example:



Volatile organics are also removed in this stage.

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