# SEM-COM SEALING GLASSES

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# SEM-COM SEALANTS

SEM-COM has glasses available which were developed to seal to a variety of materials, ranging from quartz (thermal expansion of 5 x $10^{-7}$/°C) to copper (thermal expansion of 170 x $10^{-7}$/°C). Glass to glass, glass to ceramic, glass to metal, ceramic to metal and ceramic to ceramic seals can all be made by using the proper sealant. We produce sealant glasses that seal at both high and low temperatures to meet varying application needs. Refer to Table 1 for a list of those glasses developed for sealant application.

SEM-COM's sealants fall into two general categories:

1.) Vitreous Type
2.) Crystallizing Type

The vitreous type sealants are non-crystallizing glasses which can be reworked repeatedly and still maintain the same characteristics and properties.

The crystallizing type sealants crystallize during the curing cycle and form a two phase glass-ceramic material, which is usually translucent or opaque. Upon reheating, (even to temperatures above the initial curing temperature), these glasses remain stable.

## USES AND METHODS OF APPLICATIONS

### Vitreous Type Sealant Glasses

Vitreous glasses are considerably easier to apply than the crystallizing type and require a shorter firing time to achieve complete sealing. They may be used in small seal areas where there is not much stress in the seal at higher temperature. The lower temperature vitreous sealants can be used in applications where it may be necessary to readily separate and reseal the component parts. Normally, the vitreous type sealants do not offer the strength of the devtrifying types. They also have somewhat higher electrical resistivity and a lower dielectric constant than the crystallizing type.

There are two general methods of application for vitreous type sealant glasses:

1.) **Hot Method of Application**

This "hot dip" method is generally more suitable for the lower temperature glasses. The glass is heated in a platinum crucible by either gas flame or electrical radiant heating elements to a temperature above the working point, where it is very fluid. The component parts are preheated (for glass parts preheat to about 20°C above the annealing point). Each part is dipped into the molten sealant glass for 10-20 seconds, withdrawn and allowed to "set" in air for about 5 seconds. The parts are then immediately placed in an oven set at the annealing point of the sealant material. They are held at this annealing temperature for 15-20 minutes, then cooled at a rate not to exceed 5°C per minute.

The cooled parts can be sealed together by aligning their sealing edges and placing them in an oven, under load. They are sealed according to the recommended cycle, heating at 10°C per minute and cooling to room temperature at a rate not greater than 5°C per minute. (For large glass parts with thick sections, reducing the cooling rate to as low as 1°C per minute is recommended.)

After dipping, the parts can also be sealed together immediately while the sealant glass on the sealing edges is still soft, by properly aligning and pressing the parts together. The sealed assembly then is placed in a hot annealing oven, annealed and cooled to room temperature.

Satisfactory seals can be made by having just one of the component parts pretreated with sealant, but it has been found that better seals can be obtained by coating both edges prior to sealing. Generally, seals made by the "hot dip" method show a higher seal strength than those of the "cold paste" method.

2.) **Cold Method of Application**

In the "cold paste" method, the finely ground glass powder is mixed with a vehicle to form a paste. The vehicle must readily decompose below the softening point of the glass so that it does not cause contaminated or seedy seals. It is suggested that the mixture be thoroughly blended just prior to application. The desired viscosity of the paste will be controlled by the type and amount of vehicle used. A thick paste would be suitable for spatula or extrusion application. The thinner pastes are applied by spray, roller coat or screening. The parts can also be dipped into a very thin paste. Satisfactory seals are usually obtained when the paste is applied to only one surface. However, both surfaces can be coated to insure good seals. Depending upon the vehicle used, the coated parts may have to be dried to evaporate the volatile organics before firing.

For sealing, the parts are then placed in an oven, exercising care that the sealant glass coating is not damaged. A maximum heating rate of 10°C per minute is recommended. Parts can be held at the recommended sealing temperature for just a few minutes, or up to one hour. The maximum cooling rate should be 5° per minute. In some cases it may be necessary to apply additional loading to the parts being sealed to produce proper sealant flow and proper seal line thickness. Maximum seal strength is obtained at thickness of 3 to 10 mils.

**Crystallizing Type Sealant Glasses**

Crystallizing type glasses are very stable crystalline materials. They may be compared to thermo-plastics, in that the glass, after maturing, has properties, which enable it to be reheated to a reasonably high temperature without showing any glass flow. The initial firing or curing cycle for these glasses is very important to achieve proper crystal growth and the strongest seal.

**Method of Application**

For the crystallizing type sealants, the accepted method of application is to use a finely ground glass in paste form and to make either a “cold dip” seal or to apply the paste in the same manner of application as for vitreous glasses.

When the ground form of the sealant glass is heated, it first melts to a vitreous state. In this state, the glass flows relatively well and can wet the material being sealed, thus contributing to a strong bond. On heating still further, the glass begins to devitrify and the temperature to which it is heated determines the type and size of crystals, which will be formed. This size and type of crystal in turn controls the thermal expansion of the fully matured glass, so that some variation of thermal expansion properties will occur with variation in heating conditions. Sufficient time must be allowed to completely mature the sealant glass at the temperature desired. Otherwise on reheating, stress changes will take place, which may break the seal. Seals can be made at temperatures higher than the recommended temperature, but the strength decreases considerably.

It is also necessary to observe some degree of care in heating and cooling the sealed article, because the glass seals tend to go through a region of relatively high temporary stresses at temperatures between their maturing temperature and room temperature. (See temporary stresses in seals.)

# FORMS AVAILABLE

SEM-COM can supply glasses in different forms to meet varying needs. The forms generally used are listed below:

## Ground Material

The most common form available is as a powdered or ground material. The glass is ground to a specified particle size distribution. It can be ground dry, or as a slurry in water or alcohol. Standard grinds are available or we can supply a grind to meet a customer's particular application need. The glass is classified according to the percent of material that is passed through or retained on US Sieve sizes of standard mesh screens. For example, a glass that is identified as minus 100 mesh, plus 400 mesh (-100 m, +400 m) means that all the glass will pass though a 100 mesh screen and be retained on the 400 mesh screen.

The table below lists the U.S. Sieve Series that SEM-COM uses and the mesh opening sizes for them.

**Opening Size**

| Sieve Size No. | Inches | Microns | Millimeters |
|---|---|---|---|
| 40 | .0165 | 420 | 0.420 |
| 80 | .0070 | 177 | 0.177 |
| 100 | .0059 | 149 | 0.149 |
| 120 | .0049 | 125 | 0.125 |
| 140 | .0041 | 105 | 0.105 |
| 170 | .0035 | 88 | 0.088 |
| 200 | .0029 | 74 | 0.074 |
| 230 | .0024 | 62 | 0.062 |
| 270 | .0021 | 53 | 0.053 |
| 325 | .0017 | 44 | 0.044 |
| 400 | .0015 | 37 | 0.037 |

Our standard grinds are all 100% -100m with the following percent of material passing through the 400 mesh screen:

"A" Grind 67 ± 5% -400 m
"B" Grind 87 ± 5% -400 m
"C" Grind 99.5% -400 m

## MICRO-RODS

Another form available is the micro-rod or fiber. These fibers are redrawn from a larger rod to diameters ranging from .005' to .250'. The shapes are usually round, square or rectangular. Upon request, SEM-COM can furnish information on special shapes or sizes.

## MISCELLANEOUS FORMS

SEM-COM will also furnish some of our glasses as frit or flakes or as a solid slab or rod.

# TEMPORARY STRESSES IN SEALS

## Vitreous Glasses

The stresses that develop in a glass seal during the heating and cooling associated with annealing or further processing are an important factor to consider when selecting a glass to seal to metals or other materials.

Stresses develop in a seal due to the difference in the contraction (or expansion) curves of the materials used in the seal. A vitreous glass will normally 'set up' at about 10-15°C below the annealing point and the final (room temperature) stress can be estimated by comparing the curves at the set up temperature of the lower glass.

> (To calculate the amount of stress in a seal, read the elongation values on both curves and multiply the difference of these by 50. This gives an approximate seal stress in PSI. The material with the higher value will be in tension.)

The seal may also undergo temporary stresses that build up during heating and cooling due to the different shapes of curves. These temporary stresses may be much higher than the final stress and may be in a different direction (tension or compression). A seal that goes through changes in direction (tension to compression to tension) may be more apt to fail much as a wire will break if bent back and forth. In addition to the magnitude and direction of the stress, the temperature at which the highest stress occurs is also important in order to avoid holding the sealed part at this temperature for prolonged periods of time.

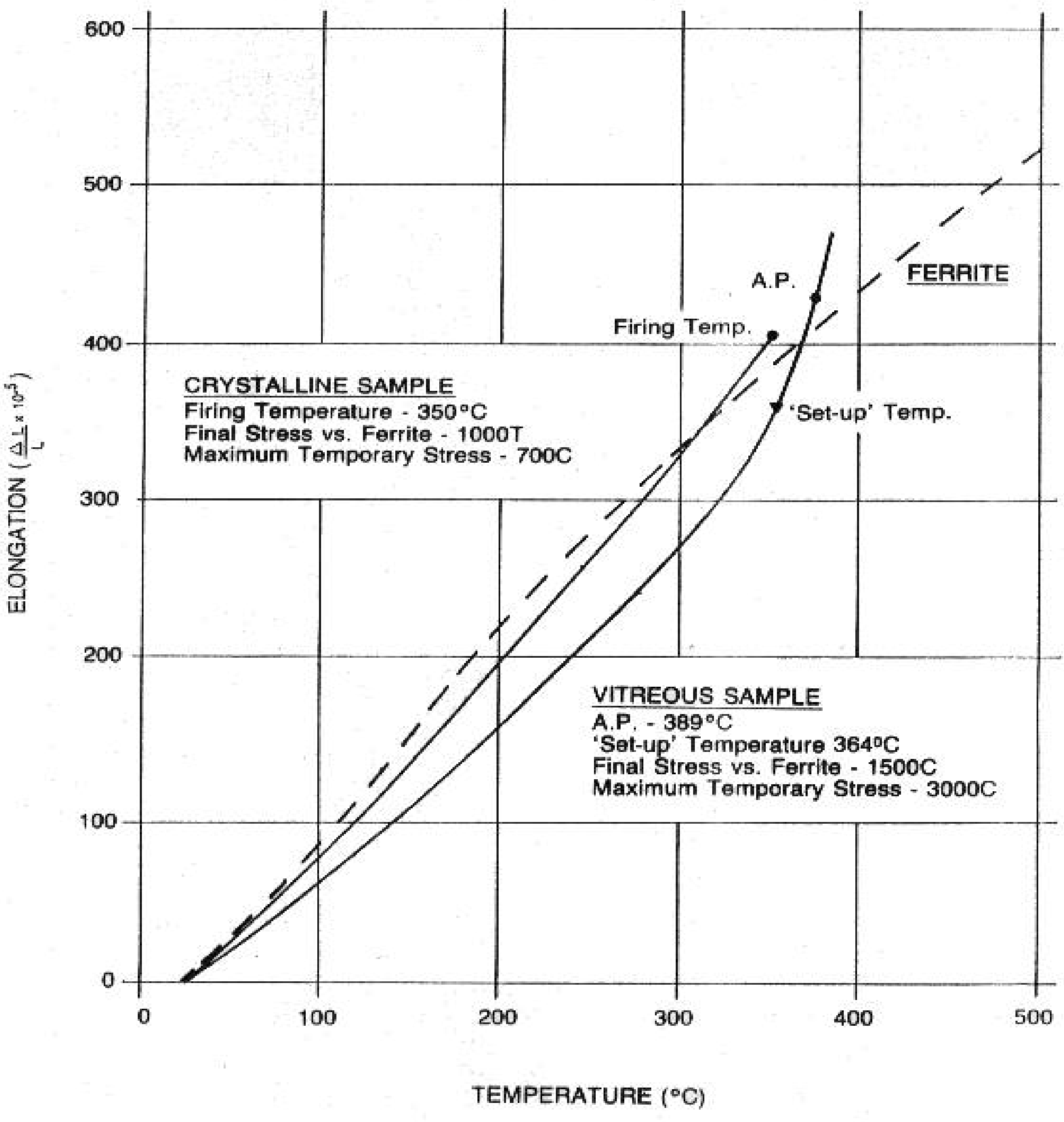
> NOTE: A temporary difference will exist between the metal and glass parts of a seal during heating and cooling. The metal 'leads' the glass due to its higher thermal conductivity, which means that the metal is warmer during heating and cooler during cooling. This temperature difference will appear as additional tension in the temporary stress during heating and additional compression during cooling. Reducing the heating and cooling rates will reduce these stresses.

## Crystalline Materials

The Crystallizing-type materials 'set up' at the firing temperature and the final stress can be approximated by comparing the curves at this temperature.

(Refer to Thermal Contraction Curves – page 7.)

# THERMAL CONTRACTION CURVES



# Table of Sealant Glasses

| SEALANT ID | EXP. / CONT.[1] (x $10^{-7}$/ °C) | | AP[2] / SP[2] | | RECOMMENDED SEALING CYCLE Temp (°C) | Time (min.) | MAXIMUM REHEAT[3] TEMP (°C) | SEALABLE TO: NON METALS | METALS[4] |
|---|---|---|---|---|---|---|---|---|---|
| SCY-1 | 3.5 | 10 | 570 | 880 | 1200 | 20 | 725 | Quartz | |
| SCY-2 | 5 | 10 | 570 | 875 | 1200 | 20 | 725 | Quartz | |
| SCE-3 | 33 | 42 | 819 | 1070 | 1350 | 30 | 1030 | Borosilicates | Silicon |
| SCQ-2 | 38 | 52 | 685 | 870 | 1050 | 30 | 700 | Aluminum Nitride | Silicon |
| **SCL-5** | **40** | | **480** | **567** | **625** | **30** | **520** | **Aluminum Nitride** | **Silicon** |
| SCY-3 | 43 | 56 | 707 | 912 | 1120 | 30 | 860 | Aluminum Nitride | Molybdenum |
| SCE-1 | 43 | 56 | 765 | 960 | 1200 | 30 | 900 | Aluminum Nitride | Molybdenum |
| **SCl-4** | **45** | | **535** | **619** | **680** | **30** | **570** | **Silicon Carbide** | **F15** |
| **SCC-1002**** | **48** | | **540** | **640** | **750** | **30** | **600** | **Silicon Carbide** | **F15** |
| SCR-2 | 49 | 62 | 510 | 710 | 1000 | 30 | 650 | Silicon Carbide | F15, Molybdenum |
| **SCL-1** | **50** | | **532** | **609** | **680** | **30** | **560** | **Silicon Carbide** | **F15, Molybdenum, Tantalum** |
| **SCL-2** | **51** | | **480** | **567** | **625** | **30** | **520** | **Silicon Carbide** | **F15, Molybdenum, Tantalum** |
| SCQ-1 | 52 | 76 | 570 | 723 | 955 | 30 | 670 | Alumina | F15, Molybdenum, Tantalum |
| SCQ-3 | 55 | 71 | 520 | 710 | 975 | 30 | 660 | Alumina | F15, F30 |
| **SCL-3** | **55** | | **480** | **567** | **625** | **30** | **520** | **Alumina** | **F15, F30** |
| SCQ-4 | 59 | 78 | 575 | 750 | 1000 | 30 | 720 | Alumina | F15 |
| **SCA-2000** | **75** | | **610** | **750** | **30** | **30** | **725** | **Alumina, soda lime** | **F15, F30 (46%), F30 (42%)** |
| **SCV-2*** | **76** | | **345** | **445** | **480** | **30** | **400** | **Alumina, soda lime** | **F15, F30 (46%), F30 (42%)** |
| **SCV-4*** | **77** | | **495** | **605** | **675** | **30** | **560** | **Alumina, soda lime** | **F30 (52%), F31** |
| **SCV-3*** | **82** | | **339** | **452** | **480** | **30** | **400** | **Soda lime** | **F30 (52%), F31, 430SS** |
| SCM-1 | 90 | 107 | 456 | 655 | 1020 | 30 | 610 | Ferrite | F256 |
| **SCZ-8**** | **95** | | **725** | **837** | **925** | **30** | **1000** | **SOFC ceramics** | **F30 (52%), F31** |
| **SCA-2002** | **95** | | **476** | **616** | **750** | **30** | **570** | **Ferrite** | **F30 (52%), F31** |
| SCS-11 | 97 | 139 | 465 | 555 | 650 | 30 | 510 | Ferrite | F30 (52%), F31 |
| SCN-1 | 99 | 119 | 495 | 685 | 1000 | 30 | 640 | Ferrite | F256, F257 |
| **SCA-2001** | **99** | | **507** | **697** | **850** | **30** | **650** | **Ferrite** | **F256, F257** |
| SCS-1 | 117 | 160 | 396 | 475 | 640 | 30 | 440 | | Iron, Inconel |
| SCU-2 | 119 | 147 | 470 | 601 | 790 | 30 | 550 | | Iron, Inconel |
| **SCV-5*** | **131** | | **348** | **433** | **470** | **30** | **390** | | **Iron, 302SS, 304SS** |
| SCU-1 | 149 | 176 | 440 | 550 | 690 | 30 | 520 | | Copper |

**Developmental Glasses**
*** Can be used as a vitreous or crystallizing glass**
**** Crystallizing**

**NOTES:**

1. For crystallizing glasses, the contraction coefficient is measured from the recommended sealing temperature to 25° C.
2. The annealing point and the softening point apply only to the vitreous glasses.
3. These temperatures can be tolerated for up to twenty-thirty (20-30) minutes. Higher temperatures may be used, depending upon the size and shape of the sealed article.
4. ASTM Metal Designations:
   - F-15: 29$^{0}$/o nickel, 170/0 cobalt, 53% iron (e.g. KOVAR® , RODAR® , THERLO$^{0}$
   - F-29: Copper clad - 42% nickel, iron (e.g. Dumet)
   - F-30: Nickel-iron alloys: 42% nickel, 44% nickel, 46% nickel, 49% nickel, 52% nickel
   - F-31: 42% nickel, 5.5 - 6% chromium, balance iron (e.g. Sylvania #4, Carpenter #426)
   - F-256: 18%chromium, 82%iron (e.g. #430 alloy)
   - F-257: 28% chromium, 72% iron (e.g. #446 alloy)