SEALING FEEDTHROUGHS INTO MACOR® MACHINABLE GLASS CERAMIC

Introduction

The excellent electrical properties of MACOR® MGC have made it a natural material for use as a precision electrical insulator. In many cases it is necessary to put a conductive material through the MACOR MGC. Although many sealing materials can be used, this bulletin will deal with frit sealing of the feedthroughs. This technique offers a seal with properties such as strength, hermeticity and high use temperature.

Sealing Glasses

Many of Ferro's solder glasses have a firing temperature and a coefficient of thermal expansion that allow them to be used with MACOR MGC. These glasses come in a powder form and are mixed with a vehicle (e.g. amyl acetate, propanol) prior to application. The following is a list of solder glasses recommended for use with MACOR Machinable Glass Ceramic.

Ferro Code[1]	Firing Cycle[2]	Continuous Use Temperature	Coefficient of Thermal Expansion
7555*	450°C-20 min.	300°C	88.5 x 10^{-7}/°C
7570*	470°C-30 min.	330°C	84.0 x 10^{-7}/°C
7572	450°C-60 min.	425°C	95.0 x 10^{-7}/°C
7575	450°C-60 min.	425°C	89.0 x 10^{-7}/°C

[1] - Ferro Corporation, PO Box 6550, 4150 E. 56th Street, Cleveland OH, 44101. Tel. (216) 641-8585, ext. 7426, Fax (412) 641-8857.

[2] - These are idealized conditions. Heating and cooling rates and soak times are dependent on the size and geometry of the parts. The time is the hold at the corresponding temperature.

* - Vitreous solder glass.

Metals

The following can be sealed to MACOR MGC:
Sylvania #4
Dumet
52% Nickel Alloys
Chrome-Iron Stainless
Platinum
Sealmet
Titanium

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www.morganadvancedceramics.com/materials/macor.htm

Procedure

1) Clean the parts to remove any grease or foreign material around the areas to be sealed. Handle with care (gloves) to keep the parts clean.

2) Mix the glass with the vehicle thoroughly and keep agitated to maintain a consistent slurry. Success has been achieved working with a ratio of frit to vehicle of between 5/1 to 14/1 and is dependent on the configuration of the seal being made. A fresh slurry should be made prior to each sealing operation as the shelf life of these slurries is relatively short.

3) The holes in the MACOR MGC should be drilled with a 0.003" – 0.005" clearance around the feedthrough to allow the frit to flow into the seal area.

4) Place feedthrough in position and then apply the frit sparingly, making sure that the entire O.D. of the part in the seal area is covered with frit. Bare spots will cause an inconsistent seal and a loss of strength and hermeticity could result. In some cases the frit can be pre-glazed to form a bead on the metal part prior to insertion to facilitate an even coating.

5) Fixture the parts so that the proper alignment is maintained throughout the firing cycle.

6) Maintain an oxidizing atmosphere in the furnace during firing.

7) Heating and cooling rates should be slow enough to minimize thermal gradients in the total assembly
(i.e. 3°C – 5°C/min.).

8) A thermocouple should be placed as close as possible to the seal for accurate temperature readings.

Precautions

These glasses have a high lead content. The following precautions should be taken to avoid the possibilities of lead poisoning:

- Wear a respirator to avoid breathing the dust
- Wash hands thoroughly before eating, drinking or smoking.
- Wear a lab coat to reduce the chance of the material being carried home in work clothes.
- Read the manufacturers MSDS's for the materials used.

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MAKING HERMETIC SEALS WITH MACOR® MACHINABLE GLASS CERAMIC

Introduction

A major requirement for a vacuum-compatible material is that it be easily and hermetically sealed to metal, ceramic or glass. MACOR Machinable Glass Ceramic (Corning Code 9658) has a high glass content and a high coefficient of thermal expansion which allows an expansion match to many metals and glasses. Thus it is capable of forming hermetic seals directly to these materials with solder glass frit. This information sheet describes a method for making vacuum-tight seals using solder glass-sealing technology.

Sealing Methodology

Solder glass frit is a finely ground powdered glass which when held in suspension by a low-viscosity vehicle forms a slurry compatible with spraying, silk screening or extrusion application techniques. The chemical composition of a frit glass is unique because the glass crystallizes at low temperatures and forms a high strength seal able to withstand temperatures within 25° C of the sealing temperature.

Ferro[1] Code 7575 is a crystallizing solder glass with a coefficient of thermal expansion that matches MACOR Machinable Glass Ceramic. It is sealed at a temperature of 450°C for 60 minutes and has a use temperature of up to 425°C. The solder glass and machinable glass ceramic can be sealed to the following materials:

Metals:	Sylvania #4
	Dumet
	52% Nickel Alloys
	Chrome-Iron Stainless
	Platinum
	Sealmet
	Titanium
Ceramic:	Fosterite

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FRIT SEALING MACOR® MACHINABLE GLASS CERAMIC

Requirements for larger and increasingly complex structures of MACOR® Machinable Glass Ceramic have led to the development of glass frits for sealing this unique material to itself. These glass frits can be applied by spraying, screen printing or extrusion techniques.

Ferro has frits with firing temperatures from 425°C to as high as 470°C that are compatible with MACOR MGC. These glasses withstand continuous operating temperatures of up to 425°C, while maintaining strong hermetic seals. The strength of these joints has been measured (M.O.R.) as high as 11,000 psi, with an average of approximately 6,000 psi. Besides offering excellent strength and reboil characteristics, these glasses maintain the outstanding electrical properties of MACOR MGC with volume resistivities averaging 10^{12}ohm-cm and
Dielectric strengths of approximately 1200 volts.mil. Many of these frits produce joints that are machinable, allowing further processing.

Sealing Techniques

A. Surface Preparation

The surfaces to be coated should be smooth and clean. A 200 grit finish on the MACOR MGC is required for optimum results. Clean the surface with acetone to remove any grease, rinse in distilled water and dry thoroughly. Take care to keep the parts clean prior to sealing.

B. Application Methods

The frits can be applied by various methods: spraying, painting, screen printing, dipping and extrusion. The frits are mixed with organic vehicles to form thixotropic suspensions for application. These organic vehicles are commercially available from Ferro Corporation[1] and Cerdec Corp./Drakenfeld[2]. Volatile solvents such as amyl acetate or denatured alcohol can also be used. When using volatile vehicles, add small amounts (1.2% by weight) of organic binders (i.e. nitro cellulose) to improve the suspension characteristics and green strength of the applied material. The frit-to-vehicle ratio is determined by the application method, but success has been achieved working in a ratio range of between 5/1 to 14/1. Mix the slurry well and continue agitation to maintain a homogeneous slurry during application.

C. Drying

After the glass slurry has been applied, drying is required. Oven drying is recommended, but air drying or heat lamps can also be used. Do not use the sealing furnace for drying and avoid temperatures greater than 350°C. Recommended drying cycle: Heat at 200°C/hr. to peak temperature of 200°C, hold for a short time (15 min.) and cool at the same rate. To fuse the glass to the part for handling, heat the glass at a rate of 3-5°C/min. to its softening point (see chart), hold for 5-10 min., and cool at the same rate. When heating and cooling MACOR MGC parts, avoid rates in excess of 5°C/min. to prevent thermal shock of the material.

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D. Sealing Cycle

Sealing cycles depend on the geometry of the seal, complexity of the parts and the solder glass used. Large, complex parts require a very slow heating and cooling rate (150°C/hr.) to maintain the integrity of the MACOR MGC parts and to allow sufficient time for the frit to seal. Because of MACOR MGC's
Low thermal conductivity it is advisable to place a thermocouple as close as possible to the area being sealed. Smaller simpler shapes can be heated at a faster rate (300°C/hr.), but again a thermocouple should be placed as close as possible to the seal area.

If cooling rates above 300°C/hr. are necessary, anneal the MACOR MGC prior to using the faster cycle. Annealing procedure: Heat the MACOR MGC at 200°C/hr. to 750°C, hold at 750°C for two hours, and cool at a rate of 25°C/hr..

Sealing Glasses

Ferro[1] Code	Softening Point (°C)	Thermal Expansion (x 10^{-7} in./in./°C)	Sealing Criteria[3] Temp. (°C)-Hold Time (min.)	Continuous Use Temp. (°C)
7555*	415	88.5	450°C – 20 min.	300°C
7570*	440	84	470°C – 30 min.	330°C
7572	370	95	450°C – 60 min	425°C
7575	370	89	450°C – 60 min	425°C

[1] - Ferro Corporation, PO Box 6550, 4150 E. 56th Street, Cleveland OH, 44101. Tel. (216) 641-8585, ext. 7426, Fax (412) 641-8857.

[2] - Cerdec Corp./Drakenfeld Products, Box 519, Washington, PA, 15301. Tel (412) 223-5900, Fax (412) 228-3170.
Cerdec France SA, Tel 05-55-31-50-50, Fax 05-55-06-05-63.
Cerdec AG Keramische Farben, Tel (069) 27 116-0, Fax (069) 116-270.

[3] -These are idealized conditions. Heating and cooling rates are dependent on the size and geometry of the parts. Keep the cooling rates as slow as possible to prevent thermal shocking of the MACOR MGC and seal, and to relieve any stresses built up during the sealing operation.

*Vitreous solder glass.

Precautions

These glasses have a high lead content. The following precautions should be taken to avoid the possibilities of lead poisoning:

- Wear a respirator to avoid breathing the dust
- Wash hands thoroughly before eating, drinking or smoking.
- Wear a lab coat to reduce the chance of the material being carried home in work clothes.
- Read the manufacturers MSDS's for the materials used.

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