

Fused Quartz Properties & Usage Guide

MPM Type 214, 214LD and 124



Momentive Quartz Plant - Willoughby, OH

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Semiconductor Grade Fused Quartz Tubing

In the semiconductor industry a combination of extreme purity and excellent high temperature properties make fused quartz tubing an ideal furnace chamber for processing silicon wafers. The material can tolerate the wide temperature gradients and high heat rates of the process. And its purity creates the low contamination environment required for achieving high wafer yields. The advent of eight inch wafers combined with today's smaller chip sizes has increased chip production by a factor of four compared to technology in place just a few years ago. These developments have impacted heavily on quartz produced, requiring both large diameter tubing and significantly higher levels of purity. GE Quartz. has responded on both counts. Quartz tubing is available in a full range of sizes, including diameters of 400mm and larger. Diameter and wall thickness dimensions are tightly controlled. Special heavy wall thicknesses are available on request. By finding new and better sources of raw material, expanding and modernizing our production facilities, and upgrading our quality control functions, GE has reduced contaminants levels in its fused quartz tubing to less than 25 ppm, with alkali levels below 1 ppm.

Grade 214LD

This is the large diameter grade of industry standard 214 quartz tubing. For all but the highly specialized operations, this low cost tubing offers the levels of purity, sag resistance, furnace life and other properties that diffusion and CVD processes require. For superior performance at elevated temperatures GE type 214 LD furnace tubing gives process engineers a better balance between the effects of higher temperatures and heavier wafer loads.

224LD - Low Alkali Quartz Tubing

As the semiconductor industry moves toward higher densities, furnace atmosphere contaminant becomes an increasingly critical factor in controlling wafer yields. One potential contaminant is sodium, which occurs naturally in the silica sand used to make fused quartz. This highly mobile ion can effectively destabilize the electrical characteristics of MOS and bipolar devices if not removed. For these critical applications GE has developed Grade 224 low alkali fused quartz tubing. It is made in a special process that eliminates up to 90 % of the naturally occurring alkalis. The process achieves a typical sodium level of 0.1 ppm (vs. a normal 0.7 ppm), greatly reduces potassium, and virtually eliminates lithium.

244LD Low Alkali/Low Aluminum Quartz Tubing

This grade has been specially developed for quartz users concerned about the aluminum level in fused quartz. 244 has a typical aluminum level of 8 ppm.

Low (OH-)

One reason that GE fused quartz tubing can withstand the wide thermal gradients and chemical environments of wafer processing operations is its (OH⁻) content of less than 10 ppm water in most grades. Low OH⁻ minimizes the sag rate at diffusion temperatures, and effectively retards the progress of devitrification. Because of its low hydroxyl content, GE Quartz tubing does not require special coatings that could potentially release contaminants at elevated temperatures.

Fused Quartz Rod & Solids

GE supplies two forms of high purity fused quartz solid shapes for fabricators of quartzware. Type 214 rod has the high purity, elevated temperature characteristics and low coefficient of thermal expansion required for wafer carriers and push rods used in semiconductor wafer processing. The material is available in diameters of 1 to 20 mm. Very tight quality control and special processing of raw materials is used to achieve low levels of trace element contamination. When larger sizes and different shaped starting materials are required, GE supplies fabricators with pieces cut from fused quartz ingots. They are up to 72 inches in diameter, two feet thick, and weigh up to 9000 pounds.

Large Ingots

GE Type 124 ingots have been the semiconductor industry's material of choice for fabricating diffusion and CVD furnace components for a number of years. The advent of larger wafer sizes, tighter device geometries, and the drive for lower contaminant levels has stimulated GE's development of an even higher purity grade. Type 144 is specially processed to reduce alkali content by up to 90%. Sodium is held to 0.2 ppm or lower, potassium is significantly reduced while lithium is about 0.2 ppm. Type 012 provides the ultra high purity of synthetic fused silica, while maintaining low (OH) at < 5 ppm.

Lamp Grade Tubing

GE Quartz is the world's leading producer of fused quartz for lighting applications. Four basic types of lamp grade quartz are available, each designed to fulfill specific performance requirements. Together, these materials cover a wide variety of applications. They include:

Type 214

The worldwide standard for clear fused quartz lamp tubing. GE 214 is a high purity, high transmittance, high temperature material with a low hydroxyl (OH-) content. It is suitable for a broad range of mercury, halogen and other quartz lamp applications.

Type 219

Known as "Ozone-Free" or "Germicidal" quartz tubing. GE 219 transmits UV-A and UV-B while blocking the deep, high energy wavelengths that cause ozone generation and pose the greatest exposure risks. Type 219 transmits the 253.7 nanometer mercury emission very efficiently, making it an ideal material for disinfection applications and various other UV treatments.

Type 254

A doped quartz material that blocks virtually all UV-B and UV-C radiation. Type 254 has a transmittance cutoff wavelength between 350 and 400 nanometers. It is ideal for lamps requiring maximum visible transmittance with nearly complete UV protection. Applications for GE 254 are those where UV exposure to people or property is undesirable, including some quartz halogen and metal halide lamps and other UV sources.

Type 021

This is a dry synthetic fused silica material providing high transmittance in the deep ultraviolet range. It combines the advantages of low hydroxyl content with ultra high purity to yield superior UV transmittance and resistance to solarization for a variety of UV lamp applications including water purification, ozone generation, paint and ink curing, and chemical processing.

Types 214A, 219A, and 254A

These are identical to the standard types but are produced with a lower hydroxyl content. "A" products contain <1 ppm (OH-) and are intended for metal halide lamps and other applications where the quartz must be devoid of hydroxyl as well as all dissolved gases.

Quartz Crucibles

In the manufacture of silicon metal for semiconductor wafer applications, polysilicon starting materials are placed in fused quartz crucibles, heated to high temperatures and pulled from the melt as a single crystal. Fused quartz is one of the few materials that can combine the high purity and high temperature properties required.

Other Compositions

To keep pace with the increasingly stringent purity requirements of the industry, GE now offers a variety of compositions in its quartz crucibles. Each type is designed to address specific micro-contamination concerns. However, other options are also available. GE's "Crucible Team" is prepared to work with you on your specific crucible designs.

Fiber Optic Tubing

GE fused quartz series as deposition tubing for one of the major methods of producing optical waveguides, the Modified chemical vapor deposition (MCVD) process. For this application, GE offers high quality quartz tubing that is virtually airline free, with tight dimensional tolerances and low (OH-). This combination of characteristics translates into excellent attenuation for the fiber manufacturer. GE produces fiber optic tubing from either naturally occurring or synthetic quartz. The synthetic grades, combined with GE's unique continuous fusion process, produces fiber optic tubing with all the advantages found in natural occurring quartz, plus the higher tensile strength required for producing long length fibers. Along with waveguide material, GE offers high quality quartz tubing and handles required by the MCVD process. Each waveguide tube produced by GE is serialized, characterized and accompanied by a data slip showing the complete geometry of the tube. If desired, a computer disc can be supplied with the shipment for direct entry into our data bank.

Guidelines for Users of Fused Quartz

Like any material that is expected to provide a design life at high temperatures, fused quartz demands some care in handling and use to achieve maximum performance from the product.

Storage

Space permitting, fused quartz should be stored in its original shipping container. If that is not practical, at least the wrapping should be retained. In the case of tubing, the end coverings should be kept in place until the product is used. This protects the ends from chipping and keeps out dirt and moisture which could compromise the purity and performance of the tubing.

Cleaning

For applications in which cleanliness is important, General Electric recommends the following procedure: The product, particularly tubing, should be washed in deionized or distilled water with a degreasing agent added to the water. The fused quartz should then be placed in a 7% (maximum) solution of ammonium bifluoride for no more than ten minutes, or a 10 vol % (maximum) solution of hydrofluoric acid for no more than five minutes. Etching of the surface will remove a small amount of fused quartz material as well as any surface contaminants. To avoid water spotting which may attract dirt and cause devitrification upon subsequent heating, the fused quartz should be rinsed several times in de-ionized or distilled water and dried rapidly. To further reduce the possibility of contamination, care should be used in handling fused quartz. The use of clean cotton gloves at all times is essential. Washing of translucent tubing is not recommended because the water or acid solution tends to enter the many capillaries in the material. This may cause the quartz to burst if the pieces are subsequently heated rapidly to very high temperatures.

Rotation Procedures For Fused Quartz Furnace Tubes

The following procedure has been used to create an even layer of crystalite on diffusion tubes in order to increase resistance to devitrification. Place the tube in a furnace at 1200 \bar{A} ,C, and rotate it 90 \bar{A} , every two hours for the first 30 hours. If the working schedule does not permit adherence to this procedure, the following suggestion is offered. Place the tube in a furnace at 1200 \bar{A} ,C and rotate it 90 \bar{A} , every two hours for the first 8 hours, then reset the furnace to operating temperature.

Solarization

Fused quartz made from natural raw material solarizes or discolors upon prolonged irradiation by high energy radiation (such as short UV, x-rays, gamma rays and neutrons). Resistance to this type of solarization increases with the purity of fused quartz. Hence, synthetic fused silica is highly resistant to solarization. Solarization in fused quartz can be thermally bleached by heating it to about 500 \bar{A} ,C.

Technical Support

An important consideration for today's users of fused quartz is the availability of technical product support. GE Quartz backs its products with fully equipped analytical and development laboratories and a staff of materials and fusion experts available to support customer requirements. State-of-the-art analytical equipment assures optimal production quality and also enables certification and subsequent verification of GE Quartz product compliance with stringent industry standards. Physical properties and other information shown on pages 14 through 24 was developed from a number of sources, including GE's technical laboratories, text books and technical publications. While GE believes that this information is accurate, it is not an exhaustive review of the subjects covered and, accordingly, GE makes no warranty as to the accuracy or completeness of the data. Customers are advised to check references to ensure that the product is suitable for the customer's particular use or requirements. Additional technical assistance from our engineering team is available by calling or faxing our world headquarters.

Table of Typical Physical Properties, Type 214 Fused Quartz

Property	Typical Values
Density	2.2x10 ³ kg/m ³
Hardness	5.5 - 6.5 Mohs' Scale 570 KHN 100
Design Tensile Strength	4.8x10 ⁷ Pa (N/m ²) (7000 psi)
Design Compressive Strength	Greater than 1.1 x 10 ⁹ Pa (160,000 psi)
Bulk Modulus	3.7x10 ¹⁰ Pa (5.3x10 ⁶ psi)
Rigidity Modulus	3.1x10 ¹⁰ Pa (4.5x10 ⁶ psi)
Young's Modulus	7.2x10 ¹⁰ Pa (10.5x10 ⁶ psi)
Poisson's Ratio	.17
Coefficient of Thermal Expansion	5.5x10 ⁻⁷ cm/cm . \bar{A} ,C (20 \bar{A} ,C-320 \bar{A} ,C)
Thermal Conductivity	1.4 W/m . \bar{A} ,C
Specific Heat	670 J/kg . \bar{A} ,C
Softening Point	1683 \bar{A} ,C
Annealing Point	1215 \bar{A} ,C
Strain Point	1120 \bar{A} ,C
Electrical Resistivity	7x10 ⁷ ohm cm (350 \bar{A} ,C)
Dielectric Properties	(20 \bar{A} ,C and 1 MHz)
Constant	3.75
Strength	5x10 ⁷ V/m
Loss Factor	Less than 4x10 ⁻⁴
Dissipation Factor	Less than 1x10 ⁻⁴
Index of Refraction	1.4585
Constringence (Nu)	67.56
Velocity of Sound-Shear Wave	3.75x10 ³ m/s
Velocity of Sound/Compression Wave	5.90x10 ³ m/s
Sonic Attenuation	Less than 11 db/m MHz
Permeability Constants (700 \bar{A} ,C)	(cm ³ mm/cm ² sec cm of Hg)
Helium	210x10 ⁻¹⁰
Hydrogen	21x10 ⁻¹⁰
Deuterium	17x10 ⁻¹⁰
Neon	9.5x10 ⁻¹⁰

Chemical Composition

Vitreous silica is the generic term used to describe all types of silica glass, with producers referring to the material as either fused quartz or as fused silica. Originally, those terms were used to distinguish between transparent and opaque grades of the material. Fused quartz products were those produced from quartz crystal into transparent ware, and fused silica described products manufactured from sand into opaque ware. Today, however, advances in raw material beneficiation permit transparent fusions from sand as well as from crystal. Consequently, if naturally occurring crystalline silica (sand or rock) is melted, the material is simply called fused quartz. If the silicon dioxide is synthetically derived, however, the material is referred to as synthetic fused silica. Controlled

Process: The performance of most fused quartz products is closely related to the purity of the material. GE's proprietary raw material bonification and fusion processes are closely monitored and controlled to yield typically less than 50 ppm total elemental impurities by weight. GE clear fused quartz varieties have a nominal purity of 99.995 W % SiO₂. Structural hydroxyl (OH⁻) impurities are also shown. The strong IR absorption of OH⁻ species in fused quartz provides a quantitative method for analysis. Beta Factor: The term Beta Factor is often used to characterize the hydroxyl (OH⁻) content of fused quartz tubing. This term is defined by the formula shown below.

Electrical Properties

Since electrical conductivity in fused quartz is ionic in nature, and alkali ions exist only as trace constituents, fused quartz is the preferred glass for electrical insulation and low loss dielectric properties. In general, the electrical insulating properties of clear fused quartz are superior to those of the opaque or translucent types. Both electrical insulation and microwave transmission properties are retained at very high temperatures and over a wide range of frequencies.

Mechanical Properties

Mechanical properties of fused quartz are much the same as those of other glasses. The material is extremely strong in compression, with design compressive strength of better than 1.1×10^9 Pa (160,000 psi). Surface flaws can drastically reduce the inherent strength of any glass, so tensile properties are greatly influenced by these defects. The design tensile strength for fused quartz with good surface quality is in excess of 4.8×10^7 Pa (7,000 psi). In practice, a design stress of $.68 \times 10^7$ Pa (1,000 psi) is generally recommended.

Permeability

Fused quartz is essentially impermeable to most gases, but helium, hydrogen, deuterium and neon may diffuse through the glass. The rate of diffusion increases at higher temperatures and differential pressures. The selective diffusion of helium through fused quartz is the basis for a method of purifying helium by essentially "screening out" contaminants by passing the gas through thin-walled quartz tubes. The diffusion of helium, hydrogen, deuterium and neon through fused quartz is accelerated with increasing temperature. According to General Electric Research Laboratory, the permeability constants for these gases through fused silica at 700 Å,C are estimated to be: Helium 2.1×10^{-8} cc/sec/cm²/mm/cm.Hg. Hydrogen 2.1×10^{-9} . Deuterium 1.7×10^{-9} . Neon 9.5×10^{-10}

Thermal Properties

One of the most important properties of fused quartz is its extremely low coefficient of expansion: 5.5×10^{-7} mm Å,C (20-320Å,C). Its coefficient is 1/34 that of copper and only 1/7 of borosilicate glass. This makes the material particularly useful for optical flats, mirrors, furnace windows and critical optical applications which require minimum sensitivity to thermal changes. A related property is its unusually high thermal shock resistance. For example, thin sections can be heated rapidly to above 1500 Å,C and then plunged into water without cracking. The residual stress or design, depending on the application, may be in the range of 1.7×10^7 to 20.4×10^7 Pa (25 to 300 psi). As a general rule, it is possible to cool up to 100Å,C/hour for sections less than 25 mm thick.

Effects Of Temperature

Fused quartz is a solid material at room temperature, but at high temperatures, it behaves like all glasses. It does not experience a distinct melting point as crystalline materials do, but softens over a fairly broad temperature range. This transition from a solid to a plastic-like behavior, called the transformation range, is distinguished by a continuous change in viscosity with temperature.

Viscosity

Viscosity is the measure of the resistance to flow of a material when exposed to a shear stress. Since the range in "flowability" is extremely wide, the viscosity scale is generally expressed logarithmically. Common glass terms for expressing viscosity include: strain point, annealing point, and softening point, which are defined as: Strain Point: The temperature at which the internal stress is substantially relieved in four hours. This corresponds to a viscosity of 10^{14.5} poise, where poise = dynes/cm² sec. Annealing Point: The temperature at which the internal stress is substantially relieved in 15 minutes, a viscosity of 10^{13.2} poise. Softening Point: The temperature at which glass will deform under its own weight, a viscosity of approximately 10^{7.6} poise. The softening point of fused quartz has been variously reported from 1500 Å,C to 1670 Å,C, the range resulting from differing conditions of measurement.

Devitrification

Devitrification and particle generation are limiting factors in the high temperature performance of fused quartz. Devitrification is a two step process of nucleation and growth. In general, the devitrification rate of fused quartz is slow for two reasons: the nucleation of the cristobalite phase is possible only at the free surface, and the growth rate of the crystalline phase is low. Nucleation in fused quartz materials is generally initiated by surface contamination from alkali elements and other metals. This heterogeneous nucleation is slower in non stoichiometric fused quartz, such as GE quartz, than in stoichiometric quartz materials.

Cristobalite Growth

The growth rate of cristobalite from the nucleation site depends on certain environmental factors and material characteristics. Temperature and quartz viscosity are the most significant factors, but oxygen and water vapor partial pressures also impact the crystal growth rate. Consequently, the rate of devitrification of fused quartz increases with increasing hydroxyl (OH⁻) content, decreasing viscosity and increasing temperature. High viscosity, low hydroxyl fused quartz materials produced by GE Quartz, therefore, provide an advantage in devitrification resistance. The phase transformation to Beta-cristobalite generally does not occur below 1000Å,C. This transformation can be detrimental to the structural integrity of fused quartz if it is thermally cycled through the crystallographic inversion temperature range (250 Å,C). This inversion is accompanied by a large change in density and can result in spalling and possible mechanical failure. Thermal Properties, cont

An Advantage

In certain applications, devitrification can be put to the user's advantage since the cristobalite tends to inhibit sag of the fused quartz. For example, if a diffusion

furnace tube is to be used at high temperatures for extended periods of time, and is not subject to thermal cycling below the cristobalite transformation, rotation procedures described on page 24 have been found to be beneficial.

Contamination

Contamination in almost any form is detrimental. Alkaline solutions, salts, or vapors are particularly deleterious. Handling of fused quartz with the bare hands deposits sufficient alkali from perspiration to leave clearly defined fingerprints upon devitrification. Drops of water allowed to stand on the surface will collect enough contamination from the air to promote devitrified spots and water marks. Surface contamination affects devitrification in two ways. First, the contaminant promotes nucleation of the cristobalite. Second, it acts as a flux to enhance the cristobalite to (high) tridymite transformation. Under some conditions, the tridymite devitrification will grow deeply and rapidly into the interior of the fused quartz. Heating fused quartz to elevated temperatures (ca. 2000 Å_C) causes the SiO2 to undergo dissociation or sublimation. This is generally considered to be: SiO2 -> SiO + 1/2 O2. Consequently, when flame-working fused quartz, there is a band of haze or smoke which forms just outside the intensely heated region. This haze presumably forms because the SiO recombines with oxygen from the air (and perhaps water) and condenses as extremely small particles of amorphous SiO2. The haze can be removed from the surface by a gentle heating in the oxy-hydrogen flame. The dissociation is greatly enhanced when the heating of fused quartz is carried out in reducing conditions. For example, the proximity or contact with graphite during heating will cause rapid dissociation of the SiO2.

Resistance To Sag

The most significant chemical factor effecting the sag resistance of fused quartz is the hydroxyl (OH-) content. GE controls the (OH-) content in its quartz to meet the specific needs of its customers. To maximize the performance of tubes used in high temperature semiconductor processes, it is important to understand the impact of changes in diameter and wall thickness. In one study using GE 214LD fused quartz tubing, it was found that the sag rate decreases as the wall thickness of the tube is increased. Generally, as the wall thickness doubles, the sag rate decreases by a factor of approximately 3. Also, it was shown that with a fixed wall thickness, the sag rate decreases as the tube diameter decreases.

Typical Trace Element Composition (ppm by weight)

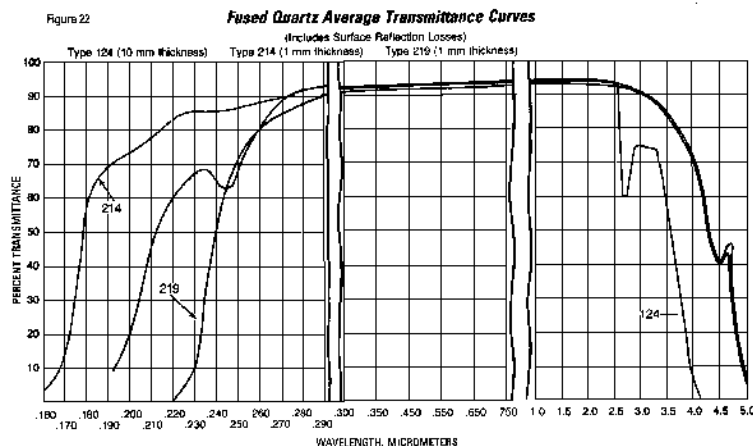
Type	Al	As	B	Ca	Cd	Cr	Cu	Fe	K	Li	Mg	Mn	Na	Ni	P	Sb	Ti	Zr	OH	Type
214	14	<0.002	<0.2	0.4	<0.01	<0.05	<0.05	0.2	0.6	0.6	0.1	<0.05	0.7	<0.1	<0.2	<0.003	1.1	0.8	<5	214
219	14	<0.01	<0.2	0.4	<0.01	<0.05	<0.05	0.2	0.6	0.6	0.1	<0.05	0.7	<0.1	<0.2	<0.003	100	0.8	<5	219
214A	14	<0.002	<0.2	0.4	<0.01	<0.05	<0.05	0.2	0.6	0.6	0.1	<0.05	0.7	<0.1	<0.2	<0.003	1.1	0.8	<1	214A
214Rod/LD	14	<0.002	<0.2	0.4	<0.01	<0.05	<0.05	0.2	0.6	0.6	0.1	<0.05	0.7	<0.1	<0.2	<0.003	1.1	0.8	10	214Rod/LD
224/Rod	14	<0.002	<0.2	0.4	<0.01	<0.05	<0.03	0.2	<0.2	<0.2	0.1	<0.03	<0.2	<0.1	<0.2	0.003	1.4	0.8	10	224/Rod
224LD	14	<0.002	<0.2	0.4	<0.01	<0.05	<0.01	0.2	<0.2	0.001	0.1	<0.05	<0.1	<0.1	<0.2	0.003	1.1	0.8	10	224LD
244/Rod	8	<0.002	<0.1	0.6	<0.01	<0.05	<0.03	0.2	<0.2	<0.2	<0.1	<0.03	<0.2	<0.1	<0.2	<0.03	1.4	0.3	10	244/Rod
244LD	8	<0.02	<0.1	0.6	<0.01	<0.05	<0.01	0.2	<0.2	0.001	<0.1	<0.03	0.1	<0.1	<0.2	<0.003	1.4	0.3	10	244LD
124	14	<0.002	<0.2	0.6	<0.01	<0.05	<0.05	0.2	0.6	0.6	0.1	<0.05	0.7	<0.1	<0.2	<0.003	1.1	0.8	<5	124
144	8	<0.002	<0.1	0.6	<0.01	<0.05	<0.05	0.2	<0.2	<0.2	<0.1	<0.03	<0.2	<0.1	<0.2	<0.03	1.4	0.3	<5	144

Optical Properties

Optical transmission properties provide a means for distinguishing among various types of vitreous silica as the degree of transparency reflects material purity and the method of manufacture. Specific indicators are the UV cutoff and the presence or absence of bands at 245 nm and 2.73 um. The UV cutoff ranges from ~155 to 175 nm for a 10 mm thick specimen and for pure fused quartz is a reflection of material purity. The presence of transition metallic impurities will shift the cutoff toward longer wavelengths. When desired, intentional doping, e.g., with Ti in the case of Type 219, may be employed to increase absorption in the UV. The absorption band at 245 nm characterizes a reduced glass and typifies material made by electric fusion. If a vitreous silica is formed by a "wet" process, either flame fusion or synthetic material, for example, the fundamental vibrational band of incorporated structural hydroxyl ions will absorb strongly at 2.73 um.

UV Cutoff

As the transmission curve in below illustrates, GE Type 214 fused quartz has a UV cutoff (1 mm thickness) at < 160 nm, a small absorption at 245 nm and no appreciable absorption due to hydroxyl ions. Type 219, which contains approximately 100 ppm Ti, has a UV cutoff at ~230 nm for a 1 mm thick sample. The IR edge falls between 4.5 and 5.0 um for a 1 mm thick sample. The chart details the percent transmittance for Types 214, 124 and 219 fused quartz, including the losses caused by reflections at both surfaces. Values represent a 1 mm thick Type 214 sample and a 10 mm thick Type 124 sample. Type 124 fused quartz is a very efficient material for the transmission of infrared radiation. Its infrared transmission extends out to about 4 micrometers with little absorption in the "water band" at 2.73 um.



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