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MINERAL INSULATION - A CRITICAL STUDY

A.A. WINER and S.B. WANG



MINERALS RESEARCH PROGRAM

MINERAL SCIENCES LABORATORIES

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MINERAL INSULATION - A CRITICAL STUDY by

A.A. Winer* and S.B. Wang*

ABSTRACT

Mineral insulation, a glass fibre product formed from melting mixtures of inorganic material, is a simple and economical component in energy conservation. Because of expected shortages in energy fuels and subsequent increases in costs, all economic sectors will be effected. The Canada Centre for Mineral and Energy Technology (CANMET) is involved in a project on mineral insulation. As part of this project, a critical study of the literature and field trips to industry have been made and discussions held with experts in insulation technology.

Composition of the inorganic mixture and temperature both influence glass structure, and structure in turn determines glass properties. Important parameters in fiberizing the glass are viscosity, temperature, surface tension and density. Phase equilibria diagrams are very useful in determining optimum composition and correct fiberizing temperature.

Most mineral producers in Canada use the cupola melting furnace and wheel spinning process but the electric furnace is becoming more popular because of its inherent advantages such as the ability to use a finer and therefore more homogeneous charge; it is also more easily controlled and inhibits environmental pollution. In electric melting, electrical conductivity is an important parameter and is related to the degree of ionization.

Tests are performed on mineral insulation products to satisfy standards established for applications in industry, building, and homes. Such standards are not all uniform across the country but may vary according to local requirements.

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ISOLATION MINERALE - UNE CRITIQUE

par

A.A. Winer* et S.B. Wang*

RESUME

Les produits isolants minéraux sont fabriqués à partir d'une fibre de verre obtenue en faisant fondre des mélanges de matériaux inorganiques. Ils constituent un élément simple et économique de conservation de l'énergie. La pénurie éventuelle des carburants et l'augmentation des coûts qui en résultera auront un effet sur la plupart des secteurs économiques. Le Centre canadien de la technologie des minéraux et de l'énergie (CANMET) est impliquée dans un project sur l'isolation minérale. En conséquence, une critique sur la documentation et des visites en industrie ont été effectées et des discussions en été entamées avec des spécialistes de la technologie de l'isolation. La composition du mélange inorganique et la température influencent la structure du verre et c'est cette structure qui détermine les propriétés du verre. Les paramètres importants pour la mise en fibre du verre sont la viscosité, la température, la tension superficielle et la densité. Les diagrammes d'équilibre de phase sont très utiles pour déterminer la composition optimale et la température adéquate de formation des fibres.

La plupart des producteurs de minéraux au Canada utilisent le cubilot et le procédé de centrifugation mais le four électrique devient de plus en plus populaire à cause de ses avantages intrinsèques notamment la possibilité d'employer une charge plus fine donc plus homogène; celui-ci est aussi plus facile à contrôler et réduit la pollution de l'environnement. Dans la fusion électrique, la conductivité électrique est un important paramètre et est fonction du degré d'ionisation.

Des essais sont effectués sur des produits isolants minéraux afin d'en vérifier leur conformité aux normes établis pour l'industrie, les immeubles et le secteur résidentiel. Ces normes diffèrent à travers le pays selon les exigences locales particulières.

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INTRODUCTION

Mineral thermal insulation is a generic term describing various glass fibre products differentiating one from another by composition of the original furnace charge. Rock wool, slag wool and glass fibre are terms describing particular products. Mineralogically, all insulating fibres made from an inorganic charge are considered to be glass in its final fibrous form. However, conventional "glass fibre insulation", is made from a mixture of industrial minerals similar to those for making glass. The term rock wool includes products made from limestone and silicious materials, and slag wool contains, as its major ingredient, slags from blast furnaces, copper smelters, etc.

Mineral insulation has a vital role to play in energy conservation which has now become very important to the Canadian economy. Because of expected shortages in energy fuels such as gas and oil, there are few sectors of the Canadian economy that will not be affected when shortfalls occur. Energy, Mines and Resources Canada has thus focused on conservation in its energy research programs.

Aware of the imminent need for energy conservation, the Canada Centre for Mineral and Energy Technology initiated a project to produce mineral insulation. This was successfully done on an experimental scale and the project is now continuing on a more intensive basis.

GLASS AND GLASS FIBRE

FUNDAMENTAL CONSIDERATIONS

Mineralogically, all mineral insulation fibres are formed as a glass, either in bulk or fibrous form, on cooling from an inorganic melt. Morey defined a glass as an inorganic substance continuous with and analogous to its liquid state, but it can be considered rigid because of a reversible change in viscosity during cooling (1).

The composition of a glass and its thermal history, e.g., temperature of the melt, rate of cooling and fiberization temperature, will govern its properties. The room temperature structure

corresponds to the equilibrium structure which this composition would have in the liquid state at some elevated temperature. This structure equivalence is referred to as the configuration temperature (2).

In the solid oxide structure, metallic cations are surrounded by oxygen ions in a three-dimensional crystalline network. Each cation is surrounded by the maximum number of oxygen anions, referred to as the coordination number which depends only on the size and charge on the ions. During melting the crystalline network is destroyed and, in the liquid phase, the bonds between ions are broken by thermal agitation. The cations are still surrounded by anions but less rigidly, and the coordination number may vary (3). Examples given by Loewenstein are:

- Si⁴⁺ surrounded by four 0²⁻ ions form the SiO₄ group;
- 2. Si⁴⁺ requires 6 fluoride ions because of smaller radii of F to form SiF₆ sufficient for adequate screening of the electrical field (2).

Important in this respect is that the screening required becomes less as the temperature is increased. For example, ${\rm TiO}_6$ at room temperature may exist as ${\rm TiO}_1$ at elevated temperatures.

Ions attempt to screen their electrical field with ions of opposite charge. This competition for different ions is related to polarizing power. The field strength of an ion, a measure of polarizing power, is given by the expression ${\rm Z/r}^2$, where Z = ion charge and r = ionic radius (2). From these considerations the following statements can be derived:

- (a) The polarizing power, varying directly as the field strength, requires surrounding ions of opposite charge in numbers related to the field strength.
- (b) From the above formula the highest field strength is when Z is high and r is small.

At high field strengths, screening requirements for ions are so strong that a positive ion surrounded by negative ions may share electrons; mobility of the ions may therefore be restricted except at very high temperatures. This type of bonding intermediate between shared elec-

trons (covalent bonding), and donated electrons (ionic bonding), is a characteristic of a glass former of which silica (SiO₂) is a prime example.

An example of bonding and attraction forces is shown in Fig. 1 where silica consists of rings of ${\rm Si0}_{\mu}$ groups with ${\rm O}^{2-}$ ions shared between ${\rm Si}^{4+}$ ions.

Fig. 1 - Bonding of $\mathrm{Si0}_{\,\mu}$ groups with shared oxygen ions. (2)

Table 1 shows the types of bonding and attraction forces for mineral oxides in slags which are of interest for mineral insulation production.

Alumina is a special type of network former because it is amphoteric. It forms ${\rm AlO}_{\downarrow }$ groups in glasses and requires positively charged ions for electrical neutrality. Network modifiers, such as the basic oxides, e.g., CaO, BaO, ${\rm Na}_2{\rm O}$, and ${\rm K}_2{\rm O}$, break Si-O-Si bridges and by doing so reduce the temperature for glass-making as shown below:

$$-\overset{1}{\underset{1}{\text{Si}}} - 0 - \overset{1}{\underset{1}{\text{Si}}} - \overset{\text{Na}_{2}0}{\underset{1}{\text{Na}}} - \overset{1}{\underset{1}{\text{Si}}} - 0 \overset{\text{Na}}{\underset{1}{\text{Na}}} - \overset{1}{\underset{1}{\text{Si}}} - 0 - \overset{\text{Na}}{\underset{1}{\text{Si}}} - 0 - \overset{\text{Na}}{\underset{1}{\text{Na}}} -$$

Each ion has an electric field related to its field strength. The ion which fits into the network is surrounded by a number of oxygen ions. The electrical field of the ion is adequately screened at the configuration temperature.

Intermediate oxides, such as Mg0, Be0, and ${\rm TiO}_2$ can take up both positions depending on the composition and configuration temperature of the glass.

GLASSES AND THEIR PROPERTIES

Silica based glasses can be made by heating the mixture to 1300-1600°C which results in a modification of the silica network, and other structurally related modifications. Figure 2 depicts the silica structure in both the solid and molten states.

Passage from solid silica to the viscous liquid has no well-defined fusion point and only prolonged heating at about 1200°C will cause crystallization. Energy levels between glass and crystalline states are small.

The properties of glasses are in general related to their structure (4). Oxides which can also influence the property and structures of a glass fall into distinct groups as shown in the last column of Table 1.

The tensile strength of a glass is much lower than the theoretical due to flaws on its surface. This has been demonstrated by removing the flaws by etching, resulting in a significant increase in strength. The tensile strength of glass also decreases with increasing temperature. Glass fibre strength measured at room temperature and humidity conditions is dependent on glass composition, fibre diameter, and the bulk glass temperature from which it was drawn.

Because of the negative charges on the surface of silica glasses, the surface attracts water molecules to which ${\rm H}^+$ ions are bonded strongly, with the ${\rm O}^{2-}$ ions end away from the surface. A temperature of 500°C is required to remove this water about 25 molecules thick (2).

Electrical properties are governed by the presence of alkali such as Na₂O and K₂O. Electrical conduction is generally via the surface of the glass and only at elevated temperatures does conduction through the bulk of the glass become important. When Na⁺ ions are present on the surface of a glass which is covered with absorbed water, these ions would likely provide an electrical path on the surface of the glass.

Density, refractive index and Young's modulus are related to the structure of the glass and, therefore, to the composition and configuration temperatures. When fiberizing the glass, the faster cooling of the melt results in the above

properties being lower than in the bulk glass - density by about 2%, refractive index by 0.15 - 0.4, and Young's modulus by 7-25%. When fibres of the same diameter are drawn at increasing temperature from the same bulk melt, they show a de-

creasing Young's modulus with increasing temperature. Tempering the fibres between 100° and 400°C decreases the difference in these properties.

Table 1 - Types of bonding and attraction forces between cations and 0^{2-} anion (3)

0xide	$z/(R_c + R_a)*$	Ionic fraction	Coordination	Character of
		of bond	number	the oxide
			solid-liquid	
Na ₂ 0	0.18	0.65	6 6 to 8	
Ba0	0.27	0.65	8 8 to 12	
Sr0	0.32	0.61	8	
Ca0	0.35	0.61	6	Network breakers
Mn0	0.42	0.47	6 6 to 8	or
Fe0	0.44	0.38	6 6	basic oxides
Zn0	0.44	0.44	6	
Mg0	0.48	0.54	6	
Be0	0.69	0.44	4	
	0. 50	0.114	11	
^{Cr} 2 ⁰ 3	0.72	0.41	4	
Fe ₂ 0 ₃	0.75	0.36	4	Amphoteric
A1 ₂ 0 ₃	0.83	0.44	6 4 to 6	oxides
TiO ₂	0.93	0.41	4	
SiO ₂	1.22	0.36	4 4	Network formers
P ₂ 0 ₅	1.66	0.28	4 4	or acid oxides

^{*}F = $2ze^2/(R_c + R_a)^2$ proportional to attraction between ions where

 $R_a + R_c = distance between ions$

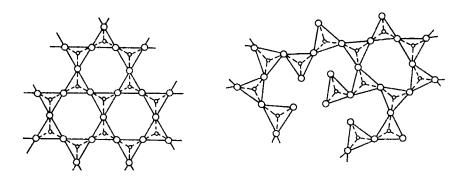


Fig. 2 - Structure of silica: (a) solid; (b) molten. (3)

F = attraction force between anions and cations

The physico-chemical properties of the charging mixture play an important role influencing the melt, the process and, therefore, the fibrous insulation product. Some of the more important material parameters are viscosity, temperature, surface tension, density and liquidus temperature. The chemical consitituents of the charge obviously will influence the melt.

The standard composition of a mineral insulation melt generally falls within the SiO₂-Al₂O₃-MgO-CaO phase diagrams. However, care must be exercised because small changes in SiO₂ content are said to cause large changes in viscosity which would require constant changes in temperature (5).

A comprehensive treatment of the science and technology of glass is contained in the proceedings of the XI International Congress on Glass (6).

VISCOSITY

The viscosity of a melt is dependent on composition and temperature. The temperature viscosity relationship is shown by the following model (3):

$$n = Ae^{(E_n/RT)}$$

where

A = a constant

 E_{η} = activation energy of slag viscous flow dependent on the composition.

Activation energy of viscous flow of a slag is large, indicated by the small decrease of viscosity with initial temperature rise. With an acid slag, the addition of a basic flux or network modifier such as Na₂O breaks the bonds of the silica tetrahedral structure, decreasing the activation energy and consequently the viscosity.

Predicting the roles of particular metal oxides in silicious melts has been attempted and for this the cationic field strength has been used. Van Der Colf et al. noted a direct relationship for the slags studied between viscosity and electrical resistivity (7). This is not surprising because viscosity is governed by the size of the anions while resistivity depends on the nature of the current-carrying cations. Increas-

ing the slag temperature thereby loosens the liquid structure and consequently decreasing viscosity and resistivity of the melt.

The slag samples studied at 1500°C by Van Der Colf with results for viscosities and resistivities are shown in Table 2 (7).

A graph of log ρ resistivity vs log η viscosity for the slags is shown in Fig. 3. Although the slopes vary, the general line equation is:

$$\ln \rho = m \ln \eta + \ln C$$
 (6)

Kozakevitch measured the change of viscosity with temperature and composition, using a rotating cylinder viscometer, for lime-aluminasilicate melt systems (8). The results of the study up to the temperature range of interest for mineral isulation is shown in Table 3.

Carron considered that the viscosity of silicate melts at any particular temperature was influenced by the relative proportions of network modifers present in the glass (9). Apart from silicon, the other network formers are the ions ${\rm Al}^{3+}$, ${\rm Fe}^{3+}$ and ${\rm P}^{5+}$. The modifiers are taken to be ${\rm Na}^+$, ${\rm K}^+$, ${\rm Ca}^{2+}$ and ${\rm Fe}^{2+}$. The chemical analysis of a silicate material is given in terms of oxides and from this information the following calculations are a mixture or feed material:

- (a) the number of network former cations (T)
- (b) the number of network modifier cations
- (c) the number of oxygen atoms associated with all the cations (0).

The number of active oxygen ions in the melt are then determined. This is defined by:

$$O_A = 2 (0 - 2T)$$
 Eq 1

The parameter related to viscosity is defined by:

$$R = \frac{O_A}{T} \times 100$$
 Eq 2

Log R is then plotted vs log viscosity to give straight lines. These calculations have been used in practice and found to be reasonably accurate (10).

Carron interpreted the results of viscosity measurements to indicate that the viscosity of a glass melt depends on the percentage of broken bonds between the alumino-silicate structure (9). He stated that results of the melt viscosity of "rocks and minerals" could be interpreted by the same model as for binary glasses SiO₂-M₂O.

Carron stated that the viscosity of any silicate melt can be estimated if the chemical composition, water content, and temperature are known. This estimate comprises the range from 10^2 to 10^{15} poises.

Saito and Kawai suggested that molten slags are considered to be ionic solutions and are composed of various networks of ${\rm Si0}_{11}$ tetra-

Table 2 - Viscosities and resistivities of slags at 1500°C

Slag N	η ₁₅₀₀ .s/m ² x 10	ρ ₁₅₀₀ Ω.m x 10 ²	^{ln η} 1500	ln P ₁₅₀₀	ρ ₁₅₀₀ (calc.) Ω.m x 10 ²
A5	0.91	0.88	-0.094	-0.128	0.79
B14	0.72	0.63	-0.329	-0.462	0.67
B13	0.66	0.66	-0.415	-0.415	0.63
B15	1.32	1.00	-0.278	-0.000	1.02
B11	0.80	0.68	-0.223	-0.386	0.72
B17	1.78	1.00			
C23	0.83	0.57			

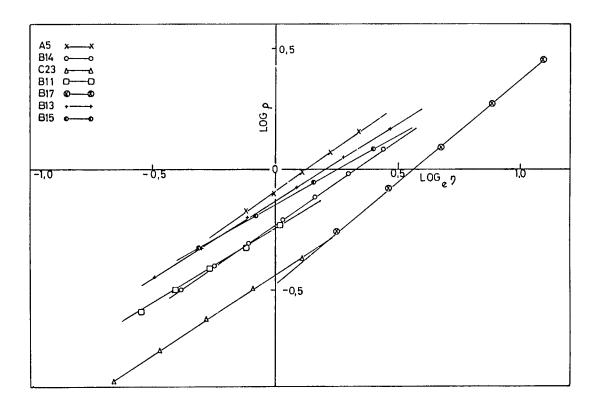


Fig. 3 - Relation between viscosity (\eta) and resistivity (p) of the same slag

Table 3 - Viscosity	of Ca0-Al ₂ 0 ₃ -Si0 ₂	melts between	1450-1600°C (8)
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SiO ₂	^{A1} 2 ⁰ 3	Ca0	1	Viscosity in	n poises at	
wt %	wt %	wt %	1450°C	1500°C	1550°C	1600°C
10	40	50	7.7	5.0	3.3	2.3
20	40	40	_	_	6.3	4.0
20	50	30	_	11.5	7.4	4.7
30	30	40	_	9.2	6.1	4.4
30	40	30	30.2	18.0	11.4	7.8
40	10	50	5.3	3.9	2.8	2.1
40	20	40	14.7	9.2	6.2	4.3
40	30	. 30	39.2	25.7	16.8	11.0
50	0	50	_	-	2.4	1.8
50	10	40	12.3	8.6	6.2	4.5
50	20	30	42.3	32.2	22.1	15.8
60	0	40	_	9.3	6.5	4.7
60	10	30	_	-	_	17.9_

hedra (11). Addition of metal oxides destroys the network and finally forms single SiO_{4} tetrahedra. Addition of small amounts of $\mathrm{Al}_2\mathrm{O}_3$ to a slag melt does not destroy the SiO_4 network but large amounts of CO_3 caused formation of AlO_3^{3-} ions with consequent large increases in viscosity. Additions of SiO_2 of less than 10 % to the CaO- $\mathrm{Al}_2\mathrm{O}_3$ system resulted in small changes in the slag melt viscosity which implied only minor formation of SiO_4 tetrahedra. The basis for this study was similar to that used by Coudurier (3) and the results could be applied to granite glasses, molten basalts, anhydrous melts, and to hydrated magmas.

Viscosities of some liquids and of rocks and minerals are shown in Table 4. The results show that viscosity of silicate melts appears to be a function of composition. Iron and manganese oxides lower the viscosity of these melts. High magnesium content slags are less viscous than calcium silicate slags but iron is a better flux than magnesium. Alumina, silica, titania and chromium oxide increase viscosity. However, the influence of these oxides is also dependent on temperature of the melt. In general, melts of acid rocks are more viscous than those of basic rocks.

Turkdogan and Bills have critically reviewed the viscosity relationships of the CaO-MgO-Al $_2$ O $_3$ -SiO $_2$ melt system (13). It is claimed that

inconsistencies of early data for the above systems were partly due to the use of graphite crucibles because alumina-silicate melts react readily with graphite at high temperatures. In other studies from this paper, it was shown that for a given temperature the viscosity and composition relationships of alkaline earth silicates were the same. The effect of the alkali oxides was similar on the viscosity of binary and ternary melts. Also, for any particular silica concentration, the vicosity will increase as more alumina replaces calcia, CaO to a molar ratio of Al₂O₃:CaO of about 1:1. Increasing the ratio by increasing Al₂O₃ results in a viscosity decrease. This is a good example of the amphoteric nature of alumina.

For slags that are neutral or basic, the effects on the viscosity of ${\rm Al_2O_3}$ and ${\rm SiO_2}$ are similar when ${\rm Al_2O_3}$:CaO ratios are low. It is therefore assumed that all the network-forming ions are Si + Al. When the ${\rm Al_2O_3}$:CaO ratios and silica concentrations are both large, the relationship becomes more complex. In this review it is shown that the alkaline earth oxides are interchangeable in their effects on the viscosity of binary melts and this behaviour is assumed to be similar for the quaternary melts. This is shown in Fig. 4.

In the system CaO-MgO-Al₂O₃-TiO₂- SiO₂,

Table 4 -	Viscosities of	selected	common	liquids,	rocks	and
	minerals (12)					

Substance	Temperature	Viscosity (η)
	t°C	poise
Water	20	1 x 10 ⁻²
Glycerine	20	10
Gelatine	20	1 x 10 ⁶ -1 x 10 ⁸
Diopside	1400	3.8
Anorthite	1400	38
Albite	1400	4 x 10 ⁴
Orthoclase	1400	1 x 10 ⁸
Diabase	1400	60 - 90
Basalt	1400	55
Nepheline basalt	1400	80
Olivine basalt	1400	1.2×10^2 -1.36 × 10^2
Andesite basalt	1400	1.38×10^2
Vesuvius lava	1400	2.5×10^{2}
Andesite	1400	1.17 x 10 ³
Obsidian	1400	1.7×10^{5}

the composition variable with the most influence on viscosity as well as resistivity was the basicity expressed as the mass ratio $(Ca0 + Mg0):Sio_2$ (7). The ratio Ca0:MgO was important only at low basicity.

The variation of viscosity with temperature for the same composition is shown in Fig. 5 and is hypothesized to be due to breakdown of Si-0 and Al-O bonds, i.e., structural changes have taken place (13). Below 1350°C, the relationship between log viscosity (η) and $\frac{1}{T}$ is linear but deviates from linearity at higher temperatures due to changes in the structure of the melt.

Turkdogan and Bills concluded that the silica equivalence of alumina varied with the Al₂O₃:CaO ratio and with the alumina concentration (13). Magnesia and lime, on a molar basis, have the same effect on viscosity.

LIQUIDUS TEMPERATURE

The melting point of a charge strongly depends on the composition which can be checked along the liquidus line and this line can be used to determine devitrification zones for a particular slag melt composition.

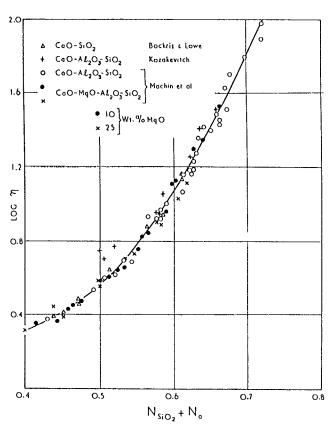


Fig. 4 - Variation of viscosity with composition in the system CaO-MgO-Al $_2$ O $_3$ -SiO $_2$ (13) Note: N $_a$ is the silica equivalence of alumina.

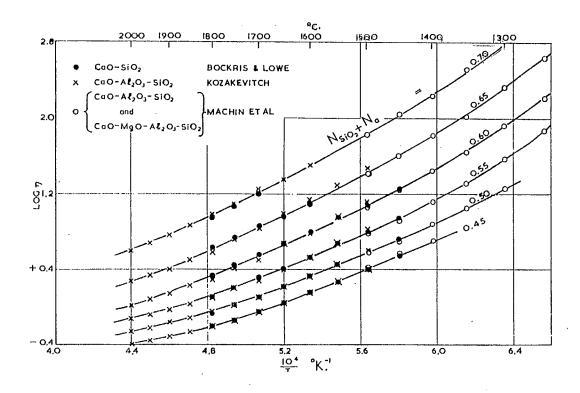


Fig. 5 - Variation of viscosity with temperature and composition of molten silicates and alumina silicates (13)

A phase equilibria diagram for the system ${\rm CaO-SiO}_2{\rm -Al}_2{\rm O}_3$ is shown in Fig. 6 (14). Components other than ${\rm SiO}_2$ and ${\rm Al}_2{\rm O}_3$ can usually be simplified to a CaO equivalence. This phase diagram can then be used to determine the slag activity from the individual component activities and the melting temperature of a charge. The slag viscosity and surface tension can also be derived from the individual component data and the composition of the melt system (15,16).

Devitrification or crystallization of a glass is avoided at all times in making glass. All glasses tend to vitrify below a temperature specific to that glass, the liquidus temperature. Rate of crystallization, although increasing below the liquidus temperature, is opposed by viscosity increases of the glass (4).

Crystallization can be calculated by the

following equation: (17)

$$R = \frac{\kappa}{n^a} (T_L - T)$$
 Eq 3

where

 η = viscosity of glass

 $T_{I.}$ = liquidus temperature

 κ , a = glass constants

R = rate of crystal growth at temperature T

Observed experimental results agreed closely with predicted data obtained by calculations using this equation.

The phases for a sodium-calcia-silica type glass are shown in Fig. 7 (14). For this particular glass type and working temperature, the area N, O, P, Q, R, represents compositions that have little difficulty with devitrification.

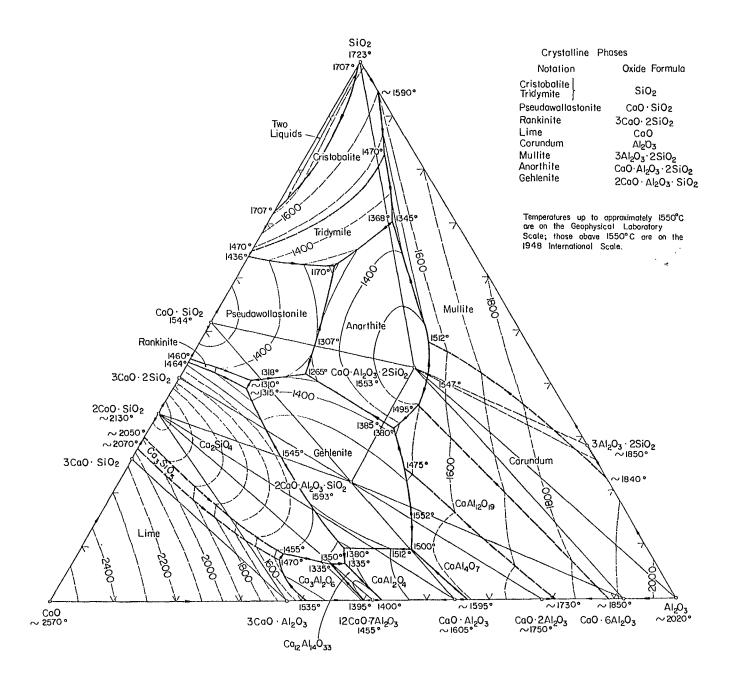


Fig. 6 - Phase equilibrium diagram for the ${\rm CaO-SiO_2-Al_2O_3}$ ternary system (14)

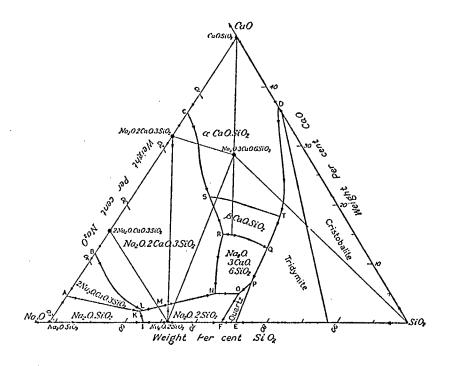


Fig. 7 - Phase equilibrium diagram of the system Na₂0-Ca0-Si0₂ (14)

DENSITY

This property of a glass reflects the structural changes in the melt. Homogeneity of a glass is extrememly important as a processing parameter including that for fiberization. Automatic machinery also requires that the composition of the melt be constant. The density of a glass is one of the simpler and more accurate methods for quality control of the above parameters. Compositional differences of glass have been shown to be more accurately determined by density than by chemical analysis (18). Densities of glass melts of varying composition for the system $CaO-SiO_2-Al_2O_3$ when plotted against temperature, resulted in straight line relationships for each of the compositions shown in Table 5, Fig. 8.

ELECTRICAL AND THERMAL CONDUCTIVITY

Electrical conductivity of slags depends on the number of ions present and their mobility, which in turn is dependent on their size and the slag viscosity. By adding CaO, FeO or MnO to a silica melt, the slag electrical conductivity is transformed from a low to a much higher value.

Conductivity values are related to the extent of ionization and silica is an ionizing solvent. The conduction of iron and manganese silicates, however, are partly electronic and partly ionic giving large conductivity values. Glass can change dramatically in electrical conductivity values from room to high temperatures (5).

Table 5 - Compositional variations of a glass melt

Sample No.	Ca0	SiO ₂	Al ₂ 0 ₃
1	39	42	19
2	35	55	10
3	34	36	30
4	30	45	25
5	30	60	10
6	29	31	40
7	25	55	20
8	25	65	10
9	23	62	15
10	15	65	20

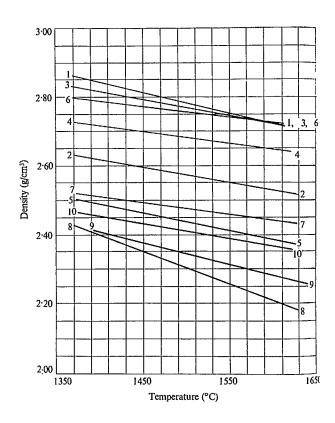


Fig. 8 - Variation of densities with temperature for the CaO-SiO₂-Al₂O₃ system (15)

Generally there is a relationship between the log viscosity and log resistivity; however, a slag with a very high silica or titania content has anomalously low electrical resistance (7).

Thermal conductivity of slags is low and because the major heat transfer in a melt is due to convection, the heat losses are greater than those which could be calculated from thermal conductivity data (3).

SURFACE TENSION

This property is important in the fiberization portion of the mineral wool process. Free movement of the melt from the furnace and from the fiberizing equipment is essential. There are methods for obtaining optimum surface tension for silicate melts. Although they are not scientifically rigorous they do serve as a guide to good practice (4,15). Improvements in homogeneity of a glass and the disappearance of cords are closely

related to surface tension. Even small differences of a few dynes/cm have a marked effect on the number of cords in glass (4).

The viscosity/surface tension value in a glass should be greater than 0.01 otherwise the melt flow is said to be reduced to droplets. Generally, the surface tension decreases with increasing temperature. The variation of composition of a melt with surface tension at 1450°C is shown in Table 6 (18).

The calculated surface tension values of selected oxides are shown in Table 7 (19). Reduction of surface tension in the slag system ${\rm CaO-MgO-SiO_2-Al_2O_3}$ by additions of ${\rm SiO_2}$ is shown in Fig. 9. Small amounts of sulphur in the slag will slightly alter these values.

Table 6 - Surface tension of melts in the systems ${\rm CaO-SiO_2-Al_2O_3} \ \ \, {\rm and} \ \ {\rm CaO-MgO-SiO_2-Al_2O_3} \\ \ \, {\rm at} \ \ \, 1450^{\circ}{\rm C} \ \, (18)$

	Composi	ition (wt	%)	Surface tension
Ca0	MgO	SiO ₂	Al ₂ 0 ₃	(dynes/cm)
30	-	55	15	486
47	-	41	12	572
40	_	40	20	530
48	-	7	45	748
47	4	35	14	591

Table 7 - Calculated surface tension values of metallic oxides (19)

	Calculated	
Oxide	surface	Temperature
	tension	(°C)
	(dynes/cm)	
SiO ₂	285	1400
A1203	640	1400
CaO	614	1400
MgO	512	1400
MnO	653	1400
Na ₂ 0	297	1400
κ ₂ ο	156	1400
B ₂ 0 ₃	96	1400

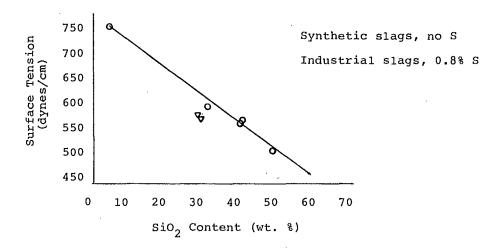


Fig. 9 - Variation of surface tension with ${\rm Si0}_2$ content at 1450°C for several synthetic slags in the CaO-MgO-SiO₂-Al₂O₃ system and two industrial slags containing sulphur (18)

Optimum surface tension can be derived from the results reported by Ustenko (20) and Zihilin (21). They have stated that at 1400°C a slag melt viscosity is 15 poise and the viscosity/surface tension value is 0.01.

RAW MATERIALS

Some of the principal raw materials and oxides supplied for making insulation, including glass and mineral wool, are shown in Table 8.

Mineral wool refers to the insulation product derived from the rocks and minerals in the feed charge. The constitutents for making glass wool are similar to those for glass but glass wool requires less alkali.

The melting points of corresponding oxides and of selected compounds are shown in Table 9.

MINERAL WOOL PROCESSING

Processes for producing mineral wool are basically simple but there can be many complex variations. All processes attempt to accomplish the following objectives:

- 1. melting of the feed charge
- 2. fiberization of the molten stream
- manufacture of various products such as batts,
 with and without vapour barrier, of various

densities and R factors* and granulated, bulk and loose wool.

In Canada and the U.S.A., except for the production of glass wool, the main type of melting furnace used for producing mineral wool is the cupola. For glass fibre insulation, gas or electric melting furnaces are generally used. In Europe, both cupola and electric resistance furnace types are used for mineral insulation manufacture.

CUPOLA TYPE FURNACE

A schematic diagram of a typical cupola furnace for the production of mineral wool is shown in Fig. 10.

The furnace is usually water-jacketed to provide cooling and the internal surface may or may not be lined with refractory material.

The cupola furnace is filled with alternate layers of fuel (coke) and raw materials up to the charging door and the air is turned on at the tuyeres. The material is normally in lump form to allow free passage of air and combustion gases, and also because fine material would be

^{*} R factor - a measure of thermal resistance of the insulation. Its value is obtained from the reciprocal of heat transfer.

Table 8 - Principal raw materials used in glass making (4)

Material	Alternative	Theoretical	Oxides	Fraction
	name	formula	supplied	
Alumina	Calcined alumina	^{A1} 2 ⁰ 3	^{A1} 2 ^O 3	1.000
Alumina hyd.	Hydrated alumina	^{А1} 2 ⁰ 3.3H2 ⁰	^{A1} 2 ⁰ 3	0.654
Aplite (typical			^{A1} 2 ^O 3	0.240
composition)			Na ₂ (K ₂)0	0.100
			SiO ₂	0.600
			CaO	0.060
Feldspar	Microcline (com-	K20. A1203	FOSTA	0.180
	position is of	6SiO ₂	K ₂ (Na ₂)0	0.130
	typical com-		sio,	0.680
	mercial spar)		-	
Nepheline syenite			Al ₂ O ₃	0.250
(typical com-			Na ₂ (K ₂)0	0.150
position)			Sio	0.600
Calumite	Calcium-aluminum	2CaO.MgO.2SiO2	SiO	0.380
	Silicate	2CaO.Al ₂ O ₃ .SiO ₂	Al ₂ O ₃	0.117
		2(CaO.SiO ₂)	CaO	0.400
		. 2.	MgO	0.080
Kyanite (90%		Al ₂ 0 ₃ .SiO ₂	Al ₂ 0 ₃	0.567
concentrate)		2-3-0-2	SiO ₂	0.433
Kaolin	China clay	Al ₂ 0 ₃ ,2Si0 ₂	Al ₂ 0 ₃	0.395
		- , -	- 3	0.465
Cryolite	Kryolith	2H ₂ O Na ₂ AlF ₆	SiO ₂	
Antimony oxide	KI YOLLUI		9h 0	1.000
Barium carbonate		Sb ₂ 0 ₃ BaCO ₂	Sb ₂ 0 ₃ Ba0	0.777
Barium oxide	Baryta	BaO BaO	BaO	
				1.000
Barium sulphate	Barytes	BaSO ₄	Ba0	0.657
Boric acid	Boracic acid	В203.3Н20	_B 20 ³	0.563
Borax		Na ₂ 0.2B ₂ 0 ₃	^B 2 ^O 3	0.365
		10н ₂ 0	Na ₂ 0	0.163
Anhydrous borax	("Pyrobor")	$^{\text{Na}_2\text{O.2B}_2\text{O}_3}$	_B 203	0.692
		A1144 A14	Na ₂ 0	0.308
Lime, burnt	Quick lime	Ca0	CaO	1.000
Lime, hydrated	Calcium hydrate	СаО.Н ₂ О	Ca0	0.757
Limestone	Calcium carb.	CaCO ₃	CaO	0.560
Calcium carb	Whiting	CaCo3	CaO	0.560
Lime, dolomitic	Burnt dolomite	CaO.MgO	CaO	0.582
			MgO	0.418
Dolomite	Raw limestone	CaO.MgO.2CO ₂	CaO	0.304
	(dolomitic)		MgO	0 218
Lime, hydrated,	Finishing lime	CaO.MgO-	CaO	0.423
Dol.		2H ₂ O	MgO	0.304
Bone ash	Calcium	3CaO.2P ₂ O ₃ +	CaO	0.372
	phosphate	CaCO ₃	P203	0.628
Potassium carb.	Calcined carbon-	K ² CO ³	κ ⁵ ο	0.681
	ate of potash	2 3	2	
Glassmaker's	Potassium	K ₂ CO ₃ .1 ¹ 2H ₂ O	к ₂ 0	0.570
potash	carbonate, hyd.	4 3 2	2	
Sand	Glass, sand,	Sio ₂	SiO ₂	1.000
	quartz	2	2	
Soda ash	Sod. carb. coml.	Na ₂ CO ₃	Na ₂ O	0.585
Salt cake	Sodium sulphate	Na ₂ SO ₁₁	-	0.437
Sodium	Sodium		Na ₂ 0	0.431
silicofluoride	fluosilicate	Na ₂ SiF ₄		

Table 9 - Melting points of some compounds of interest in glass making (4)

Name	Formula		Melting	Point
			٥F	°C
Aluminum oxide	Al ₂ 0 ₃		3720	2050
Aluminum silicate (Mullite)	3A1 ₂ 0 ₃ .2Si0 ₂	,	3290	1810
Barium carbonate	Baco ₃	d.	2640	1450
Barium sulphate	BaSO ₄		2875	1580
Boric acid	H ₂ B0 185	d.		365
Boron oxide cryst.	^B 2 ⁰ 3 490	c.		910
Calcia	Ca0	*	4660	2572
Calcium carbonate	CaCO ₃	d. E	Bright red	heat
Calcium fluoride	CaF ₂		2480	1360
Calcium pyro-phosphate	Ca ₂ P ₂ O ₇		2245	1230
Tricalcium phosphate	Ca ₃ (PO ₄) ₂		3040	1670
Calcium silicate	CaSiO ₃		2805	1540
Calcium sulphate	CaSO ₄		2640	1450
Cryolite	Na ₃ AlF ₆		1830	1000
Ferrous silicate	FeSiO4		2200	1200
Magnesium carbonate	MgCO ₃	d. I	d. Low red heat	
Magnesium oxide	Mg0		5070	2800
Magnesium silicate	MgS10 ₃		2835	1555
Magnesium sulphate	MgSO ₄		2165	1185
Phosphorus pentoxide	P ₂ 0 ₅ 563		1045	
Potassium carbonate	к ₂ со ₃		1635	891
Potassium hydroxide	кон		716	380
Potassium nitrate	KNO ₃		635	334
Potassium silicate	K2SiO3		1790	976
Potassium sulphate	κ ₂ so ₄		1970	1076
Silica	SiO ₂		3135	1725
Sodium aluminate	NaAlO ₂		3000	1650
Sodium diborate	$Na_2B_4O_7$		1365	741
Sodium carbonate	Na ₂ CO ₃		1562	851
Sodium chloride	NaCl		1480	804
		Boi]	s 2575	1413
Sodium silicate	Na ₂ SiO ₃		1990	1089
Sodium disilicate	$Na_2Si_2O_5$		1605	874
Sodium sulphate	Na ₂ SO ₄		1624	884

d. = decomposes or loses oxygen

c. = approx. soft temp. of glassy form

Sources: Chemical Rubber Co.'s "Handbook of Chemistry and physics", International Critical Tables.

lost up the stack. In practice for satisfactory operation, it has been recommended that sized material should be used to prevent excessive channeling (22). The ratio of coke to slag or other feed charge is generally between 1 and 3.5:1 (23).

The rate of burning is usually controlled by a slide gate on the air supply from the blast fan to the tuyeres. Under optimum operating conditions, an average 1.22-m (48-in.)-diameter cupola furnace could melt from about 1361 to 1814 kg (1.5 to 2 t, short) of slag per hour with a coke consumption of approximately 450 kg (0.5 t). The water circulating around the cupola cylinder is maintained at as high a temperature as possible to avoid excessive heat loss (23).

REACTIONS WITHIN A CUPOLA FURNACE

Various temperature zones within the cupola reaction or melting chamber are shown schematically in Fig. 10 (21). Figure 11 shows the principal reactions in the various zones of a

cupola furnace and Fig. 12 shows the temperature in the reaction zone.

It is apparent that the loading of a furnace charge is very important. Generally for best operating characteristics, the coke and charge materials are added in alternate layers so that mixing, melting, fluxing and zone temperature are in proper relation to one another.

The various reactions will now be dealt with in slightly more detail. They are identified as oxidation, or reduction using analogy with cupola operation in iron-steel processing.

Air enters the bottom of the furnace where it is preheated by the hot bottom layer of ash and then passes to the coke where it reacts with the carbon to form CO₂:

This major oxidation reaction in the lower coke section is exothermic.

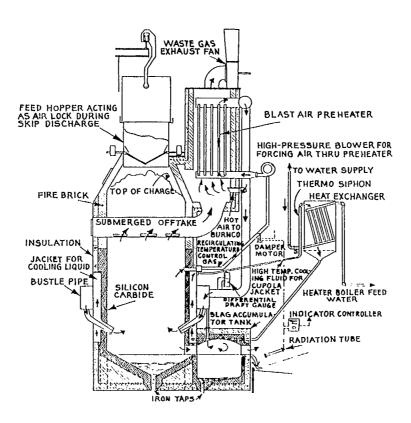


Fig. 10 - Schematic diagram of a cupola furnace used for rock wool fibre production (22)

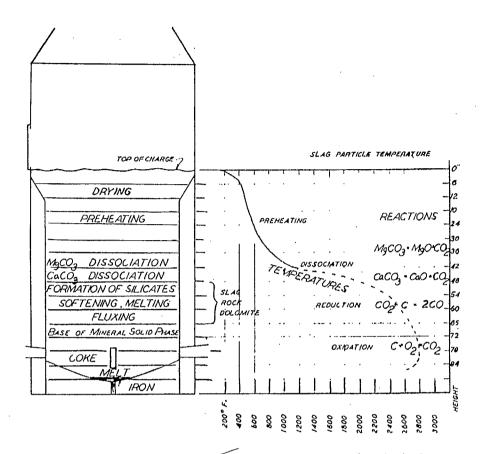


Fig. 11 - Schematic diagram of temperature zones and principal reactions in a cupola furnace (22)

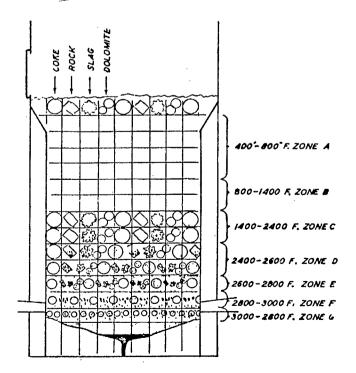


Fig. 12 - Schematic diagram showing the temperatures in the various reaction zones in a cupola furnace (22)

The ${\rm CO}_2$ proceeds upward where it reacts with more carbon to form ${\rm CO}$:

$$CO_2 + C --> 2CO + 5850$$
 Btu

This reduction reaction is endothermic.

The following oxidation-reduction water gas reactions also occur in a furnace where coke or carbon is present and are endothermic:

$$C + H_2O \longrightarrow CO + H_2$$

 $C + 2H_2O \longrightarrow CO_2 + 2H_2$

Furnace efficiency is achieved by optimizing the reactions so that as much heat as possible is available for melting the charge rather than being taken up unnecessarily in the endothermic reactions.

ELECTRIC SMELTING FURNACES

This type of furnace has recently become more popular. Increased efficiency is possible particularly where relatively small quantities of glass are required (4). Other advantages are that homogeneity and composition of the charge are more quickly and easily controlled, and air pollution is reduced because combustion gases are not swept through the furnace. When volatile components such as fluorides are used in a batch, losses are reduced considerably. Start-up time after a shutdown is relatively simple and quick, and heat loss through the sides can readily be accounted for by increasing the power. Generally, this type of furnace requires much smaller space relative to output, and continuous operation is possible (4).

A few disadvantages are: electric furnaces are restricted to locations where cheap power is available and refractory life is shorter than in other furnace types. However, a shorter down-time results in a refractory cost per rebuild that is lower than for other furnaces. Inorganic materials such as silica and limestone used as charge to the furnace do not conduct electrical current well, therefore, start-up requires a carbon source such as coke or carbon cloth. Conductivity is adequate when a melt is formed.

Figure 13 shows schematically the principles of an arc and submerged electrode type furnace operation. Electrodes are generally of two or three configurations and are operated as single or three phase.

Small furnaces of the all-electric type have more recently been adopted. Continuous operation furnaces of as little capacity as 2722 kg (3 t) per day have been making glass, and mini melters are in operation with a continuous output of as low as 9.1 kg (20 lb) per hour (4).

Electric furnace ratings range from 500 to 5000k VA, the larger type using three phase transformers and voltage taps. Electrodes are generally of the graphite type and erode in time due to oxidation.

The efficiency of a furnace can be influenced by the refractory in the lining. The U.S. Bureau of Mines has studied various types for resistance to erosion (24). The refractory also acts as a resistance to abrasion of the sides of the furnace. The buildup on the refractory by the charge can be calculated by the difference in temperature (ΔT) between the furnace and the outside wall. If all the ΔT values are known, e.g., furnace wall, insulation and refractory, then a change of total ΔT (inside to outside of furnace wall) is a function of the additional glass or slag refractory formed on the inside of the furnace.

INDUCTION OR HIGH FREQUENCY ELECTRIC FURNACE

Simply stated, heat is generated and dissipated in this furnace type by induced currents. Thus the susceptor temperature is raised by transformation of electromagnetic energy to heat energy. To be heated, the material must conduct electricity. Many materials though, such as glass, conduct electricity only when heated and in the liquid state, therefore, they must be started by a conducting material.

The heating circuit can be thought of as a transformer, the primary of which carries the high frequency current. The susceptor, the material within the primary, becomes the secondary.

The relationship of current depth and

frequency is shown by the following equation (26):

$$T = \frac{1}{2} \pi \sqrt{\frac{P}{\mu f}}$$

where

T = centimetres

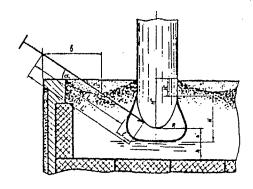
P = resistivity of conductor

μ = magnetic permeability

f = frequency

A characteristic of high frequency heating is the tendency to concentrate energy on the surface of the susceptor.

Graphite is generally used for the susceptor in atmospheric pressure furnaces and high temperatures can and have been reached. Precautions must be taken so that the graphite and refractory material do not react. Other materials used as susceptors are tungsten, molybdenum and tantalum.



(a) Submerged arc

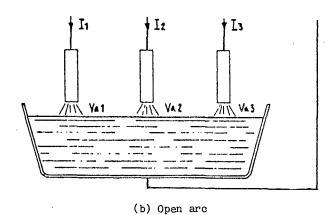


Fig. 13 - Schematic representation of the principles of operation of two types of electric-arc furnaces (24)

A schematic diagram of an induction furnace is shown in Fig. 14 (27).

Because the reaction temperature and the rate of reaction between the graphite and refractory materials are related to their degree of contact, development work had been conducted to decrease the contact (27).

OTHER FURNACE TYPES

Some furnaces used for producing glass fibre or glass marbles are similar to those used for ceramic ware such as the regenerative-fired, recuperative-fired, and direct-fired types. The raw materials are crushed, blended and fed into the furnace automatically. They are usually of large size and temperature control is easily maintained (4).

FIBERIZATION PARAMETERS

The parameters for melting outlined previously in this report are also important for the fiberization process. In addition, the rate of flow, height of drop of the melt stream before fiberization, thickness of the stream, strand or filament, must all be controlled to provide the proper length, thickness, strength, and flexibility of the fibre product.

Despite the importance of this phase of the mineral insulation process, there is a scarcity of published information. Some experimental work has been done but the results at times have been contradictory. Zaitseva suggested that for centrifugal spinning of fibres the rate of flow of the melt should be 1700-2000 Kg/h at temperatures of between 1360° and 1380°C (28). This reduces the shot to a content of 26 to 21 %. Zihilin stated that for the blowing method of fiberizing, the thickness of the molten stream should not exceed 15 to 20 mm in thickness (21). Good quality fibre and diameters up to 3 mm were obtained by Sperantov for melt streams of 15-to 20-mm diameter (29). A uniform flow rate had been determined by others as being necessary to produce good, consistent quality of fibres (30, 31, 32, 33).

It is almost intuitive and one can understand from first principles that temperature variations from any source, thickness of stream, temperature gradients, and pressure of the gas or air, all have an effect on the fiberization process and consequently on the fibre quality.

The mineral insulation industry uses two main methods of fiberization: (a) spinning, and (b) blowing, but details of the methods and the equipment design are considered by the individual companies to be confidential. Both methods produce some shot which is a small amount of bead material at the end of the formed fibre. Various techniques to reduce or eliminate this undesirable shot have been employed with some success.

SPINNING

There are many variations of the spinning technique such as horizontal, vertical and cup types. All, however, are based on the centrifugal principle. This may take the form of flat plates or discs placed horizontally, or of spinning

wheels in single or multiple configuration. The wheel is dish shaped and generally water cooled. The wheel is concave in cross section and faced with special metals to minimize wear and erosion.

The molten stream falls onto or into the spinner which, revolving at high speed, causes the melt droplets formed to be thrown out at high speed, elongating the molten glass particle into a comet-like tail with a shot or bead attached. Much of the shot is removed before the fibre product is formed. Rotational speed of the wheel has not been scientifically calculated but is based on operator skill and experience and could benefit from further research. It is easily understood that proper operation, including speed of the wheels, could have an influence on the physical properties of the fibre, e.g., its length and diameter. It could also influence the amount of shot produced. (Fig. 15,16).

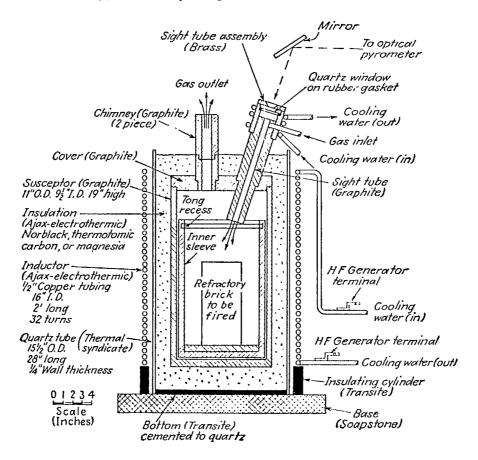


Fig. 14 - Schematic of high frequency furnace for atmospheric pressures (27)



Fig. 15 - Glass fibre manufacture from a melt (34)

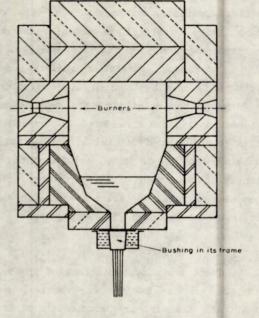


Fig. 16 - Cross section through forehearth showing bushing and glass fibre flow (2)

BLOWING OF THE MOLTEN LIQUID

A molten stream is allowed to fall from the furnace and at the proper distance from it is subjected to a blast or jet of high pressure air at right angles. The liquid forms small droplets which are propelled through the air. The droplets are elongated with a shot adhering to the end of the fibre.

Most commonly, the gas used in blowing is pressurized air but steam was also previously used. Industry has shown that no deleterious effects were experienced in switching from steam to air (35). Other gases have been used experimentally. Pressure of the gas has been said to have an effect on the shot content; it can be seen that it would also have an effect on the fibre length. Conflicting opinions about optimum gas pressure, 345 to 1380 kPa, (50 to 200 psi), indicates the need for further investigation.

Various nozzles are used or have been proposed for blowing mineral wool. Their design should allow a high gas velocity to elongate the particle. Fiberization is more complicated than appears at first glance and also requires further study.

A simplified schematic diagram of a cupola-blowing process for rock wool is shown in Fig. 17.

An innovative furnace is the Pochet which has the bottom in the form of a water cooled bowl (2). The bowl is lined with a thin refractory layer to inhibit radiation. Molybdenum electrodes at the bottom heat the melt in a three-phase mode. Capacity is 7 t of glass a day and the temperature of the melt can reach 2000°C. Power consumed is 1.2 to 2.0 Kw/kg of glass melted. Additional advantages claimed are lower weight and lower cost per tonne of product. A schematic diagram of this furnace is shown in Fig. 18.

ENVIRONMENTAL CONSIDERATIONS

A deteriorating environment has caused public concern in recent years. The regulatory agencies have become firmer in enforcing regulations and require reduced emission from all machines and production processes.

Mineral wool processing plants have been studied in this connection (37, 38, 39, 40).

A flow diagram of the cupola process from

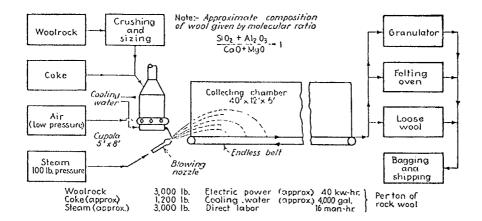
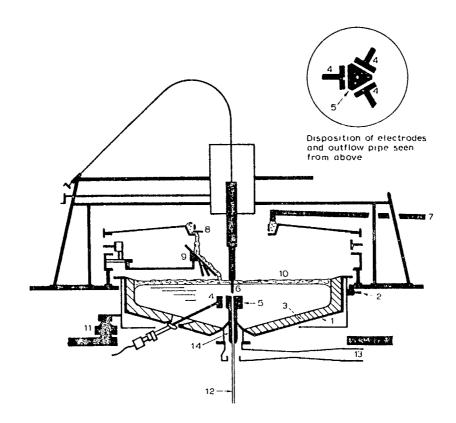


Fig. 17 - A schematic diagram of a rock wool plant (36)



1. Copper bowl surrounded by water-cooled pipes. 2. Coolingwater inlets and outlets. 3. Refractory radiation shield. 4. Electrodes. 5. Glass outflow pipe (see also insert). 6. Glass flow control rod. 7. Batch supply. 8. Rotating batch feeder. 9. Reciprocating batch distributor. 10. Permanent batch blanket. 11. Load cell. 12. Glass stream. 13. Exhaust duct. 14. Hydrogen blanket to shield molybdenum pipe.

Fig. 18 - The Pochet furnace (2)

one of these studies, is shown in Fig. 19. Emissions from the various steps in the process, must be controlled and this requires different techniques. Emissions from the various stages of operation as shown in Table 10 have been analyzed by Danielson of EPA* (37).

EVALUATION OF THERMAL INSULATION

Evaluation tests can be divided into two types:

- 1. testing during processing and
- 2. materials testing.

Tests during processing are not standard for all insulation material but have been adopted from various sources, generally from the ceramic and glass industries. For glass fibre they may include length, diameter, flexibility, thermal conductivity, density and surface tension of the melt, homogeneity of the melt and glass, and devitrification. These tests are obviously related to both processing and material quality.

Much of the second type of material testing relates to meeting standards for specific localities, and usually for buildings and homes. Most applications are covered by the American Society for Testing and Materials and the tests are generally used by Canadian authorities at

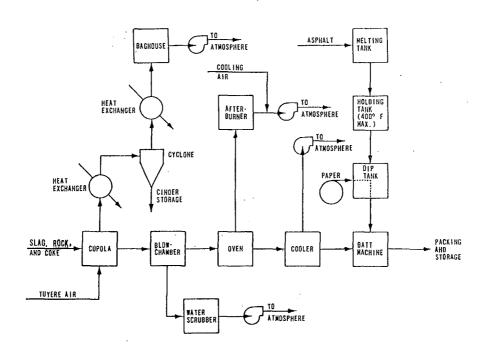


Fig. 19 - Flow diagram of a mineral wool fabrication process showing the stages venting to atmosphere (37)

Table 10 - Types of emissions in mineral insulation plants

Type of plant	Process stage	Type of emission	Remarks
Cupola	Furnace stack	Gases e.g., Co2, SO2, F	major
	Blowchamber	fine fibre, some fumes	major
	Curing oven	fine fibre, organics	medium
	Cooler	similar to cooling oven	small

^{*}EPA - U.S. Environmental Protection Agency.

least as a guide. Standard tests have also been written by the Canadian Standards Association and by the Canadian Government Specifications Board.

These tests include: thermal transmission properties, thermal conductance, specifications for batt insulation, mean specific heat, density of fibrous loose fill insulation and specifications for mineral fibre loose fill insulation. These specifications are included in ASTM C 764-73 which includes the following subject headings:

Classification
Basis of purchase
Materials and manufacture
Physical requirements
Workmanship

Test methods: Thermal resistance
Surface burning characteristics
Moisture absorption
Odour emission
Packaging and marking

For batt insulation the following tests are included: density thermal conductivity

handleability fire resistance standard sizes workmanship

Details of testing are included in the Annual Book of ASTM Standards, in ASTM special publications, in publications by the Canadian Standards Association, Toronto, and the Canadian Government Specification Board (41,42,43,44 respectively).

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