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The Georgia Institute of Technology

The George W. Woodruff School of Mechanical Engineering

ME 4192
GLASS MICROSPHERE LUBRICATION

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I. Problem Statement

In future exploration of the moon, mechanical systems will be subjected to harsh conditions not normally seen on Earth. There will be drastic temperature changes, no atmosphere, and no nearby base for support. The mechanical systems in space need to be self-supporting, durable, and able to adapt to and utilize their environment. Lubrication of bearings is the aspect that will be addressed in this paper. The drastic temperature changes will render normal bearing grease inefficient and impractical. There will also be a high probability of contamination due to the sandy surfaces. The challenge is to develop a lubricant which can withstand the severe conditions and still function efficiently.

II. Constraints and Performance Objectives

- Gravity on the moon is 1/6 that of Earth
- Moon has no atmosphere, so nothing can be burned for heat or power
- Lubricant cannot contaminate the lunar environment
- Lubricant must be able to withstand large temperature gradient
- Lubricant should utilize lunar resources
- Production of lubricant should be lunar based
- Performance of lunar lubricant must be comparable to lubricants on Earth

III. Solution Summary

The harsh lunar environment eliminated the consideration of most lubricants used on Earth. Considering that the majority of the surface of the moon consists of sand, the elements that make up this mixture were analyzed. According to previous space missions, a large portion of the moon's surface is made up of fine grained crystalline rock, about 0.02 to 0.05 mm in size. These fine grained particles can be divide into four groups: lunar rock fragments, glasses, agglutinates (rock particles, crystals or glasses), and fragments of meteorite material (rare). Analysis of the soil obtained from the missions has given chemical compositions of its materials. It is about 53 to 63 percent oxygen, 16 to 22 percent silicon, 10 to 16 percent sulfur, 5 to 9 percent aluminum, and has lesser amounts of magnesium, carbon, and sodium.

To be self-supporting, the lubricant must utilize one or more of the above elements. Considering that the element must be easy to extract and readily manipulated, silicon or glass was the most logical choice. Being a ceramic, glass has a high strength and excellent resistance to temperature. The glass would also not contaminate the environment as it comes directly from it. If sand entered a bearing lubricated with grease, the lubricant would eventually fail and the shaft would bind, causing damage to the system. In a bearing lubricated with a solid glass lubricant, sand would be ground up and have little effect on the system.

The next issue was what shape to form the glass in. Solid glass spheres was the only logical choice. The strength of the glass and its endurance would be optimal in this form. To behave as an effective lubricant, the diameter of the spheres would have to be very small, on the order of hundreds of microns or less. This would allow smaller clearances between the bearing and the shaft, and less material would be needed.

The production of glass microspheres was divided into two parts, production and sorting. Production includes the manufacturing of the microspheres, while sorting entails deciphering the good microspheres from the bad ones. Each process is discussed in detail in the succeeding sections.

IV. Microsphere Production Methods

There are two methods currently being used to produce microspheres. The more popular is the injection of particles into a flame/blower. The glass-forming materials must first be decided upon, as the properties of the produced glass are dependent upon the materials of which it is made. These materials are then melted together to produce a homogeneous glass mixture. Once the mixture has cooled, it is crushed into particles. Because the size of the particles determines the size of the produced microspheres, it is critical that only particles of the correct size are used. To insure this, the particles are sifted and those of the correct size are collected. The next step is to melt the particles by injecting them into a gas-oxygen flame. Figure 1 below, displays the flame sprayer used in the process.

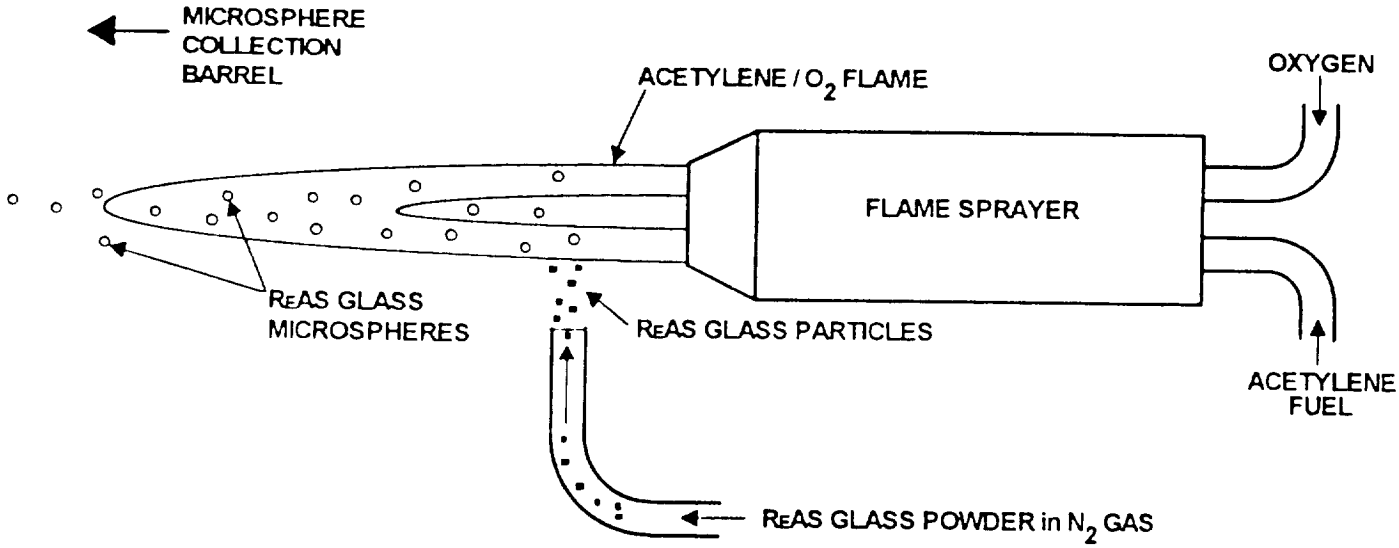


Figure 1

It is possible to inject particles into the flame from any position, but they are usually injected from the center. This is because the center of the flame is the hottest part. The temperature of the flame should be much greater than the melting temperature of the particles so that they are heated uniformly in the small amount of time that they are in the flame. (Non-uniform heating is the main problem with this method of microsphere production.) The particles are blown into and through the flame by an inert gas. They are then collected in barrels as shown in Figure 2 below.

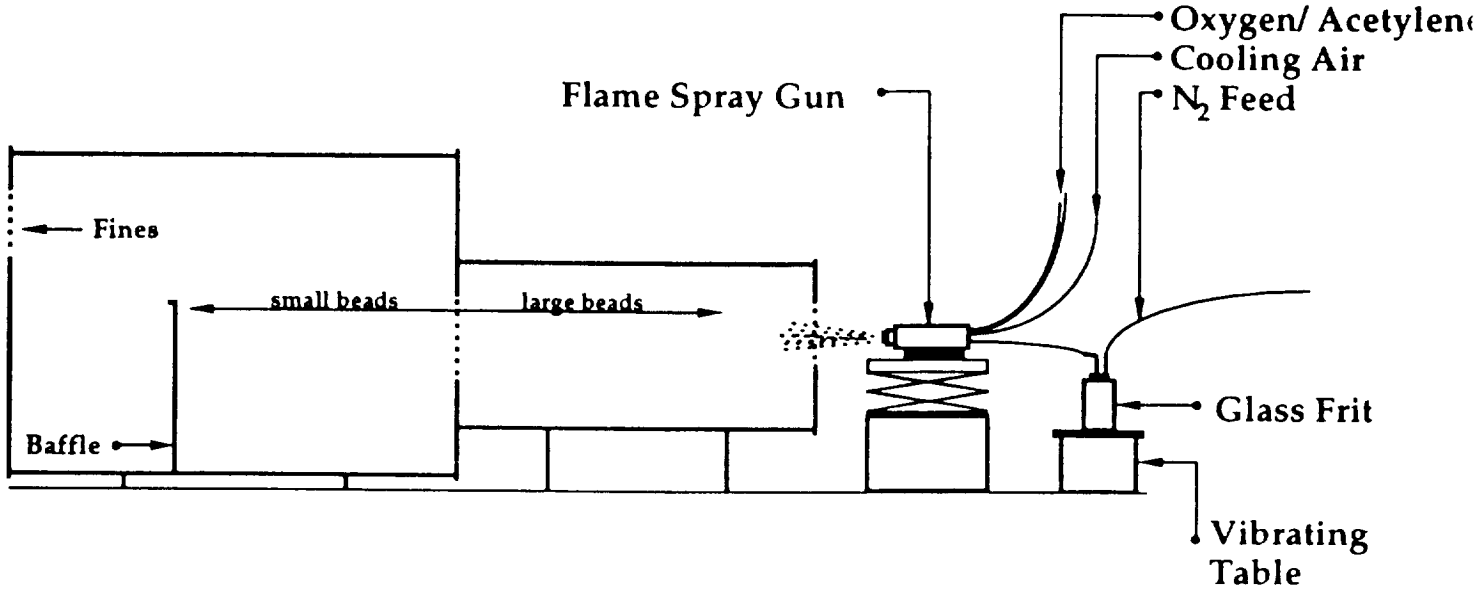


Figure 2

The baffle in the second barrel is used to reduce air flow in the barrel. The larger beads collect in the first barrel due to their higher drag coefficient, while the smaller beads travel into the second barrel. After the barrel has had a chance to cool, the glass beads can be collected by separating the two barrels.

The rate at which the particles are fed into the flame is an important criteria. Each melted particle will produce a perfect sphere due to the surface tension of the liquid glass. Gravity is not a factor due to the extremely small size of the spheres. Errors result only if the particles are fed into the flame too quickly. If this is the case, they will bump into each other while still in a non-solid state. This results in the production of either non-spherical shapes or spheres with a larger than desired volume. Therefore, the particle feed rate must be slow enough to insure that this does not happen. Due to their small volume, the spheres freeze within milliseconds of leaving the flame. The solid spheres continue to be carried by the flow of the inert gas into a collection chamber. No deformations result due to collision with the chambers walls because the spheres have reached a state of perfect elasticity by this time. The spheres should be then screened to weed out any that are not of the desired shape and size. The production of microspheres using this method was videotaped at the University of Missouri - Rolla in the School of Mines and Metallurgy. Actual electron microscope photographs of the glass microspheres produced at the University are included in the Appendix. The second picture is of the glass particles before they are fed into the flame. In this state, the particles are called the "frit".

The second method is the liquid-droplet method. This method requires the glass-forming compounds to first be mixed and in liquid form. This glass solution is then pumped through a jet that forms a mist. The jet is located at the top of a drop tower. As the droplets fall from the tower, they freeze. Once the spheres reach the bottom of the tower, they are collected and screened to insure the proper size and shape was produced. Once again, it is the surface tension of the liquid which causes the shape to be spherical. This method is not as efficient as the above method due to the higher probability of error-forming collisions. Furthermore, the size of the produceable spheres is limited by the tower height; as the microsphere volume increases the required cooling time, and hence the tower height, also increases. It is also necessary to heat the equipment (jet, tubing, pump) so that the solution does not freeze before it is ejected into the atmosphere. This method is generally used to produce hollow microspheres. When this is the case, water is added to the solution before it reaches the jet. At the elevated temperature, water vapor is trapped within the drops as a gel membrane forms on the vapor surface. The water acts as a blowing agent, forming a hollow center in the spheres. Hollow microspheres, however, are not desired for use in a lubrication process because they are not as strong as solid microspheres. Actual electron microscope photographs of microspheres produced by this method were obtained from Materials Engineering Department at Georgia Tech. This picture is located in the Appendix, and displays 200 micron soda-lime glass microspheres.

The particle injection method yields stronger microspheres than does the liquid-droplet method. There is also less chance of producing microspheres with poor spherical tolerance. As the photographs in the Appendix show, the spheres produced with the particle injection method are more uniform and spherical than those produced by the liquid-droplet method. The particle injection method is a more flexible process, allowing the production of many different microsphere sizes. For these reasons the particle injection method was chosen as the desired method for lunar microsphere production.

V. Microsphere Quality Assurance Process

The process by which acceptable microspheres are separated from the spheres that are not within the appropriate tolerances is the focus of this section. Spheres could be too large, too small, or not of the correct eccentricity to be useful as lubricants for certain processes. With this in mind a micron scale sorting apparatus in order to obtain a variety of bead sizes was designed.

After investigating various sorting methods for ball bearings, the macroscopic counterpart to the microscopic lubrication, a system for the tolerancing of a large number of spheres in a short period of time was devised. The concept of being able to separate the spheres into various classes by size and accuracy was established. Since differing needs may be served by a variety of sizes of microspheres, a system that is capable of differentiating between an indeterminate number of sizes of beads seemed necessary. In this manner a homogeneous grouping of microspheres, all of whom deviate from the desired size by less than a known amount, could be obtained.

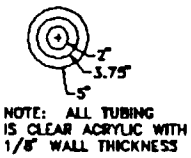
Several methods for this separation were devised and studied. Six different mechanisms of separation were designed and are listed in the Research Appendix. These designs were then weighted and judged on a variety of weighted design criteria. These criteria included: mass of apparatus, volume flowrate of microspheres, and system accuracy. This ranking process may also be found in the Research Appendix. The highest scoring design was the design which implemented concentric cylinders. This design utilized high amplitude (on the order of the diameter of the largest cylinder) vibration at a reasonably high frequency (approximately 1500 cycles per second) in order to force the microspheres through a series of holes bored through the cylinders. Much in the manner of a sieve, the microspheres would pass through the series of sieves until they are collected and all microspheres of a certain size are removed as a group.

This design allowed for easy operation in free fall, microgravity, or Earth standard gravity. Since the system is closed, there should be no losses, and accuracy is linearly dependent upon the length to diameter ratio of the cylinders. The design also does not require an atmosphere, although it is operational in one. The relatively low mass and simplicity of the system combined with the possibility of a high volume flowrate made it the ideal choice for microsphere separation.

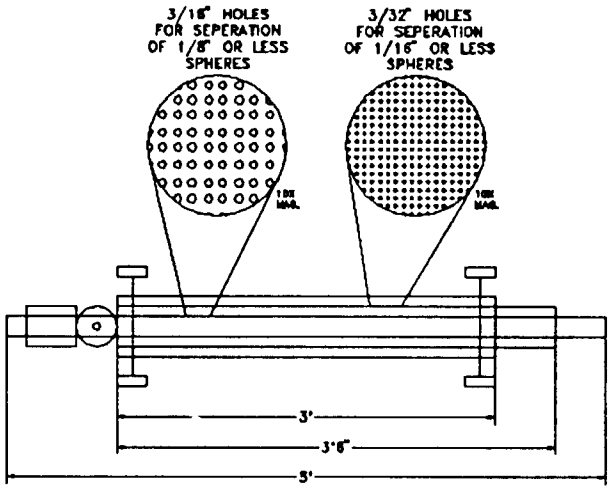
For a demonstration of the separation procedure, acrylic tubing was used. This material made up the concentric cylinders. For ease of construction, three cylinders were fabricated, although many cylinders may be added to allow for varying accuracy. Holes were drilled only on the lower half of the tubing to allow for better viewing of the demonstration model in action, and for the fact that local gravity was far too large to make full amplitude oscillations feasible.

The design was implemented by the use of an orbital sander attached to three acrylic concentric cylinders, as can be seen in Figure 3. The two inner cylinders were drilled with holes of a diameter equal to half the difference of the desired size of microspheres. After assembling the apparatus, the design was tested whereupon 2.5 degrees was determined to be the optimal down angle of the tubing. The separation process was highly accurate considering the exceedingly foreshortened length of the tubing.

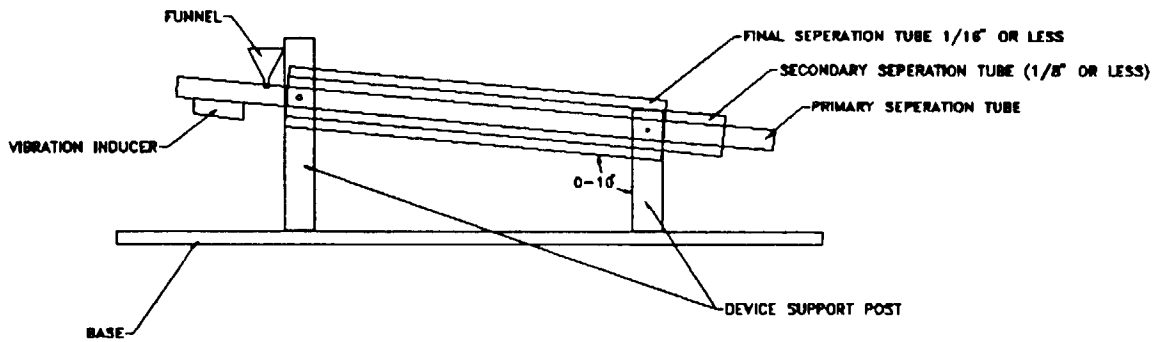
CONCENTRIC CYLINDER SPHERICAL SEPERATOR



NOTE: ALL TUBING IS CLEAR ACRYLIC WITH 1/8" WALL THICKNESS



TOP VIEW



SIDE VIEW

FIGURE 3

VI. Glass Microsphere Applications

An extensive amount of research is currently being done on glass microspheres. Much of this research deals with medical applications, such as treatment for liver cancer and arthritis. The example in this lab uses glass microspheres as a lubricant for a journal bearing. This is the kind of usage the lubricant will see in lunar exploration.

The purpose of this application was to show the effectiveness of glass microspheres as a lubricant, and to compare the glass microsphere lubricant to traditional lithium grease lubricant and a petroleum based lubricant. The setup is shown in Figure 4 below. A 1/20 hp motor drives a one inch, case hardened, steel shaft. A pillow block is utilized to stabilize the shaft. A journal bearing, fabricated in the ME Machine Shop, serves as the test site for the lubricants (see Figure 5). Two different journal bearings were fabricated due to the variance in the size of the microspheres. Since some variation in the diameter of the microspheres may be present the clearance of the journal bearing was set at two times the average diameter of the microspheres. The top of the journal bearing has a 3/16" hole through which the lubricant can be fed to the system. A one inch diameter weight was welded to the bottom of the journal bearing. When the motor is activated, the angular displacement of this weight will be used in calculations described below to determine the friction of the lubricant and the torque of the motor.

Application Setup

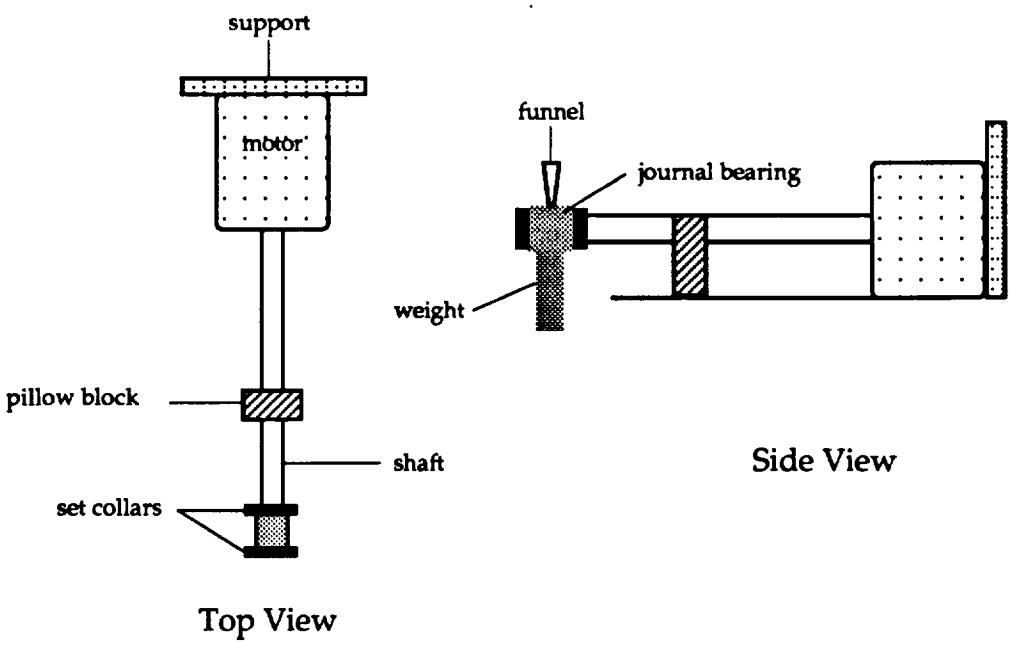
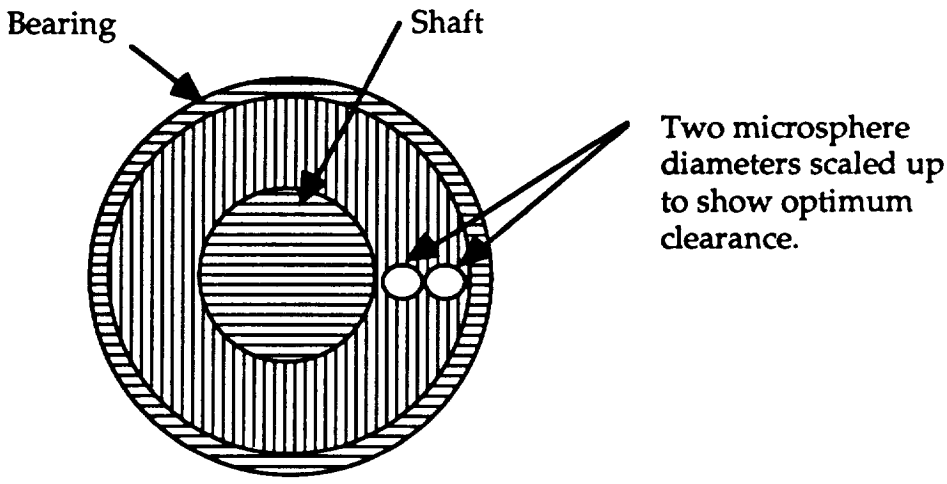


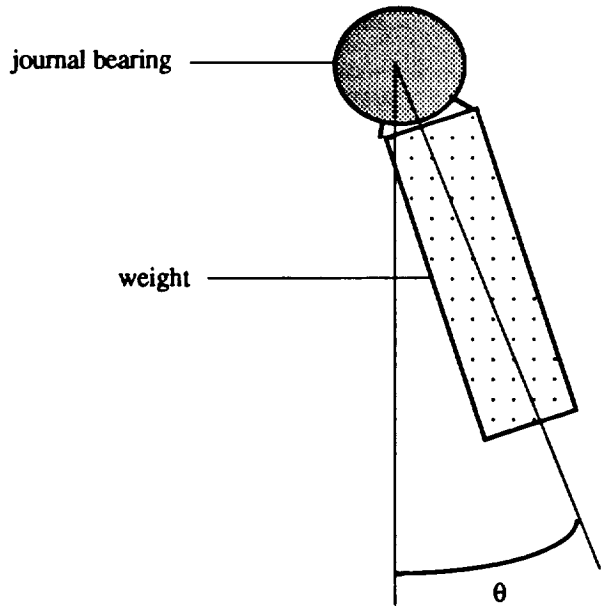
Figure 4



Journal Bearing
Figure 5

The testing was done using the same setup through two iterations to justify the data. Bearing #1 had a gap clearance of 0.012 in. Converting this to metric yields approximately 305 μm. Dividing this number by four microspheres allows for about 80μm diameters. Bearing #2 was designed to fit the 45μm spheres. Unfortunately the tolerances of this bearing were not as close to those in the first. The gap clearance would allow approximately three microspheres of this diameter side by side.

The free body diagram in Figure 6 shows the angular displacement experienced by the weight during motor operation.



Free Body Diagram
Figure 6

Results from the angular displacement testing are given below. These angles will be used to determine the frictional coefficients.

Bearing #	No Lubricant	Petroleum Based	Lithium Grease	Microspheres
1	30°	11°	15°	8° 80μm Soda-Lime
2	30°	10°	20°	19° 45μm YAS-4

Data obtained shows that using the first bearing, the angular displacement using the 80μm spheres was actually lower than either of the two other lubricants as we anticipated. On the other hand, the second experiment did not give the same results. The 45μm spheres only showed a lubricity about the same as the lithium grease.

There are two possible conclusions to these findings. One regards the hardness of the microspheres tested verses the hardness of the surrounding bearing and shaft. The soda-lime glass is a much softer material than the alumina-silica (YAS-4) and was much closer to the hardness of the steel. Because of this there was very little wear or friction between the bodies and excellent data was recorded. But, in the second application using the alumina-silica microspheres, very noticeable wear was present on both the shaft and the bearing after only about twenty seconds of

running. This was either a result of the difference in hardness of the materials or in the slightly inaccurate tolerances in the gap clearance of the second bearing.

Due to the nonlinearity of the pressure distribution across the surface of the shaft as it rotates, it is not feasible to attempt to make an accurate assessment of the coefficient of friction of the bearing being studied. However, since the differing effectiveness of the various types of lubrication studied effect the coefficient of friction of the bearing as a sine function, the relative effectiveness of the types of lubrication can be stated with accuracy. This can be done by assuming that the displacement of the weight varies linearly with the coefficient of friction. A statement of the coefficient friction for lithium grease was found in *The Handbook of Tribology, Materials, Coatings and Surface Treatment* to be 0.03. By associating this coefficient with an angular displacement of 20 degrees, the coefficient of friction for any other lubricant can be calculated, based on the angle of displacement. The following table, Figure 7, summarizes the results.

θ	μ
5°	0.008
6°	0.009
7° - 9°	0.01
10° - 16°	0.02
17° - 23°	0.03
24° - 30°	0.04

Friction Based on Angular Displacement
Figure 7

For example, based on these calculations, the coefficient of friction for the 80 μm glass microspheres, displaced at an angle of 8°, is 0.01.

Ideally, for a bearing application the race and microspheres should have the same hardness so neither wears on the other. After case hardening the shafts and bearing, it was estimated that they had a hardness of 5 on Moh's scale. The glass microspheres tested had a hardness ranging from 5.8 to 6.1 as read on Moh's scale. Moh's scale with the hardness of the materials used in the application experiment can be seen in the Appendix. In future applications, a closer tolerance on the hardness variation should be achieved. Since any substance can be formed into a glass, the properties of the microspheres are very flexible and can be chosen to meet the requirements of different applications.

VII. Theoretical Performance of Glass Microspheres

To calculate the contact stresses between the microspheres and between the bearing and microspheres the following formula for Hertzian stresses was analyzed:

$$a = \left[\frac{3F}{8} \left(\frac{\left(\frac{1-\upsilon_1^2}{E_1}\right) + \left(\frac{1-\upsilon_2^2}{E_2}\right)}{\frac{1}{d_1} + \frac{1}{d_2}} \right) \right]^{\frac{1}{3}}$$

where: F = force
d_1, d_2 = diameter of two spheres
a = area of contact
E_1, E_2 = Modulus of Elasticity
υ_1, υ_2 = Poisson's ratio

The theoretical stresses were found with the following equation:

$$\sigma_x = \sigma_y = \frac{-3F}{2\pi a^2} \left[\left(1 - \frac{z}{a} \tan^{-1} \frac{1}{\frac{z}{a}} \right) (1+\mu) - \frac{1}{2\left(1+\frac{z^2}{a^2}\right)} \right]$$

where: μ = coefficient of friction between surfaces
z = distance from point of contact

The compressive strength of commercial glass spheres ranges from 2.3 GPa to 2.8 GPa. The coefficient of friction between glass is estimated at 0.4. The Poisson's ratio for commercial glass ranges from 0.17 to 0.275 depending upon the composition.

The spreadsheet on the following page, Figure 8, shows the theoretical expected stresses and safety factors for different glasses and bearing materials for the application described above. Depending on the application, the correct combination could be determined using these results. It is important to note that the compressive strengths of both the silica glass and the borosilicate exceed the calculated experimental stresses. This yields safety factors between 88 and 140, far surpassing normal standards.

AREA OF CONTACT FOR BEARING AND MICROSPHERE

	FORCE	POISSONS RATIO 1	MOD. OF ELASTICITY 1	DIAMETER 1		POISSONS RATIO 2	MOD. OF ELASTICITY 2	DIAMETER 2	AREA OF CONTACT	MAX STRESS X, Y	MAX STRESS Z	COMPRESSIVE STRENGTH OF GLASS	FACTOR OF SAFETY
	Newtons		Pascals	meters			Pascals	meters	meter^2	Pascals	Pascals	Pascals	
glass 96% Silica	5.0E+00	1.7E-01	6.6E+06	1.0E-04	stainless steel	3.1E-01	1.9E+11	-2.6E-02	3.0E-04	-2.1E+07	-2.6E+07	2.3E+09	8.8E+01
	5.0E+00	1.7E-01	6.6E+06	1.0E-04	brass	3.2E-01	1.1E+11	-2.6E-02	3.0E-04	-2.1E+07	-2.6E+07	2.3E+09	8.8E+01
	5.0E+00	1.7E-01	6.6E+06	1.0E-04	iron	2.9E-01	2.1E+11	-2.6E-02	3.0E-04	-2.1E+07	-2.6E+07	2.3E+09	8.8E+01
	5.0E+00	1.7E-01	6.6E+06	1.0E-04	aluminum	3.3E-01	6.9E+10	-2.6E-02	3.0E-04	-2.1E+07	-2.6E+07	2.3E+09	8.8E+01
	5.0E+00	1.7E-01	6.6E+06	1.0E-04	bronze	3.5E-01	1.1E+11	-2.6E-02	3.0E-04	-2.1E+07	-2.6E+07	2.3E+09	8.8E+01
	5.0E+00	1.7E-01	6.6E+06	1.0E-04	diamond	2.0E-01	9.4E+11	-2.6E-02	3.0E-04	-2.1E+07	-2.6E+07	2.3E+09	8.8E+01
	5.0E+00	1.7E-01	6.6E+06	1.0E-04	cast steel	2.7E-01	2.0E+11	-2.6E-02	3.0E-04	-2.1E+07	-2.6E+07	2.3E+09	8.8E+01
	5.0E+00	1.7E-01	6.6E+06	1.0E-04	cold rolled steel	2.9E-01	2.0E+11	-2.6E-02	3.0E-04	-2.1E+07	-2.6E+07	2.3E+09	8.8E+01
glass Borosilicate	5.0E+00	2.0E-01	6.6E+06	1.0E-04	stainless steel	3.1E-01	1.9E+11	-2.6E-02	3.0E-04	-2.1E+07	-2.6E+07	2.8E+09	1.1E+02
	5.0E+00	2.0E-01	6.6E+06	1.0E-04	brass	3.2E-01	1.1E+11	-2.6E-02	3.0E-04	-2.1E+07	-2.6E+07	2.8E+09	1.1E+02
	5.0E+00	2.0E-01	6.6E+06	1.0E-04	iron	2.9E-01	2.1E+11	-2.6E-02	3.0E-04	-2.1E+07	-2.6E+07	2.8E+09	1.1E+02
	5.0E+00	2.0E-01	6.6E+06	1.0E-04	aluminum	3.3E-01	6.9E+10	-2.6E-02	3.0E-04	-2.1E+07	-2.6E+07	2.8E+09	1.1E+02
	5.0E+00	2.0E-01	6.6E+06	1.0E-04	bronze	3.5E-01	1.1E+11	-2.6E-02	3.0E-04	-2.1E+07	-2.6E+07	2.8E+09	1.1E+02
	5.0E+00	2.0E-01	6.6E+06	1.0E-04	diamond	2.0E-01	9.4E+11	-2.6E-02	3.0E-04	-2.1E+07	-2.6E+07	2.8E+09	1.1E+02
	5.0E+00	2.0E-01	6.6E+06	1.0E-04	cast steel	2.7E-01	2.0E+11	-2.6E-02	3.0E-04	-2.1E+07	-2.6E+07	2.8E+09	1.1E+02
	5.0E+00	2.0E-01	6.6E+06	1.0E-04	cold rolled steel	2.9E-01	2.0E+11	-2.6E-02	3.0E-04	-2.1E+07	-2.6E+07	2.8E+09	1.1E+02

FIGURE 8

AREA OF CONTACT FOR MICROSPHERES

	FORCE	POISSONS RATIO 1	MOD. OF ELASTICITY 1	DIAMETER 1	POISSONS RATIO 2	MOD. OF ELASTICITY 2	DIAMETER 2					
	Newtons		Pascals	meters		Pascals	meters					
96% Silica	5.0E+00	1.7E-01	6.6E+06	3.0E-05	1.7E-01	6.6E+06	3.0E-05	2.0E-04	-5.3E+07	-5.8E+07	2.3E+09	3.9E+01
	5.0E+00	1.7E-01	6.6E+06	8.0E-05	1.7E-01	6.6E+06	8.0E-05	2.8E-04	-2.7E+07	-3.0E+07	2.3E+09	7.6E+01
	5.0E+00	1.7E-01	6.6E+06	2.0E-04	1.7E-01	6.6E+06	2.0E-04	3.8E-04	-1.5E+07	-1.7E+07	2.3E+09	1.4E+02
Borosilicate	5.0E+00	2.0E-01	6.6E+06	3.0E-05	2.0E-01	6.6E+06	3.0E-05	2.0E-04	-5.3E+07	-5.8E+07	2.8E+09	4.8E+01
	5.0E+00	2.0E-01	6.6E+06	8.0E-05	2.0E-01	6.6E+06	8.0E-05	2.8E-04	-2.7E+07	-3.0E+07	2.8E+09	9.2E+01
	5.0E+00	2.0E-01	6.6E+06	2.0E-04	2.0E-01	6.6E+06	2.0E-04	3.8E-04	-1.5E+07	-1.7E+07	2.8E+09	1.7E+02

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VIII. Conclusions and Recommendations

Through research and design, it has been proven that glass microspheres are a viable option as an alternative lubricant for bearings. With its resilience to temperature and high strength, glass microspheres would be ideal for lunar use. The effectiveness of the spheres was proven in the results of the application.

With current technology, the glass microspheres can be manufactured to the specifications required for lubrication. That fact, combined with the numerous existing sorting methods, including the concentric tube sorter described within this report, proves the ease of selecting the appropriate size spheres for particular applications. Based on initial results with the application, glass microspheres are comparable in performance to typical lubricants. The coefficient of friction for the glass microspheres, depending on size and composition, ranged from 0.01 to 0.03. The coefficient of friction for the grease tested was 0.03, while the coefficient of friction for the petroleum based lubricant was 0.02.

Future recommendations for the production of glass microspheres are to refine the solid particle feeding method so that the feed rate can be more carefully controlled. This would produce a greater number of defect free microspheres. Methods of collecting the glass from the moon's surface should also be investigated. There is a large amount of "free" glass particles on the moon's surface that would not have to be extracted from other elements. This would make the collection process relatively easy. Addressing the separation and quality control of the glass microspheres, future work should concern the length of the concentric tubes, the angle on their incline, and the severity of the vibration. The microspheres will behave differently in one-sixth the gravity that this process was developed in.

After the conduction of the testing, several ways to improve the performance of the glass microspheres became apparent. First of all, an increase in the number of feed and release holes should be added to the bearing. In the event that one of the microspheres fractures, the bearing should be designed so that the fractured pieces fall out. This could be done by adding slots to the bearing. However, with the added slots the feed rate will need to be increased. This can be accomplished by increasing the number of feed holes in the bearing. Further research and testing should be completed in order to optimize the bearing and glass microsphere characteristics. A suggestion would be to investigate ceramic or diamond coated bearings. Further research into optimal glass composition also should be done. The size of the microspheres, hardness of materials, and bearing design should be analyzed. In addition, the endurance of the microspheres should be tested.

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X. Acknowledgments

This project has been a pain-staking quest for knowledge about an idea that was just crazy enough to work. We cannot leave out all of the people who were instrumental in our findings. The members of our group would like to thank the following people:

Mr. James W. Brazell - Instructor and project coordinator
Georgia Institute of Technology

Dr. Joe K. Cochran - Director Academic, Materials Science and Engineering
Georgia Institute of Technology

Dr. Delbert E. Day - Curators' Distinguished Professor of Ceramic Engineering and Senior Investigator, Materials Research Center
University of Missouri-Rolla

Mr. Carlos Gonzalez - Graduate Student, Ceramic Engineering
Georgia Institute of Technology

Mr. John Graham - Machine Shop Foreman, Mechanical Engineering
Georgia Institute of Technology

Ms. Amy McFutgre - Graduate Student, Ceramic Engineering
University of Missouri-Rolla

Mr. Ralph Napalitano - Graduate Student, Materials Engineering
Georgia Institute of Technology

Dr. Thomas H. B. Sanders - Professor, Materials Science and Engineering
Georgia Institute of Technology

Mr. Rob Schoenborn - Graduate Student, Textile Engineering
Georgia Institute of Technology

Mr. Sterling Skinner - Assistant to the Director, Mechanical Engineering
Georgia Institute of Technology

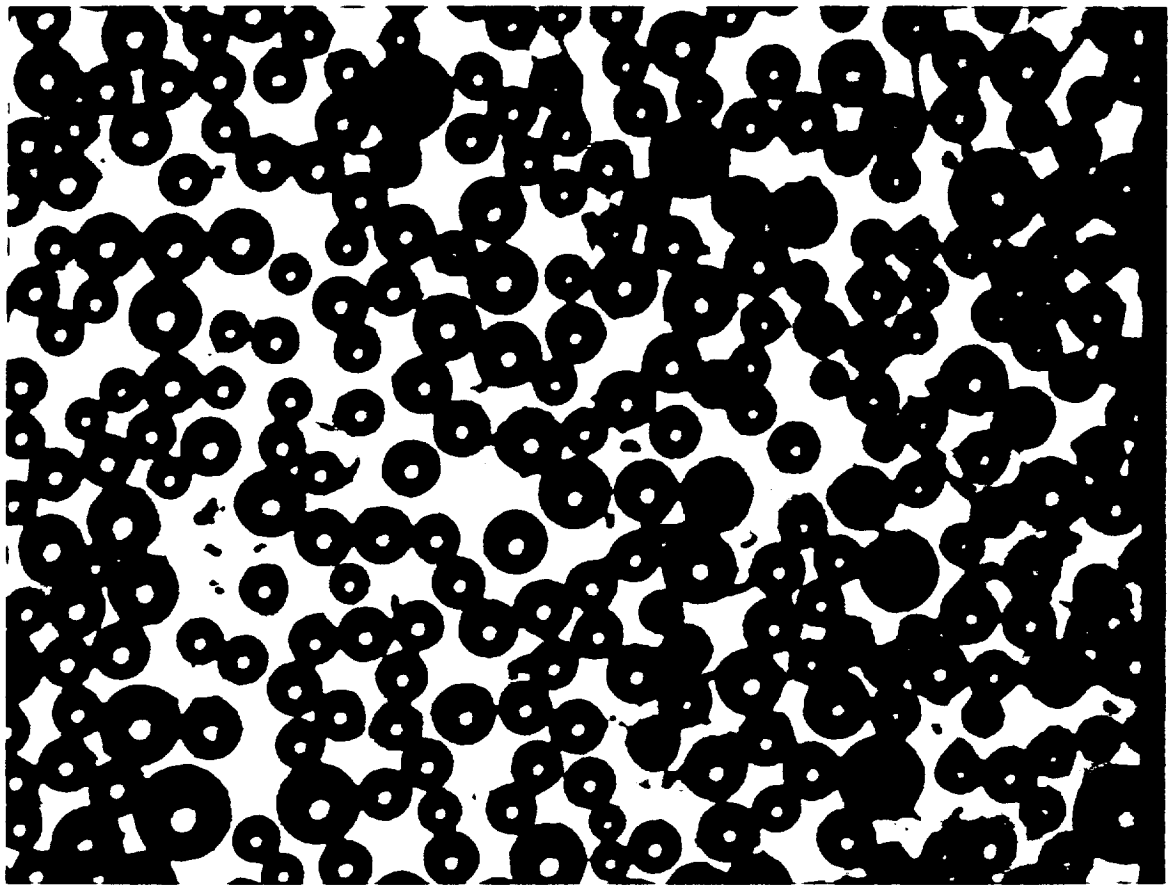
Mr. Jim White - Graduate Student, Ceramic Engineering
University of Missouri-Rolla

XI. Appendices

A. Alumina-silica based microspheres produced by particle injection method
B. 45 micron screened frit for particle injection method
C. 200 micron soda-lime microspheres produced by liquid drop method
D. Moh's table of hardness with glass microspheres and test fixture hardnesses included

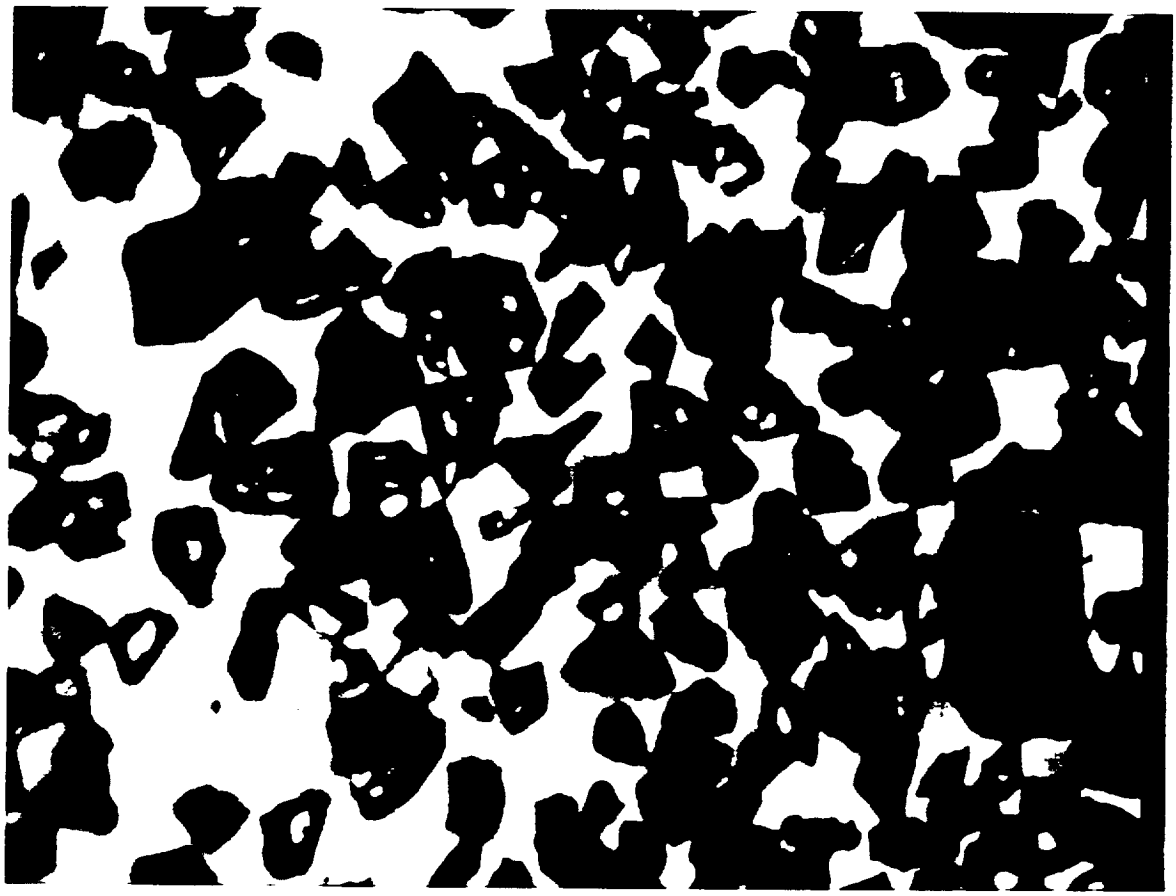
Appendix A

Alumina-silica based microspheres produced by particle injection method



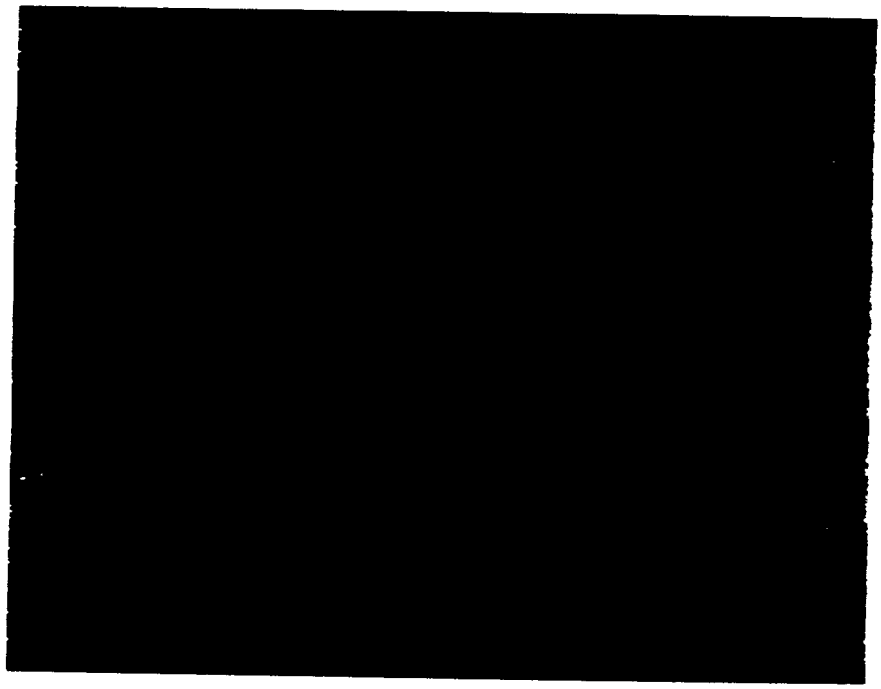
Appendix B

45 micron screened frit for particle injection method



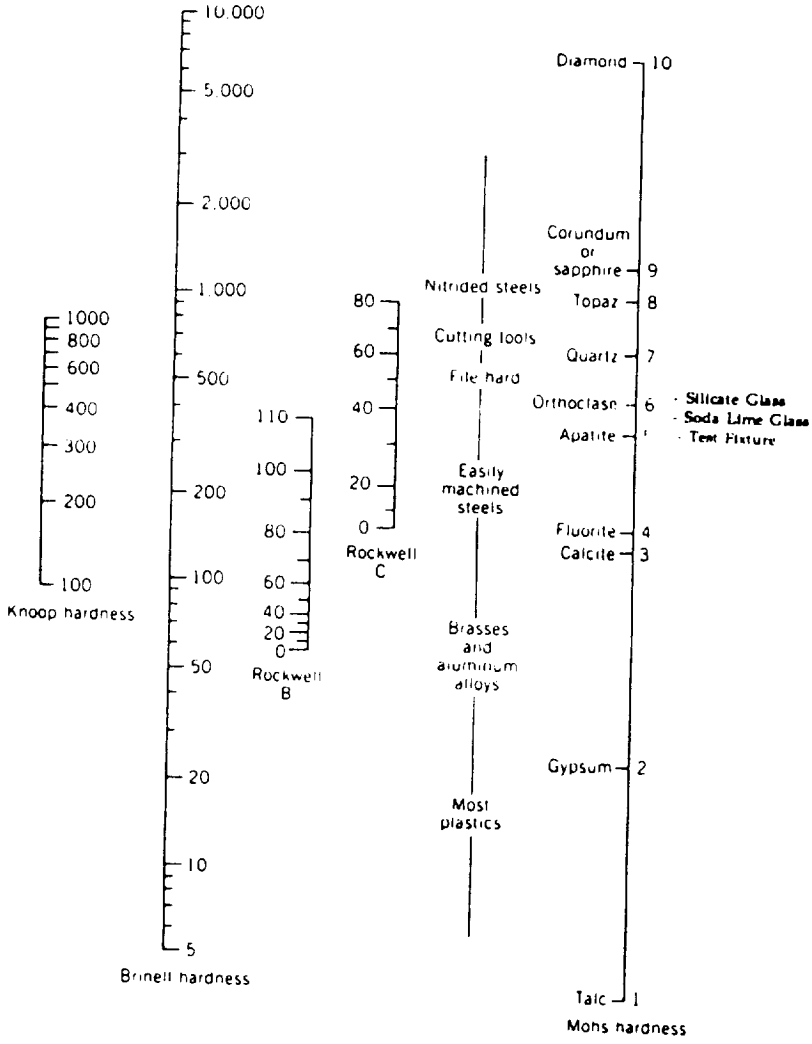
Appendix C

200 micron soda-lime microspheres produced by liquid drop method



Appendix D

Moh's table of hardness with glass microspheres and test fixture hardnesses included



Research Appendix

Future Space Resources
Interoffice Memo

TO

To: Mr. James W. Brazell
From: Rusty Goode (Group II)
Re: Visit to Univ. of Missouri-Rolla
Date: 2/23/94

We have been in touch with Dr. Day at the Univ. of Missouri-Rolla. He is scheduling us to talk to one of his T.A.'s later this evening. He does not see a problem with us coming to the university. Cindy and Michelle will be calling the T.A. and trying to schedule a time to meet. We are shooting for Mon. Feb. 28 and Tue. Mar. 1 if it fits his schedule. I have a list of goals to obtain and questions to ask and would like to review them further with you. I also have the release forms necessary for us to go. I will have a cost breakdown for you provided we receive the O.K. to go.

$1,000 BUDGET.

GOOD CHOICE OF TIMING.

GO FOR IT.

J.W.B.

Dr. Day - University of Missouri-Rolla

- Witness and film the production of glass microspheres.
    - 1 μm, 10 μm, and 50 μm microspheres
        - test for changes in friction with different sized microspheres
        - compressive strengths
        - tolerances in sphericity
        - temperature affected?

- Collect any reports or data on the production of microspheres.

- Collect any reports or data about applications of microspheres.

- Purchase microspheres if we can't obtain them for free.

- Get Dr. Day's business card to send him a copy of our report.

SENIOR INVESTIGATORS
GRADUATE CENTER FOR MATERIALS RESEARCH
UNIVERSITY OF MISSOURI-ROLLA
Research Interests

Harlan U. Anderson, Curators' Professor of Ceramic Engineering.
Nonstoichiometric oxides; sintering of oxides and metals; electronic ceramics; high purity oxides from organo-metallics; chemical corrosion of ceramics.

Richard A. Behr, Associate Professor of Civil Engineering.
Structural performance and durability of architectural glazing systems and building envelopes under environmental, wind, and earthquake effects.

Frank D. Blum, Professor of Chemistry.
Polymer-solvent/polymer-surface interactions; polymer characterization; colloid chemistry; microemulsions, liquid crystals, micelles and vesicles; NMR spectroscopy and diffusion.

Delbert E. Day, Curators' Professor of Ceramic Engineering.
Structure, chemical durability, and mass transport in vitreous solids (glasses); oxynitride glasses; ceramic biomaterials; containerless processing of glass in space; composites.

Lokesh R. Dharani, Professor of Engineering Mechanics and Aerospace Engineering.
Failure analysis and micromechanics of high temperature and structural composites; environmental effects on composites; mechanics of ceramic/metal/ceramic joints.

Gary J. Ehrhardt, (Adjunct Senior Investigator) Senior Research Scientist, Research Reactor, University of Missouri-Columbia.
Application of radioisotopes to medical, chemical, environmental, and biological problems.

Wayne Huebner, Associate Professor of Ceramic Engineering.
Dielectric and piezoelectric properties of normal and relaxor ferroelectrics; defect chemistry of high temp., conducting perovskites; synthesis/characterization of ultrasound transducers.

William J. James, Professor Emeritus of Chemistry.
Plasma deposited thin films; electrochemistry and kinetics; single crystal structures; x-ray and neutron diffraction; electrical and magnetic properties of solids.

Kenneth F. Kelton, (Adjunct Senior Investigator) Associate Professor of Physics, Washington University, St. Louis, Missouri.
Nucleation and crystallization of metallic and non-metallic glasses.

Nicholas C. Morosoff, Professor of Chemical Engineering.
Thin film technology; surface modification; plasma polymerization; transport properties of polymers; chemically reactive transition metal containing plasma polymers.

RESEARCH INVESTIGATORS
GRADUATE CENTER FOR MATERIALS RESEARCH
UNIVERSITY OF MISSOURI-ROLLA
Research Interests

Daniel W. Armstrong, Curators' Professor of Chemistry.

Separation and resolution of enantiomers by chromatographic and membrane-based processes; theory and use of secondary equilibria in field flow fractionation; gradient liquid separation of polymers; membrane separation of proteins.

Jack L. Boone, Professor of Electrical Engineering.

Physical electronics; solid state devices; wave interactions in plasmas; solar energy conversion; growth and characterization of compound semiconductors; modeling and characterization of photovoltaic devices; fabrication and characterization of barium titanate thermistors.

Roger F. Brown, Associate Professor of Life Sciences.

Therapeutic applications of synthetic biomaterials; cellular effects of non-ionizing radiation; mammalian DNA repair replication; cell adhesion factors; metabolism of cells in culture.

Douglas R. Carroll, Assistant Professor of Basic Engineering.

Mechanical properties of high temperature composite materials; processing of powder matrix composites; sintering of thin polycrystalline films; surface and grain boundary energy of polycrystalline materials.

K. Chandrashekhara, Associate Professor of Mechanical and Aerospace Engineering and Engineering Mechanics.

Finite element analysis of layered anisotropic composite plates and shells; fabrication and experimental characterization of composite materials; plasticity; variational methods and experimental mechanics.

Harvest L. Collier, Associate Professor of Chemistry.

Synthesis and characterization of inorganic and heterocycle-containing polymers; investigation of thermal and conductive properties of inorganic and polymer systems; preparation and characterization of metallomacrocycles; kinetics and mechanism of macrocycle reactivity.

Jay M. Gregg, Associate Professor of Geology & Geophysics.

Study of the mineral and rock forms of dolomite; theoretical investigations on the crystallography & solid state chemistry of natural & synthetic dolomites.

Edward B. Hale, Professor and Chairman of Physics.

Studies of ion-induced electron emission in metals using UHV high energy accelerator and surface characterization instrumentation in MRC.

Don M. Sparlin, Professor of Physics.

Electronic and magnetic properties of materials; experimental measurements of transport and magnetic properties of metals, semiconductors, and insulators; experienced with computer based instrumentation.

Michael R. Van De Mark, Associate Professor of Chemistry.

Polymer synthesis and characterization; polymer/solvent interaction; ionomeric gels; modified electrodes via polymer adsorption; corrosion inhibition through ligating polymers; organic oxidative electrochemistry.

David C. Van Aken, Associate Professor of Metallurgical Engineering.

Emphasis of the research is directed towards phase transformations. A combination of internal friction and analytical electron microscopy techniques are used in these studies.

Graduate Center for Materials Research

CERAMICS
O
METALS
P
POLYMERS
S
I
T
E
GLASS

University of Missouri-Rolla
School of Mines and Metallurgy

Graduate Center for Materials Research

The Graduate Center for Materials Research was established in 1964 for the purpose of multidisciplinary research on materials and to provide enhanced centralized laboratories and specialized equipment for faculty and students involved in materials research. The center is located in Straumanis Hall, a modern, four-story building with more than 30,000 square feet of laboratory and office space. The center has provided numerous graduate students with advanced training in materials engineering and science. The center functions as a campus resource for faculty conducting materials research, and strong interactions occur with the staffs and research programs of many departments on campus. In 1985 the past achievements and continuing importance of the UMR materials engineering and science program were acknowledged when this program was declared one of only eight areas designated for eminence in the University of Missouri System.

The center staff is composed of full-time faculty members, visiting scholars, postdoctoral fellows, graduate students, and several permanent research technicians. The permanent senior staff consists of faculty members from the departments of ceramic, chemical and metallurgical engineering; chemistry; and physics. Faculty members from other disciplines and academic departments at UM-Rolla are commonly affiliated with the center depending upon the types of research being conducted and the professional interests of the faculty. The goal is for the permanent staff to represent the widest possible spectrum of technical expertise relevant to materials research. In all, some 60 persons are involved in materials research at the center.

University of Missouri-Rolla

The University of Missouri-Rolla is one of four campuses of the University of Missouri. UMR was founded in 1870 as the University of Missouri School of Mines and Metallurgy, and, since its founding, it has been a leader in the fields of engineering and science. Degrees from B.S. to Ph.D. are offered in almost all engineering and science disciplines. UMR is among the nation's top 10 in the number of undergraduate engineering degrees granted annually.

The campus is located about 100 miles southwest of St. Louis. Also located in Rolla are many other technical and scientific agencies and small high-tech firms. Rolla is located in the pleasantly rugged terrain of the foothills of the Ozarks. The timbered hillsides and clear flowing streams make an agreeable setting and offer many forms of outdoor recreation.

Research Programs

The research conducted in the center ranges from fundamental science to applied engineering. Most all types of materials are studied, especially ceramics, metals, polymers, and composites. Research programs listed according to types of materials are:

Biomaterials

- glass microspheres for in vivo radiotherapeutic use
- orthopedic implantable ceramics
- metal adhesive intermediates for teeth
- polymer coatings for improved blood compatibility, insulating electrodes, and lens

Ceramics and Glasses

- chemical corrosion
- containerless processing of glass in space
- defects in glasses and oxides
- degradation of capacitor ceramics
- diffusion in oxides
- high-temperature conducting oxides
- low dielectric ceramic substrates
- magnetic ceramics
- mixed alkali containing glasses
- nucleation and crystallization of oxide glasses
- phosphorus oxynitride glasses
- processing of glass fibers
- sintering of ceramic oxides
- superconducting oxide materials
- thin film oxide components

Composites

- aerospace materials
- modeling
- tailored polymers

Ion-Implanted Materials

- bonding of layers to substrates
- doping of semiconductors
- improvement of wear in metals
- inhibiting corrosion of metals

Magnetic Materials

- magnetic structure of soft and permanent magnets
- magnetic properties of rare-earth-3d transition metal alloys
- properties of thin magnetic films

Metals

- coating on metals
- electrochemical corrosion of metals
- electrodeposition of metals
- electrogalvanizing
- Pb-Sn solder alloys
- surface modification of metals

Polymers

- adhesion of polymer coatings
- chemically protective polymer coatings
- paints
- plasma polymerization
- polymer thin film
- polymerization processes
- semi-permeable polymer membranes

Areas of Expertise

Each member of the staff of the center has many years of experience dealing with the development, evaluation, and application of materials. Special expertise exists in the following areas:

- adhesion
- analysis and characterization of materials
- biomaterials
- ceramics and glasses
- coatings
- composites
- corrosion
- defects in solids
- diffusion and mass transport
- electrochemistry
- ion implantation
- magnetism
- paints
- plasma polymerization
- polymers
- surface properties
- surface modification
- thin-film processing and technologies
- wear

Members of the staff have active programs in the above areas and often act as consultants and referees for grants and manuscripts in these areas.

Laboratory Facilities and Major Equipment

The center features modern equipment commonly needed for research in materials development, characterization and evaluation, and for measuring common mechanical, thermal, electrical, and optical properties. In addition, the center has specialized laboratories such as (1) a high-temperature laboratory containing numerous furnaces, some capable of 1900°C in air or controlled atmospheres, for melting and heat treating metals, glass, and ceramics; (2) a laboratory

for handling radioactive substances; (3) an X-ray laboratory with several diffractometers and camera units for ambient and high temperature use; and (4) a surface characterization laboratory for detailed studies of surface layers of solids. Other major items of research equipment include a JEOL JSM-35CF scanning electron microscope with both EDX and WDX X-ray analyzing attachments; a scanning Auger microprobe; an XPS (ESCA) spectrometer; UV, visible and IR spectrometers; optical microscopes with an image/feature analysis computer; and a thermal analyzer with DSC, TGA, and DTA capabilities.

The center has very specialized and adaptable experimental facilities for the plasma deposition of polymers and other materials. A unique tandem semi-continuous plasma polymerization coater for fibers and films allows different processes to be accomplished in a single pass. A system for coating the inside of tubing has the special advantage of providing a nonthrombogenic barrier coating on the inner surface of small diameter tubing used in many biomedical applications. In addition, the center has a variety of laboratory scale plasma polymerization reactors which are suited for academic research on the preparation processes of ultrathin films by plasma polymerization. Three tubular glow discharge reactors are used to synthesize polymers, composites, carbides, nitrides, and thin metal films at ambient temperature.

An extensive electrochemistry laboratory also is housed in the center. Equipment for the deposition and evaluation of electrolytically produced metals, polarization equipment, both standard and computer controlled, power supplies, RDE apparatus, pulse and periodic reverse platers, and analytical apparatus are available for specialized research.

The center has a scanning Auger microprobe and an XPS (ESCA) spectrometer which are used for the detailed analysis of all types of surfaces. These instruments can provide monolayer information on the elemental composition and bonding in a surface layer or at a surface layer/bulk interface. This information, along with that obtained from SEM measurements using the EDX and WDX X-ray spectrometers, gives an elaborate and detailed picture of the composition and properties of surfaces and surface layers.

Interaction With Industry

UMR has a long tradition of working with private industry on materials research and development problems. Industrially sponsored research is encouraged by a flexible University policy which gives industry timely access to specialized research equipment and allows for licensing agreements with private industry. For certain types of research, the state of Missouri will contribute to the cost under the Missouri Research Assistance Act.

The center has an active interest in industrial research which is suitable for graduate student education and which falls within the technical expertise of the staff. Examples of industrially sponsored R&D projects conducted in the Center are (1) ion-implanted metals for improved wear and corrosion resistance, (2) chemical durability and thermal performance of refractory concretes used in coal gasifiers, (3) evaluation and optimization of metal electrolyte quality using cyclic voltammetry techniques, (4) metal alloy coating of bearings, (5) development of polymer membranes for separation processes and coatings for corrosion protection and leakage, (6) surface modification of polymers and polymeric water vapor barriers, (7) development of chemically durable oxynitride glasses, (8) high temperature and special composites for aerospace applications, (9) determination of surface composition and imperfections in stainless steel, electronic circuit boards, polymers, photocells, and other materials, and (10) development of paints for special applications.

Many companies also use the center's facilities for materials characterization, especially surface analysis. The arrangements for equipment use are very flexible, and can be tailored to satisfy many special needs. A fee is charged for operator time, supplies, and equipment usage.

Materials Research in Other Units at UMR

Materials research also is conducted in the following institutes and academic departments.

Institute for Chemical and Extractive Metallurgy

The purpose of this institute is to enhance interaction among researchers in different disciplines whose work is concentrated on extractive metallurgy. Particular emphasis is placed on the metals produced in the central United States. Many active projects in minerals processing, hydrometallurgy, electrometallurgy, and pyrometallurgy arise from current industrial problems of process efficiency, pollution control or both.

The institute staff has close interaction with the major nonferrous metal producers in both the U.S. and Canada and with the Rolla Research Center of the U.S. Bureau of Mines. Many of these organizations support the research being conducted by the institute.

Institute for Thin Film Processing Science

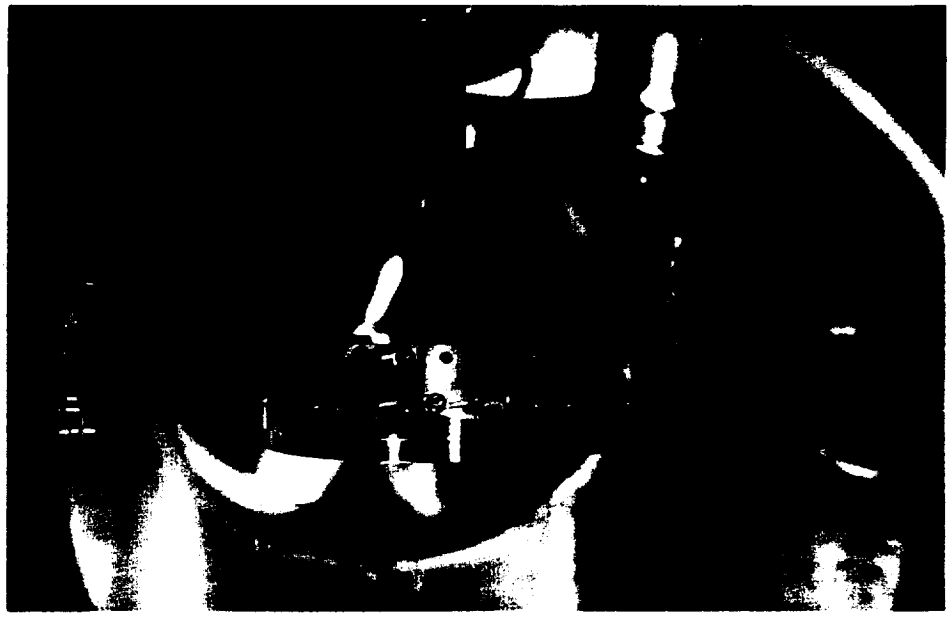
The purpose of this institute is to investigate thin films that can be used to improve properties of an exposed surface or to improve the interfacial region between two bulk materials. Emphasis is placed on film and substrate material selection and for a variety of film deposition techniques. Expertise from several disciplines is used to study films and substrates made from a combination of materials including metal, ceramic, glass, and especially polymers.

Electronic Materials Processing and Characterization Institute

The purpose of this institute is to conduct research and educate students in electronic materials, especially those involving ceramics. Scientists from several disciplines are studying nonmetallic, semiconducting, and metallic materials with funding from several federal agencies and private companies.

Ceramic Engineering

Major areas of materials research are electronic ceramics, especially for capacitor and electrode applications; corrosion of refractory oxides by slags/glasses; transformation toughening of ceramic composites; fracture mechanics of semiconductor silicon and polyphase, polycrystalline ceramics; single crystal growth (Si) in controlled atmospheres; development and properties of high-alumina cements and refractory concretes; chemical reaction of refractory oxides with high-pressure-temperature gases; and defect chemistry of ferroelectric materials.



Chemistry

Materials research is conducted in the areas of surface coatings and treatment, corrosion prevention, renewable resources for polymer precursors and characterization and dynamics of polymers. Samples are studied using a variety of spectrometers and other major scientific instruments. Materials related research is also conducted in formulation science; polymerization processes of foams and films; microemulsion polymerizations; and in the physical and chemical properties of microemulsion, vesicles, colloids, and liquid crystals.

Electrical Engineering

Research projects include the growth kinetics and evaluation of thin film photovoltaic materials (CdTe), theoretical modeling of photovoltaic devices, electrical and optical characteristics of semiconductor devices, and the use of fiber optics, laser diodes, and semiconductor materials in information processing.

Metallurgical Engineering

Research relevant to materials R&D include nonlinear mechanical properties and phase transformations of alloys, mechanical working of electroplated wires, prediction of metals failure from the early stages of metal fatigue, acoustic analysis of ore milling operations, improved wear resistant metals, and pyrometallurgical processing of metals.

Physics

Areas of major activity are materials for nonlinear optics, optical properties of ion-implanted surfaces of semiconducting solids and of materials in the far infrared and mm range, properties of piezoelectric solids and characterization of materials using surface electromagnetic waves and ion beams.

Generic Mineral Technology Center for Pyrometallurgy

This center was established by the U.S. Bureau of Mines in 1982, with UMR acting as the lead university and assisted by two other universities. The research conducted falls into the following main areas: smelting and refining processes in liquid systems, gas-solid reactions in roasting processes, innovative and complex processes in refining, process mineralogy of smelter feeds and products, and worldwide information collection and exchange on pyrometallurgy research.

For More Information

To obtain more information about the research projects, arrangements for equipment use, specialized apparatus, or other programs of the Graduate Center for Materials Research, contact:

Director
Graduate Center for Materials Research
101 Straumanis Hall
University of Missouri-Rolla
Rolla, MO 65401-0249
Telephone: (314) 341-4873

Persons interested in pursuing graduate degrees in any of the academic disciplines which are part of the center are invited to write for complete admission requirements to:

Director of Admissions
102 Parker Hall
University of Missouri-Rolla
Rolla, MO 65401-0249
Telephone: (314) 341-4164

Lunar Lubricants — *Concentric Cylinders*

Separation Team — **BILL OF MATERIALS**

Micronic Glass Sphere Simulants	- Color	**Simulates**
~2 cups - Spheres Dia. ~ 1/4th"	- Red	**Spheres Too Small**
~2 cups - Spheres Dia. ~ 1/8th"	- Blue	**Acceptable Spheres**
~2 cups - Spheres Dia. ~ 1/16th"	- White	**Spheres Too Large**
~2 cups - Tylenol Gel Caps	- Green	**Spheres Too Eccentric**

Concentric Cylinders Separation Apparatus

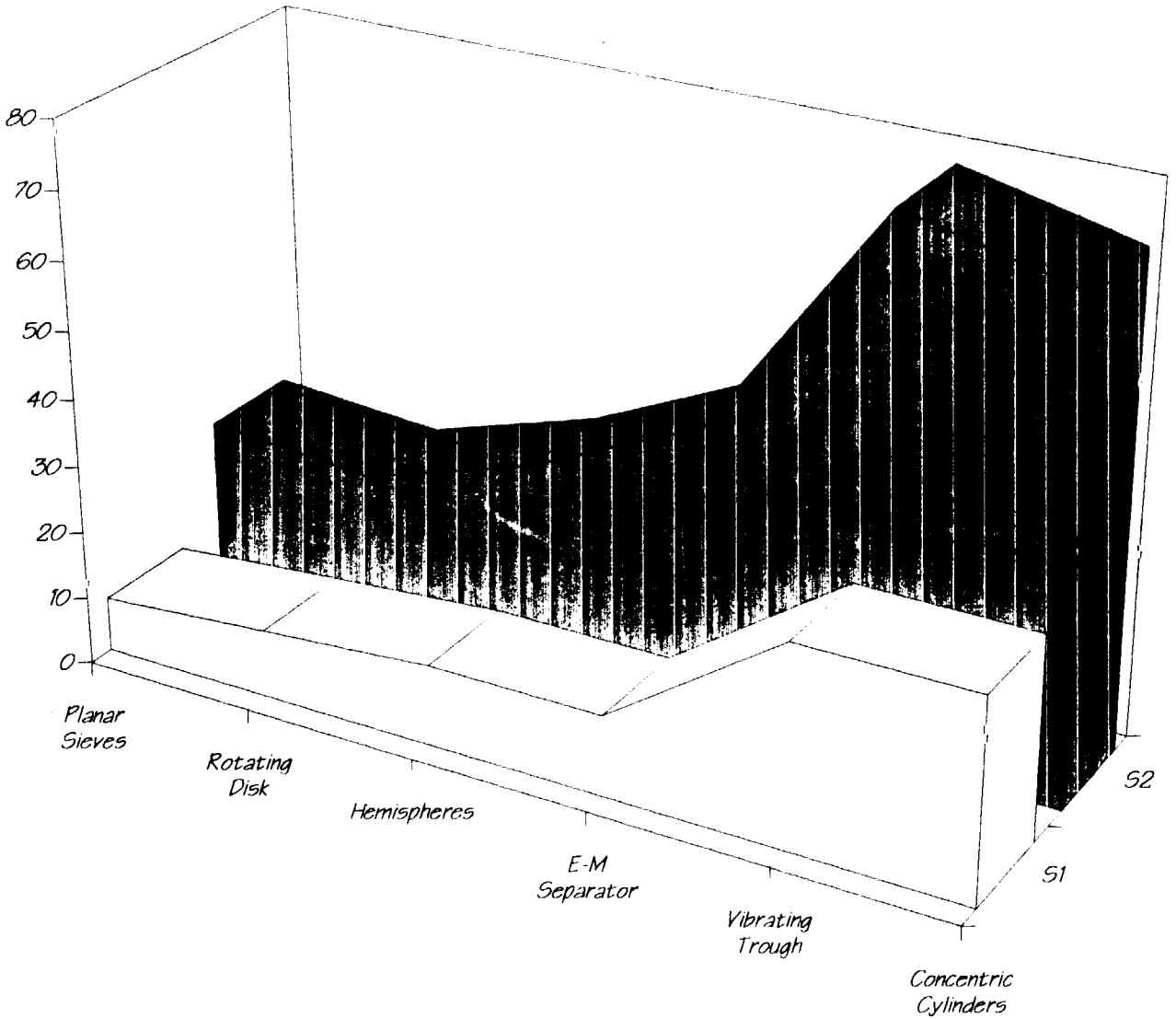
Inner Cylinder	**~2" OD x 3' long (clear)**
Middle Cylinder	**~3" OD x 3' long (clear)**
Outer Cylinder	**~4" OD x 3' long (clear)**

Mounting Apparatus and Other Necessary Paraphenalia

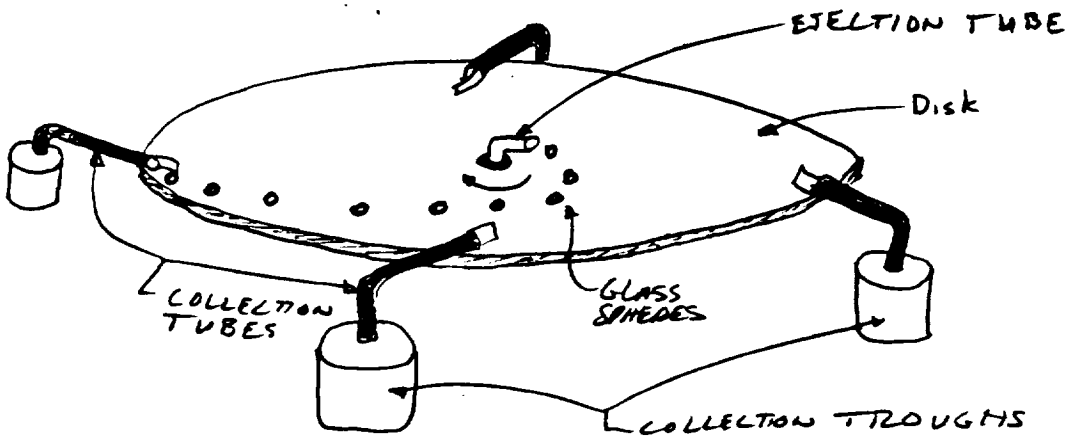
- One Surplus AC Motor (probably from a pawn shop)
- One Sheet Plywood
- One 2"x4"x8'
- One Roll Duct Tape
- Various Screw, Nuts and/or Bolts.

Separation Mechanism Design Evaluation

	Accuracy	Flowrate	Mass	Simplicity	Total	Weighted Sum
Weighting	4	2	3	1	X	X
Planar Sieves	3	1	3	1	8	24
Rotating Disk	1	3	3	3	10	22
Hemispheres	3	3	3	3	12	30
E-M Separator	9	1	1	1	12	42
Vibrating Trough	9	3	9	9	30	78
Concentric Cylinders	9	9	3	9	30	72


80
70
60
50
40
30
20
10
0
Planar Sieves
Rotating Disk
Hemispheres
E-M Separator
Vibrating Trough
Concentric Cylinders
S1
S2

ROTATING DISK METHOD



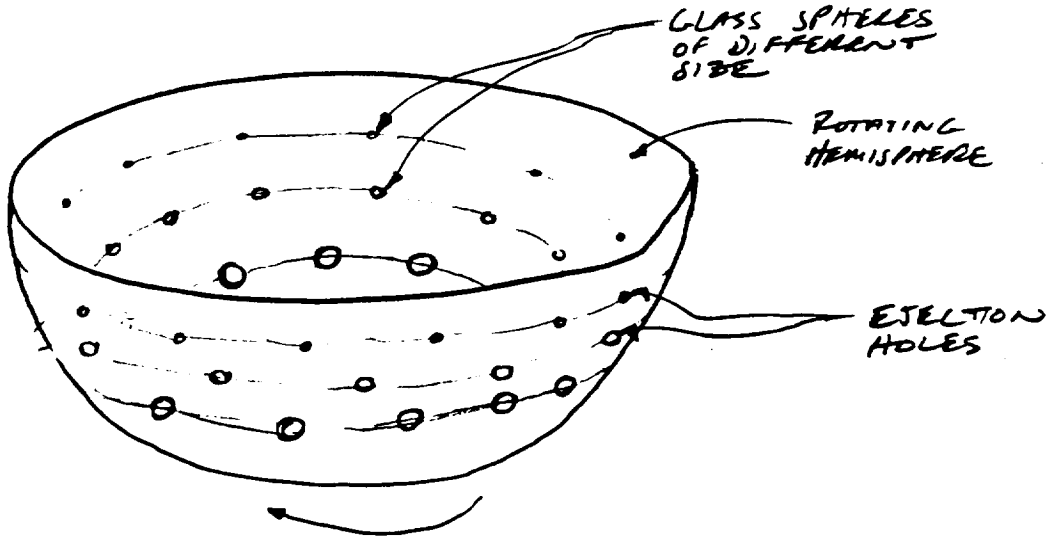
- BASED ON PRINCIPAL OF SPECIFIC ACCELERATIONS FOR OBJECTS WITH CERTAIN SIZE AND MASS

1) GLASS SPHERES ARE EJECTED FROM EJECTION TUBE.

2) WITH A CERTAIN ACCELERATION THE SPHERES MOVE TOWARDS THE OUTSIDE OF THE DISK.

3) THE SPHERES EITHER HIT A COLLECTION TUBE FOR THEIR SPECIFIC GEOMETRY OR MISS AND ARE REMELTED AND REFORMED.

ROTATING HEMISPHERE METHOD



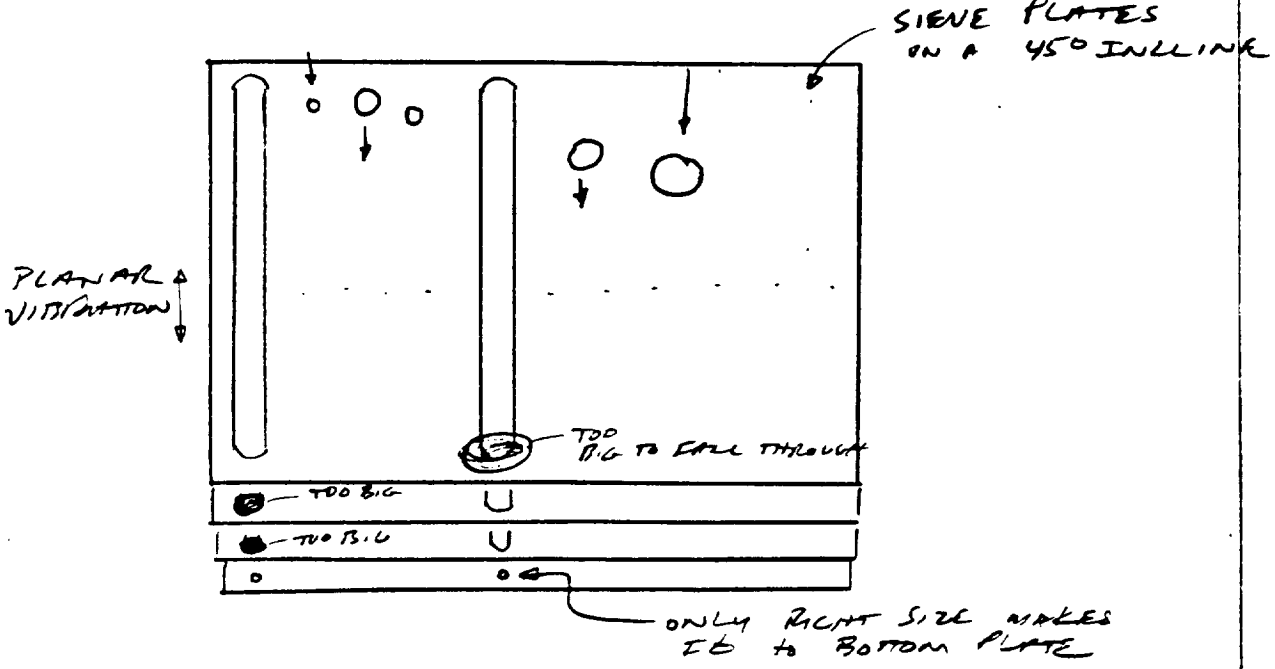
- BASED ON PRINCIPAL OF OBJECTS OF DIFFERENT MASS AND SIZE WILL TRAVEL UP TO A SPECIFIC ORIENTATION ON DISK

1) GLASS SPHERES EJECTED FROM CENTER OF HEMISPHERE

2) SPHERES TRAVEL UP THE SIDES OF THE HEMISPHERE TO A SPECIFIC ROTATION POINT.

3) SPHERES FALL THROUGH HOLES OF CERTAIN ORIENTATION AND SIZE AND ARE COLLECTED

SIEVE METHOD



— BASED ON PRINCIPAL OF DIFFERENT SIZED SLOTS ON A SERIES OF PLATES TO SEPERATE DIFFERENT SIZED GLASS SPHERES.

1) ALL GLASS SPHERES ARE RELEASED ON TOP PLATE

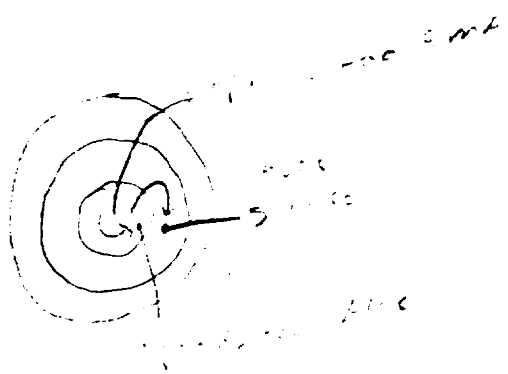
2) SPHERES OF CERTAIN SIZES FALL TO CERTAIN PLATES AND ARE COLLECTED.

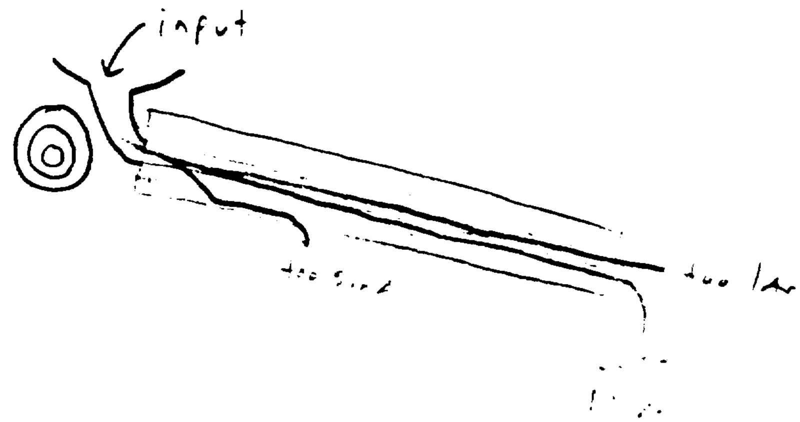
Design: Concentric Cylinders

Description: [illegible] A separation device similar to Sieve [illegible] Concentric cylinders are fixed on a vibrating [illegible], the innermost cylinder has been drilled [illegible] with a diameter equal to the smallest acceptable diameter for spheres. The second cylinder is [illegible] drilled [illegible] diameter equal to the size of acceptable diameter. The third cylinder is sealed [and] acts as a environmental containment unit.

Advantages: high flowrate, ease of construction, Accuracy, low mass

Disadvantages: accuracy of construction + shipment, ease of construction.





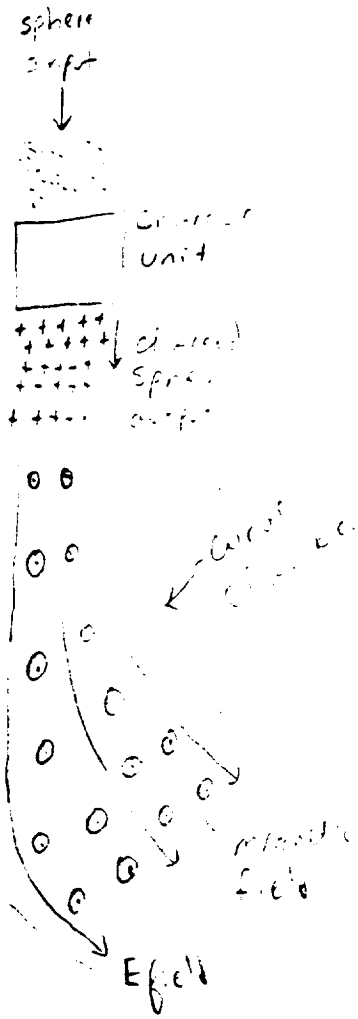
Note: rotation of the cylinders would maintain a centripetal force that may increase jam frequency. Large amplitude vibration [illegible] spheres will greatly reduce jamming of sieve holes.

Design: E·M· Separator

Description: By [illegible] charge on each of [illegible] spheres [illegible] sphere through an Electromagnetic [illegible] curved path, highly accurate [illegible] spherical separation can be achieved.

Advantages: [illegible]

Disadvantages: very slow, very expensive, requires accurate current + circuitry



Charged particles will curve in the E.M. field.
Their curvature will be dependant upon [illegible] mass accelerated + the spherical distribution of charge.

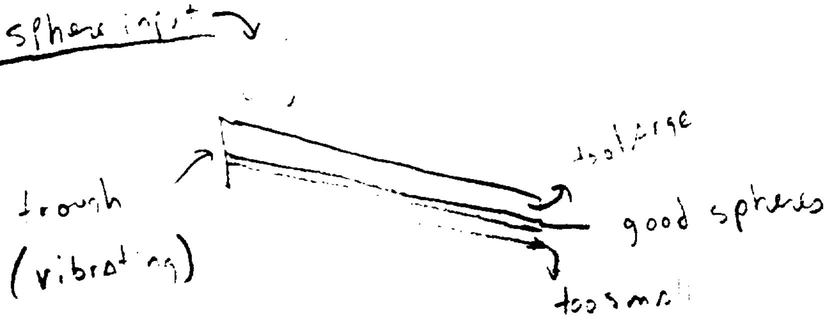
ORIGINAL PAGE IS
OF POOR QUALITY

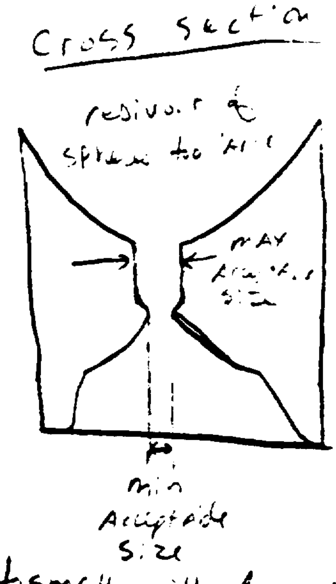
Design: Vibrating Trough

Description: By pouring the spheres onto a vibrating trough (with a number of ... constriction in cross section to ... radius) separation of size ... estimate of eccentricity ..., be made

Advantages: Accurate radius separation. Easy to construct. reasonably flow rates. ~~good~~ OK durable

Disadvantages: lack of effective eccentricity measure, large jamming possibility. ↗ increased flow rate/more





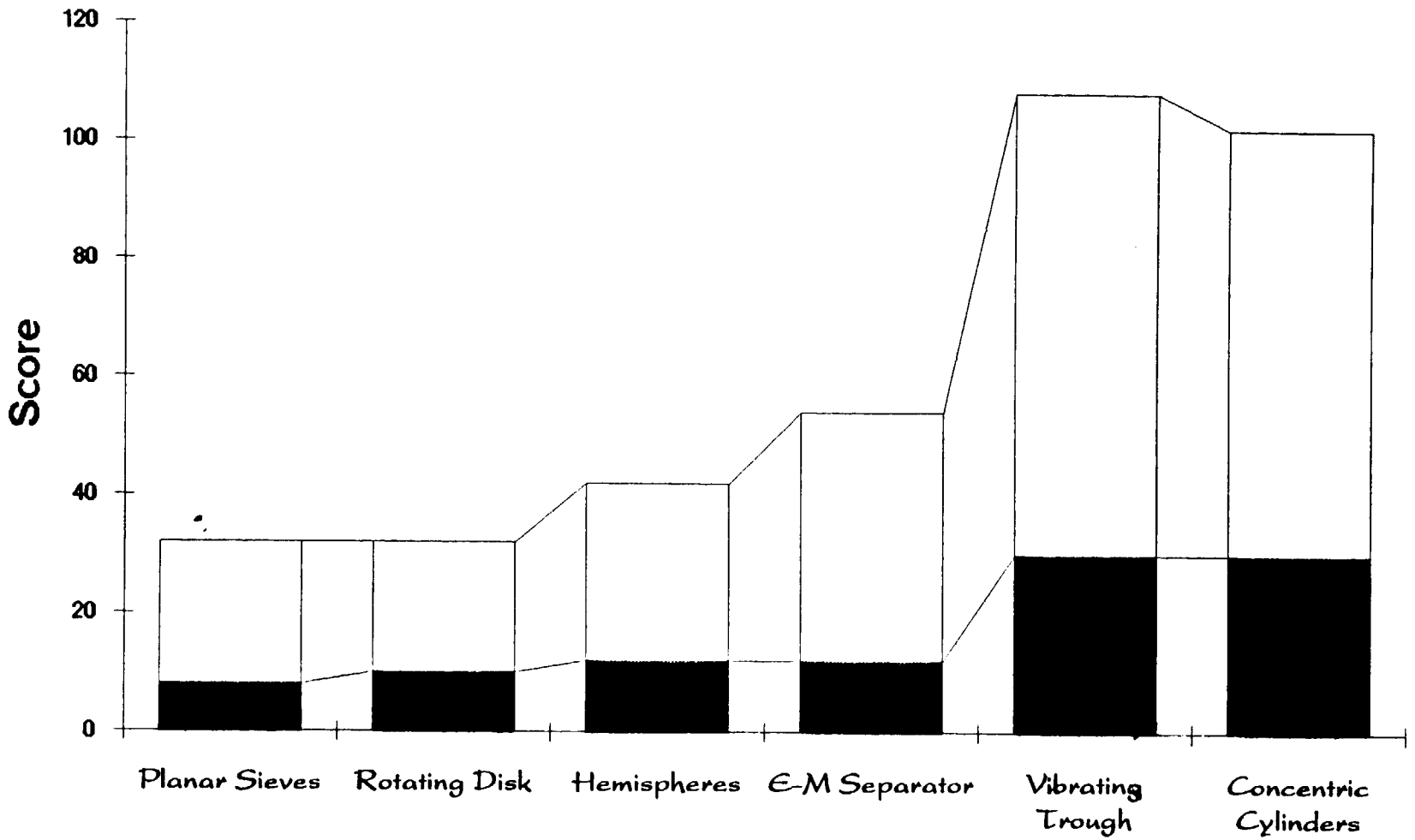
note many troughs may be paralleled for an increased flow rate

operation: All spheres are dropped onto trough. spheres too small will fall all the way through to the bottom. Sphere too large will be unable to fall through the slots and ... to the End of the trough where they are recycled. Spheres of the correct dimensions will fall through the first section but will be unable to fall further & will be separated from all other sized spheres.

Separation Mechanism Design Evaluation

	Accuracy	Flowrate	Mass	Simplicity	Total	Weighted Sum
Weighting	4	2	3	1	X	X
Planar Sieves	3	1	3	1	8	24
Rotating Disk	1	3	3	3	10	22
Hemispheres	3	3	3	3	12	30
E-M Separator	9	1	1	1	12	42
Vibrating Trough	9	3	9	9	30	78
Concentric Cylinders	9	9	3	9	30	72

Design Evaluation Results



Production and separation of small glass spheres

By W. Paul, M.A., Ph.D., and Professor R. V. Jones, C.B., C.B.E., D.Phil., F.Inst.P., Natural Philosophy Department, University of Aberdeen

[*Paper received* 22 *May*, 1952]

Glass spheres free from gaseous inclusions have been made in the size range 1–40 μ. They have been separated with relatively high accuracy into narrow ranges of size by a new sedimentation method using a liquid column containing a gradient of density.

1. Introduction

Small uniform spheres of diameters between 1 and 40 μ have many possible applications as standard particles. For example, experimental investigations of the scattering of radiation by particles require small spheres of uniform size and of known refractive index and absorption coefficient. These investigations enter into such diverse fields as the transmission of light through mist and fog, the measurement of droplet size in a Wilson chamber, the covering power of pigment particles, and the optical characteristics of colloids. Spheres of uniform size and of known optical and mechanical properties provide the best means of testing and calibrating apparatus intended for the size analysis and surface area determination of powders, particularly when the apparatus depends on the phenomena of light scattering and absorption. The spheres can also be used in experiments on flocculation and adhesion, and on fluid and heat flow through packings.

The main purpose of this paper is to describe how spherical glass particles can be made and separated into fractions having a small spread in size, in the hope that the technique may be generally useful. A subsidiary purpose will be to describe the method of separation, which may have general application as a method of size analysis with a relatively high resolving power.

2. Preparation of the Spheres

(*a*) Methods for the production of glass spheres have been described by Sklarew,[1] Sollner,[2] and Bloomquist and Clark.[3] After the glass has been ground and powdered to the desired size, each individual particle is raised to fusion temperature while free to take up spherical shape, and kept in that shape until it has cooled to a rigid solid. We have extended the methods of the above workers towards producing an apparatus which is compact and easy to operate, which is efficient in the avoidance or elimination of particles that are non-spherical or have impurity inclusions, and which collects a high proportion of the original powdered glass in spherical form.

(*b*) *Experimental method*

Prior to fusion, the glass is ground to the desired size and dried for 12–24 h in an oven at 300° C to remove most of the moisture so that the particles do not lump together or stick to the walls of the container. The apparatus shown in Fig. 1 is used. A blowpipe T is fed by ordinary coal gas and an oxygen-air mixture which becomes loaded with glass particles on passing through the container A. The flame is first enclosed in a cylinder C_1 and at its tip is placed a baffle B having a circular hole concentric with the flame and of about the same diameter. Behind B is placed the cylinder C_2; a cold air stream S at right angles to this cylinder at its far end ejects the fused glass particles into a water trough R.

The oxygen-air ratio in the feed to the blowpipe is adjusted to give a "roaring" flame; this imparts sufficient velocity to the glass to carry it into the receiver and gives sufficient heat to fuse most of the particles. The bottle A and the connecting tubes to the blowpipe are thoroughly dried before operation to prevent the glass powder clogging. The inlet to A from the oxygen-air source is by way of a glass tube bent into a semi-circle to create a swirl loading the stream with glass powder. The formation of pockets of powder that may shoot through the flame without dispersion into individual particles must be prevented. The blowpipe should deliver a broad, long flame. The glass particles are injected centrally into the

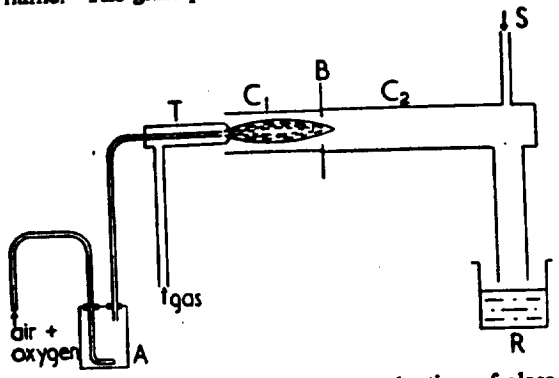


Fig. 1. Sketch of apparatus for production of glass spheres

flame from the air-oxygen tube. The cylinder C_1 confines the flame and creates a path to the receiver that is more uniformly heated than if the flame were in the open.

A glass particle might be ejected sideways from the flame so quickly that it does not fuse. To prevent this, the particles might be guided through the whole length of the flame; this would be very difficult experimentally. Alternatively, they might be made to stay longer in the flame by reducing the flame velocity or they might be heated more quickly by increasing the flame temperature. Some of these requirements are mutually exclusive, and, in practice, fusion time and fusion temperature are adjusted to give the maximum number of spherical particles and any non-spherical ones are prevented from entering the collecting apparatus by the baffle B. The efficiency of collection is thereby reduced, but the non-spherical component is completely eliminated.

After ejection from the flame the spheres are confined to the cylinder C_2 and allowed to cool before final extraction. The air stream S cools them still further and pumps them along C_2 into the receiver R.

(*c*) *Results*

When soda and Pyrex glasses are fused, inclusions are often noticed under microscope examination (see Fig. 2). These inclusions have a lower refractive index than glass, as observed by the Becké line test, and a lower density, as is easily verified in separation of the spheres by centrifugation in a liquid of density near to the glass density. The inclusions are probably gaseous and may be caused by the "freezing in" of expanded bubbles of gas in the glass or by the trapping of air when several small particles fuse into one large one.

The production of gas bubbles could be reduced by lowering the flame temperature to the lowest usable value; however, there would be some variation in temperature in a cross-section perpendicular to the length of the flame so that

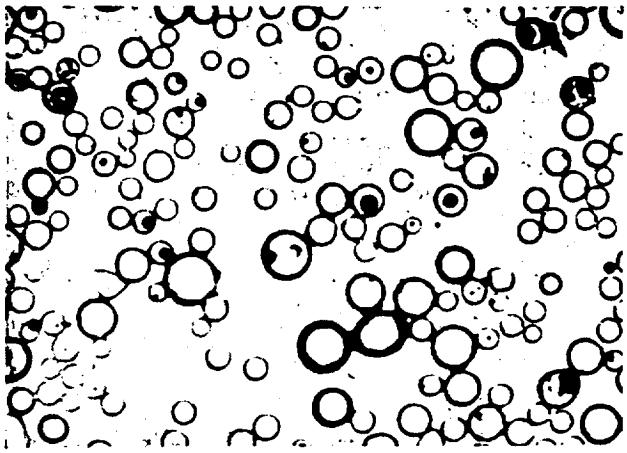


Fig. 2. Photomicrograph of soda glass spheres, immersed in water, showing inclusions. Diameter 1–40 μ

differently situated particles would be differently treated. The trapping of air between particles would be lowered by reducing the density of particles in the air-oxygen stream. When these precautions are applied in the case of soda glass, some improvement ensues, but a wholly inclusionless sample has not yet been obtained (cf. Figs. 2 and 3). The final product, when Hysil glass is used, contains uniformly spherical particles of diameters ranging from less than 1 μ to greater than 40 μ (see Fig. 3). There are few particles that are seriously a-spherical. Measurements of the worst-shaped member of a random sample of twenty particles gave a maximum to minimum diameter ratio of 1·02. No extra difficulty is anticipated in fusing other materials by a similar technique.

Experiments were carried out on the production of spheres

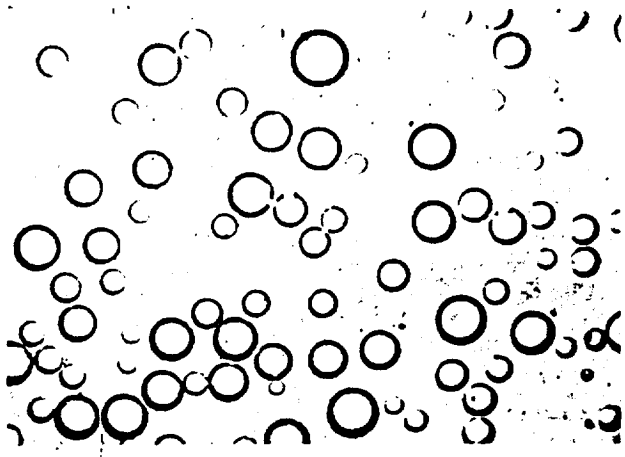


Fig. 3. Photomicrograph of Hysil glass spheres, diameter 1–40 μ, immersed in water

by dropping glass through a silica furnace 30 cm long run at 1 000° C. A long, wide furnace that can be maintained at a steady high temperature would fuse most types of glass. Further, the better control and estimation of temperature would allow the exact determination of the best conditions of fusion to avoid inclusions and strains. A more effe cooling device could be established and a higher percent of the ultimate product collected. The preliminary exp ments carried out were only moderately successful, the m difficulty lying in the initial dispersion of the powder sam In the present method of separation Hysil glass spheres fu in a flame as described above are used.

3. SEPARATION OF THE SPHERES INTO CLOSELY-SIZED FRACTIONS

Different definitions of the term "size" are appropriat different methods of particle separation and analysis. this discussion the size is measured by the actual sph diameter.

(a) *Accepted methods*

There exist several established methods of separat particles of the size required; however, none of these g very uniform fractions even with considerable expenditur effort. The sieves available are unsuitable for produc sharply defined graded fractions, the proportional variat in sieve opening increasing as the opening becomes smal A possible modification of the sieve, a slit mechanism narrow aperture, was constructed with a slit length approximately 1 cm and adjustable down to slit widths approximately 2 μ. The variation of slit width over whole length was of the order of 5% at 5 μ. Glass sphe

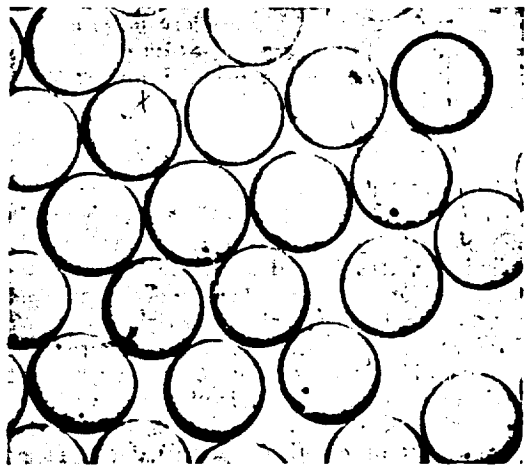


Fig. 4. Photomicrograph of Chance glass spheres immersed in water. Diameter 216–224 μ

were vibrated vertically in a suitably shaped closed cav above the horizontal slit. The relative velocity of spheres a slit was probably less than 100 cm/sec and there was observable deterioration in the condition of the slit jaws d to abrasion by the glass. It was found that the yield spheres smaller than 40 μ was very low owing to clogg of the slit by spheres suffering electrostatic attracti Spheres larger than these were separated quite efficient Successive separations at slightly different slit widths succeed in giving a sample inside a narrow size range. Fig. 4 sho spheres of Chance glass, size 216–224 μ, separated by t means. The size corresponds exactly with the slit settin In the size range greater than 40 μ there seems to be lit difficulty in thus producing monodisperse samples.

Small homogeneous spheres settle in a fluid with limiti

velocities dependent on some power of their diameter. The usual applications of this in separation work are in the well-known techniques of sedimentation with decantation, in elutriation, and in centrifugation. Sedimentation followed by decantation is a laborious process involving large quantities of liquid, which never gives complete separation. It is a useful way of producing a wholly "under-size" fraction. The elutriator tube, which uses an upward fluid flow to balance the fall with limiting velocity under Stokes' Law, has several inherent disadvantages—variations in fluid velocity in different parts of the tube, wall effects, temperature effects—that spoil its performance. The ordinary centrifuge method faces difficulties similar to those of the decantation procedure, and in any case is usually employed in the size range below 1 μ.

Electrostatic methods of separation are theoretically possible, but the experimental difficulties are considerable.

(*b*) *Method used*

(i) The density gradient. The equation of motion of a particle falling under streamline flow in a fluid medium is

$$m\ddot{x} = (m - m')g - kd\eta\dot{x}$$

where mg = weight of particle, $m'g$ = upthrust of liquid, d = diameter of particle, η = viscosity of liquid, k = constant. The particle, starting from rest, accelerates until the resistance due to the fluid viscosity balances the resultant gravitational pull, whence it moves with a terminal velocity v given by

$$v = \frac{k'(\rho - \rho_0)d^2}{\eta}$$

where ρ, ρ_0 = densities of particle and liquid, k' = constant. In our experiments the conditions necessary for the application of Stokes' Law to the fall of the particles may be assumed to be satisfied.

It is necessary in the ordinary sedimentation procedure to settle and decant a large number of times to produce reasonably good size separation. If the particles could be released simultaneously at the top of the sedimentation vessel and allowed to fall with their terminal velocities, they would reach the bottom in order of size. In practice, the particles swirl downwards like a blob of ink, large and small mixed, and no application of grids and baffles reduces the convection. The instability of the column is caused by the larger mean density of the layer containing the particles over the layer below.

Dr. F. C. Frank suggested that if the particles were made to settle against a density gradient there would be no tendency for a swirl to develop, and the particles would settle with their limiting velocity under Stokes' Law.* That this is borne out in practice has already been summarily reported.(4) In choosing liquids to form a density gradient several considerations have to be borne in mind:

(*a*) the liquids should mix over a considerable range of proportionate volumes;

(*b*) the liquids should have a density difference sufficiently high to make that between successive layers greater than a certain minimum. This minimum is dependent on the liquids used, and is just large enough to prevent the layers mixing immediately on contact;

(*c*) none of the liquids used should coagulate the particles to be separated;

(*d*) the liquids should not react with the container, producing precipitated particles or giving any product likely to coagulate the particles to be separated or interfere with their free fall.

Mixtures of alcohol and water are found to be satisfactory in the separation of glass particles. Mercuric chloride was usually added to the alcohol-water mixture to prevent organic growths, which were a troublesome feature of initial experiments with the density gradient column. Diffusion at the interfaces between layers provides a gradual change-over in density. The layers can be made thin and numerous so that a sensibly continuous gradation of density is obtained, especially at the top of the sedimentation column. The gradient persists for a long time and is not upset by small oscillational movements or temperature fluctuations. Convection currents are damped out and the column obtains an equilibrium state with a gradient of density from 0·86 to 1·0 g/c.c. from top to bottom.

The densities of the mixtures used in setting up the gradient are in steps of 0·01 g/c.c. from 1·0 to 0·86 g/c.c. The containing vessel is filled with water, and a tube with a flanged end lowered on to the water surface. Liquid of density 0·99 g/c.c. is slowly pipetted down the side of the tube. At the flange the downward velocity is reduced and a flow outwards across the water surface produced. With a density difference of 0·01 g/c.c. it is relatively easy to create a step in density at the water surface. The procedure is repeated with further layers. The thickness of the different layers is adjusted to provide a steep gradient at the top of the sedimentation vessel. A typical gradient is shown in Fig. 5.

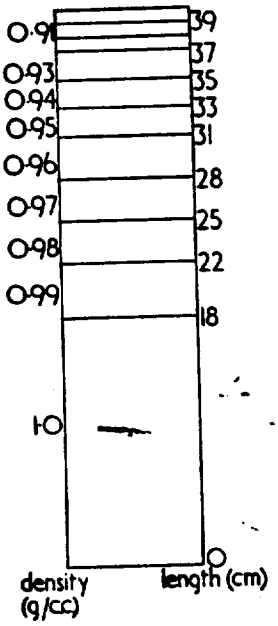


Fig. 5. Typical construction of alcohol-water column having a gradient of density

(ii) The instrument. The gradient is incorporated in the instrument of Fig. 6. The sedimentation vessel *A* is locked to the detachable top *C* of a large cylindrical container *B* at a point near the circumference. A number of small glass dishes are mounted round the circumference of a plate *D* which is rotatable about a central stem *E* in such a way as to bring the dishes successively beneath *A*, which may be of any length and may have a wide variation in diameter. The length is determined by the size of particles being separated (the larger the particles, the greater the length required) by the resolution required in the separation, by the time considered necessary for the completion of a separatory run and by the time taken to change the dishes in position without introducing any tendency for the liquid in *B* to swirl. The vessels used varied in length from 20 cm to 100 cm and in diameter from 6 cm upwards. The bottom part of *A* is wax-sealed into a cylindrical brass container *H* which in turn is locked securely through a hole in the Perspex plate *C* by the ring *J*, screwed tightly to the threaded bottom portion *K* of *H*. Rubber washers are used to perfect the liquid-tight

* Mr. W. H. Walton has independently developed the same method—private communication.

seal between Perspex and brass. A stopcock F empties A as required. The top Perspex plate C is fixed to B by a brass ring L and twelve bolts and nuts M spaced round B. B is constructed of brass and has a stopcock G.

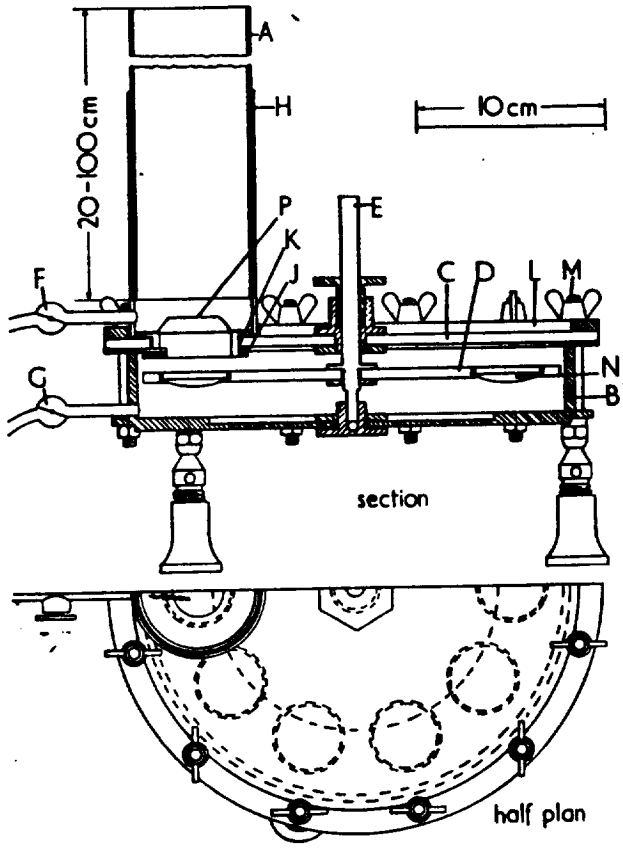


Fig. 6. Apparatus for separation of glass spheres

Through the centre of C is sealed the stem E which has attached to its lower end a Perspex plate D carrying a number of small, concave glass dishes N fixed in position by circlips. The size of D and position of the dishes are such that they can be revolved in turn, by E, into position under the vessel A. The height of D is adjusted until the dishes are under, and as close as possible to, the brass end H, to ensure that any particles falling out of A will settle in a glass receiving dish. The instrument is levelled by mounting the three feet of B on adjustable jacks.

When the vessel B is being filled it is advisable to prevent the trapping of air bubbles on any part of B or the apparatus inside B. An air bubble released during a run which rises through the sedimentation column spoils the separation by causing swirls along its path. This difficulty can be avoided in several ways, e.g. by coning part of the underside of the top C and trapping the air in the cone apex, or by providing an escape port by suitably slotting the locking ring J and the bottom threaded part of H.

(iii) Use of instrument. When the instrument has been assembled and the liquid column with its gradient of density established, a dilute suspension of spheres in pure alcohol is run down on to the top of the column using the flanged tube as before. The dishes are then rotated under the sedimentation vessel at times determined by the size of particles required and their velocity of fall as calculated from Stokes' Law and empirical calibration of the gradient for density and viscosity. Several precautions improve the quality of the separation.

(*a*) The instrument and the liquids used must be as free as possible from extraneous particles.

(*b*) The glass introduced in suspension must be completely deflocculated and must stay dispersed during the time of the separation. A combination of mechanical dispersion and suitably chosen liquids provides good deflocculation. Water-alcohol mixtures are especially suitable for glass.

(*c*) Particles which stick to the walls and are dislodged may eventually fall into the "wrong" receiving dish. They are eliminated by inserting, at the bottom of the vessel A, a baffle ring P designed to present a sharp edge to the falling spheres at such an angle that they are unlikely to attach themselves to the edge itself.

(iv) Results and conclusions. Spheres with diameters up to 40 μ have been separated in one operation giving over 90% of all those in one dish within $\pm$ 5% of the mean size. Fig. 7 shows a typical separation. The separated particles can be used in any application requiring a small amount of uniformly spherical particles of the same size. The amount of material separated in each operation is small, being of the order of 0·3 g, but it is possible to make at least 3 runs in the same column without destroying the density gradient.

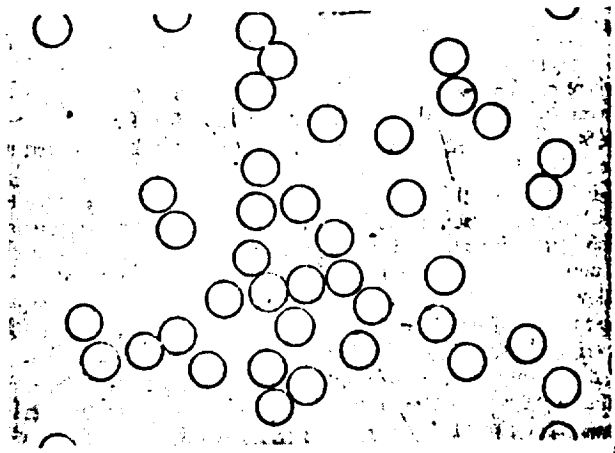


Fig. 7. Photomicrograph of Hysil glass spheres immersed in water. Mean diamater 14 μ

The method may be used for any material if two liquids can be found that maintain dispersion of the particles and are of suitable density and viscosity. The method of separation may be applied directly to the size distribution analysis of powders and has been used in a method depending on the scattering of light by the particles as they cross a horizontal section of the sedimentation vessel.

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(4) Paul, W., and Jones, R. V. *Research*, 3, p. 98 (1950).

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September 24, 1993

Material Specifications for Duke Glass Products

Soda Lime Glass:
Chemical Composition: See Over

Temperatures: **Strain Point: 505C**
Annealing Point: 550C
Softening Point: 730C
Working Point: 980C

Refractive Index: 1.51 - 1.52
Specific Gravity: 2.45 - 2.55
Linear Coefficient of Thermal Expansion (0 - 300C): 8.5 - 9.3 x 10^{-6} /C
Young's Modulus: 1 x 10^{7} psi
Rigidity Modulus: 4.3 x 10^{6} psi
Poisson's Ratio: 0.21
Dielectric Constant: 12.1 (23C, 1KHz), 7.0 - 7.6 (20C, 1MHz)
Power Factor (1MHz at 20C): 0.004 - 0.011
Volume Resistivity @ 250C: 10^{7} Ohms
Hardness: DPH 50g Load: 540 kg/mm²
Knoop 100g Load: 515 kg/mm²
Speed of Sound: 5.8 km/s at 23C
Mean Specific Heat: 0.18 cal/gmC at 20C, 0.28 cal/gmC at 1000C
True Specific Heat: 0.18 cal/gm/C at 20C, 0.32 cal/gm/C at 1000C
Thermal Conductivity: 0.002 cal/sec/cmC at 0C, 0.0036 cal/sec/cmC at 500C
Coefficient of Friction: 0.18 to 0.24 (glass on glass)
Dielectric Strength: 4500 Kv/cm
Thermal Diffusivity at Room Temperature: 0.005 cm²/sec
Emissivity: 10% at 2μm
58% at 3μm
90% at 4.5μm
98% at 8μm
78% at 9.5μm
85% at 12.0μm
Spectral Emissivity of a 1/8" thick piece: 72%
Chemical Durability--Powder Tests
In water, 4 hours at 90C: 0.05% Na_2O extracted
In N/50 H2SO4, 4 hours 90C: 0.03% Na_2O extracted
Absorption Coefficient: 0.069/cm in the visible region

Chemical Composition of Glass

Glass is very rich in Si and it can also contain large amounts of other elements such as Fe, Na, K, Ca, Mg, and Al. The primary compound in glass is SiO_2. Pure SiO_2 glass is very similar to quartz except that it does not have the long range crystalline structure of quartz. Such glass has the following characteristics:

excellent chemical durability
can withstand large temperature shocks
transparent to a wide range of wavelengths of light
very high melting point (1723C)
difficult to shape

Adding other elements lowers the glass m.p. and viscosity so it is easier to work with although not as durable.

Soda - Lime Glass: Soda lime glass is a mixture of Na_2O, CaO, and SiO_2 along with other trace elements. Color can be added with trace amounts of transition metals. The colors are a result of transitions of electrons in the 3d orbital.

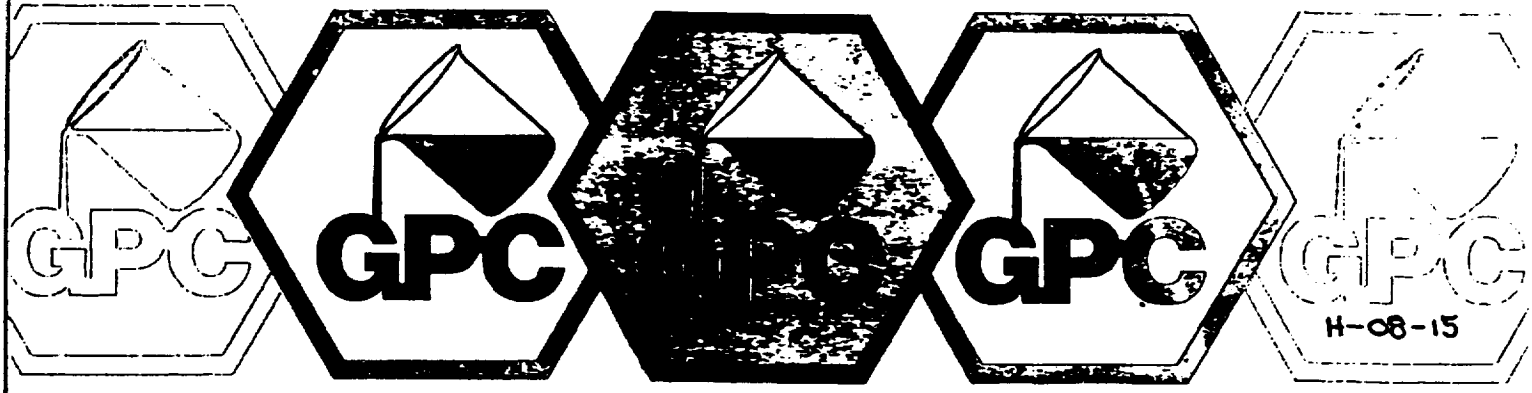
Fe^{3+}	green color you see when looking at the edge of a window pane
Co^{2+}	blue
Mn^{3+}	purple
Cr^{3+}	greens

In acidic solution, H^+ exchanges with alkali ions on the surface. This has little effect on the structural integrity of the glass, so it holds up quite well under acidic conditions.

In basic solution, OH^- ions disrupt the structure and can actually dissolve the glass. Formation of a white film on the glass is an indicator of this effect.

Chemical Analysis of a Typical Duke Soda-Lime Glass

SiO_2	67-75%
Na_2O	13%
CaO	9.7%
MgO	3.3%
Al_2O_3	0.4%
K_2O	0.1%
Iron Oxide	≤0.2%



CHAIN/CONVEYOR LUBRICANT

A group of high temperature lubricants containing graphite or molybdenum disulfide with additives in a variety of carriers designed to reduce friction without forming objectionable residues over wide temperature ranges.

TYPICAL BENEFITS

- Extended life of moving parts
- Extended lubrication cycles
- Function over wide temperature ranges
- Reduced energy consumption
- Reduced downtime from chain freeze-up

TYPICAL APPLICATIONS

- Oven and furnace chains or bearings
- Rails in annealing furnaces
- Metal lithograph lines
- Fiberboard and carton lines
- Conveyor chains and trolley wheel bearings
- Dryer ovens — Tobacco — Plywood
- Paint lines
- Can lines

	Product	Pigment	Carrier	Flash Pt[1]	Temperature[2]
(A) (C)	GP-250*	Graphite	Synthetic	380°F.	1600°F.
(A) (C)	GP-251	Graphite	Synthetic	490°F.	1000°F.
(A) (C)	GP-751	Moly	Synthetic	490°F.	750°F.
(A) (C)	LS-1350*	Graphite	Synthetic	490°F.	1000°F.
(A) (B) (C)	LS-2527	Moly	Hydrocarbon	130°F.	750°F.
(A) (B) (C)	LS-2574	Moly	Hydrocarbon	130°F.	750°F.
(A)	LS-2593	Graphite	Synthetic	470°F.	1800°F.
(A)	LS-3101	Graphite	Synthetic	570°F.	1000°F.
(C)	LS-3118	Moly/Graphite	Oil	350°F.	1000°F.
(B) (C)	LS-3111	Moly/Graphite	Hydrocarbon	265°F.	1000°F.
(A) (B) (C)	LS-3110	Moly	Synthetic	280°F.	750°F.
(A) (B) (C)	LS-3145	Graphite	Synthetic	250°F.	1800°F.

(1) Flash point of carrier.
(2) Maximum continuous operating temperature after the carrier has evaporated.
(A) Synthetic carriers will not form objectionable residues, i.e., carbon and varnish deposits.
(B) After evaporation of carrier, products will function as dry film lubricants.
(C) Suitable for automatic lubricating systems.
(*) Require some agitation.

symplastics INC.

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SYMPOXY SP-1, SP-2 and SP-3

HOLLOW MICROSPHERES FOR LIGHTWEIGHT SYNTACTIC RESINS FOAMS, ETC. IN EPOXY, POLYESTERS, URETHANES ETC.

DESCRIPTION:

Sympoxy SP-1, SP-2 and SP-3 are hollow glass microspheres of different qualities and specific gravities.

Sympoxy SP-1 is a thinner walled bead that has been acid etched, washed, dried to take out all broken beads and has controlled specific gravity assuring fixed specific gravities when mixed with liquid or powdered systems. These beads have excellent moisture resistance providing high insulation resistance.

Sympoxy SP-2 has a higher specific gravity variance but has higher compressive strengths and higher specific gravity at similar loadings in the carrier. A higher loading, by volume, gives reduced shrinkage, higher hardness, better abrasion resistance while maintaining lower viscosity and better flow than SP-1. SP-2's are also lower in cost.

Sympoxy SP-3 has a very thick wall and much higher specific gravity range but does well in systems where larger fillings are required and no lower than .85 specific gravity is required. Higher compressive strengths, lower system costs, better filler suspension and easier processing are the application requirements fulfilled by SP-3.

All of the above microspheres increase flame retardancy by lowering the thermal conductivity of the system.

TYPICAL PROPERTIES:	SP-1	SP-2	SP-3
Specific gravities	.16 to .18	.20 to .25	.70 to .75
Oil absorption (ASTM D281)	30	9	6
Bulk density, lbs/cubic foot	4.3-5.0	5.9-7.5	25-27
Color	white	light gray	gray
Hiding power	good	excellent	excellent
Electricals	excellent	good	good
Costs	high	low	low
Vehicle processing:			
by hand	yes	yes	yes
by mill	no	no	no
by shear	no	no	no

CAUTION:

Since all three microspheres are of a fine particle size and low density, caution should be used in working with them. A face mask, long sleeve and long legged clothing should be used and good personal hygiene followed by washing exposed skin with soap and water. Launder clothing before reusing. Avoid flame or sparks in unventilated areas. Use mechanical exhaust in work area after using.

3630

AMERICAN TECHNICA Inc.
118 DENTON AVENUE
NEW HYDE PARK, NY 11043

TEL: 516-741-7140
FAX:516-741-7321

J-03-16

MOST SUITABLE DIMENSIONS OF BALLS

*0.127mm	2.50	7.50
*0.15	2.788 = 7/64"	7.938 = 5/16"
0.2	3.00	8.00
0.25	3.175 = 1/8"	8.50
0.30	3.50	8.731 = 11/32"
0.35	3.969 = 5/32"	9.00
0.40	4.00	9.525 = 3/8"
0.45	4.50	10.00
0.50	4.762 = 3/16"	10.319 = 13/32"
0.6	5.00	10.50
0.7	5.50"	11.00
0.8	5.556 = 7/32"	11.112 = 7/16"
0.9	6.00	11.50
1.00	6.350 = 1/4"	11.906 = 15/32"
1.50	6.50	12.00
1.588 = 1/16"	6.747 = 17/64"	12.50
2.00	7.00	12.700 = 1/2"
2.381 = 3/32"	7.144 = 9/32"	

All sizes in metric except where noted.

Other sizes are available upon request.

Ball are available in Synthetic, Ruby, Sapphire, Quartz, Optical glasses, Ceramics, Zirconia, Sodalime, Borosilicate and Tungsted Carbide.

*Only available in Synthetic Ruby

3/90

Glass Balls

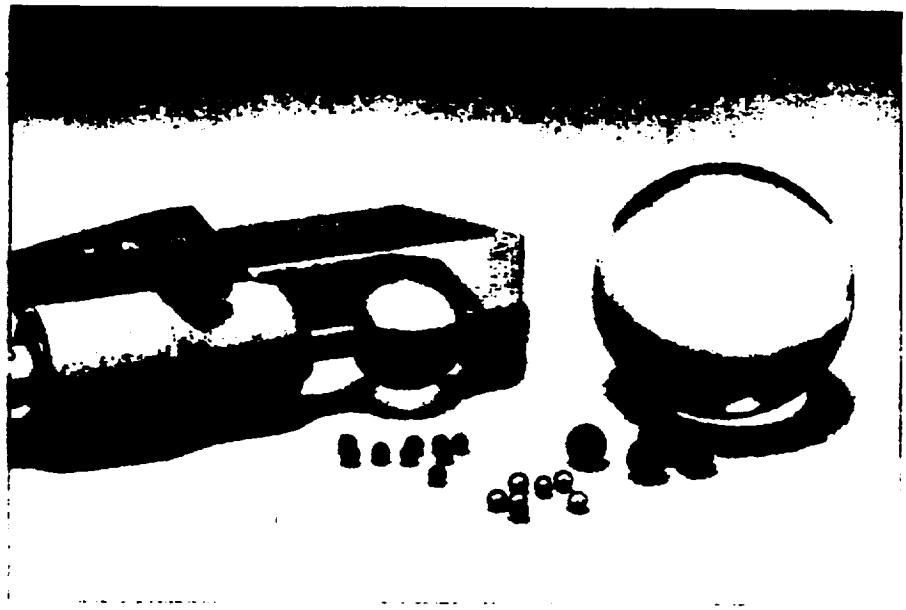
When it comes to low cost flow control and high heat applications, design engineers find glass balls hard to beat. For good reason.

Glass balls are dimensionally stable. They resist corrosion and chemical absorption well. Plus, they can withstand temperatures up to 600°F.

Glass balls also vary in density, depending on the type of glass they're made from. They are widely used in applications requiring a specific gravity.

Let's take a look at why these unique characteristics make glass balls ideal for flow control, instrumentation and fiber optic applications.

Food processing, pharmaceutical, and photographic processing equipment engineers select glass balls for check valves because they provide



Glass Properties	Soda-Lime	Borosilicate	'Black Glass'
Density: gm./cm.^{3}	2.47	2.23	2.64
Hardness: Knoop-KHN$_{100}$	465.0	418.0	405.0
Softening Point: °C.	695.0	820.0	650.0
Maximum Working Temperature* (annealed glass) Normal °C.	110.0	230.0	110.0
Extreme °C.	460.0	490.0	380.0
Young Modulus: 10^6 lb./5 sq. in.	10.0	9.1	9.8
Poisson's Ratio	0.24	0.20	0.21
Thermal Expansion cal. cm./300°C. - 10^{-7} in./in./°C.	92.0	33.0	89.0
Thermal Conductivity cal. cm./cm.^{2} sec. deg. C.			
- 148°F./- 100°C. (x 10^{-3})	1.99	2.13	—
+32°F./0°C. (x 10^{-3})	2.43	2.71	—
+212°F./+100°C. (x 10^{-3})	2.65	3.12	—
Thermal Stress Resistance	17°C.	53°C.	18°C.
Dielectric Properties at 1 MHz - 20°C.			
Power Factor %	0.9	0.5	0.17
Dielectric Constant	7.2	4.6	6.3
Loss Factor	6.5	2.6	1.1
Log$_{10}$ of Volume Resistivity: ohm-cm.			
25°C.	12.4	15.0	—
250°C.	6.4	8.1	8.9
350°C.	5.1	6.6	7.0
Refractive Index Sod. D Line (.5893 microns)	1.512	1.474	1.507

*Mechanical considerations only.

NOTE: The physical properties will vary between raw glass manufacturers.

AMERICAN TECHNICA Inc.
418 DENTON AVENUE
NEW HYDE PARK, NY 11043

TEL: 516-741-7440
FAX:516-741-7321

A.F.B.M.A. * Grade Tolerances and Terminology

A.F.B.M.A. Grade	Diameter Tolerance per Ball	"V" Block Out-of-Round in 120° Angle	Diameter Tolerance per Unit Container	Basic Diameter Tolerance	Marking Increments	Maximum Surface Roughness Micro-Inch "AA"**
	Inch	Inch	Inch	Inch	Inch	
3	.000003	.000003	.000005	±.00003	.000003	.5**
5	.000005	.000005	.00001	±.00005	.000005	.7**
10	.000010	.000010	.00002	±.0001	.000010	1.0**
15	.000015	.000015	.00003	±.0001	.000015	1.2**
25	.000025	.000025	.00005	±.0001	000025	1.5**
50	.00005	.00005	.0001	±.0002	00005	3.0
100	.0001	.0001	.0002	±.0005	0001	5.0
200	.0002	.0002	.0004	±.0010	0002	8.0

*"AA" Arithmetical Average.
**These grades may carry waviness requirements.

Terminology:
Ball industry terminology has been interpreted by the A.F.B.M.A. for use by the industry. Listed below are the terms which conform to the A.F.B.M.A. standard definitions, unless otherwise specified. Please call us if any of these terms need further clarification.

Ball Roughness:
Surface irregularities, finely spaced, direction, height and width of which demonstrate the overall surface pattern.

Ball Waviness:
Geometrical irregularity of the ball surface where wave lengths are longer than the roughness.

Basic Diameter:
The Basic Diameter is specified by a fraction plus a decimal only to the sixth place e.g. 1/4" + .0002" or .2502"; 15/64" + .0003", or .223575".

Basic Diameter Tolerance:
The maximum deviation allowed of any ball diameter from the basic diameter, in any shipments to satisfy orders for that basic diameter.

Diameter Tolerance Per Ball:
The allowable difference between the largest diameter and the smallest diameter measurable on one ball.

Diameter Tolerance Per Unity Container:
The allowable range of the average diameter of single balls within any one unit container.

Grade:
Numerical value of the Diameter Tolerance Per Ball shown in millionths of an inch.

Hardness:
Measure of resistance of balls to penetration as figured by the methods indicated in this standard.

Lot:
A lot is made up of all balls, same grade, specific diameter (marking increment), hardness and material, submitted for approval as an undivided whole at the same time.

Marking Increments:
Standard unit steps in millionths of an inch to show the Specific Diameter.

Nominal Diameter:
SIZE which is used for the purpose of general identification, e.g., 1 m/m 1/8", 3/16", 7/32", 15/64", etc.

Quality of Geometry:
Degree of precision indicated by dimensional tolerances.

Quality of Surface:
Degree of refinement of the surface characteristics as indicated by waviness, roughness and appearance.

Specific Diameter:
The unit container diameter as marked, shown in the grade's standard marking increment closest to the average diameter of the balls in that unit container.

Unit Container:
Any single container marked as having balls of the same material, specific diameter and grade.

"V" Block Out of Round:
Occasionally referred to as three-point or multiple point out-of-roundness. It is the maximum variation in the rise of the ball made possible through changing the position while the ball is supported in a "V" block.

Visual Inspection:
A macroscopic inspection of the ball surface for imperfections.

*Anti Friction Bearing Manufacturers Association (A.F.B.M.A.)

GP ceramic precoats are designed for use in metalforming operations to provide lubrication and protection against oxidation at elevated temperatures.

TYPICAL BENEFITS

- Improve metal flow
- Provide oxidation protection
- Provide added lubrication
- Reduce friction and pressures on dies
- Reduce heat loss of workpiece
- Improve surface finish of workpiece

SURFACE PREPARATION

Workpieces should be chemically or mechanically cleaned and rinsed with water prior to coating. Acid etching followed by a clean rinse is recommended. For water-base coatings, preheat workpiece to 150°F to 300°F (66°C to 149°C) after cleaning and before coating application.

METHOD OF APPLICATION

The products may be used full strength for blocking/extruding operations. For mixing or diluting to a desired level, thoroughly mix to a uniform consistency, using a method that minimizes air entrapment. Hand mixing or agitation with a slow speed mixer is recommended.

Work pieces may be coated by dipping or spraying, followed by air drying from 10 to 30 minutes depending upon carrier and room temperature.

For optimum results, a smooth uniform coating, free of cracks, runs or pinholes is necessary.

Dipping Procedure

Clean and thoroughly dry parts before dipping. Position parts after dipping to minimize buildup of coating while drying. A coating thickness of 0.001" to 0.002" after drying is recommended.

Spraying Procedure

Conventional spray or electrostatic systems are suitable for coating parts. Experience will dictate proper patterns and technique. Dry-film coating thickness should be 0.001" to 0.002". If necessary make additional applications only after initial film is thoroughly dry.

DILUTION RATIOS

Concentrated product may be diluted for thinner coating or spray system application. Start with 8 parts concentrate by volume to 1 part proper carrier. Dilute further if necessary. Slowly add diluent to product while gently stirring. Avoid excessive agitation which causes air entrapment.

COATING REMOVAL

Residual coatings may be removed by sandblasting or hot salt bath treatment.

HANDLING AND STORAGE

Shelf life of products is from six months to one year in original unopened containers. Materials with flammable solvents should be kept away from open flame or sparks. Seal containers when not in use to prevent contamination and evaporation. Use adequate ventilation with all solvent based coatings.

For additional information of safe use and handling refer to the MSDS of each product.
For information on Graphite Die Lubricants and Precoats please request PB-2 Bulletin.

Graphite Products Corporation

P.O. Box 29, Brookfield, Ohio 44403
Phone: (216) 394-1617
FAX: (216) 394-2389

PDS

PB-8

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GRAPHITE POWDERS

A selection of three grades of graphite powder in three different ranges of particle sizes. The black boundary lubricant powders have no melting point and are thermally stable at temperatures in excess of 1000°F. in the presence of air.

TYPICAL CHARACTERISTICS

- Adheres to most surfaces.
- Lubricates over a wide temperature range up to 1000°F. in the presence of air.
- Readily mixes with greases, oils, and fluids.
- Maintains a very low coefficient of friction in high temperature environments.

AVERAGE PARTICLE SIZE[1] OF POWDER

GP 600 98% by weight through 325 sieve. Average particle diameter determined by sieve analysis.

GP 601 96% to 98% by weight through 325 sieve. Average particle diameter determined by sieve analysis.

GP 603 .7 to .85 microns average particle diameter by Fisher Sub Sieve Sizer.

[1]Not to be used as a specification.

INDUSTRIAL USES

- Metalworking for hot forming, hot extrusion applications.
- Additive for plastics, rubber to reduce or modify friction.
- Additive for plastics, rubber, as an electrically conductive pigment.
- Tumbling/burnishing application to "O" Rings, packings and seals.
- Maintenance lubricant in powder form.

APPLICATION METHODS

- Tumbling for metalworking or small component coating.
- Burnishing or rubbing surfaces with cloth.
- Dusting or small applicator bottle for penetrating narrow areas.
- Powder ingredient mix for plastics, elastomers, plastics and powder metals.

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(see Inspection Devices)

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READE ADVANCED MATERIALS, EASTERN REGION SALES OFFICES P.O. Drawer 15039 (ZIP 02915-0039) (Mfr. & Dist. Of High Purity Metal, Ceramic & Intermetallic Powders, Fibers & Whiskers To Specs.) (401—433-7000) ...5M+
(See Our Company Profile In Volume 18)

SEE OUR CATALOG IN CATALOG FILE SECTION

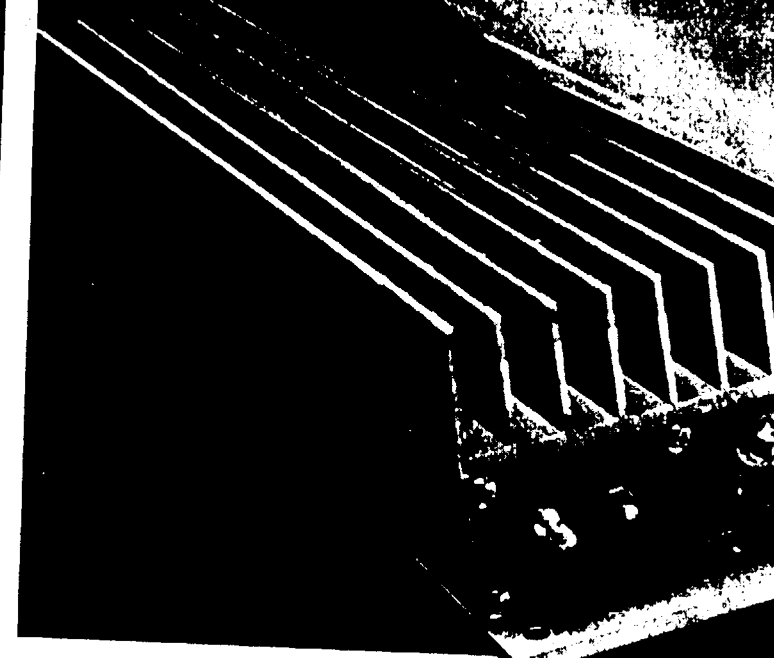
VA: SPRINGFIELD

Versar, Inc. ...25M+

WA: KINGSTON

Particle Information Services, Inc. 34467-T Hood Canal Dr., N.E. ...NR

MICROTOMES



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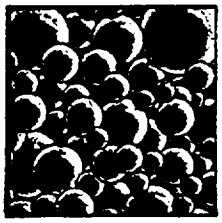
Hollow Microspheres



PQ's hollow microspheres are your best choice when you need a lightweight filler. We currently offer glass, ceramic and plastic hollow microspheres.

To the naked eye microspheres look like a fine white power. If you looked at them through a microscope, you would see micron-sized, spherical particles filled with air.

These tiny spheres provide a surprisingly wide range of benefits in finished products:

- low density
- improved flow
- reduced shrinkage and warpage
- better impact resistance
- cost reduction when more expensive materials are displaced
- easier finishing and working characteristics
- sound and thermal insulation

Microspheres were first introduced as lightweight fillers for plastics, explosives and cements. Because they have a low specific gravity, a relatively small quantity of microspheres can displace more costly materials and reduce overall weight and costs. In water gel and emulsion explosives, microspheres function as sensitizers.

The number of industries that use microspheres has grown considerably over the years. Today our products are found in lightweight cements, explosives, auto parts and underbody sealants, bowling balls, marine industry products, building materials, autobody fillers, cultured marble, paints, grinding wheels, friction compounds and refractories. They are compatible with polyesters, epoxies, plastisols, urethanes, thermoplastic, latex and phenolic resins.

Our products are available with surface functional coatings, including silane, metals and pigments for specialty applications.

We manufacture microspheres at four locations on three continents. Our network of distributors circles the world. PQ's technical service representatives are available to advise you on formulating or processing questions.

The PQ Corporation
Your single source for microspheres—anywhere in the world.

Applications for Microspheres

End Use	Matrix	Advantages
Cultured marble Simulated-wood furniture Decorative casting	Polyester	Density reduction Resistance to thermal shock Casting quality
Marine decks and hull coring Marine putty Fiberglass-reinforced plastic coring	Polyester Epoxy	Cost reduction Improved stiffness Easier finishing
Bowling-ball cores	Polyester	Density control
Plywood-patching compounds Insulative pipe covering	Polyester Epoxy Polyurethane	Improved shaping and finishing Thermal insulation
Automotive sound-deadening pads	Asphaltic	Density reduction Acoustic insulation
Automotive sealants	PVC plastisol	Density reduction Reduced sag tendency Chip resistance
Auto and truck brake pads	Phenolic	Increased pad life Density reduction
Syntactic-foam flotation devices	Epoxy	Density reduction
Hi-mil coatings Textured paints	Solvent-based resin Water-borne binder	Density reduction Improved rheology
Grouts Roof coatings Synthetic stucco Concrete	Latex Elastomeric latex Latex/cement Cement	Density reduction Enhanced trowelability Improved sprayability
Industrial explosives	Water gel Emulsion	Sensitivity
Refractory coating/bricks	Ceramic composite	Thermal insulation
Grinding wheels	Ceramic composite Phenolic	Antislump in green state Controlled porosity Weight reduction

PA102/1092

The PQ Corporation
Specialty Products Group
P.O. Box 840, Valley Forge, PA 19482 U.S.A.

BRASS

TYPE ANALYSIS – Cabra 260
Copper 68.5-71.5%; Lead .07 max; Iron .05 max; Zinc, remainder.

GENERAL INFORMATION
For use primarily in valve applications.

MATERIAL CHARACTERISTICS
Very tough. Very good corrosion resistance at lowest cost.

MACHINABILITY – 30.

CORROSION RESISTANCE – Good.

HARDNESS
Rockwell B75-87 measured on parallel flats.

NAVAL BRASS*

TYPE ANALYSIS – Cabra 464.
Copper 60%; Tin 0.75%; Zinc 39.25%.

GENERAL INFORMATION
Desirable for use primarily in valve applications.

MATERIAL CHARACTERISTICS
Extremely tough. Offers good corrosion resistance at relatively low cost.

MACHINABILITY – 30.

CORROSION RESISTANCE – Good.

HARDNESS
Rockwell B75-98 measured on parallel flats.

*On special order only.

HIGH CARBON CHROME ALLOY

TYPE ANALYSIS – AISI-52100
Carbon .95-1.1%; Chromium 1.3-1.6%.

GENERAL INFORMATION
This steel is the result of years of experimenting with various analyses in search of the best material for bearing use.

MATERIAL CHARACTERISTICS
High hardness and consequent resistance to deformation with excellent wear resistance. Manufactured from the highest quality chrome alloy electric furnace steel in accordance with our standard specifications and uniformly hardened by our latest methods of controlled heat treating throughout their entire diameter, assuring maximum strength and long life.

HARDNESS
Rockwell C – 60 to 66 measured on parallel flats.

DRILLED BALLS AND SPECIAL BALL PRODUCTS

Hartford manufactures drilled balls in various materials, to customer blueprint specifications. Critical hole diameter tolerance and hole concentricity are achievable. All drilled balls are accurately lapped to finished surface and geometric specifications.

We offer tapered, counterbored and countersunk holes, as well as other secondary operations. Also, variable modifications can be made on a vast selection of materials.

AID IN NEW DEVELOPMENTS
To obtain aid for the solution of difficult problems, where special balls are required, we encourage you to contact our Sales and Engineering Departments. We are willing at all times to provide technical information relative to the adaptation of special balls.

Should you wish to use our technical experts for a development project, arrangements can be made accordingly.

It is our sincere desire to help you with your special needs, therefore, do not hesitate to consult our experienced and knowledgeable staff.

GLASS*

SODA LIME
Will not stand thermal shock; can mechanically give continual service at 230 degrees F.; stands limited static and torque load; good electric and corrosion resistant properties.

SURFACE
Ground – 20-30 Microinches R.M.S.

Polish – 10-20 Microinches R.M.S.

PYREX BRAND – (Corning 7740)*

Will stand high thermal shock; can mechanically give continual service at 450 degrees F. with extreme temperature limit at 900 degrees F; very high electric resistivity and dielectric strength; high chemical stability, and will withstand high applied torque loads.

SURFACE
Ground – 20-30 Microinches R.M.S.

Polish – 10-20 Microinches R.M.S.

*On special order only.

PLASTIC*

NYLON-zytel 101
CORROSION RESISTANCE
Nylon is insoluble in common solvents, alkalies, dilute mineral acids and most organic acids. Nylon is particularly outstanding in resistance to alkalies, petroleum oil and greases at temperatures up to 300 degrees F. Acids such as lactic acids in milk, photographic solutions, etc., have little or no effect. Nylon balls are used where the requirement calls for lightweight, resilient material and resistance to abrasion. Used without lubrication.

HARDNESS – Rockwell R-118.

TEFLON
CORROSION RESISTANCE
No known industrial acids or caustics will attack TEFLON, and there is no know solvent for it. Recommended for applications where lightweight, nonmetallic, and erosion resistant properties are required.

HARDNESS – Durometer – 65-70.

*On special order only.

TUNGSTEN CARBIDE*

GENERAL INFORMATION
Typical industry applications are: Instrumentation; valves in high precision hydraulic systems; high load bearings; inertial navigation systems; ball screws; linear bearings in slideways; gaging and checking; meters; and for ballizing.

MATERIAL CHARACTERISTICS
The characteristic properties of Tungsten carbide make it highly suitable for precision balls in applications requiring high hardness; resistance to wear, impact elevated temperature, corrosion, humidity, abrasion, and poor conditions of lubrication.

TYPE ANALYSIS
94% Tungsten, 6% Cobalt

MECHANICAL PROPERTIES

Ultimate Tensile Strength	220,000 psi
Ultimate Compressive Strength	643,000 psi
Transverse Rupture Strength	228,000 psi
Hardness (Rockwell A)	90.5-91.5
Modulus of Elasticity	98,000,000
Density (Approx.)	0.54 lb./in.
Specific Gravity	14.85-15.05

*Available through Specialty Ball Co.

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D. F. Herman
National Lead Company

MICROSCOPY. See ELECTRON MICROSCOPY; RESINOGRAPHY

MICROSPHERES

Microspheres are thin-walled hollow spheres with diameters in the micron range, and made of plastic or glass (Fig. 1). Wall thickness is 2–3 μ. In appearance, bulk microspheres are a free-flowing powder resembling fine sand. Bulk density ranges from 6 to 12 lb/ft^3. They are used in bulk form to control evaporation of liquids and as a filler in the manufacture of low-density filled plastics. See also CELLULAR MATERIALS.

Microspheres were developed somewhat over fifteen years ago by The Standard Oil Company (Ohio), which holds the basic patents for their manufacture (1). They are produced and marketed by several companies in the United States under license from Sohio. Suppliers include Emerson & Cuming, Inc. (Eccospheres), Union Carbide Plastics (Phenolic Microballoons), and Sohio Chemical Co. (glass Microballoon spheres). Minnesota Mining and Manufacturing Co. has recently introduced a type of hollow glass sphere.

Originally designed for use as an evaporation retardant for oil tanks, microspheres are becoming increasingly popular as a filler for plastics in the field of commercial structure foams, in marine buoyancy applications, and in the production of lightweight casting resins with "tailored" properties, molding compounds, and dielectric materials for electrical and electronic uses (2–5).

Evaporation control still represents the largest single outlet for plastic microspheres, but the proportion of total microsphere output going into this area is steadily decreasing in favor of applications in the field of commercial and specialty foams.

Types. The field of plastic microspheres is dominated by the phenolic-based variety. Low cost of raw materials contributes to this. At bulk densities of 6–8 lb/ft^3 phenolic microspheres for general-purpose applications sell for just below $1.00/lb. Hollow epoxy spheres (Eccospheres EP), with diameters of $\frac{1}{16}$ in. and up, have re-

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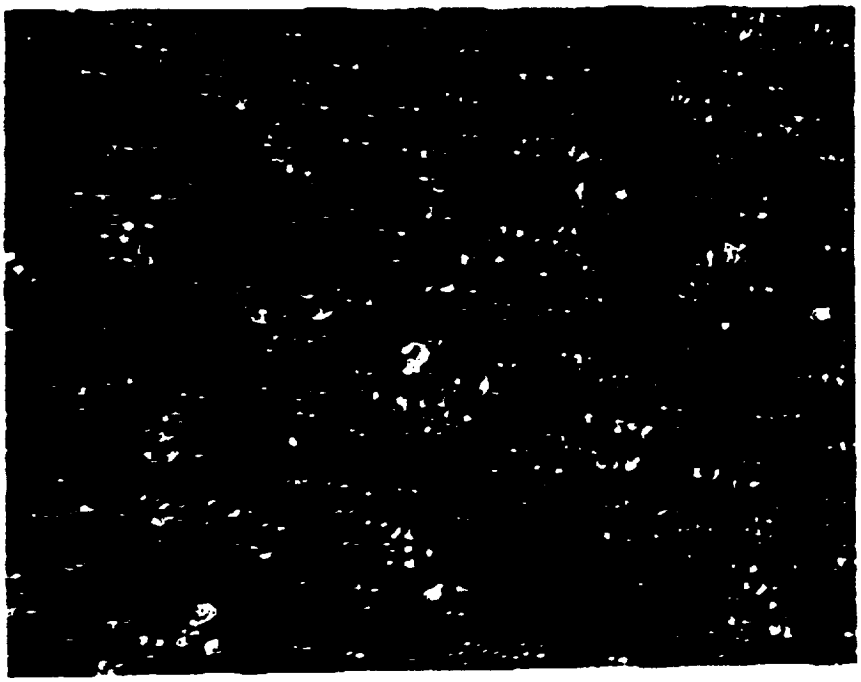

Fig. 1. Enlarged view of glass microspheres (Eccospheres R). Courtesy Emerson & Cuming, Inc.

cently been developed. Bulk density range is 7.5 to about 15 lb/ft³. One application of the epoxy type is in foams with high compressive strength.

General-purpose glass microspheres are made from low-cost glass. Bulk densities as low as 10 lb/ft³ are available. At somewhat lower prices per pound than the phenolic type, they could compete with plastic microspheres for applications using the bulk powder, and also as a resin filler for commercial foams.

Electrical and specialty-grade microspheres of the glass type are made from specially formulated and processed glasses. Compositions consisting of over 95% pure silica are available (Eccospheres SI), as well as surface-treated types for maximum compatibility with bonding resins (Eccospheres VT).

Manufacture. Plastic microspheres are prepared from a solution of the plastic in a volatile solvent containing a blowing agent (qv). The solution is introduced at the top of a spray-drying chamber. Solvent evaporation in the drying process produces a tough skin on the tiny droplets of spray. Simultaneously, an entrapped blowing agent is released within the sphere. Pressure of the entrained gas prevents collapse of the spheres.

In making glass spheres the proper components are mixed, dried, pulverized, and screened. Particles of the appropriate screened fraction are subjected to the blowing reaction by introduction at the bottom of a chamber into an ascending column of hot furnace gas. Further screening, purification, and subsequent chemical treatments produce the various specialty- and electrical-grade products.

Uses. Phenolic microspheres are used in the bulk powder form for control of oil evaporation. When poured on the surface of crude oil the spheres spread out in a ¾ to 1 in. layer to form a vapor barrier. Phenolic spheres in crude oil service have

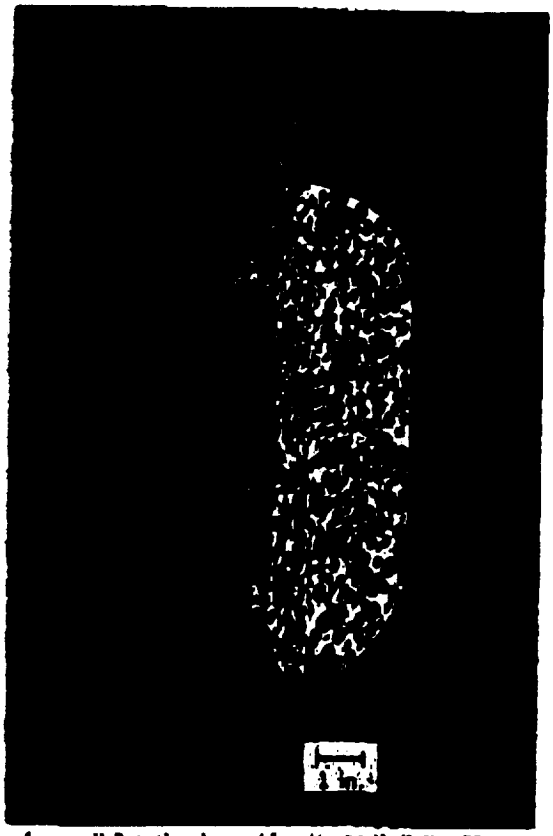

Fig. 1. Cutaway view of a small flotation buoy (density 30 lb/ft³). Hollow epoxy spheres are embedded in a syntactic foam based on glass microspheres. Courtesy Emerson & Cuming, Inc.

been observed to reduce up to 90% of normal evaporation. Savings in evaporation losses of more than two million dollars in ten years have been reported at a single tank farm.

A primary aspect of the use of microspheres as a filler in plastics technology is that they yield products of reduced density. These products, termed syntactic foams, are true foams, but they have some unique qualities, both in structure and in properties. Foams incorporating microspheres have closed, microscopic-size cells, and are outstandingly uniform in structure. Density can be controlled accurately according to the proportion of microspheres added to the resin mix. Compared with conventional blown or frothed foams they have markedly greater strength, particularly under compressive load. Formulations containing microspheres are supplied either as casting or molding compounds for processing by conventional techniques or as mixtures with the consistency of damp sand, which can be tamped into place and cured. Epoxy, silicone, polystyrene, and phenolic resins are the common matrixes.

General-purpose applications of microsphere-based foams employ large quantities of industrial-grade plastic and glass types. As a structural void filler and buoyancy material in submarines, ships, and small boats, the foam prevents water from collecting in nonfunctional void spaces. In addition to the buoyancy effect, the foam helps prevent corrosion of metal craft. Microsphere foams are used in cores of sandwich

Table 1. Properties of Electronic-Grade Microspheres

	Eccospheres R[a]	Eccospheres SI[a]	Eccospheres VT[a]
bulk density, lb/ft³; g/cm³	14; 0.22	11; 0.18	11; 0.18
true particle density, lb/ft³; g/cm³	26; 0.42	17; 0.28	17; 0.28
particle size, μ	30–300	30–125	30–125
wall thickness, μ	about 2	about 2	about 2
composition	borosilicate glass	over 95% SiO_2	SiO_2 plus coating
temperature capability, °F	1000	2500	600
thermal conductivity of loosely packed material, Btu/hr/ft²/ft	0.04	0.03	0.03
dielectric constant (dry), 1×10^6 to 8.5×10^9 cps (approx)	1.3	1.2	1.2
dissipation factor (dry), 1×10^6 to 8.6×10^9 cps (approx)	0.001	0.0005	0.0005

[a] Trademark of Emerson & Cuming, Inc.

structures, sheets, and panels for decks, rudders, and cabin tops of pleasure craft; in subflooring of airplanes (eg, Douglas DC-8); and in the construction field for thermal insulation as wall, roof, and floor panels. A light-weight fabric of asbestos filler and glass microspheres is used in making resin laminates having a thermal conductivity less than half that of conventional materials (Johns Manville Corp.).

Deep-water floats for oceanographic research use glass-microsphere foams with controlled density (Fig. 2). A 42 lb/ft³ foam with an epoxy resin binder passes tests at 20,000 ft depth and 10,000 psi compression.

Electronic and aerospace applications embody special-property glass and silica microspheres in casting resins, syntactic foam formulations, and dielectric materials with "tailored" properties. Low density, low dielectric loss, high temperature capability, ruggedness, and stability of properties are among the requirements for the finished foams. See also ENCAPSULATING.

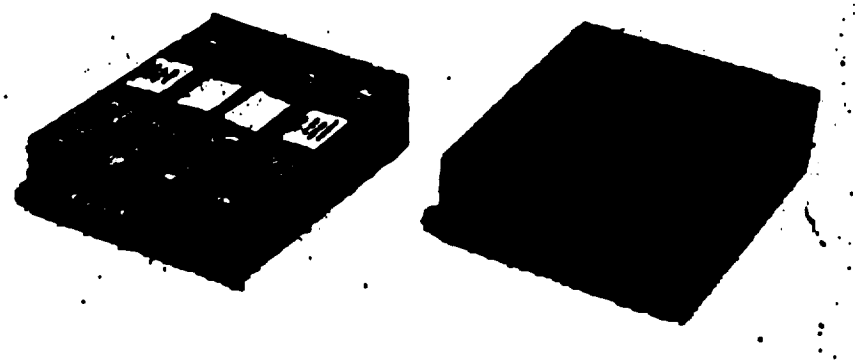

Fig. 3. Typical electronic circuit for Midas III satellite before and after encapsulation in glass microsphere-filled epoxy resin (Stycast 1090) (5).

Table 2. Foams Based on Hollow Glass Microspheres

	Epoxy resin-bonded[a]	Polystyrene-bonded[a]	Silicone resin-bonded
weight, lb/ft³	23	39	36
dielectric constant, 10^3 to 10^{10} cps	1.55	1.67	1.6
dissipation factor, 10^3 to 10^{10} cps	0.01	0.001	0.002
compressive strength, psi	1800	6000	750
operating temperature range, °F	−70 to +500	−70 to +350	−70 to +800
commercial designation	Eccofoam *DPT*	Styrast LoK	Eccofoam LM

[a] Crosslinked.

The properties of a selection of microspheres for electronic applications are given in Table 1.

Properties of typical glass microsphere (Eccospheres R) foams based on three widely used resin systems are shown in Table 2. Notable are the reduction in density

Fig. 6. Microwave lens from glass-microsphere foams. Left, and center, artificial dielectric elements (Eccofoam Hik 616D); right, finished lens housed in matched-property syntactic foam. Courtesy Emerson & Cuming, Inc.

to about half that of the corresponding solid plastic, low dielectric constant, and low dissipation factor. The use of silica microspheres results in foams of still lower dielectric loss and higher temperature capability. Encapsulation of circuitry of a Midas satellite (5) is shown in Figure 3.

Artificial dielectric foams with dielectric properties tailored to cover a broad range of specifications for electronic and microwave applications, from ultra-low-loss materials with dielectric constant close to that of air to high-loss, conductive, electromagnetic energy absorbers, are made by incorporating metal, carbon, or ferromagnetic particles in the microsphere foam. These light-weight dielectric materials with adjusted properties find application in microwave lenses, radomes, electromagnetic windows, dielectric supports, microwave absorbers, waveguide loads, and antennas (Fig. 4). See also ELECTRICAL APPLICATIONS.

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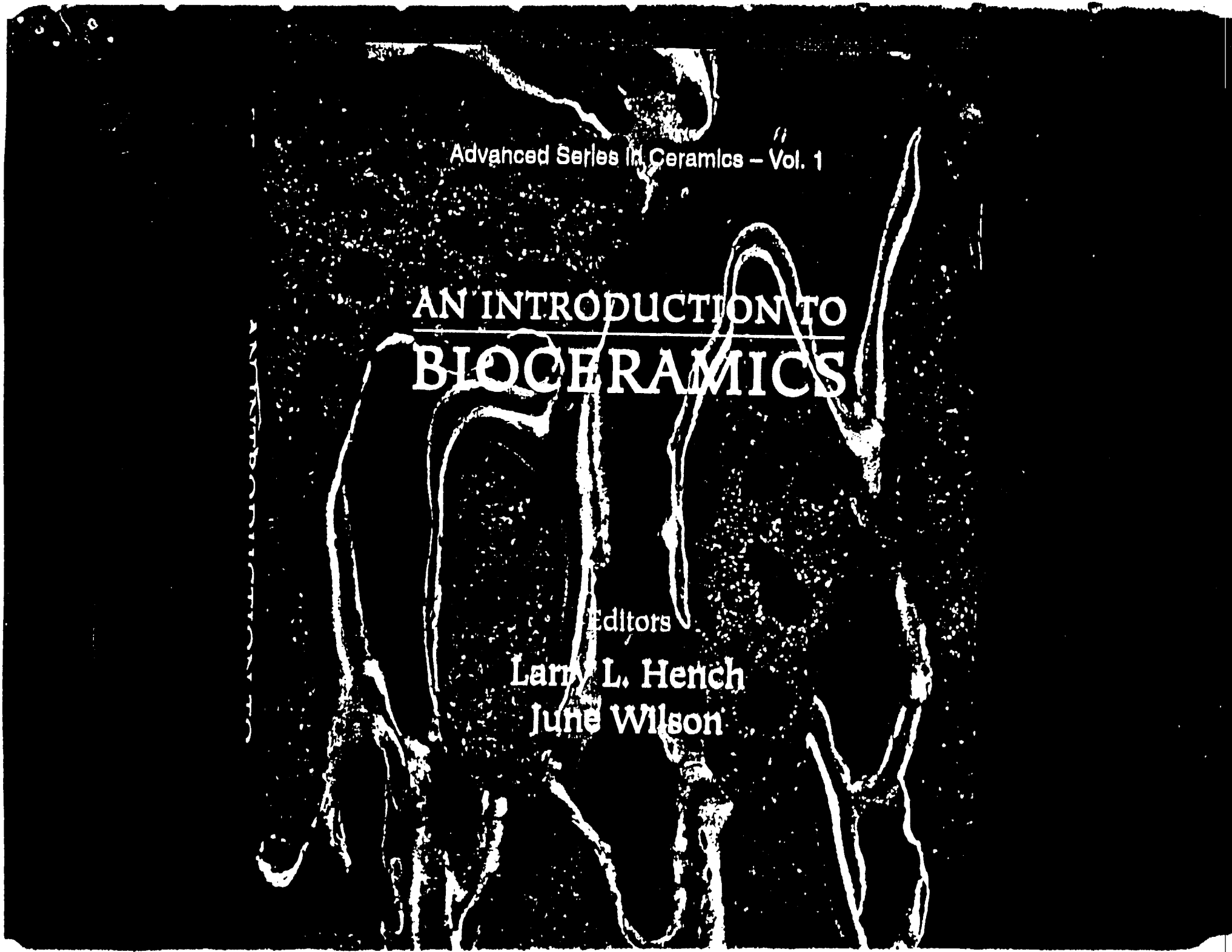
MICROTACTICITY

This article deals with the experimental characterization of stereoregular polymers and, more specifically, with the determination of microtacticity.

Almost concurrently with the establishment of the macromolecular hypothesis due to Staudinger, the important effect of stereochemistry on polymer properties was perceived by Meyer and Mark (1), who studied natural rubber and gutta percha. In spite of this early demonstration of the influence of geometrical isomerism, and the subsequent formulation of the relation between stereoisomerism and the development of crystallinity (2), over twenty five years passed before procedures were developed for producing α-olefin, vinyl, and vinylidene polymers having regular configurations of the substituents about the asymmetric carbon atoms. This belated development is rather surprising, since the prevalence of head-to-tail enchainment had been experimentally established (3) by the mid-1940s, and the failure of many polymers to crystallize had been correctly ascribed to the random sequence of asymmetric carbon atom configurations along the chain (4). In the post-war years little attention was paid to developing stereoregular order per se, though Schildknecht and co-workers (5) showed that poly(vinyl ethers) could be prepared which displayed either rubbery or crystalline behavior, and discussed these results in terms of isomerism (6). However, the spectacular preparation by Natta et al. (7) of highly crystalline polymers using Ziegler catalysts unleashed a wave of interest in stereospecific polymerizations. The creation of this new class of linear polymers stimulated considerable activity in polymer characterization and in the study of structure–property relationships, as described in a recent review (8), and with which this article will be concerned. Details concerning stereospecific polymerization conditions and mechanisms can be found in an excellent review article of Bawn and Ledwith (9) and in the appropriate articles in this Encyclopedia, eg. COORDINATE POLYMERIZATION; STEREOREGULAR–LINEAR POLYMERS; ZIEGLER–NATTA CATALYSTS.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Chapter 17

RADIOTHERAPY GLASSES

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INTRODUCTION

Radiotherapy glasses are defined as radioactive glasses used for *in situ* irradiation, beta or gamma radiation, of targeted organs inside the body. Glasses used for this purpose must not only be biocompatible, but also chemically insoluble in the body during the time that the glass is radioactive to prevent the unwanted release of the radioisotope from the targeted site. The development of radiotherapy glasses was motivated by the need to deliver large (> 10,000 rads), localized doses of beta radiation to diseased organs in the body in such a way as to minimize, and ideally avoid, damage to adjacent healthy tissue. Irradiating malignant tumors inside the body by external beam radiation is limited in several important ways. A major limitation is that the maximum dose which can be safely delivered is constrained by the need to protect surrounding healthy tissue and is usually too small ($\leq$ 3,000 rads) to be therapeutic. Furthermore, external irradiation with energy radiation such as gamma, which is needed (often causes damage to healthy tissue) . The lower energy beta radiation is not well suited for delivery by external means because its smaller range in tissue may be too small for it to reach the target site. Since beta radiation is preferred in many cases, a means of *in situ* radiation would be advantageous.

For use as *in vivo* radiation delivery vehicles, radiotherapy glasses should be (1) biocompatible and nontoxic to the body, (2) chemically insoluble during the time the glass is radioactive, and (3) have high chemical purity. Aluminosilicate glasses containing yttrium and rare earth (RE) cations such as Sm, Ho, Re, and Dy satisfy these criteria. Furthermore, they have the advantage that radioisotopes such as Y-90, Sm-153, and Ho-166, can be made by neutron activation as the last step in the manufacturing process so that the glass can be manufactured in the normal way avoiding the handling of radioactive materials.

Yttrium aluminosilicate (YAS) glasses have been successfully used in clinical trials for more than five years. This is the only commercial use at this time, and uses YAS glass microspheres, containing Y-90, to irradiate malignant tumors in the liver. Depending upon the size of the liver, the desired dose, and the diameter of the microspheres, from 1 to 15 million microspheres of radioactive YAS glass, 15 to 35 μm in diameter, are injected into the hepatic artery which is the primary blood supply for the target tumors. The microspheres are sized so that the blood carries them into the

capillary bed of the liver, but they are too large to pass completely through the liver and enter the circulatory system. Since the distribution of the radioactive microspheres follows the blood flow, the microspheres will concentrate in the tumor which has a greater than normal blood supply, and irradiate the tumor with β rays. In one case, 80% of the dose reached the tumor. Since Y-90 has a half life of 64.1 hrs, the radioactivity decays to a negligible level in about 21 days.

Radiotherapy glasses can be made in a variety of shapes, such as irregular particles, fibers, or spheres. Microspheres are the present shape of choice since the diameter can be carefully controlled and the smooth spherical surface helps with easy delivery of the particles to the target.

The RE aluminosilicate glasses are currently being evaluated for applications such as the irradiation of diseased kidneys prior to surgical removal, radiation synovectomy of arthritic joints, and the irradiation of malignant tumors in the liver. This chapter focuses on YAS glass microspheres which have been in commercial use in Canada since 1991.

PROCESSING

A typical manufacturing sequence for preparing radiotherapy glass microspheres is given in Fig. 1. The first step is the melting of a homogeneous mixture of high purity powders, such as Y_2O_3, Al_2O_3, and SiO_2, in a platinum crucible. Melting typically occurs at 1550 to 1650°C for the RE aluminosilicate compositions inside the glass forming areas depicted in Fig. 2. After melting, the chemically homogeneous melt is quenched to room temperature and crushed to a powder of the desired size. This powder is spheriodized by passing the particles through a gas/oxygen flame where each particle is melted, forms a sphere by surface tension forces, and becomes solid during cooling. The microspheres are then screened to obtain microspheres of the desired size. An example of the uniform and highly spherical microspheres made in this way is shown in Fig. 3. The final step is the irradiation of the glass microspheres with neutrons so as to form the desired quantity of radioisotope. YAS glasses are easily irradiated, forming Y-90, to a specific activity up to 5 mCi/mg of glass. After irradiation the microspheres are ready for packaging and shipment. Naturally, it is important that high purity raw materials, free of neutron activatable impurities, be used and that care be taken during the various manufacturing steps to avoid chemical contamination of the glass.

In addition to preparing glasses by conventional melting as just described, YAS glasses have been made by sol-gel processing. Property measurements made on a sol-gel derived YAS glass indicate that it should be acceptable for human use.

COMPOSITIONS

As evident from Fig. 2, glasses can be obtained from a wide range of Y, Sm, and Ho aluminosilicate compositions which melt below 1600°C. At this time, these are the only RE aluminosilicate systems where the boundaries for glass formation have been determined. However, glasses have been prepared from isolated aluminosilicate

1. GLASS MELTING

- **a.** Select chemically pure raw materials (oxides) which do not contain any impurities that would form undesirable radioisotopes during neutron irradiation.
- **b.** Mix raw materials to form a homogeneous mixture of powders.
- **c.** Melt raw materials to form homogeneous glass.

2. SPHERIODIZATION (MICROSPHERE FORMATION)

- **a.** Crush glass to particles of desired size.
- **b.** Inject particles into gas-oxygen flame to melt each particle and form solid glass sphere (flame spray powder).
- **c.** Collect microspheres in suitable container.

3. SIZING -- screen or separate microspheres into desired size range.

4. NEUTRON ACTIVATION -- irradiate microspheres in nuclear reactor (several days) until desired level of radioactivity is achieved. Package microspheres for delivery to physician.

Fig. 1. Steps in manufacturing radiotherapy glass microspheres.

compositions which contain ReO_2, Dy_2O_3, or Er_2O_3. Since a large range of β-emitting RE radioisotopes can be incorporated into aluminosilicate glasses, it is possible to select one which is best suited to the particular type and size of the target organ. This compositional flexibility is an inherent advantage of radiotherapy glasses. In cases where some amount of gamma radiation is desired, neutron activatable gamma emitting radioisotopes, such as Na-24, K-42, or P-32, can also be incorporated into the aluminosilicate glass matrix.

An aluminosilicate glass is well suited for radiotherapy use since (a) no unwanted radioisotopes are formed by the neutron activation of Al, Si, or O, (b) these glasses have a high chemical durability, being essentially insoluble in the body, (c) microspheres with a high specific activity can be easily obtained because of the large amount (40 to 70 wt%) of RE oxide which can be present in the glass, (d) homogeneous melts can be prepared at reasonable temperatures (< 1600°C), and (e) particles of the glass are easily spheriodized in a flame because of the viscosity characteristics of the glass.

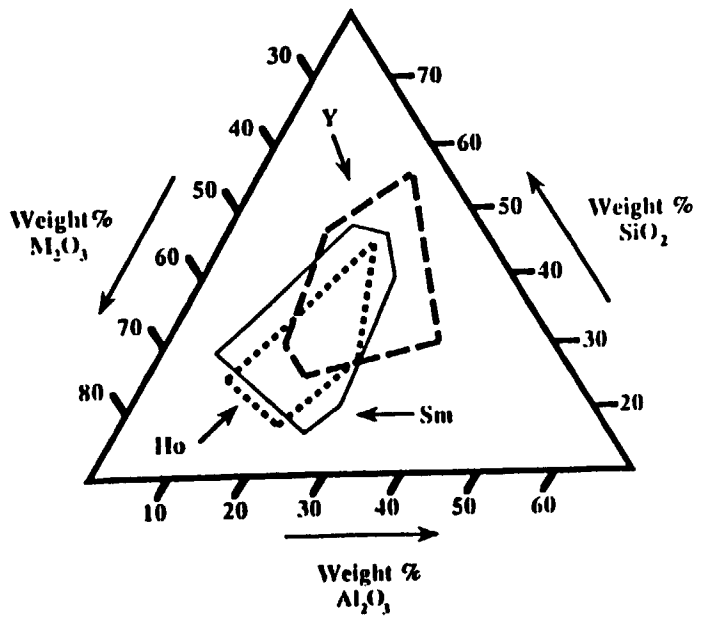

Fig. 2. Glass formation region (< 1600°C) for Y_2O_3, Ho_2O_3, or Sm_2O_3 aluminosilicate glasses.

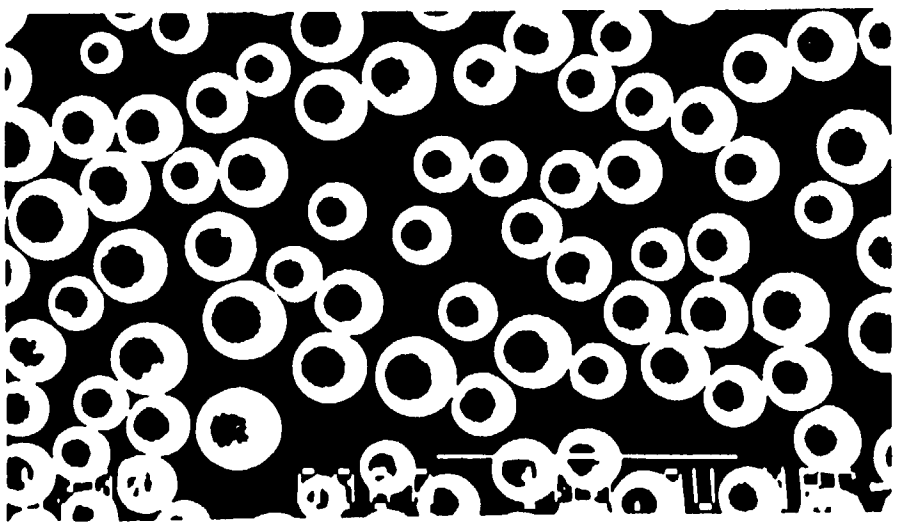

Fig. 3. Appearance of "typical" glass microspheres made according to the procedure described in Fig. 1. White bar in lower right hand corner is 100 μm.

PROPERTIES

Chemical Durability

Glasses used for radiotherapy purposes need to be highly durable during the time they are radioactive, since the means of confining the radioisotope to the target organ is to keep it inside a chemically insoluble microsphere. *In vitro* and clinical tests on radioactive YAS glasses have demonstrated their superior chemical durability. More than 100 patients have been injected with radioactive YAS glass microspheres over the past five years, with no reports of any premature or unwanted release of radioactive Y-90 in the body.

In vitro tests on a wide range of YAS glasses, containing from 9 to 30 Y_2O_3, 11 to 35 Al_2O_3, and 48 to 72 SiO_2, mol%, have shown that these glasses have an excellent chemical durability in deionized water and in saline at 37°C; this durability varies only slightly with chemical composition. An example of the small amount of yttrium released from a typical YAS glass, is shown in Fig. 4. The only data of practical interest is that for the first three weeks since the glass is no longer radioactive after that time. Slightly more yttrium is leached from YAS glasses at higher temperatures, 50°C, or in the HCl solution (both of which are used for accelerated testing), but the amount present in deionized water at three weeks, < 5 ppm/cm^2 of glass, is too small to be of concern.

A comparison of the small amount of yttrium released from a glass in either bulk form or as glass microspheres or powder is shown in Table 1. The results for microspheres, which are relevant to the use of such glasses in the body, show that little

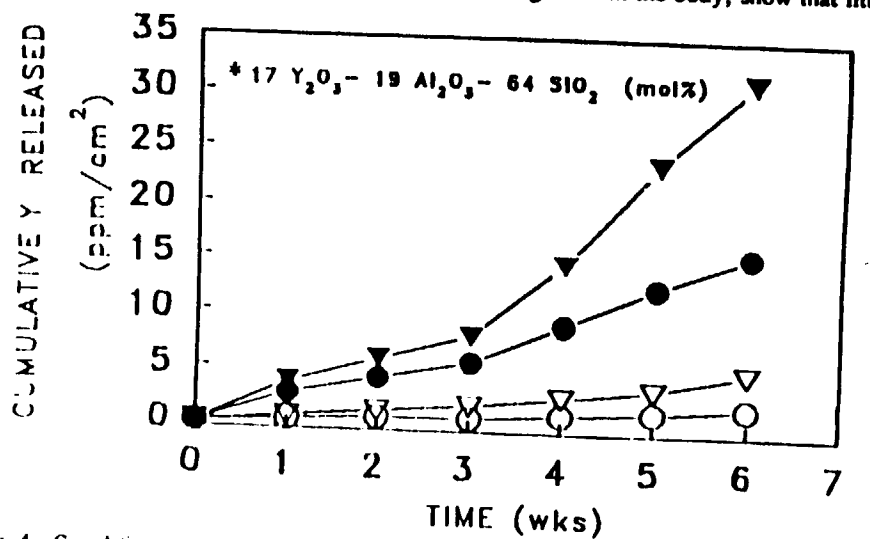

Fig. 4. Cumulative concentration of Y dissolved from YAS-4 glass and present in 100 ml of solution. Open circles and triangles are for DI water at 37 and 50°C, respectively, while closed circles and triangles are for 12M HCl (pH = 2) at 37 and 50°C, respectively. (Ref. Erbe, 1991).

Table 1. Weight Percent Yttrium Released Per gm of $17Y_2O_3$-$19Al_2O_3$-$64SiO_2$, Mol%, Glass. (Ref. Erbe 1991).

Conditions	% Y Related/gm of Glass 3 wks	4 wks
DI Water at 37°C		
CM* Bulk Glass	0.02	0.04
CM Microspheres (25 to 35 μm)	0.06	0.09
CM Powder (20 to 38 μm)	0.20	0.27
SG* Powder (20 to 38 μm)	0.11	0.20
Saline at 37°C		
CM Bulk Glass	0.03	0.07**
CM Microspheres	0.04	0.11**
SG Powder	0.81	1.19

*CM means conventionally melting glass while SG means a glass prepared by sol-gel techniques.
**Measured at 6 weeks.

yttrium is released from the microspheres or powder even though the surface area of these samples is 300 times larger than that of the bulk sample. There is no significant difference in the amount of yttrium released from microspheres tested in either deionized water or saline at 37°C.

The *in vitro* test data in Table 1 for the YAS microspheres have been used to calculate the amount of radioactive Y-90 that would be released in a patient injected with 300 mCi of Y-90. The solid data points in Fig. 5 show the calculated amount of radiation released to the body due to the very slight chemical attack (dissolution) of the YAS glass beads. The calculation takes into account the decay of the Y-90 (half life of 64.1 hrs). The solid line labeled C in Fig. 5 was calculated assuming that all of the radioactive Y-90 dissolved from the microspheres was absorbed in the most susceptible tissue, bone marrow. Even in this worse case scenario, the total dose to the bone marrow is estimated at less than 5 mrads which is roughly equivalent to a chest x-ray or about the same dose that a person living in Leadville, Colorado receives in one year from cosmic radiation. All of the *in vitro* tests to date in deionized water and saline up to 50°C indicate that the YAS glass microspheres should have an extremely good chemical durability in the body. The lack of any detectable release of radioactive Y-90 from YAS glass microspheres that have been injected into humans that is, no depression of bone marrow activity, substantiates the *in vitro* test results and demonstrates the suitability of these glasses for use in humans.

Overall, the RE aluminosilicate glasses have excellent durability in deionized water and saline, but their durability should be expected to vary somewhat with temperature and with the RE concentration and the specific RE cation in the glass. In

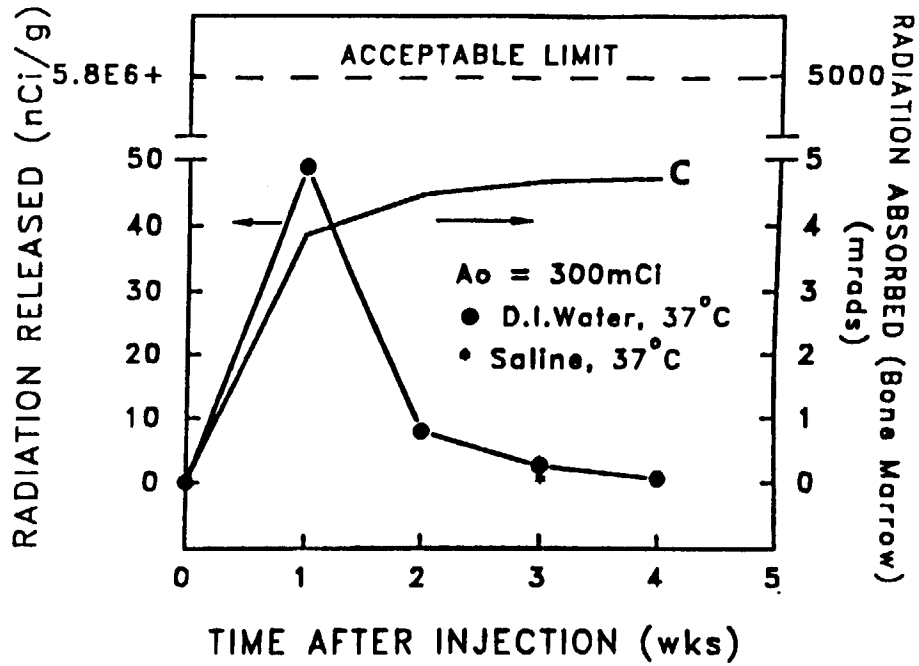

Fig. 5. Calculated amount of Y-90 radiation released (nCi/g) from YAS-4 microspheres (25 to 35 μm) immersed in (•) DI water (pH 6.9) at 37°C for up to 4 wks or in (*) isotonic saline (pH 6.2) at 37°C for 3 wks. Calculated from data (Table 1), assuming an initial injected dose of 300 mCi and taking into account the decay of the radioactive Y-90. Curve C represents cumulative absorbed dose (mrads) assuming that all radiation from released Y-90 is absorbed by bone marrow. (Ref. Erbe, 1991).

general, the durability in deionized water or saline tends to decrease slightly with increasing concentration of the RE cation in the glass as shown in Fig. 6 where slightly more yttrium is dissolved from YAS glasses of higher yttrium content, YAS-9 and -11 contain 27.4 and 30 mol% Y_2O_3, respectively, while the YAS-4 glass contains 17 mol% Y_2O_3. Samarium aluminosilicate (SmAS) glasses are also highly durable in deionized water at 37°C, their dissolution rate ranging from about 30×10^{-9} gm/cm^2/min to 2×10^{-9} gm/cm^2/min, which is quite similar to the dissolution rate for YAS glasses. While SmAS glass microspheres have not been used in humans at this time, the chemical durability of these glasses is considered acceptable for human use and there has been no reported release of Sm-153 from SmAS glass microspheres injected into the kidneys of rabbits.

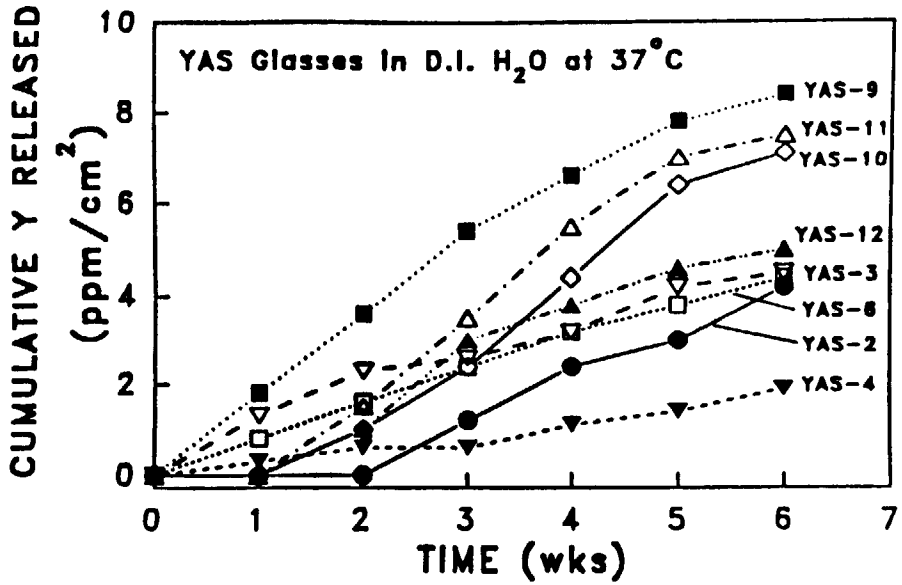

Fig. 6. Cumulative concentration (ppm) of Y released into 100 ml of DI water at 37°C per surface area (cm^2) of bulk glass. The YAS-4, YAS-12, and YAS-9 glasses contain 17.0, 30.0, and 27.4 mol% Y_2O_3, respectively. Experimental error ± 2.0 ppm. (Ref. Erbe, 1991).

The excellent chemical durability of RE aluminosilicate glasses is attributed to the absence of alkali and alkaline earth oxides in these glasses, which typically lower the chemical durability of silicate glasses, and to the presence of small highly charged cations which can form strong chemical bonds with oxygen. The RE aluminosilicate glasses have a strongly bonded, three dimensional network structure which is not easily attacked by aqueous solutions having a pH between 6 and 8. *In vitro* measurements of the chemical durability of Y, Sm, Ho, and a few Re aluminosilicate glasses indicate that most RE aluminosilicate glasses should have a chemical durability satisfactory for *in vivo* use.

Density and Refractive Index

Since the molecular weight of the rare earth oxides is much higher than that of Al_2O_3 and SiO_2, the density of the RE aluminosilicate glasses depends primarily on the concentration of the RE oxide. As shown in Fig. 7, the density of YAS glasses ranges from about 2.8 gm/cm^3 at 10 mol% Y_2O_3 to about 4.0 gm/cm^3 for glasses containing 30 mol% Y_2O_3. Comparable SmAS glasses have a higher density, ranging from about 3.4 to 4.6 gm/cm^3, which is consistent with the higher molecular weight of Sm_2O_3.

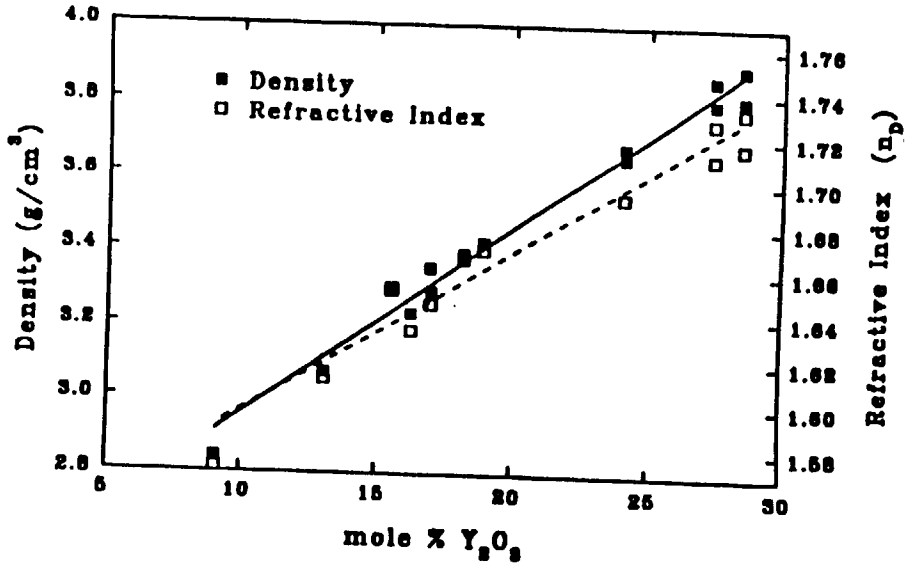

Fig. 7. Density (solid line) and refractive index (dashed line) of YAS glasses. Lines are least squares fit to data points.

The density of the RE aluminosilicate glasses is obviously considerably higher than that of blood, but this has not caused any problems in the injection of these glasses into humans or test animals. During injection, precautions are necessary to insure that the microspheres do not settle out of solution, but simple agitation is adequate to keep the microspheres suspended.

The refractive index of the glasses is not important to their use for radiotherapy purposes, but this is another property which depends primarily upon the concentration of the RE oxide, see Fig. 7. The relative amounts of alumina and silica in these glasses is of lesser importance to properties such as refractive index, density, and thermal expansion coefficient.

CLINICAL RESULTS

The tissue response to radiotherapy glasses varies according to the tissue being irradiated and clinical data relating tissue response to radiotherapy glasses exists only for the liver and kidney. This chapter discusses effects only in the liver.

The liver is referred to as a reverting post-mitotic cell type. This means that the liver does not normally divide or renew itself, as does the skin or lining of the digestive tract, but has the capability to do so. If the capacity of the liver to function is decreased

by some type of injury (chemical, trauma, etc.), it will be stimulated to renew itself in order to maintain normal body function.

Liver tumors, like other tumor types, undergo rapid mitotic division and are highly sensitive to ionizing radiation. All tissues that are rapidly dividing cell types are extremely sensitive to the effects of ionizing radiation. This is due to the large percentage of the time that the cells' genetic material is condensed in the nucleus. Ionizing events reaching the condensed genetic material in the nucleus of the cell will lead to cell death. Since the normal liver is not usually in a dividing state, it is resistant to the effects of low to moderate levels of ionizing radiation.

The "average" doses that have been delivered with radiotherapy glasses to liver tumors have ranged from 5,000 to 15,000 rads. This would normally be considered a large human dose and it is difficult to predict the exact dose delivered to just the tumor. As previously mentioned, the distribution of the glass microspheres in the liver is believed to depend on the blood flow. Many hepatic tumors are classified as hypervascularized, which means that the blood flow to the tumors exceeds that to the normal surrounding tissue, and, consequently, a larger than normal fraction of microspheres will be transported and deposited in the tumor. This increases the radiation dose to the tumor while minimizing the exposure of the surrounding normal tissue. This localization of the radiation explains why patients, treated with radioactive Y-90 glass microspheres can tolerate much higher doses than those treated by whole liver radiation methods.

One study used dogs as a model for hepatic arterial injection of both non and radioactive YAS glass microspheres. Doses exceeding 30,000 rads were delivered to the livers of these dogs. Delivered doses of non and radioactive microspheres (143 to 562 mg) were measured in units of mCi per gm of liver tissue and ranged from one to twelve times the anticipated human dose. The dogs were grouped by varying dose levels and a control group that received nonradioactive microspheres was used to determine the physical impact of the microspheres alone on liver function.

Doses of nonradioactive microspheres delivered were up to six times that of the anticipated human dose. Minimal changes were detected, such as changes within the walls of the central veins, in the appearance of the hepatocytes, and in the tissue architecture. Hepatocellular function and damage were within normal limits. There were no signs of portal fibrosis or cirrhosis.

Changes seen in the liver injected with radioactive YAS microspheres, were similar to the findings of other irradiation studies in dog liver. These changes included histologic changes in the portal areas of the liver. Doses as high as 35,000 rads were delivered, but did not cause total necrosis and were judged by clinical standards as compatible with survival. Doses up to 15,000 rads were well tolerated and showed little change in liver function. No microspheres were found in the bone marrow. Even in dogs receiving more than 15,000 rads, no bone marrow suppression occurred. At doses above 25,000 rads, the consequences of hepatic cirrhosis would probably pose significant problems.

In a preliminary study, a transient increase in body temperature has been noted, but this lasted only a few days. In some patients with a history of previous liver disease (chronic alcoholism), ulcerations of the lower stomach and upper small intestine have occurred. When treated, these conditions were self-limiting. In almost all patients receiving YAS radiotherapy glasses, liver enzymes were mildly elevated. This effect was not dose-related and lasted from a few days to weeks.

Clinical applications of radiotherapy glasses have been on liver and kidney tumors. Most of this work has been with liver tumors, since patients with liver cancer can enter the terminal phase within four to six months of diagnosis. This has sparked a major effort in investigating ways of delivering ionizing radiation *in vivo* to treat these malignant tumors.

Work is currently underway to discover whether very large doses delivered to the kidneys will reduce the shedding of malignant cells, which can spread the tumor, during surgical removal of a diseased kidney.

In summary, any tissue that is relatively insensitive to low or moderate amounts of ionizing radiation in which these unique microspheres can be deposited, by either the blood flow or surgical implantation, is a potential candidate for this new form of radiotherapy.

SUMMARY

Rare earth aluminosilicate glass microspheres have proved to be well suited for radio-therapeutic use in humans. These microspheres provide a new and unique method of irradiating diseased internal organs with beta radiation, in amounts which exceed those that can be delivered by other means. YAS glass microspheres have been safely used for more than five years to irradiate, up to 15,000 rads, malignant tumors in the liver in more than 100 patients. Since the glass microspheres tend to distribute themselves in the liver in proportion to the blood flow, the actual dose to the tumors is believed to be much larger than the average dose to the entire liver since the microspheres tend to concentrate in the tumors, because of their vascularity. In one case, 80% of the YAS microspheres were estimated to lodge in the tumor vascular bed, giving and estimated dose of 32,000 rads. Samarium aluminosilicate glass microspheres have been used to irradiate, up to 15,000 rads, the kidneys in rabbits without any harmful side effects or detectable damage to adjacent tissue.

A major advantage of the RE aluminosilicate glass microspheres is excellent chemical durability in the body. Since the glasses are insoluble in body fluids, the radioactive RE isotope is confined to the target organ and prevented from entering the circulation. The maximum time which these glass microspheres will remain in the body is currently unknown, but YAS microspheres have been in one patient for more than four years with no reported problems. If needed, it should be possible to develop RE containing glasses which will gradually degrade in the body when they are no longer radioactive.

The use of RE aluminosilicate glass microspheres is still at an early stage in treating liver cancer, but results are promising. The currently recommended dose range

is 8,000 to 15,000 rads. In one group of 39 adenocarcinoma patients treated with 5,000 to 11,000 rads in a phase I-II study, the average survival time was 9.7 months from the date of treatment with the microspheres. This compares with a median survival time of 12 months from the time of diagnosis for patients treated with conventional chemotherapy. All of the patients treated with the YAS glass microspheres had undergone one chemotherapy treatment and diagnosis may have occurred several months prior to injection with the YAS microspheres. Treatment with glass microspheres takes about one hour, followed by a few hours in the hospital for observation; treatment as "day-patient". Chemotherapy requires several repeated treatments over several weeks. Increased liver enzymes are a common side effect following treatment with the glass microspheres, and transient fever, increased pain, and nausea and vomiting are less common side effects.

Radiotherapy glass microspheres are being considered for treating other diseases and other types of cancer. Using radiotherapy glasses to kill cancer cells in diseased kidneys prior to surgical removal has already been mentioned. Ideally, it should be possible to use radiotherapy glass microspheres to irradiate any diseased organ having a capillary bed. The *in situ* irradiation of arthritic joints with beta emitting RE aluminosilicate glass microspheres is also under study. The stifle joints in rabbits have been injected with usable quantities of glass microspheres without any noticeable physical damage to the joint for periods up to one year. In rabbits the glass microspheres were found imbedded in a layer of the synovial tissue. In this application, the radioactive glass microspheres were used to perform a radiation synovectomy of the diseased joint

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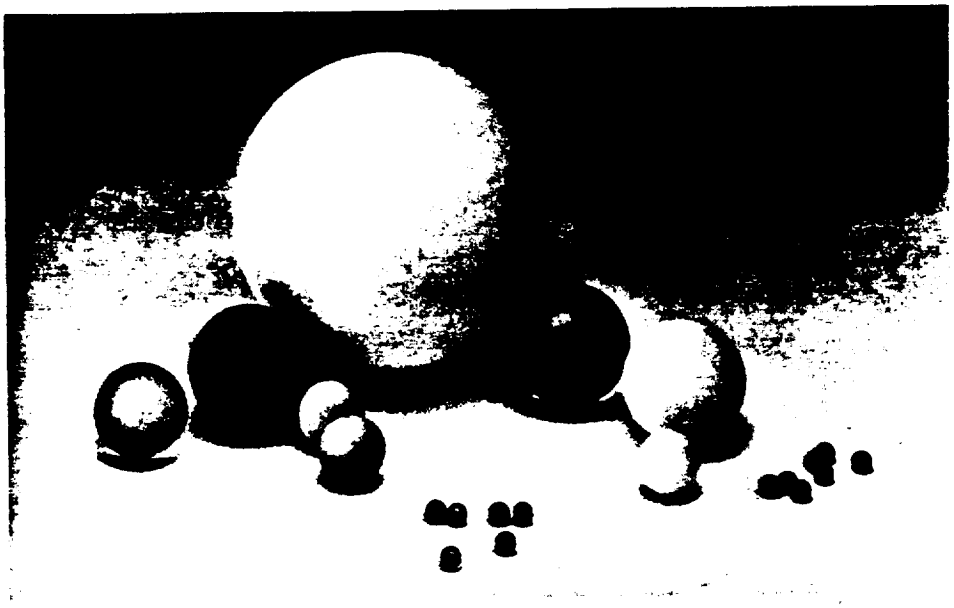
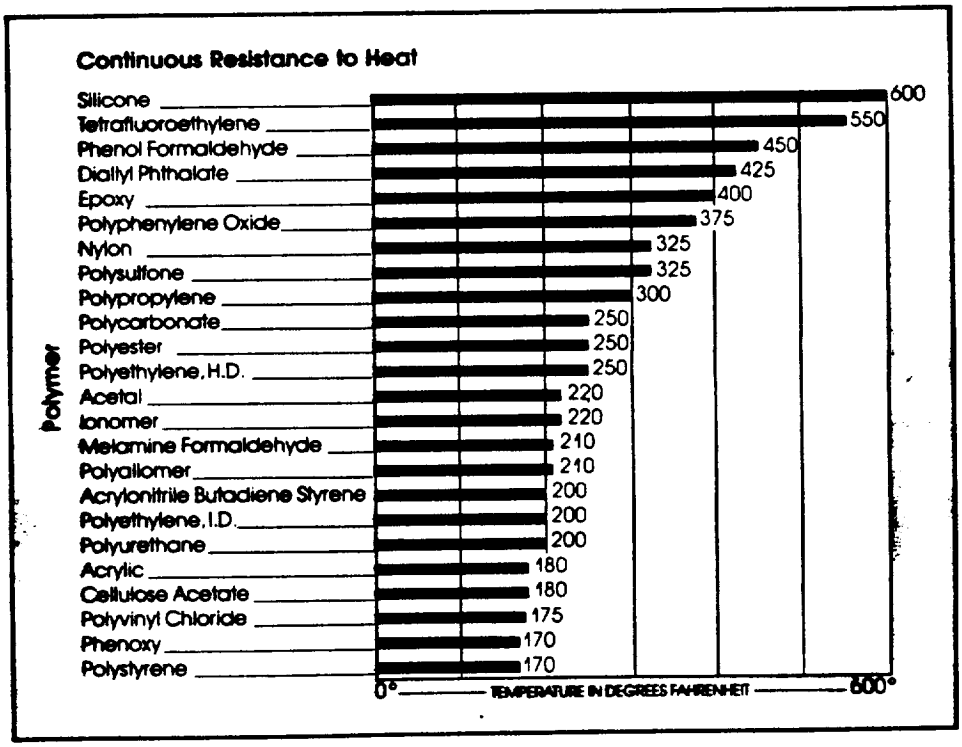
Selection and Application Guide

Plastic, Glass and Ceramic Balls

Plastic Balls





More and more design engineers have been specifying plastic balls to take the place of more expensive materials, such as stainless steel. Let's take a look at what's to be gained by designing with plastic balls.

First of all, plastic balls resist corrosion and abrasion very well. They make durable components even in highly corrosive environments, and compared to metals having comparable corrosion resistance, plastics are generally less expensive.

Next, you can choose from a wide range of materials, including common resins and new engineered plastics. Each performs differently, so you can specify the polymer that best fits your application. And your budget.

If your design is weight-sensitive, plastic balls are ideal. They're much lighter than metal balls.

Finally, many plastics are extremely resistant to heat. Silicone, for example, withstands temperatures up to 600°F and tetrafluoroethylene withstands almost equally high temperatures. The bar graph at left shows the degree of continuous heat resistance you can expect from different plastics.

Now that we've touched on the basic design advantages plastic balls offer, let's see what types of applications suit them best.

Plastic balls are ideal for light-load bearings and flow control applications. Here's why.

Plastic balls have low friction and require virtually no lubrication. Also, since plastic balls are quiet, they're often used in office furniture, bearings, medical products and

(continued on page 4)

Polychloro-trifluoro-ethylene	Phenolic, Wood Flour Filled	Polyamide (Nylon)	Poly-carbonate	Polyethylene High Density	Polyethylene Low Density	Polypropylene (Impact)	Polystyrene
3.5	0.24-0.34	0.9-2.0	8-16	1-10	No break	1.0-7.0	0.25-0.70
6	6.5-8.5	8.5-11	9-10.5	2.5-5.0	1.1-1.7	3.3-5.2	5.5-8.0
150	800-1200	210-410	320	85-160	14-38	100-170	400-500
28-36	–	60-300	60-100	5-10	20-40	50-550	1-2.5
8	8-12	14.6	11-13	2-3	–	5.5-10.0	8-15
175	800-1200	210-410	375	90-150	–	175	400-500
12	24-36	13	11	–	–	4.0-8.0	11.5-16
180	600-1000	–	240	50-100	–	–	300-560
–	290-340	150	280-290	–	–	2.0	175-195
390	300-350	325	250-275	250	200	290-320	150-170
7	3.0-4.5	10	7	15-30	15-30	4.0-8.5	6-8
6	4-7	5.8	4.6	8	8	0.048-0.098	2.4-3.3
10^{18}	10^{11}-10^{13}	4.5×10^{13}	2.1×10^{16}	$>10^{15}$	$>10^{15}$	6.5×10^{16}	10^{17}-10^{21}
2.65	5.0-9.0	3.9-7.6	3.17	2.3	2.3	2.2-2.3	2.5-2.65
450	200-425	385	400	480	480	450-660	500-700
0.015	0.04-0.30	0.01-0.09	0.0009	<0.0005	<0.005	0.0005	0.0001-0.0005
>360	5	140	10-11	melts	melts	185	60-100
nil	0.3-0.8	1.5	0.3	<0.02	<0.02	0.01-0.03	0.03-0.05
R112	M100-120	R108-118	M70, R118	R30-50	R10	R45-95	M70-80
nil	self-exting.	self-exting.	self-exting.	slow burning	slow burning	slow burning	0.5-2.0
2.1	1.32-1.55	1.14	1.2	0.94-0.96	0.91-0.92	0.90-0.92	1.05-1.08
red	brown-black	off-white	clear	milky white	milky white	milky white	clear
limited	blue & brown	unlimited	unlimited	unlimited	unlimited	unlimited	unlimited
opaque	opaque	translucent to opaque	transparent	translucent to opaque	translucent to opaque	translucent	transparent
Kel-F	Plenco	Zytel	Lexan	Super Dylan	Dylan	Himont	Styron
	Valite	Vydyne	Merlon	Marlex	Petrothene	Dypro	Lustrex
		Nylatron		Fortiflex	Eastman	Tenite	Dylene
				Dow	Northern Pet		
				Norchem	Norchem		
				U.S.I.	Dow		

Glass Balls

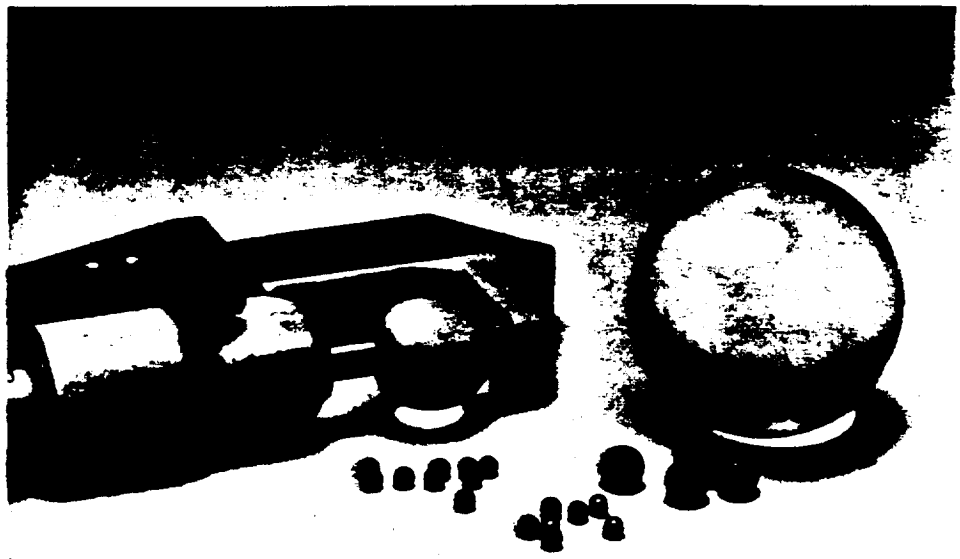
When it comes to low cost flow control and high heat applications, design engineers find glass balls hard to beat. For good reason.

Glass balls are dimensionally stable. They resist corrosion and chemical absorption well. Plus, they can withstand temperatures up to 600°F.

Glass balls also vary in density, depending on the type of glass they're made from. They are widely used in applications requiring a specific gravity.

Let's take a look at why these unique characteristics make glass balls ideal for flow control, instrumentation and fiber optic applications.

Food processing, pharmaceutical, and photographic processing equipment engineers select glass balls for check valves because they provide



Glass Properties	Soda-Lime	Borosilicate	'Black Glass'
Density: gm./cm.^{3}	2.47	2.23	2.64
Hardness: Knoop-KHN_{100}	465.0	418.0	405.0
Softening Point: °C.	695.0	820.0	650.0
Maximum Working Temperature* (annealed glass) Normal °C.	110.0	230.0	110.0
Extreme °C.	460.0	490.0	380.0
Young Modulus: 10^6 lb./5 sq. in.	10.0	9.1	9.8
Poisson's Ratio	0.24	0.20	0.21
Thermal Expansion cal. cm./300°C. - 10^{-7} in./in./°C.	92.0	33.0	89.0
Thermal Conductivity cal. cm./cm.^{2} sec. deg. C.			
- 148°F./- 100°C. (x 10^{-3})	1.99	2.13	—
+32°F./0°C. (x 10^{-3})	2.43	2.71	—
+212°F./+100°C. (x 10^{-3})	2.65	3.12	—
Thermal Stress Resistance	17°C.	53°C.	18°C.
Dielectric Properties at 1 MHz - 20°C.			
Power Factor %	0.9	0.5	0.17
Dielectric Constant	7.2	4.6	6.3
Loss Factor	6.5	2.6	1.1
Log_{10} of Volume Resistivity: ohm-cm.			
25°C.	12.4	15.0	—
250°C.	6.4	8.1	8.9
350°C.	5.1	6.6	7.0
Refractive Index Sod. D Line (.5893 microns)	1.512	1.474	1.507

*Mechanical considerations only.

NOTE: The physical properties will vary between raw glass manufacturers.

Ceramic Balls

Just as there are thousands of plastics available, ceramics are also available in a host of specific compositions and formulations. Each has its own distinct properties, but in this section we'll only look at the general characteristics of ceramic balls.

As a general rule, ceramic balls resist corrosion and abrasion extremely well. Plus, they have low thermal conductivity.

Ceramic balls also have excellent resistance to heat. Some ceramics, like ruby sapphire, withstand extended exposure to temperatures in excess of 3250°F.

Combined, these features make ceramic balls great for flow control and bearing applications.

As check valves, ceramic balls resist wear and corrosion, within limits. (Don't use ceramics with either strong acids, such as hydrochloric or hydrofluoric, or with strong alkaline solutions.)

Some ceramic balls are used in flowmeters where the ball position on a scale indicates flow rate. Ruby

sapphire balls are ideal for these applications because of their red opaque color. Ruby sapphire balls are also used in gaging devices because of their great resistance to wear and their minimal thermal expansion.

A low coefficient of thermal expansion makes other ceramics attractive as alternatives to metals in certain bearing applications. Compared to steel, their coefficient of thermal expansion is just 25%. So, ceramic balls are less likely to increase bearing friction as heat increases.

Secondly, since ceramic balls absorb less heat, cooling requirements for ceramic ball bearings are much lower.

ITI produces standard ceramic balls from alumina oxide, silicon nitride, silicon carbide, ruby sapphire and zirconia in sizes ranging from 0.125" to 3.0". However, both smaller and larger sizes can be specified.

Since they are used in both valves and bearings, ITI produces ceramic balls in both valve and bearing grades. Refer to our Ball Grade Table for the exact tolerances available.

These levels of precision, coupled with ceramic balls' great wear and heat resistance, make them integral components in many designs.

ITI would be glad to help you specify the best ceramic material or any other material for designs which use standard or modified balls. ■

Ceramic Properties*	Zirconia (MgO partially stabilized)	Silicon Carbide	Alumina Oxide	Ruby Sapphire	Silicon Nitride
Max. Useful Temperature	1800°F.	2500°F.	3180°F.	3250°F.	2552°F.
Density	5.5g/cm³ .199 lbs/in³	3.1 gm/cm³ .112 lbs/in³	3.86 gm/cm³ .139 lbs/in³	3.98 gm/cm³ .144 lbs/in³	3.2 gm/cm³ .116 lbs/in³
Compressive Strength	280,000 psi	400,000 psi	330,000 psi	300,000 psi	341,300 psi
Young's Modulus of Elasticity	29×10^6 psi (@70°F.) 26×10^6 psi (@1800°F.)	53×10^6 psi	54×10^6 psi	50×10^6 – 55×10^6 psi	47×10^6 psi
Hardness	2100 Vickers	2400 Vickers	1365 Vickers	1570–1800 Vickers	1500–2000 Vickers
Thermal Conductivity	2 W/m °C. 1.5 BTU/hr ft – °F.	145 W/m °C. 85 BTU/hr ft – °F.	35.6 W/m °C. (@20°C.) 6.3 W/m°C. (@800°C.)	.086 Cal/sec cm°C.	.07 Cal/sec cm°C.
Thermal Expansion	5.5×10^{-6}/°F.	2.5×10^{-6}/°F.	4.6×10^{-6}/°F.	3.2×10^{-6}/°F.	1.78×10^{-6}/°F.
Electrical Resistivity	10^{11} Ohm/cm (@25°C.) 200 Ohm/cm (@1000°C.)	Data not available	$> 10^{14}$ Ohm/cm (@25°C.) 2.5×10^6 Ohm/cm (@900°C.)	10^9 Ohm/cm (@500°C.) 10 Ohm/cm (@2000°C.)	10^6 Ohm/cm (@500°C.) 10^5 Ohm/cm (@2000°C.)
Corrosion Resistance	Inert except for hydrofluoric and hot concentrated sulfuric acids.	Inert to most substances.	Inert to most substances, not recommended for environments of hydrochloric or hydrofluoric acids or strong alkaline solutions.	Inert to most substances even at very high temperatures.	Inert to most substances.

*Data from various industrial literature. Exact properties will vary from supplier to supplier.