

National Bureau of Standards

Report of Investigation

Research Material 8531

Glass Fibers for Microanalysis

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This Research Material (RM) was developed for use with microanalysis techniques such as electron probe microanalysis (EPMA) and secondary ion mass spectrometry (SIMS). RM 8531 consists of five types of glass in the form of fibers ranging from 10-100 μm in diameter and 50-60 mm long. The fibers are suited for use in particulate analyses for which no well-characterized standards of complex composition and simple shape are available.

Preparation of the Glasses:

Each glass melt was prepared by thoroughly mixing weighed quantities of the selected oxides together; where this was not possible, the carbonates, nitrates, or phosphates were used. The major constituents of the glasses were selected so that a variety of matrices of differing average atomic numbers would be represented. For each of the five matrices, there are two glasses - one with and one without the low-concentration additives. All materials used were reagent grade materials or the equivalent.

The materials were separately melted in 300 mL platinum crucibles in electrically heated furnaces, in an oxidizing atmosphere (air). To ensure homogeneity, the melts were stirred from four to six hours with a motor-driven, double-bladed, propeller-type stirrer made of a platinum-rhodium alloy. Melting and stirring temperatures ranged from 900-1400 $^{\circ}\text{C}$ depending upon the glass being prepared. After completion of the melting and stirring operations, each glass was cast into a rectangular block and annealed to remove residual strain.

The bulk glasses from which these fibers were prepared are being certified as SRM's (Glasses for Microanalysis SRM's 1871-1875). The SRM's should be available with the certified values early in 1984. The homogeneity and composition of only the major components will be certified. There is no guarantee that the compositions of the fibers are identical to the bulk glasses in the SRM's.

Composition:

The compositions, in weights percent, of the glasses are listed in the attached table. These are nominal values calculated from the weights of the components added to the melt during the manufacture of the glasses.

Preparation of Fibers:

Prior to drawing fibers, a glass specimen was melted in a platinum boat that was heated directly by an electric current. The fiber was formed by drawing a thread of molten glass through an orifice in the bottom of the platinum boat. The continuous glass fiber was wound onto a 25.4 cm (10 inch) diameter metal drum driven by a variable speed electric motor (1000-4000 rpm). Different orifice sizes and drum speeds were used in drawing the fibers, depending upon the viscosity and surface tension of each glass. This procedure requires a minimum amount of working time and low temperatures, therefore minimizing compositional changes due to vaporization.

1. Fiori, C.E.; Heinrich, K.F.J.; Marinenko, R.B.; Darr, M.M.; Blackburn, D.H.; Newbury, D.E.; and Small, J.A.; "An Overview of the Glass Standards Program for Microanalysis at the National Bureau of Standards," Proceedings of the Eleventh Annual Conference of the Microbeam Analysis Society, Miami Beach, Fla., August 1976.
2. Small, J.A.; Heinrich, K.F.J.; Fiori, C.E.; Myklebust, R.L.; Newbury, D.E.; and Dillmore, M.F.; "The Production and Characterization of Glass Fibers and Spheres for Microanalysis," Scanning Electron Microscopy, 1978, Vol. 1, SEM Inc., Ill., U.S.A.

Glasses for Microanalysis

Melt No.	K-456	K-493	K-453	K-491	K-458	K-489	K-495	K-490	K-496	K-497
<u>Composition in Weight Percent</u>										
SiO ₂	28.77	27.89	---	0.19	49.38	46.76	---	0.42	---	0.27
PbO	71.23	69.08	58.72	59.35	---	1.28	---	1.55	---	.99
GeO ₂	---	---	41.28	37.98	---	---	---	---	---	---
BaO	---	---	---	---	46.80	43.88	---	---	---	---
ZnO	---	---	---	---	3.82	3.72	---	---	---	---
P ₂ O ₅	---	---	---	---	---	---	---	---	79.54	76.03
MgO	---	---	---	---	---	---	---	---	9.03	8.64
Al ₂ O ₃	---	0.20	---	0.16	---	0.29	20.00	18.68	11.43	10.92
B ₂ O ₃	---	.14	---	.11	---	.20	75.00	70.00	---	0.15
ZrO ₂	---	.49	---	.40	---	.70	---	0.85	---	.54
TiO ₂	---	.32	---	.26	---	.46	---	.55	---	.35
CeO ₂	---	.68	---	.56	---	.98	---	1.19	---	.76
Ta ₂ O ₅	---	.88	---	.72	---	1.26	---	1.53	---	.98
Fe ₂ O ₃	---	.32	---	.26	---	.046	---	0.55	---	.35
Li ₂ O	---	.001	---	.001	---	.002	5.00	4.67	---	.001