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Property Data for Simulated Americium/Curium Glasses

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October 1999

Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

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UNITED STATES DEPARTMENT OF ENERGY
under Contract DE-AC06-76RLO 1830

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Summary

We studied the properties of mixed lanthanide-alumino-borosilicate glasses. Fifty-five glasses were designed to augment a previous, Phase I, study by systematically varying the composition of Ln_2O_3 and the concentrations of Ln_2O_3 , SiO_2 , B_2O_3 , Al_2O_3 , and SrO in glass. These glasses were designed and fabricated at the Savannah River Technology Center and tested at the Pacific Northwest National Laboratory. The properties measured include the high-temperature viscosity (η) as a function of temperature (T) and the liquidus temperature (T_L) of Phase II test glasses.

The T_L s of the 55 Phase II glasses were between 1153°C (AC2-52) and 1405°C (AC2-04). Only two glasses had T_L s above the 1350°C limit, AC2-04 and AC2-18. Two crystal types, rare earth silicates (RESs) and aluminum oxide (Al_2O_3), were identified as the primary crystalline phases in the Phase II glasses using a combination of X-ray diffraction (XRD), scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDS), and optical microscopy (OM). This differs from Phase I glasses in which RES and aluminosilicate (ALS) crystals were found and Al_2O_3 was not. These data are summarized in Table S.1. Appendix A (Glass Property Measurements) contains the detailed explanation of T_L measurements for each glass, including T_L measurement data, OM and SEM micrographs, and analyses results from XRD and EDS.

RES was the primary phase in 47 of the 55 glasses; however, we observed four distinct morphologies of the RES when identifying these crystals: needle-like (thin and straight), hair-like (thin and wavy), plate-like, and rectangular cap. Crystals with each of the different morphologies were analyzed using SEM/EDS. EDS data showed Si, La, Nd, and O as major components and Ce, Pr, Sm, Gd, and Er as minor components in the crystals. Al is present as a minor component in some crystals, but not in others. Chemically, the RES crystals are the same regardless of morphology. Samples analyzed by XRD matched the crystal structure of neodymium germanium borate silicate ($\text{Nd}_3\text{BGe}_{1.08}\text{Si}_{0.92}\text{O}_{10}$). In this crystal structure, Ge could be replaced by Si and Nd could be replaced by rare earth elements to form $\text{RE}_3\text{BSi}_2\text{O}_{10}$, the crystal type that is probably present in these samples. Generally, the glasses within the RES primary phase field showed massive crystallinity (i.e., > 30 vol % RES) after a 24 h heat treatment at temperatures between 3°C and 30°C below T_L . As RES crystals are difficult to redissolve once crystallized, this observation should be considered when planning for off-normal operating scenarios.

Aluminum oxide crystals were found as the primary phase in eight glasses. Two distinct Al_2O_3 crystals formed concurrently, a plate-like crystal and a garnet shaped crystal. SEM/EDS identified both crystal types with high aluminum and oxygen and did not make any differentiation as to chemical composition. The structures of these crystals were not determinable by XRD; therefore, it is not understood whether there is more than one Al_2O_3 crystal structure.

The η - T relationship was measured for Phase II glasses using a high-temperature spindle viscometer. Generally, the data did not conform to the standard η - T relationship as found in most silicate glasses (described by the Vogel-Tamman-Fulcher model). The viscosity values at

1350°C (η_{1350}) for Phase II glasses were estimated by a linear interpolation of $\ln[\eta_{1350}]$ vs $1/T$ for either 1) the entire data set for glasses displaying nearly linear $\ln[\eta]-1/T$ behavior or 2) the data points taken on either side of 1350°C for glasses displaying non-linear $\ln[\eta]-1/T$ behavior and the data was available.

The η_{1350} values of Phase II test glasses range from 0.57 Pa·s for AC2-03 to 2.21 Pa·s for AC2-07, both exterior vertex glasses. All Phase II glasses passed the 0.3 Pa·s to 2.4 Pa·s limit on η_{1350} . The η_{1350} values are listed in Table S.1 for these glasses. A roughly linear relationship between Ln_2O_3 concentration in glass and the $\ln[\eta_{1350}]$ value was shown for Phase II glasses consistent with that found with Phase I glasses.

Table S.1. T_L (°C), Primary Crystalline Phase, and η_{1350} (Pa·s) for Test Glasses

Glass	T_L	Phase	η_{1350}	Glass	T_L	Phase	η_{1350}
AC2-01	1308	RES	1.44	AC2-29	1300	RES	0.66
AC2-02	1257	Al_2O_3	0.81	AC2-30	1227	RES	1.35
AC2-03	1313	RES	0.57	AC2-31	1305	RES	1.49
AC2-04	1405	RES	⁽¹⁾	AC2-32	1264	RES	1.18
AC2-05	1241	RES	2.20	AC2-33	1271	RES	1.17
AC2-06	1309	RES	0.70	AC2-34	1232	RES	1.44
AC2-07	1209	RES	2.21	AC2-35	1289	RES	1.08
AC2-08	1242	RES	1.70	AC2-36	1294	RES	1.03
AC2-09	1323	RES	0.79	AC2-37	1305	RES	0.96
AC2-10	1255	RES	1.29	AC2-38	1308	RES	0.95
AC2-11	1292	Al_2O_3	0.96	AC2-39	1318	RES	0.91
AC2-12	1333	RES	1.42	AC2-40	1325	RES	0.91
AC2-13	1181	RES	1.86	AC2-41	1324	RES	0.87
AC2-14	1250	Al_2O_3	0.89	AC2-42	1180	RES	1.75
AC2-15	1279	RES	1.33	AC2-43	1227	RES	1.32
AC2-16	1298	Al_2O_3	1.09	AC2-44	1305	RES	0.94
AC2-17	1273	RES	1.14	AC2-45	1259	RES	1.10
AC2-18	1366	RES	⁽¹⁾	AC2-46	1333	RES	0.80
AC2-19	1249	RES	1.39	AC2-47	1272	RES	1.17
AC2-20	1343	RES	⁽¹⁾	AC2-48	1181	RES	1.73
AC2-21	1242	Al_2O_3	0.86	AC2-49	1220	RES	1.46
AC2-22	1284	RES	1.34	AC2-50	1272	RES	1.12
AC2-23	1282	Al_2O_3	1.00	AC2-51	1305	RES	0.95
AC2-24	1319	RES	0.61	AC2-52	1153	RES	2.17
AC2-25	1305	Al_2O_3	1.18	AC2-53	1192	RES	1.76
AC2-26	1208	RES	2.17	AC2-54	1326	RES	0.86
AC2-27	1242	RES	1.93	AC2-55	1255	RES	1.14
AC2-28	1260	Al_2O_3	0.86				

⁽¹⁾ Insufficient data to determine η_{1350}

Glossary

η	viscosity
ρ	density
AIS	aluminosilicate
DNFSB	Defense Nuclear Facility Safety Board
EDS	energy dispersive spectroscopy
MPPF	Multi-Purpose Processing Facility
NBS	National Bureau of Standards
NIST	National Institute for Standards and Technolog
OM	optical microscopy
ORNL	Oak Ridge National Laboratory
Pa·s	pascal seconds
PNNL	Pacific Northwest National Laboratory
QA	quality assurance
RES	rare-earth silicate
SEM	scanning electron microscopy
SRM	Standard Reference Material
SRS	Savannah River Site
SRTC	Savannah River Technology Center
T	temperature
T_A	lowest temperature at which samples did not form crystals
T_C	highest temperature at which samples did form crystals
T_L	liquidus temperature
VTF	Vogel-Tamman-Fulcher (model)
XRD	X-ray diffraction
ZAF	corrections for atomic number, absorption, and fluorescence effects

Acknowledgments

Pacific Northwest National Laboratory is operated for the U.S. Department of Energy by Battelle under Contract DE-AC06-76RLO 1830.

The authors wish to acknowledge the guidance and funding for this multi-institutional task by the Department of Energy's Environmental Management Program. Funding to perform this task was provided by a subcontract from Savannah River Technical Center and we would like to thank this institution for their selection of this team to do this challenging and demanding task.

The following individuals from Pacific Northwest National Laboratory were key in helping complete the task:

Wayne C. Cosby – for his tireless efforts, late nights, and long weekends to make this document a well written report.

Orie Barnes – for his expertise as a Quality Assurance representative and ensuring quality work is passed on to the client.

Loni M. Peurrung – for her helpful support as line manager and for her thoughtful review of this document.

Stacey A. Hartley – for the mathematical calculations she did to define the measured viscosity data.

James E. Coleman and James S. Young – who worked very hard providing scanning electron microscopy data, sometimes under very demanding conditions.

Jarrod V. Crum – who helped us with sample heat treatments, XRD analyses, and furnace profiling and who volunteered for weekend sample changes.

Renee L. Russell – for her help in preparation of glass for T_L measurement.

Harry D. Smith – for review of all the calculations performed for this study.

Virginia A. Rhoades – who supported us as secretary and records custodian.

Pavel Hrma, Hong Li, and Greg Piepel – for their helpful comments and suggestions on this document.

Contents

1.0	Introduction.....	1.1
1.1	Americium/Curium Project.....	1.1
1.2	Phase I Study.....	1.2
1.2.1	Density	1.2
1.2.2	Liquidus Temperature.....	1.3
1.2.3	Viscosity	1.4
1.3	Phase II Plan	1.5
2.0	Experimental Procedures	2.1
2.1	Liquidus Temperature.....	2.1
2.2	Viscosity	2.2
2.3	Quality Assurance.....	2.2
3.0	Calibration and Validation.....	3.1
3.1	Liquidus Temperature.....	3.1
3.2	Viscosity	3.2
4.0	Discussion of Results.....	4.1
4.1	Liquidus Temperature.....	4.1
4.1.1	Rare Earth Silicate Crystals	4.2
4.1.2	Aluminum Oxide Crystals	4.5
4.1.3	“As-Received” Glasses	4.8
4.1.4	Sample “Budding”	4.8
4.1.5	Quantitative Analysis of RES Crystals by SEM.....	4.11
4.1.6	Glass Composition Effects.....	4.13
4.2	Viscosity	4.15
5.0	Recommendations.....	5.1
6.0	References.....	6.1

Figures

Figure 1.1. Schematic of the Vitrification Process Steps.....	1.2
Figure 1.2. Specific Volume as a Function of Ln_2O_3 Concentration for Phase I Glasses	1.3
Figure 1.3. T_L as a Function of Ln_2O_3 Concentration for Phase I Glasses	1.4
Figure 1.4. $\text{Ln}(\eta_{1350})$ as a Function of Ln_2O_3 Concentration for Phase I Glasses	1.5
Figure 1.5. Ternary Projection of Phase I and II Glasses in $\text{SiO}_2 - \text{Al}_2\text{O}_3 - \text{B}_2\text{O}_3$ Space.....	1.6
Figure 3.1. Comparison of Measured (Val-02) and Certified η for SRM 710a Glass as a Function of Temperature.....	3.3
Figure 4.1. Example of Needle-Like RES Crystal Phase	4.3
Figure 4.2. AC1-31, Plate-Like RES Crystals	4.3
Figure 4.3. AC2-44 (1302°C) Plate-Like RES Crystals	4.4
Figure 4.4. AC2-44 (1302°C) 200×	4.4
Figure 4.5. AC2-41 (1313°C) 200×	4.4
Figure 4.6. Rectangular Cap RES Crystals on Surface of AC2-46 (1321°C).....	4.5
Figure 4.7. Aluminum Oxide Crystals.....	4.6
Figure 4.8. SEM Analysis of AC2-21 (1229°C).....	4.6
Figure 4.9. AC2-14 (1232°), Garnet Shaped Al_2O_3 Crystal	4.7
Figure 4.10. Scan of Crystal in Figure 4.9	4.7
Figure 4.11. Photograph of Buds on Crucible Edges.....	4.8
Figure 4.12. AC2-16 (1297°C) Buds on Crucible Wing of Platinum Fold	4.9
Figure 4.13. Magnification of Figure 4.12.....	4.9
Figure 4.14. AC2-29 (1299°C), Small Buds on Wing of Crucible.....	4.10
Figure 4.15. AC2-29 (1299°C) after Crucible Wing is Polished (note: platinum wall exposed on left side of sample).....	4.10
Figure 4.16. AC2-29 (1299°C), Outer Surface of Bud.....	4.10
Figure 4.17. EDS Scan of Light Gray Crystals in Figure 4.16	4.11
Figure 4.18. EDS Scan of Black Crystals in Figure 4.16	4.11
Figure 4.19. T_L as a Function of Ln_2O_3 Concentration in Phase I and II Glasses (S-Alumino- Silicate Crystals in Phase I Glasses, *-RES Crystals in Phase I Glasses, +-RES Crystals in Phase II glasses, and A- Al_2O_3 Crystals in Phase II Glasses).....	4.14
Figure 4.20. Composition Projection of Phase I and II Glasses in a $\text{SiO}_2 - \text{Al}_2\text{O}_3 - \text{Ln}_2\text{O}_3$ Space with Primary Crystalline Phases Noted as Symbols: S-Alumino-Silicate, +-RES, and A- Al_2O_3	4.15
Figure 4.21. Comparison of $\ln[\eta]-1/T$ Behavior of (a) AC2-14 and (b) Val-01	4.16
Figure 4.22. Effect of Ln_2O_3 Concentration on $\ln[\eta_{1350}]$ for Phase II Glasses (symbol numbers represent glass numbers).....	4.19
Figure 4.23. Effect of Ln_2O_3 Concentration on $\ln[\eta_{1350}]$ for Phase I and II Glasses (symbols represent: A-Phase I glasses, B-Linear Phase II glasses, and N- Nonlinear Phase II glasses).....	4.20

Tables

Table 1.1. Phase II Glass Compositions in Mass Fractions of Oxide Components.....	1.7
Table 3.1. T_L Validation Data.....	3.1
Table 3.2. Viscosity-Temperature Data for Validation Glasses	3.2
Table 4.1. T_L and Primary Crystalline Phase for 55 Am/Cm Phase II Glasses	4.1
Table 4.2. Quantitative Analysis of Crystals in Glass	4.12
Table 4.3. Quantitative Analysis of Glass Matrix Compared to Target Composition	4.13
Table 4.4. Summary of Phase II Viscosity Data.....	4.16

1.0 Introduction

1.1 Americium/Curium Project

Approximately 11,000 liters (3,600 gallons) of solution containing isotopes of Am and Cm are stored in Tank 17.1 in the Savannah River Site's (SRS) F-Canyon. These isotopes were recovered during plutonium-242 production campaigns in the mid and late 1970s. The continued storage of these isotopes was identified as an item of primary concern in the Defense Nuclear Facility Safety Board's (DNFSB) Recommendation 94-1. The process selected to treat the contents of this tank includes

- pretreating the tank contents in existing canyon vessels to separate actinides and lanthanides from other impurities (primarily iron, aluminum, and sodium)
- vitrifying the actinide/lanthanide fraction of the tank contents in the Multi-Purpose Processing Facility (MPPF) in the F-Canyon
- shipping the actinide containing glass to Oak Ridge National Laboratory (ORNL) for storage and separation of actinides from glass
- irradiating the separated Cm to form heavy elements such as Cf for civilian and military applications.

The proposed process is described in detail by Fellingner et al. (1998a and 1998b) and Marra et al. (to be published 1999). A schematic of the vitrification step is displayed in Figure 1.1. In the vitrification process, the feed from pretreatment is transferred to a reaction vessel. Actinide and lanthanide oxalates are precipitated from solution and washed. Oxalate precipitate is then transferred to a Pt/Rh induction melter, which is preloaded with fritted glass-making additives. The mixture is dried and heated to approximately 1450°C to produce a molten glass. The glass is then poured through a drain tube into a stainless steel can.

A lanthanide-alumino-borosilicate glass has been developed specifically for this process (Fellinger et al. 1998a and 1998b): The 25SrABS glass frit (33.68 mass% SiO₂, 25.00 La₂O₃, 24.87 Al₂O₃, 13.54 B₂O₃, and 2.91 SrO), when combined with 20 to 50 mass percent lanthanide/actinide (Ln₂O₃)^a oxides, produces a glass with adequate properties for this process. It was shown that properties critical to the success of the proposed process, including viscosity (η) and liquidus temperature (T_L), vary with glass composition (Fellinger et al. 1998a and 1998b). Property limits were set to ensure adequate processability of the melter feed (Martin 1999 and Peeler and Edwards 1999). These limits include $T_L \leq 1350^\circ\text{C}$ and $0.3 \text{ Pa}\cdot\text{s} \leq \eta$ at 1350°C (η_{1350}) $\leq 2.4 \text{ Pa}\cdot\text{s}$. Therefore, we studied the impact of total Ln₂O₃ concentration and composition on the properties of the proposed glass.

^a In this study, Ln₂O₃ represents the total concentration of three valent lanthanide oxides. Which simulates the total concentration of three valent lanthanide and actinide oxides in the radioactive glass?

Figure 1.1. Schematic of the Vitrification Process Steps

1.2 Phase I Study

In the first phase of this study, the concentration of Ln_2O_3 in glass was systematically varied between 38 and 54 mass percent along with its composition (Peeler 1998 and Peeler and Edwards 1999). Thirty-one glasses were fabricated and characterized in a joint Savannah River Technology Center (SRTC) and Pacific Northwest National Laboratory (PNNL) effort. The results of this study are described in detail by Peeler and colleagues (Peeler et al. 1999 and Peeler et al., to be published, 2000). It was found that the variation in properties, such as density (ρ), T_L , and η , could be described to first order by the variation in Ln_2O_3 concentration in glass.

1.2.1 Density

The ρ of Phase I glasses, measured by gas pycnometry, ranged from 3.435 g/cm^3 to 4.103 g/cm^3 , corresponding to specific volumes (v) of $0.291 \text{ cm}^3/\text{g}$ and $0.244 \text{ cm}^3/\text{g}$. A linear decrease in v with Ln_2O_3 is shown in Figure 1.2. The trend suggests a partial specific v from Ln_2O_3 of $0.1023 \text{ cm}^3/\text{g}^2$ with a Ln_2O_3 free-glass partial specific v of $0.4091 \text{ cm}^3/\text{g}^2$. These values extrapolate to effective ρ s for Ln_2O_3 and Ln_2O_3 free glass of 9.775 g/cm^3 and 2.444 g/cm^3 , respectively. The v of $\text{SrO-Al}_2\text{O}_3\text{-B}_2\text{O}_3\text{-SiO}_2$ glasses can be expected to fall reasonably close to $0.41 \text{ cm}^3/\text{g}$. The ρ of lanthanide oxide minerals range from 6.51 g/cm^3 for La_2O_3 to 9.42 g/cm^3 for Lu_2O_3 ($v = 0.106 \text{ cm}^3/\text{g}$ to $0.154 \text{ cm}^3/\text{g}$). This suggests that the local density of Ln_2O_3 in this

glass is similar to that in the oxide minerals. Furthermore, the similarity in densities between the end members suggest a low heat of mixing.

1.2.2 Liquidus Temperature

Two primary crystalline phases were found to precipitate from Phase I glasses, an aluminosilicate (AIS) with T_L values ranging from 1164°C to 1255°C and a rare-earth silicate (RES) with T_L values ranging from 1163°C to 1298°C. For Ln_2O_3 concentrations less than 43 mass percent, AIS was found to be the primary phase; glasses with higher Ln_2O_3 concentrations were in the RES primary phase field. A weak correlation, based on low R^2 values, was found between T_L and Ln_2O_3 concentrations for Phase I glasses in both primary phase fields as shown in Figure 1.3. A minimum in T_L is found at approximately 44 mass percent Ln_2O_3 , with increasing T_L at both higher and lower Ln_2O_3 concentrations.

Figure 1.2. Specific Volume as a Function of Ln_2O_3 Concentration (in mass fraction) for Phase I Glasses

Figure 1.3. T_L as a Function of Ln_2O_3 Concentration for Phase I Glasses

1.2.3 Viscosity

The η s of Phase I glasses were measured as functions of temperature (T). The η - T data were then fitted to a Vogel-Tamman-Fulcher (VTF) model:

$$\ln \eta = A + \frac{B}{T - T_0} \quad (1)$$

where A , B , and T_0 are temperature independent coefficients. Equation (1) was then used to calculate a η at 1350°C , which was considered the nominal melter operating temperature during the Phase I study. The η_{1350} values ranged from 1.24 pascal seconds (Pa·s) to 4.01 Pa·s. The effect of Ln_2O_3 concentration on $\ln[\eta_{1350}]$ is shown in Figure 1.4. The $\ln[\eta_{1350} \text{ (Pa·s)}]$ is decreased by 7.28 per mass fraction of Ln_2O_3 to the glass. For a glass with $\eta_{1350} = 2.5 \text{ Pa·s}$, adding one mass percent Ln_2O_3 will reduce the η_{1350} by 0.2 Pa·s to 2.3 Pa·s.

Figure 1.4. $\ln(\eta_{1350})$ as a Function of Ln_2O_3 Concentration for Phase I Glasses

1.3 Phase II Plan

Phase II was designed to systematically vary the composition of Ln_2O_3 and the concentrations of Ln_2O_3 , SiO_2 , Al_2O_3 , B_2O_3 , and SrO in glass (Peeler and Edwards 1999). Fifty five glasses were fabricated and characterized in a joint SRTC and PNNL effort. Table 1.1 lists these glass compositions. The concentration of Ln_2O_3 varied between 48.9 and 64.1 mass percent. The ratios of SiO_2 , Al_2O_3 , B_2O_3 , and SrO remained constant and equal to those in the Phase I study in 27 of the 55 glasses, at 46.7, 34.5, 18.8, and 0.39 mass percent, respectively. The ratios of these components varied systematically in the remaining 28 glass compositions. Figure 1.5 shows the normalized projections of the 55 glass compositions in $\text{SiO}_2 - \text{Al}_2\text{O}_3 - \text{B}_2\text{O}_3$ space. The center point of the ring contains the 27 Phase II and 32 Phase I glasses with constant ratios of frit components. The data points on the near circle surrounding the center point contain the additional 28 glass compositions. The ratios of SiO_2 , Al_2O_3 , B_2O_3 , and SrO fall into categories of low (L) and high (H) for each component and a central or middle (M) for those ratios consistent with Phase I glasses. In the discussion of glass properties, the L, M, and H will be used to describe the relative concentrations of these components in glass rather than the absolute concentrations. See Peeler and Edwards (1999) for further detail on test matrix design.

Figure 1.5. Ternary Projection of Phase I and II Glasses in SiO_2 - Al_2O_3 - B_2O_3 Space

The results of T_L and η testing on Phase II glasses are described in this report. Section 2.0 describes the experimental procedures, Section 3.0 describes how test calibration and validation were performed, Section 4.0 discusses results, and Section 5.0 gives recommendations for the use of these data and further studies. Appendix A shows the measurement results by glass.

Table 1.1. Phase II Glass Compositions in Mass Fractions of Oxide Components

ID	La ₂ O ₃	Ce ₂ O ₃	Pr ₂ O ₃	Nd ₂ O ₃	Sm ₂ O ₃	Eu ₂ O ₃	Gd ₂ O ₃	Er ₂ O ₃	Al ₂ O ₃	B ₂ O ₃	SiO ₂	SrO	minor ^(a)	Ln ₂ O ₃
AC2-01	0.2300	0.0840	0.0730	0.0840	0.0200	0.0050	0.0110	0.0760	0.1190	0.0650	0.2190	0.0140	N	0.5830
AC2-02	0.2300	0.0840	0.0730	0.0920	0.0200	0.0050	0.0280	0.0300	0.1700	0.0930	0.1610	0.0140	N	0.5620
AC2-03	0.2300	0.0840	0.0290	0.1900	0.0200	0.0130	0.0110	0.0300	0.1190	0.0930	0.1610	0.0200	N	0.6070
AC2-04	0.2300	0.0340	0.0730	0.1850	0.0500	0.0050	0.0280	0.0300	0.1190	0.0650	0.1610	0.0200	N	0.6350
AC2-05	0.2300	0.0340	0.0290	0.1020	0.0500	0.0130	0.0280	0.0300	0.1700	0.0650	0.2290	0.0200	N	0.5160
AC2-06	0.2300	0.0340	0.0290	0.1780	0.0500	0.0050	0.0110	0.0760	0.1190	0.0930	0.1610	0.0140	N	0.6130
AC2-07	0.2300	0.0340	0.0290	0.1090	0.0200	0.0130	0.0110	0.0760	0.1700	0.0650	0.2290	0.0140	N	0.5220
AC2-08	0.1840	0.0840	0.0730	0.0840	0.0500	0.0130	0.0110	0.0300	0.1700	0.0650	0.2160	0.0200	N	0.5290
AC2-09	0.1840	0.0840	0.0730	0.1330	0.0500	0.0130	0.0280	0.0760	0.1190	0.0650	0.1610	0.0140	N	0.6410
AC2-10	0.1840	0.0840	0.0290	0.0840	0.0500	0.0050	0.0280	0.0760	0.1190	0.0930	0.2280	0.0200	N	0.5400
AC2-11	0.1840	0.0840	0.0290	0.1970	0.0500	0.0050	0.0110	0.0300	0.1700	0.0650	0.1610	0.0140	N	0.5900
AC2-12	0.1840	0.0340	0.0730	0.2070	0.0200	0.0050	0.0110	0.0330	0.1190	0.0650	0.2290	0.0200	N	0.5670
AC2-13	0.1840	0.0340	0.0730	0.1070	0.0500	0.0050	0.0110	0.0300	0.1700	0.0930	0.2290	0.0140	N	0.4940
AC2-14	0.1840	0.0340	0.0730	0.1450	0.0200	0.0130	0.0110	0.0760	0.1700	0.0930	0.1610	0.0200	N	0.5560
AC2-15	0.1840	0.0340	0.0290	0.2070	0.0200	0.0130	0.0280	0.0300	0.1190	0.0930	0.2290	0.0140	N	0.5450
AC2-16	0.1840	0.0350	0.0290	0.2070	0.0200	0.0050	0.0280	0.0760	0.1700	0.0650	0.1610	0.0200	N	0.5840
AC2-17	0.2064	0.0580	0.0504	0.1361	0.0347	0.0090	0.0194	0.0524	0.1439	0.0787	0.1941	0.0170	N	0.5664
AC2-18	0.1700	0.0970	0.0220	0.2420	0.0580	0.0150	0.0080	0.0230	0.1190	0.0650	0.1610	0.0200	N	0.6350
AC2-19	0.2400	0.0970	0.0250	0.0630	0.0150	0.0030	0.0080	0.0880	0.1190	0.0930	0.2290	0.0200	N	0.5390
AC2-20	0.2400	0.0970	0.0850	0.1340	0.0150	0.0150	0.0320	0.0230	0.1190	0.0650	0.1610	0.0140	N	0.6410
AC2-21	0.2400	0.0970	0.0220	0.0810	0.0580	0.0030	0.0320	0.0230	0.1700	0.0930	0.1610	0.0200	N	0.5560
AC2-22	0.2400	0.0250	0.0830	0.0630	0.0580	0.0150	0.0320	0.0230	0.1190	0.0930	0.2290	0.0200	N	0.5390
AC2-23	0.2400	0.0250	0.0850	0.1850	0.0150	0.0030	0.0080	0.0230	0.1700	0.0650	0.1610	0.0200	N	0.5840
AC2-24	0.2400	0.0250	0.0220	0.2420	0.0150	0.0030	0.0320	0.0340	0.1190	0.0930	0.1610	0.0140	N	0.6130

ID	La ₂ O ₃	Ce ₂ O ₃	Pr ₂ O ₃	Nd ₂ O ₃	Sm ₂ O ₃	Eu ₂ O ₃	Gd ₂ O ₃	Er ₂ O ₃	Al ₂ O ₃	B ₂ O ₃	SiO ₂	SrO	minor ^(a)	Ln ₂ O ₃
AC2-25	0.2400	0.0250	0.0220	0.1040	0.0580	0.0150	0.0320	0.0880	0.1700	0.0650	0.1610	0.0200	N	0.5840
AC2-26	0.2400	0.0280	0.0220	0.0630	0.0580	0.0150	0.0080	0.0880	0.1700	0.0650	0.2290	0.0140	N	0.5220
AC2-27	0.1680	0.0970	0.0850	0.0630	0.0580	0.0030	0.0320	0.0230	0.1630	0.0650	0.2290	0.0140	N	0.5290
AC2-28	0.1680	0.0970	0.0850	0.0630	0.0150	0.0150	0.0320	0.0810	0.1700	0.0930	0.1610	0.0200	N	0.5560
AC2-29	0.1680	0.0250	0.0850	0.1780	0.0580	0.0030	0.0080	0.0880	0.1190	0.0930	0.1610	0.0140	N	0.6130
AC2-30	0.1680	0.0250	0.0220	0.2180	0.0150	0.0150	0.0080	0.0230	0.1700	0.0930	0.2290	0.0140	N	0.4940
AC2-31	0.1680	0.0250	0.0220	0.2140	0.0150	0.0030	0.0320	0.0880	0.1190	0.0650	0.2290	0.0200	N	0.5670
AC2-32	0.2031	0.0601	0.0528	0.1298	0.0364	0.0090	0.0198	0.0546	0.1443	0.0788	0.1943	0.0170	N	0.5656
AC2-33	0.2098	0.0559	0.0489	0.1394	0.0331	0.0081	0.0183	0.0507	0.1442	0.0785	0.1953	0.0169	N	0.5642
AC2-34	0.2088	0.0573	0.0501	0.1427	0.0338	0.0083	0.0187	0.0519	0.1418	0.0772	0.1920	0.0166	N	0.5716
AC2-35	0.2079	0.0586	0.0512	0.1460	0.0346	0.0085	0.0192	0.0532	0.1393	0.0758	0.1886	0.0163	N	0.5792
AC2-36	0.2069	0.0599	0.0524	0.1493	0.0354	0.0087	0.0196	0.0544	0.1368	0.0745	0.1852	0.0160	N	0.5866
AC2-37	0.2060	0.0613	0.0535	0.1526	0.0362	0.0089	0.0201	0.0556	0.1343	0.0731	0.1819	0.0157	N	0.5942
AC2-38	0.2050	0.0626	0.0547	0.1559	0.0370	0.0091	0.0205	0.0568	0.1318	0.0718	0.1785	0.0154	N	0.6016
AC2-39	0.2041	0.0639	0.0559	0.1593	0.0378	0.0093	0.0209	0.0580	0.1293	0.0704	0.1751	0.0151	N	0.6092
AC2-40	0.2031	0.0653	0.0570	0.1626	0.0386	0.0095	0.0214	0.0592	0.1268	0.0691	0.1718	0.0148	N	0.6167
AC2-41	0.2022	0.0666	0.0582	0.1659	0.0394	0.0097	0.0218	0.0604	0.1244	0.0677	0.1684	0.0146	N	0.6242
AC2-42	0.2194	0.0426	0.0372	0.1062	0.0252	0.0062	0.0140	0.0387	0.1691	0.0921	0.2290	0.0198	N	0.4895
AC2-43	0.2146	0.0493	0.0431	0.1228	0.0291	0.0072	0.0161	0.0447	0.1567	0.0853	0.2122	0.0183	N	0.5269
AC2-44	0.2002	0.0693	0.0605	0.1725	0.0409	0.0101	0.0227	0.0628	0.1194	0.0650	0.1617	0.0140	N	0.6390
AC2-45	0.1988	0.1087	0.0407	0.1155	0.0276	0.0069	0.0154	0.0507	0.1442	0.0785	0.1953	0.0169	N	0.5643
AC2-46	0.2050	0.0626	0.0547	0.1560	0.0370	0.0091	0.0205	0.0568	0.1319	0.0718	0.1785	0.0154	N	0.6017
AC2-47	0.2098	0.0559	0.0489	0.1394	0.0331	0.0081	0.0183	0.0507	0.1442	0.0785	0.1953	0.0169	N	0.5642
AC2-48	0.2194	0.0426	0.0372	0.1062	0.0252	0.0062	0.0140	0.0387	0.1691	0.0921	0.2290	0.0198	N	0.4895
AC2-49	0.2146	0.0493	0.0431	0.1228	0.0291	0.0072	0.0161	0.0447	0.1567	0.0853	0.2122	0.0183	N	0.5269
AC2-50	0.2098	0.0559	0.0489	0.1394	0.0331	0.0081	0.0183	0.0507	0.1442	0.0785	0.1953	0.0169	Y	0.5642
AC2-51	0.2050	0.0626	0.0547	0.1559	0.0370	0.0091	0.0205	0.0568	0.1318	0.0718	0.1785	0.0154	Y	0.6016

ID	La ₂ O ₃	Ce ₂ O ₃	Pr ₂ O ₃	Nd ₂ O ₃	Sm ₂ O ₃	Eu ₂ O ₃	Gd ₂ O ₃	Er ₂ O ₃	Al ₂ O ₃	B ₂ O ₃	SiO ₂	SrO	minor ^(a)	Ln ₂ O ₃
AC2-52	0.2194	0.0426	0.0372	0.1062	0.0252	0.0062	0.0140	0.0387	0.1691	0.0921	0.2290	0.0198	Y	0.4895
AC2-53	0.2146	0.0493	0.0431	0.1228	0.0291	0.0072	0.0161	0.0447	0.1567	0.0853	0.2122	0.0183	Y	0.5269
AC2-54	0.2002	0.0693	0.0605	0.1725	0.0409	0.0101	0.0227	0.0628	0.1194	0.0650	0.1617	0.0140	Y	0.6390
AC2-55	0.2100	0.1120	0.0489	0.0835	0.0331	0.0082	0.0183	0.0508	0.1444	0.0786	0.1955	0.0169	N	0.5648

(a) N – Indicates no minor component addition, Y - Indicates that the glass has been spiked with a mixture of minor components (0.01 mass % Fe₂O₃, 0.005 NiO, 0.005 Cr₂O₃, 0.005 CaO, 0.005 Na₂O, 0.005 K₂O, and 0.05 mass % MnO)

2.0 Experimental Procedures

2.1 Liquidus Temperature

T_L was measured following Method B of PNNL technical procedure GDL-LQT, “Standard Test Methods for Determining the T_L of Waste Glasses and Simulated Waste Glasses,” latest revision. A run order was not used for determining T_L for individual samples due to time constraints, but samples were grouped together in furnaces operating at a specific temperature. The final measurements used to determine T_L were confined to one of three validated furnaces to prevent differences of temperature profiles between furnaces from biasing results. In many cases, additional heat treatments in alternate furnaces are reported (See Appendix A); however, these measurements were not used in final T_L determination.

As glass samples were received from SRTC, they were logged and analyzed by optical microscopy (OM) for crystalline material or inhomogeneities. Approximately 300 g of each glass was split out to measure η (See Section 2.2). The remaining glass was for T_L testing. The T_L test samples were crushed in a tungsten carbide milling chamber, sieved to capture 4.0 to 0.5 mm grains, ultrasonically washed for 2 minutes in ethanol, and dried at 90°C following the technical procedure. Each glass sample had its own platinum crucible (approximately 1.27 cm³) and lid, which were cleaned between heat treatments. Samples were heat treated for 24 ± 2 h in calibrated and temperature-profiled furnaces.

Each glass was initially tested with three to nine preliminary heat treatments, which narrowed the temperature range in which T_L would be determined and reported. The first heat treatments were at roughly 1200°C, and new samples were heat treated at progressively higher (or lower) temperatures in roughly 20°C to 50°C intervals until the heat-treated glass sample appeared crystal free by low magnification OM. Based on these results, the temperature difference between the lowest temperature at which samples did not form crystals (T_A) and the highest temperature at which samples did form crystals (T_C) was narrowed by additional heat treatments. Data were then collected on the samples tested near the temperature region of T_L . Some of the samples that were used for XRD and scanning electron microscopy (SEM) came from the initial heat treatments because the samples near T_L did not have sufficient crystal-number density and crystal size for useful microstructural analysis. Care was taken to ensure that the crystals analyzed matched those found at T_L .

Three Del TechTM furnaces were used for final T_L determination. The furnaces were profiled for temperature differences in the hot zone where samples were heat treated using certified thermocouples and calibrated thermocouple readouts. The method was validated by heat treating SRM-773 T_L Glass from the National Institute of Standards and Technology (NIST) at the beginning, middle, and end of the test period.

Crystals were identified by OM (at magnifications as high as 500×), X-ray diffraction (XRD), and SEM, accompanied by energy dispersive spectroscopy (EDS), to characterize the chemical composition of analyzed samples. XRD and SEM/EDS were used for phase

identification of those crystals observed by OM. Familiar phases observed by OM were not repeatedly characterized with XRD or SEM.

2.2 Viscosity

Glass η s were measured as functions of T using a spindle viscometer. The measurements were obtained in accordance with the procedure ASTM C 965, "Standard Practice for Measuring Viscosity of Glass Above the Softening Point," using PNNL procedure GDL-VIS, Rev. 1, "Standard Viscosity Measurement for Vitrified Nuclear Waste." The η data were evaluated and fit to either single or multiple Arrhenius functions:

$$\ln\eta = E + F/T \quad (2)$$

where E and F are T -independent coefficients. Many $\ln\eta$ - $1/T$ curves showed nonlinear behavior, which is rare in silicate glasses and was not observed in the Phase I study. Therefore, great care was taken in the analyses of η - T data for each glass. Any η data points obtained at temperatures below the measured T_L were not considered in the calculation due to the potential impact of crystals on η . To obtain the most data possible on these glasses, data were collected at η s below the limit for the viscometer's current setup. Viscosity values below 0.549 Pa·s, $\ln[\eta(\text{Pa}\cdot\text{s})] = -0.600$, are listed for information only and were not used in any calculations. Finally, the $\ln\eta$ - $1/T$ curve was evaluated for linear trends. In many cases, a single line accurately described the $\ln\eta$ - $1/T$ behavior (the glass behaved in an Arrhenian fashion), and in other glasses (nonlinear), it did not. For the nonlinear glasses, the η_{1350} was interpolated from the measured data at T closest to and on either side of 1350°C with Equation (2). In some cases, there were insufficient data to interpolate the η_{1350} ; in these cases, the value was obtained by extrapolation of the available data or was not estimated at all if less than two data points were available. The exact steps used to determine the η - T function and η_{1350} are described in Section Appendix A for each glass.

2.3 Quality Assurance

All data were collected and reported under the PNNL Spent Nuclear Fuel Quality Assurance (QA) Program, SNF-70-001. The SNF QA Program is an NQA-1-based program that also conforms to the requirements of DOE/RW-0333P, Quality Assurance Requirements and Description. Quality assurance oversight within PNNL was provided by a Quality Engineer matrixed to this work from the independent Process Quality Department. Additional surveillance was provided by the SRTC QA staff as documented in Surveillance Report 99-SUR-11-020.

3.0 Calibration and Validation

3.1 Liquidus Temperature

The three Del Tech[™] furnaces were checked initially, in the middle of the testing period (see note below for Furnace #8), and at the end of the testing period using Standard Reference Material number 773 (SRM-773) glass to validate the accuracy of the furnaces, thermocouples, and thermocouple readouts used for T_L measurement. SRM-773 glass has a reported T_L of $988^\circ\text{C} \pm 3^\circ\text{C}$ for Method A, the trough-type platinum container test, and $991^\circ\text{C} \pm 5^\circ\text{C}$ Method B, the perforated platinum plate test (NBS 1980). A different method was used than what is described in the SRM-773 certificate. All of the validation test measurements are summarized in Table 3.1.

Table 3.1. T_L Validation Data

Furnace	IN	T ($^\circ\text{C}$)	OUT	Observations
3	3:00pm 5/3	988	3:00pm 5/4	crystals
3	3:00pm 5/4	993	2:55pm 5/5	no crystals
3	3:00pm 5/5	990	2:55pm 5/6	no crystals
3	3:00pm 5/6	986	2:55pm 5/7	crystals $T_L = 989^\circ\text{C}$
3	11:58am 6/7	986	10:52am 6/8	crystals $T_L = 988^\circ\text{C}$
3	3:45pm 8/23	990	2:00pm 8/24	no crystals
3	3:45pm 5/25	985	3:25pm 8/26	few crystals $T_L = 987^\circ\text{C}$
8	3:00pm 6/11	993	2:20pm 6/12	no crystals
8	5:00pm 6/15	990	3:20pm 6/16	no crystals
8	5:00pm 6/16	986	3:00pm 6/17	few crystals
8	5:00pm 6/17	982	3:00pm 6/18	crystals $T_L = 988^\circ\text{C}$
8	11:03am 8/4	992	1:14pm 8/5	no crystals
8	2:00pm 8/9	989	3:30pm 8/10	no crystals
8	4:15pm 8/10	984	4:00pm 8/11	few crystals
8	4:30pm 8/12	980	5:00pm 8/13	crystals $T_L = 986^\circ\text{C}$
12	3:00pm 4/13	1001	1:00pm 4/14	no crystals
12	3:00pm 4/14	991	3:00pm 4/15	no crystals
12	3:00pm 4/20	982	3:00pm 4/21	crystals
12	3:00pm 4/21	988	3:00pm 4/22	crystals $T_L = 990^\circ\text{C}$
12	2:45pm 7/9	987	1:00pm 7/10	crystals
12	5:15pm 7/18	992	3:15pm 7/19	no crystals $T_L = 989^\circ\text{C}$
12	12:00pm 8/4	994	1:14pm 8/5	no crystals
12	2:00pm 8/9	992	3:30pm 8/10	no crystals
12	4:15pm 8/10	988	4:00pm 8/11	no crystals
12	4:30pm 8/12	984	5:00pm 8/13	one crystal
12	4:30pm 8/17	980	5:00pm 8/18	crystals $T_L = 985^\circ\text{C}$

To summarize the validation tests in each furnace with SRM-773 glass, Furnace #3 had a T_L of 989°C (beginning), 988°C (mid), and 987°C (final), Furnace #8 had T_L of 988°C (beginning) and 986°C (final), and Furnace #12 had a T_L of 990°C (beginning), 989°C (mid), and 985°C (final). Furnace #8 was used mainly for preliminary tests; therefore, only two validation tests were performed during its six weeks of use for final T_L measurements.

The gradual decline in T_L temperatures for the validation tests (e.g., Furnace #3, $T_L = 989^\circ\text{C}$ in May 1999 and by late August 1999, $T_L = 987^\circ\text{C}$) could be attributed to the evaporation of rhodium from the “Type S” thermocouple wire used in the thermocouples for temperature measurements. Phase II testing was at much higher temperatures than Phase I, and the constant use of the furnaces at these high temperatures is known to slowly degrade the thermocouples, causing the electrical signal from the wire to drop slightly in voltage output, which would cause the measured temperature to decline.

3.2 Viscosity

To validate the glass η data, the η of a standard glass was repeatedly measured. Each measured η - T relationship of the SRM for high-temperature η (SRM-710a) (NIST 1991) was referred to as a run. Samples of this glass were tested at the beginning (Val-01) and end of this study (Val-03) and twice during the study (Val-01a and Val-02) for a total of four runs. The η - T data from these runs are listed in Table 3.2.

Table 3.2. Viscosity-Temperature Data for Validation Glasses

Val-01		Val-01a		Val-02		Val-03	
T (°C)	η (Pa·s)	T (°C)	η (Pa·s)	T (°C)	η (Pa·s)	T (°C)	η (Pa·s)
1136	227.405	1135	248.029	1137	256.169	1137	235.003
1185	122.914	1184	133.711	1186	137.310	1186	126.375
1234	70.454	1233	76.683	1235	78.898	1235	72.807
1283	43.182	1282	47.495	1284	47.553	1284	44.115
1332	28.157	1283	46.737	1333	30.447	1333	28.404
1382	17.915	1283	46.737	1382	19.153	1382	17.946
1431	12.346	1331	29.313	1431	13.152	1431	12.411
1480	8.381	1381	19.308	1480	8.736	1480	8.413
1529	6.254	1430	13.442	1529	6.576	1529	6.576
1530	6.197	1479	9.219			1529	6.302
		1529	6.616				

The η - T data from each run were fitted to the VTF model (Equation 1), and the within-run data variability was estimated using the uncertainties associated with these fits. The between-run variability was estimated by pooling η data from similar temperatures (within 1°C). To estimate the correct degrees of freedom, a method called the “Satterwaithe” procedure was employed for each model (Neter et al. 1990). This procedure pools the estimates and their

degrees of freedom to provide an estimate such that the uncertainties can be combined. The estimate of total variance is the combination of the between-run and within-run variances.

Once the total estimates of uncertainty were estimated, 95% confidence intervals were constructed to compare the measured PNNL glasses to the NIST standards (function of η). An asymptotic t-distribution is appropriate for estimating the intervals, using the degrees of freedom derived from the Satterwaithe procedure and the combined estimate of within-run and between-run uncertainty from the variance-covariance matrix and the between-run variability. The measured η interval corresponded with that reported by the National Bureau of Standards (NBS) for three of the four validation runs over the entire measured-temperature range. However, the measured η was slightly higher than the certified values in one of the validation runs (Val-02) at temperatures below 1282°C. Figure 3.1 compares the $\ln[\eta] - T$ curves for the measured values from run Val-02 (dotted lines) with those reported with the SRM-710a certificate (solid lines) with associated uncertainties. The uncertainty associated with this run (Val-02) was also significantly higher than in the other three runs, giving unusually broad error bands.

Figure 3.1. Comparison of Measured (Val-02) and Certified η for SRM 710a Glass as a Function of Temperature

As the uncertainty of any η -temperature data point is a combination of the within-run and between-run uncertainties, it is difficult to accurately estimate the uncertainties at any measured point for Phase II test glasses. The within-run uncertainty is estimated using a fit of the VFT data (an estimate of the variability seen that cannot be explained by the model), while the between-run uncertainty is calculated separately.

4.0 Discussion of Results

Detailed results for each glass are described in Appendix A. These descriptions include a discussion of each measurement performed to identify the η -T behavior, T_L , and primary crystalline phase of each glass. The results of the study as a whole are discussed in the following sections. General trends in T_L data are given in Section 4.1, and η -T data are summarized in Section 4.2.

4.1 Liquidus Temperature

The T_L s of the 55 Phase II glasses were between 1153°C (AC2-52) and 1405°C (AC2-04). Only two glasses had T_L s above the 1350°C limit, AC2-04 and AC2-18. Two crystal types, rare earth silicates (RESs) and aluminum oxide (Al_2O_3), were identified as the primary crystalline phases in the Phase II glasses using a combination of XRD, SEM/EDS, and OM. This differs from Phase I glasses in which aluminosilicate crystals were found and Al_2O_3 was not. These data are summarized in Table S.1. The primary crystalline phase, T_L , T_A , and T_C are summarized in Table 4.1 along with notations regarding primary crystalline phase and budding, described in Section 4.1.4. Appendix A (Glass Property Measurements) contains the detailed explanation of T_L measurements for each glass, including T_L measurement data, OM and SEM micrographs, and analyses results from XRD and EDS.

Table 4.1. T_L and Primary Crystalline Phase for 55 Am/Cm Phase II Glasses

Glass ID	T_C (°C)	T_A (°C)	T_L (°C)	Phase	buds	Glass ID	T_C (°C)	T_A (°C)	T_L (°C)	Phase	buds
AC2-01	1306	1315	1308	RES		AC2-29	1296	1306	1300	RES	yes
AC2-02	1252	1262	1257	Al_2O_3		AC2-30	1225	1236	1227	RES	
AC2-03	1312	1315	1313	RES	yes	AC2-31	1301	1306	1305	RES	
AC2-04	1401	1409	1405	RES		AC2-32	1260	1265	1264	RES	yes
AC2-05	1240	1241	1241	RES		AC2-33	1265	1274	1271	RES	yes
AC2-06	1306	1314	1309	RES	yes	AC2-34	1225	1235	1232	RES	
AC2-07	1204	1213	1209	RES		AC2-35	1284	1289	1289	RES	
AC2-08	1241	1252	1242	RES		AC2-36	1289	1295	1294	RES	yes
AC2-09	1320	1327	1323	RES	yes	AC2-37	1302	1307	1305	RES	
AC2-10	1252	1261	1255	RES		AC2-38	1306	1312	1308	RES	
AC2-11	1289	1295	1292	Al_2O_3	yes	AC2-39	1314	1321	1318	RES	
AC2-12	1328	1335	1333	RES		AC2-40	1321	1328	1325	RES	yes
AC2-13	1179	1185	1181	RES	yes	AC2-41	1320	1327	1324	RES	yes
AC2-14	1246	1252	1250	Al_2O_3		AC2-42	1179	1191	1180	RES	
AC2-15	1275	1283	1279	RES		AC2-43	1225	1236	1227	RES	
AC2-16	1295	1299	1298	Al_2O_3	yes	AC2-44	1302	1307	1305	RES	yes
AC2-17	1265	1275	1273	RES		AC2-45	1255	1260	1259	RES	
AC2-18	1364	1374	1366	RES		AC2-46	1328	1335	1333	RES	yes
AC2-19	1242	1252	1249	RES		AC2-47	1265	1274	1272	RES	yes
AC2-20	1336	1349	1343	RES	yes	AC2-48	1179	1185	1181	RES	

Glass ID	T _C (°C)	T _A (°C)	T _L (°C)	Phase	buds	Glass ID	T _C (°C)	T _A (°C)	T _L (°C)	Phase	buds
AC2-21	1240	1249	1242	Al ₂ O ₃		AC2-49	1219	1226	1220	RES	
AC2-22	1283	1287	1284	RES		AC2-50	1265	1274	1272	RES	yes
AC2-23	1278	1284	1282	Al ₂ O ₃	yes	AC2-51	1300	1307	1305	RES	
AC2-24	1313	1321	1319	RES		AC2-52	1150	1158	1153	RES	
AC2-25	1299	1314	1305	Al ₂ O ₃		AC2-53	1191	1197	1192	RES	
AC2-26	1204	1210	1208	RES		AC2-54	1321	1328	1326	RES	yes
AC2-27	1241	1242	1242	RES		AC2-55	1249	1257	1255	RES	yes
AC2-28	1249	1264	1260	Al ₂ O ₃							

4.1.1 Rare Earth Silicate Crystals

RES was the primary phase in 47 of the 55 glasses; however, we observed four distinct morphologies of the RES when identifying these crystals: needle-like (thin and straight), hair-like (thin and wavy), plate-like, and rectangular cap. Each crystal shape was analyzed using SEM. EDS coupled to SEM showed Si, La, Nd, and O as major components and Ce, Pr, Sm, Gd, and Er as minor components in the crystals. Al is present as a minor component in some crystals, but not in others. Chemically, the RES crystals are the same regardless of morphology. Samples analyzed by XRD matched the crystal structure of neodymium germanium borate silicate (Nd₃BGe_{1.08}Si_{0.92}O₁₀). In this crystal structure, Ge could be replaced by Si and Nd could be replaced by rare earth elements to form RE₃BSi₂O₁₀, the crystal type that is probably present in these samples. Generally, the glasses within the RES primary phase field showed massive crystallinity (i.e., > 30 vol % RES) after a 24 h heat treatment at temperatures between 3°C and 30°C below T_L. As RES crystals are difficult to redissolve once crystallized, this observation should be considered when planning for off-normal operating scenarios.

The RES crystals observed as needle-shaped crystals were found individually and clustered at a focal point with the needles pointing out in all directions, both found in Figure 4.1. The size of the crystals had a wide range in length from <0.1 mm to >1.0 mm, and were approximately 1 µm to >10 µm thick.

Associated with the needle crystals are hair-like crystals, found within or near the needle clusters (Figure 4.2a). These crystals are very thin (<0.5 µm) and long (>100 µm) and curved like an eyebrow. Occasionally they are observed in a coil. These crystals may be straight also, but they are so thin that they are difficult to observe by OM. However, they are very distinct when they curve or coil. SEM/EDS scans indicated a similar chemical composition for both needle and hair-like RES crystals.

Plate-like, RES crystals (Figure 4.2b and Figure 4.3) were found in many of the samples. Unlike the plate-like RES in Phase I, some of the plate-like crystals in Phase II developed unusual structures at the narrow end of the rectangular or lathe shaped crystal (Figure 4.4 and Figure 4.5). SEM/EDS analysis found no change in chemical composition of the crystals even with the unique shapes.

Figure 4.1. Examples of Needle-Like RES Crystals

Figure 4.2. Examples of Needle-Like, Hair-Like, and Plate-Like RES Crystals

Figure 4.3. SEM Micrograph of Plate-Like RES Crystals in AC2-44 (1302°C)

Another unique crystal shape, observed only in the Phase II study, is the rectangular cap (Figure 4.6) found in seven samples: AC2-09 (1314°C), AC2-29 (1288°C), AC2-40 (1321°C), AC2-41 (1321°C), AC2-44 (1321°C), AC2-46 (1321°C), and AC2-54 (1321°C). This RES crystal shape is found in samples with dense crystal numbers, crystals settling on the bottom, and

the top smooth glass surface. The rectangular cap crystals are found floating on the surface in the middle of the sample, not near other crystal types. Many samples were thin-sectioned in an attempt to analyze this odd-shaped crystal type, but the rectangular cap was never found below the surface. AC2-46 was sent as a bulk sample, and the surface was analyzed by SEM/EDS. The EDS scan on these crystals had a similar profile to that of RES crystals with other shapes.

Figure 4.6. SEM Micrograph of Rectangular Cap RES Crystals on the Surface of AC2-46 (1321°C)

4.1.2 Aluminum Oxide Crystals

Aluminum oxide was identified as the primary phase in 8 Phase II glasses (see Table 4.1). Two distinct Al_2O_3 crystal morphologies formed in the same melt, a plate-like crystal (Figure 4.7a) and a garnet shaped crystal (Figure 4.7b). SEM/EDS identified high aluminum and oxygen concentrations in both crystal types (Figure 4.8, along with Figure 4.9 and Figure 4.10). No chemical difference between the two differently shaped crystals could be detected. Many samples of glasses crystallizing Al_2O_3 were analyzed by XRD, with no conclusive results. Therefore, the structure(s) of the Al_2O_3 crystals was not identified.

In glasses with Al_2O_3 as the primary phase, RES formed as a secondary phase at 30°C to 40°C below T_L . In samples that were 50 to 100°C < T_L , RES crystals would be very high (in both size and number density of crystals), and Al_2O_3 crystals would not be found. The Al_2O_3 crystal was never found in samples where RES was the primary phase, except when budding was observed (see Section 4.1.4 for a description of budding and Appendix A for further details) where Al_2O_3 crystals were detected by SEM/EDS in the buds.

Figure 4.7. Aluminum Oxide Crystals

Figure 4.8. SEM Analysis of AC2-21 (1229°C)

Figure 4.9. SEM Micrograph of a Garnet Shaped Al₂O₃ Crystal in AC2-14 (1232°),

Figure 4.10. EDS Spectrum of a Crystal in Figure 4.9

4.1.3 “As-Received” Glasses

“As-Received” glasses were analyzed by OM to determine if crystalline or immiscible liquid phases existed. Three glasses, AC2-04, -18, and -19, were found to have RES crystals present in streaks as minor inclusions. The crystals appear very similar to those found in AC2-04 (see Figure 4.1). The crystal fraction is not determined, but was estimated to be well less than 1 percent. Only a few scattered crystals were found in AC2-19. All other samples appeared to be free of crystals based on OM analyses.

4.1.4 Sample “Budding”

Some of the heat-treated samples displayed a phenomenon coined “budding.” In these samples, nodes or buds would form at the folds of the box crucible as shown in Figure 4.11. This phenomenon was thought to be caused when a portion of the melt was pulled by capillary force to the top of the Pt folds and exposed to the open atmosphere. This phenomenon was not consistent. In other words, no pattern for the behavior was determined. If budding occurred, it would happen once, sometimes twice, in the series of heat treatments for the glass, usually at one of the highest temperatures runs, but not consistently at the highest heat-treatment temperature. This phenomenon was also observed in some glasses during Phase I testing, but only with a few samples. Forty percent of the Phase II glasses experienced budding in some heat-treated samples (see Table 4.1 and the “Comments” section of the Heat-Treatment Summary for Determination of T_L for each glass in Appendix A).

Figure 4.11. Photograph of Buds on Crucible Edges

Buds that formed on heat-treated crucible walls were dull, opaque, and rough, having lost the appearance of the smooth, highly reflective glass surface. AC2-16, heat treated at 1297°C, had buds form on the crucible folds. Figure 4.12 is an SEM micrograph of the wing of the platinum box crucible of this sample. The platinum wall of the fold can be seen as a smooth metallic section at the base of the picture. The buds were found on the top of the platinum folds

and appear as undulating mounds. Figure 4.13 is a magnified area of Figure 4.12 and shows the crystals densely packed on the surface of the bud.

Glass AC2-29 heat treated at 1299°C also formed small buds. This sample was examined by SEM (Figure 4.14), polished with 600-grit polishing paper, and re-examined (Figure 4.15). SEM micrographs show densely packed crystals at the surface of the bud (see magnified area of bud surface—Figure 4.16), but within 50 microns of the surface, the crystal number density diminishes. The surface layer contains both RES (light gray crystal phase justified in Figure 4.17) and Al_2O_3 crystals (black phase justified in Figure 4.18), but below this dense crystal layer, Al_2O_3 crystals did not exist. RES crystals observed in the remaining cross section of the polished sample were estimated at 3 to 5 volume percent by visual inspection.

99S186-1(AC2-16:1297 °C) OVERALL VIEW;GLASS ON CRUCIBLE WING

Figure 4.12. SEM Micrograph of Buds on Crucible Wing of Platinum Fold in AC2-16 (1297°C)

99S186-2(AC2-16:1297 °C)

Figure 4.13. Magnification of Figure 4.12

Budding was not found to affect T_L measurement. Several samples were tested in crucibles where the wings were folded in tightly, and a platinum lid covered the entire top of the crucible during sample heat treatment. No differences existed between these samples and those heat treated at the same temperatures that produced buds. Budding is a localized effect that does not appear to change conditions in the bulk glass.

99S183-1(AC2-29:1299°C) CRUCIBLE WING

Figure 4.14. SEM Micrograph of Small Buds on Wing of Crucible in AC2-29 (1299°C),

99S183-3(AC2-29:1299 °C) OVERALL VIEW.POLISHED WING

Figure 4.15. SEM Micrograph of AC2-29 (1299°C) after Crucible Wing is Polished (note: platinum wall exposed on left side of sample)

99S183-4(AC2-29:1299 °C) UPPER RIGHT IN 99S183-3

Figure 4.16. SEM Micrograph of the Bud Outer Surface in AC2-29 (1299°C)

4.1.5 Quantitative Analysis of RES Crystals by SEM

Quantitative SEM/EDS analyses^b were performed to determine if differences in chemical composition occurred in the RES crystals. Glasses AC2-04 heat-treated at 1398°C and AC2-05 heat treated at 1223°C were chosen for the analysis since there was a large difference in T_L values, 1405°C for AC2-04 and 1241°C for AC2-05. In addition, the bulk chemical composition was quite different between the two glasses. Therefore, we expect a difference in the chemical composition of the RES crystals formed in the two glasses. The RES crystals in the two glass samples were different sizes. The RES crystals found in AC2-04 were 5 to 10 times longer than those found in AC2-05, though they were both needle-shaped crystals.

^b Analysis was performed on an elemental basis using Al_2O_3 , SiO_2 (quartz), LaB_6 , CeO_2 , PrF_3 , NdF_3 , GdF_3 , and ErF_3 as instrument calibration standards for their representative elements. To correct for X-ray absorbency and fluorescence, a mathematical iteration (quantitative ZAF correction method) was applied. The comparison between SEM glass-matrix composition analysis and the normalized target composition (mole percent) had an error margin of less than ± 10 percent for all major elements.

The analyzed crystal compositions are listed in Table 4.2, and glass compositions are listed in Table 4.3. On a mole-percent basis, Si was the major element in the RES crystal structure (40.5% in AC2-04 and between 43.6 to 46.4% for AC2-05). The Si concentration in glass appears to influence the RES crystal composition. As Si increased in the glass composition from 30.6 mole percent in AC2-04 glass to 37.6 mole percent in AC2-05 glass, so did its concentration in the crystal structure (again 40.5 to about 45 mole percent). Lanthanum, the element with the second highest concentration in both the crystal structure and glass, also increased from 23.65 mole percent in AC2-04 to about 26 mole percent in AC2-05. This is not intuitive since the concentration of La in the glass decreases from 18.2 mole percent in AC2-04 to 14.3 mole percent in AC2-05 (measured by EDS) and from 16.1 mole percent to 13.9 mole percent (target composition) for AC2-04 and AC2-05, respectively. Similarly, Ce concentration in the RES crystal increased while its concentration in the base glass decreased. Praseodymium and Nd followed the expected pattern of increasing in concentration in the crystal with higher concentration in the base glass. The most notable difference is in the concentration of Al in the two crystals. This evidence, however, does not suggest a different crystal type as Al is only a minor element in both spectra, and the total rare earth concentration in both crystals is similar.

Table 4.2. Quantitative Analysis of Crystals in Glass (in mole percent metals normalized to 100)

	AC2-04		AC2-05	
	Spectrum 1	Spectrum 2	Spectrum 1	Spectrum 2
Al	2.19	2.63	0.42	0.22
Si	40.41	40.62	43.67	46.37
La	23.72	23.63	26.50	25.14
Ce	2.54	2.69	3.50	3.35
Pr	6.20	6.75	3.26	3.15
Nd	20.99	21.25	12.11	11.43
Sm	not analyzed	not analyzed	5.07	4.87
Gd	1.50	0.67	2.84	2.77
Er	2.44	1.78	2.63	2.70
Total	100.00	100.00	100.00	100.00

Table 4.3. Quantitative Analysis of Glass Matrix Compared to Target Composition (in mole percent metals normalized to 100)

	AC2-04		AC2-05	
	SEM Analysis	Normalized Target Comp	SEM Analysis	Normalized Target Comp
Al	27.76	26.64	33.21	32.90
Si	33.56	30.58	39.84	37.60
La	18.23	16.11	14.28	13.93
Ce	2.62	2.25	1.93	1.95
Pr	4.03	5.05	1.34	1.74
Nd	11.64	12.55	4.85	5.98
Sm	not analyzed	3.27	2.07	2.83
Gd	0.74	1.76	1.07	1.52
Er	1.42	1.79	1.41	1.55
Total	100.00	100.00	100.00	100.00

4.1.6 Glass Composition Effects

A linear relationship between Ln_2O_3 concentration in glass with T_L and with a primary-phase boundary was shown for Phase I glasses in Section 1.2.2. The effect of Ln_2O_3 concentration on T_L of Phase I and II glasses is shown in Figure 4.19. This comparison shows a reasonably close match in Ln_2O_3 effect on T_L for Phase I and Phase II glasses, despite the fact that SiO_2 , Al_2O_3 , B_2O_3 , and SrO concentrations varied independently in Phase II glasses and not in Phase I glasses. There is a clear primary-phase boundary between alumino-silicate and RES fields. However, the glass compositions in the Al_2O_3 primary phase field are intermixed with those in the RES primary phase field. Further examination of compositions was required to separate the two primary-phase fields. Figure 4.20 shows a composition projection of Phase I and II glasses in a $\text{SiO}_2 - \text{Al}_2\text{O}_3 - \text{Ln}_2\text{O}_3$ space with primary crystalline phases noted as symbols. A clear distinction between the RES and Al_2O_3 primary-phase fields can be seen by the $\text{Al}_2\text{O}_3/\text{SiO}_2$ ratio. All glasses with a ratio above 1 are in the Al_2O_3 primary phase field.

Figure 4.19. T_L as a Function of Ln_2O_3 Concentration (in mass fraction) in Phase I and I Glasses (S-Alumino-Silicate Crystals in Phase I Glasses, *-RES Crystals in Phase I Glasses, +-RES Crystals in Phase II glasses, and A- Al_2O_3 Crystals in Phase II Glasses)

Figure 4.20. Composition Projection of Phase I and II Glasses in a $\text{SiO}_2 - \text{Al}_2\text{O}_3 - \text{Ln}_2\text{O}_3$ Space with Primary Crystalline Phases Noted as Symbols: S-Alumino-Silicate, +RES, and A- Al_2O_3

4.2 Viscosity

The η - T relationship was measured for Phase II glasses using a high-temperature spindle viscometer. Generally, the data did not conform to the standard η -T relationship as described by the VTF model (Equation 1). Figure 4.21 compares the $\ln[\eta]$ -1/T behavior of a “typical” silicate glass with that of AC2-14. The $\ln[\eta]$ of AC2-14 is nearly piecewise linear with variation in 1/T. For Val-01, the variation of $\ln[\eta]$ with 1/T is nearly linear with a positive curvature, which is typical of silicate glasses.

Figure 4.21. Comparison of $\ln[\eta]$ - $1/T$ Behavior of (a) AC2-14 and (b) Val-01

For glasses with linear $\ln[\eta_{1350}] - 1/T$ relationship the η_{1350} value of Phase II glasses was estimated by a linear interpolation of $\ln[\eta_{1350}]$ vs $1/T$ for the entire data set. For the nonlinear glasses, the η_{1350} was interpolated from the measured data at T closest to and on either side of 1350°C with Equation (2). In some cases, there was insufficient data to interpolate the η_{1350} , in these cases the value was obtained by extrapolation of the available data or wasn't estimated at all if less than two data points were available. The exact steps used to determine the η - T function and η_{1350} are described in Section Appendix A for each glass. This was in contrast to the VTF model (Equation 1), which was used to estimate η_{1350} values for glasses in the Phase I study. Table 4.4 summarizes the linear η - T parameters (E and F in Equation 2), η_{1350} , and the method used to derive these values for the Phase II glasses.

Table 4.4. Summary of Phase II Viscosity Data

Glass ID	E	F (K)	η_{1350} (Pa.s)	Comments/Method
AC2-01	-17.449	28,908	1.44	linear
AC2-02	-20.390	32,760	0.81	nonlinear
AC2-03	-19.528	30,769	0.57	two data points
AC2-04				fewer than two data points
AC2-05	-15.642	26,669	2.20	nonlinear
AC2-06	-9.177	14,314	0.70	two data points
AC2-07	-19.963	33,688	2.21	nonlinear
AC2-08	-12.177	20,627	1.70	nonlinear
AC2-09	-18.999	30,452	0.79	linear
AC2-10	-8.973	14,981	1.29	nonlinear
AC2-11	-15.613	25,279	0.96	nonlinear
AC2-12	-4.052	7,147	1.42	nonlinear
AC2-13	-13.098	22,267	1.86	nonlinear
AC2-14	-15.750	25,380	0.89	nonlinear

Glass ID	E	F (K)	η_{1350} (Pa-s)	Comments/Method
AC2-15	-15.571	25,731	1.33	linear
AC2-16	-17.671	28,821	1.09	nonlinear
AC2-17	-15.154	24,804	1.14	linear
AC2-18				fewer than two data points
AC2-19	-14.837	24,619	1.39	linear
AC2-20				fewer than two data points
AC2-21	-14.596	23,443	0.86	linear
AC2-22	-15.045	24,893	1.34	linear
AC2-23	-14.288	23,182	1.00	linear
AC2-24	-19.831	31,376	0.61	two data points
AC2-25	-15.537	25,492	1.18	two data points
AC2-26	-19.470	32,858	2.17	nonlinear
AC2-27	-13.273	22,608	1.93	nonlinear
AC2-28	-18.947	30,512	0.86	nonlinear
AC2-29	-16.871	26,717	0.66	linear
AC2-30	-9.360	15,680	1.35	nonlinear
AC2-31	-7.207	12,340	1.49	nonlinear
AC2-32	-19.993	32,717	1.18	nonlinear
AC2-33	-14.740	24,182	1.17	linear
AC2-34	-11.883	19,877	1.44	nonlinear
AC2-35	-15.684	25,573	1.08	two data points
AC2-36	-15.187	24,702	1.03	linear
AC2-37	-17.594	28,495	0.96	linear
AC2-38	-16.272	26,319	0.95	linear
AC2-39	-16.986	27,416	0.91	linear
AC2-40	-18.659	30,139	0.91	two data points
AC2-41	-17.554	28,261	0.87	linear
AC2-42	-11.555	19,663	1.75	nonlinear
AC2-43	-8.382	14,049	1.32	nonlinear
AC2-44	-15.956	25,803	0.94	linear
AC2-45	-14.669	23,967	1.10	linear
AC2-46	-16.753	26,832	0.80	linear
AC2-47	-14.938	24,506	1.17	linear
AC2-48	-11.809	20,058	1.73	nonlinear
AC2-49	-10.729	18,031	1.46	nonlinear
AC2-50	-14.338	23,449	1.12	linear
AC2-51	-16.473	26,651	0.95	linear
AC2-52	-18.357	31,054	2.17	nonlinear
AC2-53	-10.844	18,520	1.76	nonlinear

Glass ID	E	F (K)	η_{1350} (Pa·s)	Comments/Method
AC2-54	-17.617	28,349	0.86	linear
AC2-55	-15.600	25,530	1.14	nonlinear

The η_{1350} values of Phase II test glasses range from 0.57 Pa·s for AC2-03 to 2.21 Pa·s for AC2-07, both exterior vertex glasses. All Phase II glasses passed the 0.3 Pa·s to 2.4 Pa·s constrain on η_{1350} established by Martin (1999). However, the odd η -T behavior of these glasses warrants further study.

A linear relationship between the Ln_2O_3 concentration in glass and the $\ln[\eta_{1350}]$ value was shown for Phase I glasses in Section 1.2.3. The effect of Ln_2O_3 concentration on $\ln[\eta_{1350}]$ of Phase II glasses is shown in Figure 4.22. This relationship is compared to that for Phase glasses in Figure 4.23. This comparison shows a reasonably close match in the Ln_2O_3 effect on $\ln[\eta_{1350}]$ for Phase I and Phase II glasses, despite the fact that SiO_2 , Al_2O_3 , B_2O_3 , and SrO concentrations varied independently in Phase II glasses and not in Phase I glasses. Specifically, the Ln_2O_3 partial specific $\ln[\eta_{1350}]$ for Phase I and Phase II glasses are -3.1 and -3.0 , respectively. Generally, some of the Phase I glasses and some of the nonlinear Phase II glasses fell off of the linear trend.

Figure 4.22. Effect of Ln_2O_3 Concentration on $\ln[\eta_{1350}]$ for Phase II Glasses (symbol numbers represent glass numbers)

Figure 4.23. Effect of Ln_2O_3 Concentration (in mass fraction) on $\ln[\eta_{1350}]$ for Phase I and II Glasses (symbols represent: A-Phase I glasses, B-Linear Phase II glasses, and N- Nonlinear Phase II glasses)

5.0 Recommendations

Based on the glass-property measurement results from Phase I and Phase II glasses, we recommend the following in considering in plant operation and planning for future testing.

1. Based on the following information,

- the glass property criteria set by Martin (1999) and Peeler and Edwards (1999)
- the favorable measured η_{1350} and T_L values
- the assumption that all other property restrictions are met by a majority of Phase I glasses,

we recommend that the Am/Cm process proceed to design and implementation using the composition regions studied in Phases I and II. In other words, no critical glass-property flaws were identified in this study, and only two glasses failed to meet the $T_L < 1350^\circ\text{C}$ criteria.

2. The anomalous η -T behavior is indicative of glass structural changes occurring at high temperatures. These changes may be due to medium (5 to 50 Å) range-order reconfigurations in the melt or may be due to nano-crystal (e.g., crystal nuclei) formation. In either case, these changes could have a pronounced impact on melter operation and the current scientific understanding of rare earth-alumino-borosilicate melts. We recommend further study aimed at identifying the cause for the anomalous η -T behavior and its impact on properties critical to plant operation.
3. The composition region studied in Phase II is generally more fragile than that studied in Phase I. In other words, Phase II glasses showed a higher tendency to grossly crystallize at temperatures slightly below T_L than Phase I glasses. Since massive crystallization in the melter could be detrimental to plant operation, we recommend that studies be performed to understand gross crystallization observed in many Phase II glasses. These studies should focus on determining the composition regions most susceptible to and the conditions (T-t) that allow massive crystallization.
4. The crystalline phases formed under the T_L of Phase I and Phase II glasses are highly refractory minerals, alumino-silicates, RESs, and aluminum oxide. If a large fraction of these crystalline phases were to form in the melter, it would be difficult to redissolve the crystals and return to a homogeneous melt. We recommend that plans be made to mitigate such an event if it were to occur during off-normal operations. This planning should include study of 1) methods for redissolving the crystals, 2) characterization of the properties of a crystallized melt body, and 3) methods for disposition of a full melter body and melter replacement.
5. The rare earth-alumino-borosilicate melts have been shown to compositionally segregate at standard plant-operating temperatures into a higher and lower density melt. It is recommended that the process of conversion of the frit and simulated oxalate feed be studied. These studies should continue to develop an understanding of the conversion chemistry, the

phenomena responsible for melt segregation, and mitigation strategies to ensure that melt segregation does not occur or is controllable in plant operation.

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Waste Management Technologies in Ceramic and Nuclear Industries, Ceramic Transaction series.

Appendix A

Glass Property Measurements

Contents

1.0 AC2-01.....	1
2.0 AC2-02.....	5
3.0 AC2-03.....	11
4.0 AC2-04.....	15
5.0 AC2-05.....	19
6.0 AC2-06.....	23
7.0 AC2-07.....	27
8.0 AC2-08.....	31
9.0 AC2-09.....	35
10.0 AC2-10.....	37
11.0 AC2-11.....	41
12.0 AC2-12.....	45
13.0 AC2-13.....	49
14.0 AC2-14.....	51
15.0 AC2-15.....	55
16.0 AC2-16.....	57
17.0 AC2-17.....	59
18.0 AC2-18.....	61
19.0 AC2-19.....	65
20.0 AC2-20.....	67
21.0 AC2-21.....	69
22.0 AC2-22.....	73
23.0 AC2-23.....	75
24.0 AC2-24.....	79
25.0 AC2-25.....	81
26.0 AC2-26.....	83
27.0 AC2-27.....	85
28.0 AC2-28.....	87
29.0 AC2-29.....	91
30.0 AC2-30.....	95
31.0 AC2-31.....	97
32.0 AC2-32.....	99
33.0 AC2-33.....	101
34.0 AC2-34.....	103
35.0 AC2-35.....	105
36.0 AC2-36.....	109
37.0 AC2-37.....	113
38.0 AC2-38.....	117
39.0 AC2-39.....	119
40.0 AC2-40.....	121
41.0 AC2-41.....	123
42.0 AC2-42.....	125
43.0 AC2-43.....	127
44.0 AC2-44.....	129

45.0 AC2-45.....	133
46.0 AC2-46.....	135
47.0 AC2-47.....	137
48.0 AC2-48.....	139
49.0 AC2-49.....	141
50.0 AC2-50.....	143
51.0 AC2-51.....	145
52.0 AC2-52.....	147
53.0 AC2-53.....	149
54.0 AC2-54.....	151
55.0 AC2-55.....	153

Appendix A

Glass Property Measurements

1.0 AC2-01

Glass AC2-01 is an extreme vertex on the inner level of glass compositions. This glass targets 58.3 mass percent Ln_2O_3 . The ratio of SiO_2 to other frit components is higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are high, low, low, and low, respectively in comparison to the standard frit composition.

1.1 Liquidus Temperature

In the final T_L measurements, four heat treatments were performed in Furnace #12 between 1296°C and 1315°C (see Table 1.1). Heat treatments at 1296°C and 1301°C had a thin crystal layer on the surface with crystals precipitating to a thick layer of settled crystals on the crucible bottom; all crystals were rare earth silicates (RES). At 1306°C, only a few crystal clusters remained on the bottom of the sample after heat treatment, and at 1315°C, the heat-treated sample appeared to be crystal free by OM. The T_L was estimated to be 1308°C with RES as the primary phase. Both XRD (Figure 1.1) and SEM (Figure 1.2) analyses were performed on a sample heat treated at 1296°C. An EDS spectrum of the glass matrix is shown in Figure 1.3. The EDS spectrum in Figure 1.4 shows the relative concentration of components in the RES crystal.

Eight preliminary heat treatments were performed between 1153°C and 1343°C at approximately 50°C intervals. Abundant crystals were observed at 1248°C and 1296°C, which matches the observations in the final heat treatments. Samples at 1319°C and 1343°C appeared to be crystal free by OM. A sample, heat treated in Furnace #3 at 1313°C, appeared to be free of crystals when examined by OM.

Table 1.1. Heat-Treatment Summary for Determination of T_L for AC2-01

Crucible	Furnace	Time/Date IN	T (°C)	Time/Date OUT	Observations	Comments
L	8	2:00pm 04/20	1198	1:00pm 04/21	Crystals	Preliminary
1	3	3:00pm 06/18	1313	1:30pm 06/19	No crystals	different furnace
1	12	2:00pm 06/23	1306	3:00pm 06/24	Few clusters	T_C
1	12	3:00pm 06/25	1296	1:20pm 06/26	Crystals	XRD/SEM
1	12	8:52am 07/06	1301	10:50am 07/07	Crystals	
1	12	4:05pm 07/17	1315	3:22pm 07/18	No crystals	T_A

Figure 1.1. XRD Scan of AC2-01 (1299°C)

Figure 1.2. SEM Micrograph of AC2-01 (1296°C) at 500×

1.2 Viscosity

Table 1.2 lists the measured η -T values for AC2-01 in chronological order. These data are shown on an Arrhenius plot ($\ln \eta$ vs. $1/T$) in Figure 1.5. With a T_L of 1308°C, all the data in Table 1.2 are valid for calculations. Equation (2) was fitted to the data to yield E and F values of -17.449 and 28908 K, respectively. This fit, which accurately reflects the data, was then used to calculate a η_{1350} value of 1.44 Pa·s.

Table 1.2. Viscosity-Temperature Data for AC2-01

T (°C)	η (Pa·s)	$10000/T$ (K ⁻¹)	$\ln[\eta(\text{Pa}\cdot\text{s})]$
1383	1.017	6.039	0.017
1404	0.827	5.963	-0.190
1424	0.643	5.893	-0.441
1384	1.004	6.035	0.004
1364	1.228	6.109	0.206
1344	1.515	6.184	0.415
1384	1.004	6.035	0.004

Figure 1.5. Measured $\ln$ Viscosity vs. $10000/T$ for AC2-01

2.0 AC2-02

Glass AC2-02 is an extreme vertex on the inner level of glass compositions. This glass targets 56.2 mass percent Ln_2O_3 . The ratios of Al_2O_3 and B_2O_3 to other frit components are higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are low, high, high, and low, respectively, in comparison to the standard frit composition.

2.1 Liquidus Temperature

The final T_L measurements were performed in Furnace #3 between 1232°C and 1271°C with seven heat treatments (see Table 2.1). Aluminum oxide was the only crystalline phase observed in this temperature range. Aluminum oxide was abundant in the 1232°C and 1237°C samples checked by OM with both plate-like and garnet-shaped crystals; both crystal types were transparent to visible light. SEM was used to analyze the sample at 1232°C (Figure 2.1 and Figure 2.2), but when several measurements were performed on the same sample with XRD, including a 48-h scan, no distinct peaks characterized the crystalline phase (Figure 2.3).

Aluminum oxide crystal number density diminished in the three samples between 1243°C and 1252°C, with only a few crystals observed at 1252°C. The T_L was estimated to be 1257°C with aluminum oxide as the primary phase. The RES phase was observed concurrently with the Al_2O_3 phase at < 1232°C. A 48-h XRD scan was obtained on the heat-treated sample at 1198°C with both phases matching peaks observed in the scan along with a SiO_2 phase.

Six preliminary heat treatments were performed between 1153°C and 1248°C at approximately 50°C intervals. Abundant crystals were observed at 1232°C, which matches the observations in the final heat treatments. The sample at 1248°C appeared to be crystal free by OM. One measurement was performed in Furnace #12 at 1259°C, which contained only a few crystals.

Table 2.1. Heat-Treatment Summary for Determination of T_L for AC2-02

Crucible	Furnace	Time/Date IN	T (°C)	Time/Date OUT	Observations	Comments
P	8	2:00pm 04/20	1198	1:00pm 04/21	Surface crystals	Preliminary/XRD
2	3	2:30pm 05/18	1246	2:40pm 05/19	Few crystals	
2	3	3:20pm 05/20	1232	3:35pm 05/21	Crystals on surface	
2	3	3:45pm 05/25	1237	1:45pm 05/26	Crystals on surface	
2	3	4:30pm 06/09	1243	3:00pm 06/10	Few small crystals	
2	3	4:30pm 06/10	1252	2:30pm 06/11	Few bulk crystals	T_C
2	12	2:17pm 06/20	1259	1:00pm 06/21	Few crystals	different furnace
2	3	3:20pm 07/27	1262	2:25pm 07/28	No crystals	T_A , Buds
2	3	4:40pm 07/28	1271	3:40pm 07/29	No crystals	Buds

Operator : Jim Young
Client : Mike Schweiger
Job : Job number 354 L0885 AC2-02
Spectrum 2 (6/2/99 09:56)

Figure 2.1. EDS Spectrum of the Glass Matrix of AC2-02 (1232°C)

Operator : Jim Young
Client : Mike Schweiger
Job : Job number 354 L0885 AC2-02 (1232°C)
Spectrum 1 (6/2/99 09:46)

Figure 2.2. EDS Spectrum of an Al_2O_3 Crystal in AC2-02 (1232°C)

(2) [072499a.rdl] AC2-02 1232, SCAN: 5.075,0.05/123(sec), Cu, I(cps)=3471, 07/26/99 11:40
 (1) [072499a.rdl] AC2-02 1232, SCAN: 5.075,0.05/123(sec), Cu, I(cps)=74, 07/26/99 11:40

Figure 2.3. XRD Scan of AC2-02 (1232°C) Al₂O₃ Crystal Containing Glass

2.2 Viscosity

Table 2.2 lists the measured η -T values for AC2-02 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 2.4. Six data points from Table 2.2 were excluded from calculations. Two of these points, at 1383°C and 1384°C, had measured η values below the instrument's range while four data points, at 1187°C, 1206°C, 1226°C, and 1246°C, were taken at T_s below T_L . The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -15.245 and 24410 K, respectively. The data appear to be nonlinear. Therefore, a η_{1350} value of 0.81 Pa·s was obtained by interpolation between the data points at 1345°C and 1364°C using Equation (2).

Table 2.2. Viscosity-Temperature Data for AC2-02

T (°C)	η (Pa·s)	10000/T (K ⁻¹)	$\ln[\eta(\text{Pa}\cdot\text{s})]$
1383	0.532	6.039	-0.631
1364	0.685	6.109	-0.378
1345	0.867	6.180	-0.143
1325	1.056	6.258	0.054
1306	1.306	6.333	0.267
1285	1.476	6.418	0.390
1266	1.818	6.498	0.598
1246	2.437	6.583	0.891
1226	3.352	6.671	1.210
1206	4.690	6.761	1.545
1187	6.979	6.849	1.943
1384	0.513	6.035	-0.668

Figure 2.4. Measured $\ln$ Viscosity vs. $10000/T$ for AC2-02

3.0 · AC2-03

Glass AC2-03 is an extreme vertex on inner level of glass compositions. This glass targets 60.7 mass percent Ln_2O_3 . The ratios of B_2O_3 and SrO to other frit components are higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are low, low, high, and high, respectively, in comparison to the standard frit composition.

3.1 Liquidus Temperature

Table 3.1 summarizes the final heat treatments performed on this glass. The T_L of this glass was estimated at 1313°C using Furnace #12 since only one small crystal remained during the 1312°C heat treatment, and the sample heat treated at 1315°C appeared to be crystal free by OM. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphology of the crystal as seen in Figure 3.1 and by an XRD scan of a sample heat treated at 1198°C shown in Figure 3.2.

During the 1306°C heat treatment of AC2-03, small nodules or buds of glass formed at the upper edge of the Pt/Rh box folds. Figure 4.11 (body of report) shows an example of these buds. The buds are thought to be formed by capillary forces drawing glass to the top of the Pt/Rh foil folds. Further analysis of these buds was performed and is reported in Section 4.1.4 of this report.

Eight preliminary heat treatments were performed between 1153°C and 1343°C at approximately 50°C intervals. Abundant crystals were observed at 1248°C and 1296°C, which matches the observations in the final heat treatments. The sample at 1343°C appeared to be crystal free by OM.

Table 3.1. Heat-Treatment Summary for Determination of T_L for AC2-03

Crucible	Furnace	Time/Date IN	T (°C)	Time/Date OUT	Observations	Comments
A	8	2:00pm 04/20	1198	1:00pm 04/21	Crystals	Preliminary/XRD
3	12	3:40pm 06/21	1312	3:30pm 06/22	One small crystal	T_C
3	12	2:00pm 06/23	1306	3:00pm 06/24	Few prisms	Buds
3	12	4:05pm 07/16	1315	3:22pm 07/17	No crystals	T_A , Buds

Figure 3.1. Optical Micrograph of RES Crystals in AC2-03 (1296°C) at 100×

Figure 3.2. XRD Scan of a AC2-03 (1198°C) Sample Containing RES Crystals

3.2 Viscosity

Table 3.2 lists the measured η -T values for AC2-03 in chronological order. These data are shown on an Arrhenius plot ($\ln \eta$ vs. $1/T$) in Figure 3.3. Four data points from Table 3.2 were excluded from calculations. These points, two at 1384°C and one at 1364°C, had measured η values below the instrument's range while a datum, at 1305°C, was at a T below T_L . The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining two data, plotted as filled circles, to yield E and F values of -19.528 and 30769 K, respectively. This fit, based on only two valid data points, was then used to calculate a η_{1350} value of 0.57 Pa-s.

Table 3.2. Viscosity-Temperature Data for AC2-03

T (°C)	η (Pa-s)	10000/T (K ⁻¹)	$\ln[\eta(\text{Pa-s})]$
1384	0.355	6.035	-1.04
1364	0.456	6.109	-0.78
1344	0.609	6.184	-0.50
1324	0.774	6.262	-0.26
1305	0.964	6.337	-0.04
1384	0.335	6.035	-1.09

Figure 3.3. Measured $\ln$ Viscosity vs. $10000/T$ for AC2-03

4.0 AC2-04

Glass AC2-04 is an extreme vertex on inner level of glass compositions. This glass targets 63.5 mass percent Ln_2O_3 . The ratio of SrO to other frit components is higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are same, same, same, and high, respectively, in comparison to the standard frit composition.

4.1 Liquidus Temperature

The “as-received” glass had thin, hair-like crystals observed by OM. The glass was not remelted before testing, but every effort was performed to use representative samples for T_L measurements. Nine preliminary tests starting at 1451°C down to 1268°C indicated the T_L to be approximately 1400°C. Subsequent tests between 1401°C and 1344°C produced no crystals. Another glass sample was requested from Savannah River Technology Center (SRTC), and in three measurements between 1380°C and 1409°C, the T_L was estimated to be at 1405°C with the primary phase as RESs, as seen in Table 4.1.

A few needle-like crystals, probably RESs, were observed by OM on the melt surfaces of the sample heat treated at 1409°C and the preliminary sample heat treated at 1451°C. Because of the high testing temperatures of this sample (the only glass with $T_L > 1405^\circ\text{C}$), volatilization of glass components was evident and caused surface crystals to form. These crystals were easily distinguished from those formed just below T_L . The crystals formed at temperatures just below T_L were observed below the glass surface and on the crucible bottom.

Nine preliminary heat treatments were performed between 1268°C and 1451°C at approximately 50°C intervals. Abundant crystals were observed at 1344°C and 1387°C, which matches the observations in the final heat treatments. Unusually long needle-like crystals (4 to 5 mm length) were observed at 1398°C and characterized by SEM as RESs (Figure 4.1 and Figure 4.2).

Table 4.1. Heat-Treatment Summary for Determination of T_L for AC2-04

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
4	8	9:25am 06/30	1401	9:25am 07/01	Few crystals	T_C
4	8	3:50pm 07/26	1390	3:00pm 07/27	RES on bottom	
4	8	3:20pm 07/27	1380	1:12pm 07/28	Crystals throughout	T_A
4	8	3:07pm 07/28	1409	1:12pm 07/29	Only one crystal caused by volatility

Figure 4.1. SEM Micrograph of AC2-04 (1398°C)

Figure 4.2. EDS Spectrum of RES Crystal in AC2-04 (1398°C)

4.2 Viscosity

Table 4.2 lists the measured η -T values for AC2-04 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 4.3. All four data points from Table 4.2 were excluded from calculations. All but one of these points, at 1384°C, had measured η values below the instrument's range while all data points were taken at T_s below T_L . The excluded data are plotted as open circles and listed in strike-through font. With no valid data for calculations, Equation (2) was not fitted for this glass.

Table 4.2. Viscosity-Temperature Data for AC2-04

T (°C)	η (Pa·s)	10000/T (K ⁻¹)	ln[η (Pa·s)]
1384	0.585	6.035	-0.536
1404	0.380	5.963	-0.967
1384	0.496	6.035	-0.700
1404	0.484	5.963	-0.727

Figure 4.3. Measured ln Viscosity vs. 10000/T for AC2-04

5.0 AC2-05

Glass AC2-05 is an extreme vertex on the inner level of glass compositions. This glass targets 51.6 mass percent Ln_2O_3 . The ratios of SiO_2 , Al_2O_3 , and SrO to other frit components are higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are high, high, low, and high, respectively, in comparison to the standard frit composition.

5.1 Liquidus Temperature

Table 5.1 summarizes the final heat treatments performed on this glass. The T_L of this glass was estimated at 1241°C since one sample appeared to be crystal free after a heat treatment at 1241°C , and a second sample contained only a few small crystals after a heat treatment at 1240°C . RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified as RESs by SEM (Figure 5.1 and Figure 5.2) and by the characteristic morphology seen in Figure 5.3. See Section 4.1.5 in this report for a comparison of the EDS spectra of the crystals in a 1223°C heat-treated sample with that of the glass/matrix. The crystal appears to be composed of Ln_2O_3 and SiO_2 ; the ratio of Ln_2O_3 to SiO_2 appears to be higher in the crystal than in the glass.

Six preliminary heat treatments were performed between 1153°C and 1248°C at approximately 50°C intervals. Abundant crystals were observed at 1223°C and 1233°C , which matches observations from final heat treatments. The sample at 1240°C appeared to be crystal free by OM.

Table 5.1. Heat-Treatment Summary for Determination of T_L for AC2-05

Crucible	Furnace	Time/Date IN	T ($^\circ\text{C}$)	Time/Date OUT	Observations	Comments
C	8	2:45pm 04/23	1223	4:53pm 04/24	Long crystals	Preliminary, SEM
5	3	4:00pm 05/28	1241	2:00pm 05/29	No crystals	T_A
5	3	4:00pm 06/01	1233	2:30pm 06/02	Very few crystals	
5	3	3:30pm 06/02	1240	2:30pm 06/03	Few small crystals	T_C
5	3	1:45pm 06/23	1251	2:00pm 06/24	No crystals

Figure 5.1. SEM Micrograph of AC2-05 (1223°C)

Figure 5.2. EDS Spectrum of a RES Crystal in AC2-05 (1223°C)

Figure 5.3. Optical Micrograph of AC2-05 (1198°C) at 100×

5.2 Viscosity

Table 5.2 lists the measured η -T values for AC2-05 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 5.4. Three data points from Table 5.2 were taken at T s below T_L , two at 1234°C and one at 1185°C, and were excluded from calculations. The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -17.764 and 30253 K, respectively. The data appear to be nonlinear. Therefore, a η_{1350} value of 2.20 Pa·s was obtained by interpolation between the data points at 1332°C and 1382°C using Equation (2).

Table 5.2. Viscosity-Temperature Data for AC2-05

T (°C)	η (Pa·s)	10000/T (K ⁻¹)	$\ln[\eta(\text{Pa}\cdot\text{s})]$
1283	5.472	6.427	1.700
1234	12.298	6.636	2.509
1185	31.760	6.859	3.458
1234	13.120	6.636	2.574
1283	5.488	6.427	1.703
1332	2.650	6.231	0.974
1382	1.604	6.042	0.472
1432	1.049	5.865	0.048
1284	5.512	6.423	1.707

Figure 5.4. Measured $\ln$ Viscosity vs. $10000/T$ for AC2-05

6.0 AC2-06

Glass AC2-06 is an extreme vertex on the inner level of glass compositions. This glass targets 61.3 mass percent Ln_2O_3 . The ratio of B_2O_3 to other frit components is higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are low, low, high, and low, respectively, in comparison to the standard frit composition.

6.1 Liquidus Temperature

Table 6.1 summarizes the final heat treatments performed on this glass. The T_L of this glass was estimated using Furnace #3 at 1309°C, since one sample appeared to be crystal free after a heat treatment at 1314°C, and a second sample contained only a few small crystals after a heat treatment at 1306°C. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-01, AC2-03, and other glasses that were shown by SEM and/or XRD to be RES crystals. Figure 6.1 shows the morphology of the crystals observed in AC2-06.

During the 1295°C heat treatment of AC2-06, small nodules or buds of glass formed at the upper edge of the Pt/Rh box folds. The buds are thought to be formed by capillary forces drawing glass to the top of the Pt/Rh foil folds. Further analysis of these buds was performed and is reported in Section 4.1.4 of this report.

Six preliminary heat treatments were performed between 1153°C and 1343°C at approximately 50°C intervals. Abundant crystals were observed at 1248°C and 1296°C, which matches observations from final heat treatments. The sample at 1319°C appeared to be crystal free by OM. An additional sample, heat treated in Furnace #8 at 1288°C, contained crystals.

Table 6.1. Heat-Treatment Summary for Determination of T_L for AC2-06

Crucible	Furnace	Time/Date IN	T (°C)	Time/Date OUT	Observations	Comments
6	3	3:00pm 06/11	1314	2:20pm 06/12	No crystals	T_A
6	3	2:56pm 06/27	1295	1:00pm 06/28	Full of crystals	Buds
6	3	11:00am 06/29	1306	9:20am 06/30	Few corner crystals	T_C
6	8	8:54am 07/07	1288	10:50am 07/07	Full of crystals	different furnace, Buds

6.2 Viscosity

Table 6.2 lists the measured η -T values for AC2-06 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 6.2. Four data points from Table 6.2 were excluded from calculations. Three of these points, two at 1382°C and one at 1363°C, had measured η values below the instrument's range while a datum, at 1304°C, was at a T below T_L . The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining two data, plotted as filled circles, to yield E and F values of -9.177 and 14314 K, respectively. This fit, based on only two valid data points (both below 1350°C), was then used to calculate a η_{1350} value of 0.70 Pa·s.

Table 6.2. Viscosity-Temperature Data for AC2-06

T (°C)	η (Pa·s)	$10000/T$ (K ⁻¹)	$\ln[\eta(\text{Pa}\cdot\text{s})]$
1382	0.411	6.042	-0.889
1363	0.524	6.113	-0.647
1343	0.727	6.188	-0.319
1323	0.812	6.266	-0.208
1304	1.085	6.341	0.081
1382	0.422	6.042	-0.862

Figure 6.2. Measured $\ln$ Viscosity vs. $10000/T$ for AC2-06

7.0 AC2-07

Glass AC2-07 is an extreme vertex on the inner level of glass compositions. This glass targets 52.2 mass percent Ln_2O_3 . The ratios of SiO_2 and Al_2O_3 to other frit components are higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are high, high, low and low, respectively, in comparison to the standard frit composition.

7.1 Liquidus Temperature

Table 7.1 summarizes the heat treatments performed on this glass. The T_L of this glass was estimated using Furnace #8 at 1209°C since one sample appeared to be crystal free after a heat treatment at 1213°C, and a second sample contained only a few small crystals after a heat treatment at 1204°C. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by their characteristic morphologies. The morphology of crystals observed in AC2-07 (micrograph shown in Figure 7.1) matched those in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals.

Seven preliminary heat treatments were performed with AC2-07 glass starting at 1153°C and increasing at approximately 50°C intervals to 1233°C where the sample appeared to be crystal free by OM. The sample that was heat treated at 1198°C had fewer crystals compared to the 1153°C sample, indicating that the glass was near T_L . An additional sample, heat treated in Furnace #3 at 1215°C, appeared to be crystal free by OM.

Table 7.1. Heat-Treatment Summary for Determination of T_L for AC2-07

Crucible	Furnace	Time/Date IN	T (°C)	Time/Date OUT	Observations	Comments
7	3	2:30pm 06/12	1215	3:00pm 06/13	No crystals	different furnace
7	8	9:20am 06/28	1204	11:00am 06/29	Very few crystals	T_C
7	8	11:15am 07/03	1213	9:15am 07/04	No crystals	T_A

Figure 7.1. Optical Micrograph of RES Crystals in AC2-07 (1150°C) at 100×

7.2 Viscosity

Table 7.2 lists the measured η -T values for AC2-07 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 7.2. Two data points from Table 7.2 were taken at T_s below T_L , at 1204°C and 1184°C, and were excluded from calculations. The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -19.528 and 33144 K, respectively. The data appear to be nonlinear. Therefore, a η_{1350} value of 2.21 Pa·s was obtained by interpolation between the data points at 1334°C and 1382°C using Equation (2).

Table 7.2. Viscosity-Temperature Data for AC2-07

T (°C)	η (Pa.s)	10000/T (K ⁻¹)	ln[η (Pa.s)]
1383	1.499	6.039	0.405
1403	1.412	5.967	0.345
1453	0.886	5.794	-0.121
1384	1.438	6.035	0.363
1334	2.717	6.223	1.000
1283	5.536	6.427	1.711
1264	7.479	6.506	2.012
1244	10.638	6.592	2.364
1224	15.570	6.680	2.745
1204	22.185	6.770	3.099
1184	32.926	6.863	3.494
1382	1.480	6.042	0.392

Figure 7.2. Measured ln Viscosity vs. 10000/T for AC2-07

8.0 AC2-08

Glass AC2-08 is an extreme vertex on the inner level of glass compositions. This glass targets 52.9 mass percent Ln_2O_3 . The ratios of SiO_2 , Al_2O_3 , and SrO to other frit components are higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are high, high, low, and high, respectively, in comparison to the standard frit composition.

8.1 Liquidus Temperature

Table 8.1 summarizes the heat treatments performed on this glass. The T_L of this glass was estimated using Furnace #3 at 1242°C since one sample appeared to be crystal free after a heat treatment at 1240°C, a second sample contained only a few small crystals after a heat treatment at 1241°C, and only a few small rounded crystals remained in a sample heat treated at 1243°C. These are likely caused by small glass composition inhomogeneities. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphology of the crystal as seen in Figure 8.1. Figure 8.3 shows the EDS spectrum of a crystal in a 1233°C heat-treated sample (indicated in Figure 8.2). The crystal appears to be composed of Ln_2O_3 and SiO_2 ; the ratio of Ln_2O_3 to SiO_2 appears to be higher in the crystal than in the glass, similar to those seen in samples of AC2-05.

Five preliminary heat treatments were performed with AC2-08 glass starting at 1153°C and increasing at approximately 50°C intervals to 1247°C where the sample appeared to be crystal free by OM. The sample heat treated at 1240°C had fewer crystals the 1233°C sample, which indicated that the glass was near T_L .

Table 8.1. Heat-Treatment Summary for Determination of T_L for AC2-08

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
8	3	4:00pm 05/28	1241	2:00pm 05/29	Few crystals	T_C
8	3	4:00pm 06/01	1233	2:30pm 06/02	Small crystals	SEM
8	3	3:30pm 06/02	1240	2:30pm 06/03	No crystals	T_A
8	3	4:30pm 06/09	1243	3:00pm 06/10	Few round crystals	
8	3	4:30pm 06/10	1252	2:30pm 06/11	No crystals

8.2 Viscosity

Table 8.2 lists the measured η -T values for AC2-08 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 8.4. Two data points from Table 8.2 were taken at T_s below T_L , at 1236°C and 1187°C, and were excluded from calculations. The

excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -15.776 and 26567 K, respectively. The data appear to be nonlinear. Therefore, a η_{1350} value of 1.70 Pa·s was obtained by interpolation between the data points at 1335°C and 1384°C using Equation (2).

Table 8.2. Viscosity-Temperature Data for AC2-08

T ($^{\circ}\text{C}$)	η (Pa·s)	$10000/T$ (K^{-1})	$\ln[\eta(\text{Pa}\cdot\text{s})]$
1384	1.289	6.035	0.254
1335	1.928	6.219	0.656
1285	3.989	6.418	1.384
1335	1.908	6.219	0.646
1384	1.326	6.035	0.283
1434	0.840	5.858	-0.175
1384	1.320	6.035	0.278
1236	8.800	6.627	2.175
1187	24.908	6.849	3.215

Figure 8.4. Measured $\ln$ Viscosity vs. $10000/T$ for AC2-08

9.0 AC2-09

Glass AC2-09 is an extreme vertex on the inner level of glass compositions. This glass targets 64.1 mass percent Ln_2O_3 . The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

9.1 Liquidus Temperature

Table 9.1 details the final heat treatments near T_L . A T_L of 1323°C was estimated using Furnace #8 because the sample at 1327°C appeared to be crystal free by OM, and at 1320°C , crystals were observed in the corners and edges of the glass sample. An EDS spectrum of a crystal in the 1320°C sample identified the primary phase as RES. Several distinct crystal morphologies were observed near T_L . At 1314°C , a crystal that is only seen on the surface of samples with an abundance of RES in the bulk glass and just below T_L was observed. A sample from AC2-46 with similar morphology was analyzed by SEM/EDS (Figure 4.6 of the main report) and observed to be RES. A few samples near T_L , like the sample at 1320°C , had dendritic ends on the RES crystals (Figures 4.4 and 4.5 of the main report). The EDS spectra matched those of other RES crystals.

Seven preliminary samples were heat treated between 1151°C and 1352°C at approximately 50°C intervals with samples full of crystals from 1151°C to 1315°C , indicating gross crystallization occurred at temperatures slightly below T_L . Four additional samples were heat treated in Furnaces 3 and 12 between 1314°C and 1328°C , all containing crystals.

Table 9.1. Heat-Treatment Summary for Determination of T_L for AC2-09

Crucible	Furnace	Time/Date IN	T ($^\circ\text{C}$)	Time/Date OUT	Observations	Comments
9	3	3:00pm 06/11	1314	2:20pm 06/12	Crystals	different furnace, XRD
9	8	4:30pm 06/23	1327	3:00pm 06/24	No crystals	T_A
9	12	5:00pm 06/24	1320	3:00pm 06/25	Crystals	different furnace, Buds
9	8	1:15pm 07/08	1320	11:15am 07/09	Crystals	T_C , Buds
9	3	1:45pm 07/14	1321	3:05pm 07/15	Crystals	different furnace
9	3	3:34pm 07/16	1328	3:44pm 07/17	Crystals	different furnace

9.2 Viscosity

Table 9.2 lists the measured η -T values for AC2-09 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 9.1. Two data points from Table 9.2 had measured η values below the instrument's range, 1383°C and 1384°C , and were excluded from calculations. The excluded data are shown as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, shown as solid circles, to yield E and F values of -18.999 and 30452 K, respectively. This fit, which accurately portrays the data, was then used to calculate a η_{1350} value of 0.79 Pa-s.

Table 9.2. Viscosity-Temperature Data for AC2-09

T (°C)	η (Pa.s)	10000/T (K ⁻¹)	$\ln[\eta(\text{Pa.s})]$
1383	0.521	6.039	-0.653
1364	0.672	6.109	-0.397
1344	0.846	6.184	-0.167
1325	1.059	6.258	0.057
1384	0.535	6.035	-0.625

Figure 9.1. Measured $\ln$ Viscosity vs. 10000/T for AC2-09

10.0 AC2-10

Glass AC2-10 is an extreme vertex on the inner level of glass compositions. This glass targets 54.0 mass percent Ln_2O_3 . The ratios of SiO_2 , B_2O_3 and SrO to other frit components are higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are high, low, high, and high, respectively, in comparison to the standard frit composition.

10.1 Liquidus Temperature

Table 10.1 summarizes the heat treatments performed on this glass. The T_L of this glass was estimated using Furnace #3 at 1255°C since a sample heat treated at 1261°C appeared to be crystal free, and the sample heat treated at 1252°C contained only a few small crystals. The sample at 1243°C had an abundance of crystals throughout, indicating that gross crystallization occurred at temperatures slightly below T_L . RES crystals formed in samples of this glass heat treated at temperatures below T_L . This phase was identified by the characteristic morphologies and EDS analyses of the crystals as seen in Figure 10.1. Figure 10.2 shows the EDS spectrum of a crystal in the 1243°C heat-treated sample. The crystal appears to be composed of Ln_2O_3 and SiO_2 .

Five preliminary heat treatments were performed between 1212°C and 1268°C at approximately 25°C intervals with an abundance of crystals in the samples at 1212°C and 1229°C. One additional sample, heat treated at 1259°C in Furnace #12, appeared to be crystal free.

Table 10.1. Heat-Treatment Summary for Determination of T_L for AC2-10

Crucible	Furnace	Time/Date IN	T (°C)	Time/Date OUT	Observations	Comments
10	3	4:30pm 06/09	1243	3:00pm 06/10	Lots of crystals	SEM
10	3	4:30pm 06/10	1252	2:30pm 06/11	Few crystals	T_C
10	12	2:17pm 06/20	1259	1:00pm 06/21	No crystals	different furnace
10	3	8:53am 07/06	1261	10:50am 07/07	No crystals	T_A

10.2 Viscosity

Table 10.2 lists the measured η -T values for AC2-10 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 10.3. A data point from Table 10.2 was taken at T's below T_L , at 1237°C, and was excluded from calculations. The excluded data is plotted as an open circle and listed in strike-through font. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -13.516 and 22451 K, respectively. The data appear to be nonlinear. Therefore, a η_{1350} value of 1.29 Pa·s was obtained by interpolation between the data points at 1335°C and 1384°C using Equation (2).

Table 10.2. Viscosity-temperature data for AC2-10

T (°C)	η (Pa·s)	10000/T (K ⁻¹)	$\ln[\eta(\text{Pa}\cdot\text{s})]$
1384	1.067	6.035	0.065
1335	1.405	6.219	0.340
1285	2.701	6.418	0.994
1334	1.425	6.223	0.354
1384	1.078	6.035	0.075
1434	0.695	5.858	-0.364
1384	1.069	6.035	0.066
1237	5.891	6.623	1.773

Figure 10.3. Measured $\ln$ viscosity vs. $10000/T$ for AC2-10

11.0 AC2-11

Glass AC2-11 is an extreme vertex on the inner level of glass compositions. This glass targets 59.0 mass percent Ln_2O_3 . The ratio of Al_2O_3 to other frit components is higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are low, high, low, and low, respectively, in comparison to the standard frit composition.

11.1 Liquidus Temperature

Table 11.1 summarizes the heat treatments performed on samples of AC2-11. The T_L of this glass was estimated at 1292°C using Furnace #3. A few large crystals remained in the sample near the surface in the 1289°C heat treatment, and no crystals were observed in samples heat treated at 1295°C. Al_2O_3 crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphology of the crystal as seen in Figure 11.1 and by an XRD scan of a sample heat treated at 1175°C, shown in Figure 11.2. During the 1286°C heat treatment of AC2-11 (in Furnace #8), small nodules or buds of glass formed at the upper edge of the Pt/Rh box folds. These buds were similar to those seen in AC2-03 and AC2-06 and those discussed in Section 4.1.4.

Six preliminary heat treatments were performed between 1151°C and 1296°C with approximately 50°C intervals. Crystals were abundant in samples between 1151°C and 1248°C, indicating that gross crystallization occurred at temperatures slightly below T_L . Two additional samples, heat treated at 1275°C in Furnace #12 and at 1286°C in Furnace #8, contained crystals.

Table 11.1. Heat-Treatment Summary for Determination of T_L for AC2-11

Crucible	Furnace	Time/Date IN	T (°C)	Time/Date OUT	Observations	Comments
11	12	3:00pm 06/18	1275	1:20pm 06/19	Crystals throughout	different furnace
11	8	5:00pm 06/24	1286	3:00pm 06/25	Small crystals	different furnace, Buds
11	3	2:56pm 06/27	1295	1:00pm 06/28	No crystals	T_A
11	3	9:30am 07/01	1289	10:45am 07/02	Few crystals	T_C

Figure 11.1. Optical Micrograph of AC2-11 (1248°C) 100x

Figure 11.2. XRD Scan of AC2-11 (1175°C)

11.2 Viscosity

Table 11.2 lists the measured η -T values for AC2-11 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 11.3. Four data points from Table 11.2 were excluded from calculations. One of these points, at 1403°C, had a measured η value below the instrument's range while three data points, at 1284°C, 1264°C, and 1244°C, were taken at T_s below T_L . The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -15.887 and 25700 K, respectively. The data appear to be nonlinear. Therefore, a η_{1350} value of

0.96 Pa·s was obtained by interpolation between the data points at 1344°C and 1364°C using Equation (2).

Table 11.2. Viscosity-Temperature Data for AC2-11

T (°C)	η (Pa·s)	10000/T (K ⁻¹)	ln[η (Pa·s)]
1383	0.682	6.039	-0.383
1403	0.540	5.967	-0.616
1383	0.685	6.039	-0.378
1364	0.843	6.109	-0.171
1344	1.020	6.184	0.020
1324	1.272	6.262	0.240
1304	1.454	6.341	0.374
1284	1.766	6.423	0.569
1264	2.360	6.506	0.858
1244	4.102	6.592	1.411
1383	0.690	6.039	-0.371

Figure 11.3. Measured ln Viscosity vs. 10000/T for AC2-11

12.0 AC2-12

Glass AC2-12 is an extreme vertex on the inner level of glass compositions. This glass targets 56.7 mass percent Ln_2O_3 . The ratios of SiO_2 and SrO to other frit components are higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are high, low, low, and high, respectively, in comparison to the standard frit composition.

12.1 Liquidus Temperature

Five final heat treatments were performed in Furnaces #8 and #3 (see Table 12.1) from 1314°C to 1335°C, with 1314°C being heavily crystallized. Long needle-like crystals were observed at 1227°C and 1228°C, and both samples measured at 1335°C appeared to be crystal free by OM. The T_L was estimated at 1333°C using two independent measurements (one in Furnace #8 and the other in Furnace #3). The RES crystals were identified by the characteristic morphology of the crystals as shown in (Figure 12.1).

Seven preliminary heat treatments were conducted between 1151°C and 1352°C with an approximately 50°C difference from sample to sample. Glasses were heavily crystallized in samples from 1151°C and 1315°C, indicating that gross crystallization occurred at temperatures slightly below T_L .

Table 12.1. Heat-Treatment Summary for Determination of T_L for AC2-12

Crucible	Furnace	Time/Date IN	T (°C)	Time/Date OUT	Observations	Comments
12	3	3:00pm 06/11	1314	2:20pm 06/12	Very crystalline	
12	8	4:30pm 06/23	1327	3:00pm 06/24	Crystals	$T_C - 1$
12	3	1:30pm 06/26	1335	2:52pm 06/27	No crystals	$T_A - 2$
12	8	1:15pm 07/10	1335	2:47pm 07/11	No crystals	$T_A - 1$
12	3	3:34pm 07/16	1328	3:44pm 07/17	Crystals	$T_C - 2$

12.2 Viscosity

Table 12.2 lists the measured η -T values for AC2-12 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 12.2. A datum from Table 12.2 was taken at T_s below T_L , at 1324°C, and was excluded from calculations. The excluded data point is plotted as an open circle and listed in strike-through font. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -15.760 and 26240 K, respectively. The data appear to be nonlinear. Therefore, a η_{1350} value of 1.42 Pa·s was obtained by interpolation between the data points at 1344°C and 1364°C using Equation (2).

Table 12.2. Viscosity-Temperature Data for AC2-12

T (°C)	η (Pa·s)	10000/T (K ⁻¹)	$\ln[\eta(\text{Pa}\cdot\text{s})]$
1383	1.114	6.039	0.108
1403	0.930	5.967	-0.073
1422	0.766	5.900	-0.267
1433	0.627	5.862	-0.467
1384	1.115	6.035	0.109
1364	1.368	6.109	0.314
1344	1.444	6.184	0.368
1324	1.837	6.262	0.608
1384	1.127	6.035	0.119

Figure 12.2. Measured $\ln$ Viscosity vs. $10000/T$ for AC2-12

13.0 AC2-13

Glass AC2-13 is an extreme vertex on the inner level of glass compositions. This glass targets 49.4 mass percent Ln_2O_3 . The ratio of SrO to other frit components is lower in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are same, same, same, and low, respectively, in comparison to the standard frit composition.

13.1 Liquidus Temperature

Table 13.1 summarizes the heat treatments performed on this glass. The T_L of this glass was estimated using Furnace #3 at 1186°C since one sample appeared to be crystal free after a heat treatment at 1191°C, and a second sample contained only a few crystals or crystal clusters after a heat treatment at 1179°C. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals. During the 1179°C heat treatment, small nodules or buds of glass formed at the upper edge of the Pt/Rh box folds as discussed in Section 4.1.4.

Five preliminary heat treatments were conducted between 1151°C and 1200°C with approximately 20°C difference from sample to sample. The glass was heavily crystallized in the sample at 1151°C, indicating that gross crystallization occurred at temperatures slightly below T_L .

Table 13.1. Heat-Treatment Summary for Determination of T_L for AC2-13

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
13	3	2:18pm 06/20	1173	1:00pm 06/21	Crystals throughout	
13	3	1:05pm 06/28	1185	11:00am 06/29	One single crystal	
13	3	9:25am 06/30	1179	9:25am 07/01	Few clusters	
13	3	8:45am 07/04	1179	8:45am 07/05	Few crystals	T_C , Buds
13	3	3:30pm 07/12	1191	2:45pm 07/13	No crystals	T_A
13	3	4:30pm 07/15	1150	2:37pm 07/16	Very crystalline	

13.2 Viscosity

Table 13.2 lists the measured η -T values for AC2-13 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 13.1. Two data points from Table 13.2 were taken at T_s below T_L , at 1137°C and 1088°C, and were excluded from calculations. The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -20.323 and 33987 K, respectively. The data appear to be nonlinear. Therefore, a η_{1350} value of 1.86 Pa·s was obtained by interpolation between the data points at 1334°C and 1383°C using Equation (2).

Table 13.2. Viscosity-Temperature Data for AC2-13

T (°C)	η (Pa·s)	10000/T (K ⁻¹)	ln[η (Pa·s)]
1284	4.263	6.423	1.450
1235	8.929	6.631	2.189
1186	21.999	6.854	3.091
1235	8.994	6.631	2.197
1285	4.223	6.418	1.440
1334	2.134	6.223	0.758
1383	1.417	6.039	0.348
1285	4.215	6.418	1.439
1137	62.841	7.092	4.141
1088	238.531	7.348	5.474

Figure 13.1. Measured ln Viscosity vs. 10000/T for AC2-13

14.0 AC2-14

Glass AC2-14 is an extreme vertex on the inner level of glass compositions. This glass targets 55.6 mass percent Ln_2O_3 . The ratios of Al_2O_3 , B_2O_3 and SrO to other frit components are higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are low, high, high, and high, respectively, in comparison to the standard frit composition.

14.1 Liquidus Temperature

Table 14.1 summarizes the final heat treatments performed on this glass. The T_L of this glass was estimated using Furnace #3 at 1250°C since the sample at 1252°C appeared to be crystal free, and samples heat-treated at 1232°C , 1237°C , 1243°C , and 1246°C formed crystals. Aluminum oxide crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by SEM/EDS of a crystal observed in the 1232°C heat-treated sample as shown in Figure 14.1 and Figure 14.2. The crystal appears to be composed of aluminum and oxygen alone.

Five preliminary heat treatments were conducted from 1201 to 1247°C with a combination of RES and Al_2O_3 crystals filling the three samples between 1201 and 1223°C , and Al_2O_3 crystals only in the samples at 1233 and 1247°C (just a few crystals at 1247°C).

Table 14.1. Heat-Treatment Summary for Determination of T_L for AC2-14

Crucible	Furnace	Date/Time IN	T ($^\circ\text{C}$)	Date/Time OUT	Observations	Comments
14	3	2:30pm 05/18	1246	2:40pm 05/19	Few crystals	T_C
14	3	3:20pm 05/20	1232	3:35pm 05/21	Crystals on surface	SEM
14	3	3:45pm 05/25	1237	1:45pm 05/26	Crystals on surface	
14	3	4:30pm 06/09	1243	3:00pm 06/10	Crystals throughout	
14	3	4:30pm 06/10	1252	2:30pm 06/11	No crystals	T_A

Figure 14.1. SEM Micrograph of AC2-14 (1232°C)

Figure 14.2. EDS Spectrum of an Al_2O_3 Crystal in AC2-14 (1232°C)

14.2 Viscosity

Table 14.2 lists the measured η -T values for AC2-14 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 14.3. Four data points from Table 14.2 were taken at Ts below T_L , at 1244°C, 1225°C, 1205°C, and 1185°C, and were excluded from calculations. The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -16.124 and 25960 K, respectively. The data appear to be nonlinear. Therefore, a η_{1350} value of 0.89 Pa·s was obtained by interpolation between the data points at 1343°C and 1363°C using Equation (2).

Table 14.2. Viscosity-Temperature Data for AC2-14

T (°C)	η (Pa·s)	10000/T (K ⁻¹)	$\ln[\eta(\text{Pa}\cdot\text{s})]$
1382	0.614	6.042	-0.488
1363	0.790	6.112	-0.236
1343	0.957	6.188	-0.044
1324	1.183	6.262	0.168
1304	1.473	6.341	0.387
1284	1.615	6.423	0.479
1264	2.150	6.506	0.766
1244	2.911	6.592	1.068
1225	4.054	6.676	1.400
1205	5.730	6.766	1.746
1185	8.172	6.859	2.101
1382	0.643	6.042	-0.441

Figure 14.3. Measured $\ln$ Viscosity vs. $10000/T$ for AC2-14

15.0 AC2-15

Glass AC2-15 is an extreme vertex on the inner level of glass compositions. This glass targets 54.5 mass percent Ln_2O_3 . The ratios of SiO_2 and B_2O_3 to other frit components are higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are high, low, high, and low, respectively, in comparison to the standard frit composition.

15.1 Liquidus Temperature

Three final heat treatments were performed in Furnace #3 on AC2-15 between 1265°C and 1283°C (see Table 15.1). The RES crystals were abundant at 1265°C, filling the sample. Only a few needle-like RES were observed in the corners at 1275°C, and the sample at 1283°C appeared to be crystal free by OM. The T_L was estimated at 1279°C with a primary crystalline phase of RES. The crystals were identified by the characteristic morphologies of the crystals that formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals.

Preliminary heat treatments were accomplished on five samples from 1223 to 1295°C. An additional sample, heat treated in Furnace #12 at 1275°C, appeared to be crystal free.

Table 15.1. Heat-Treatment Summary for Determination of T_L for AC2-15

Crucible	Furnace	Time/Date IN	T (°C)	Time/Date OUT	Observations	Comments
15	12	3:00pm 06/18	1275	1:20pm 06/19	No crystals	different furnace
15	3	3:00pm 06/25	1265	1:20pm 06/26	Crystals throughout	
15	3	1:30pm 07/09	1275	1:00pm 07/10	Few crystals	T_C
15	3	4:06pm 07/17	1283	3:22pm 07/18	No crystals	T_A

15.2 Viscosity

Table 15.2 lists the measured η -T values for AC2-15 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 15.1. A datum from Table 15.2 had a measured η value below the instrument's range, at 1482°C, which was excluded from calculations. The excluded point is shown as an open circle and listed in strike-through font. Equation (2) was fitted to the remaining data, shown as solid circles, to yield E and F values of -15.571 and 25731 K, respectively. This fit, which accurately portrays the data, was then used to calculate a η_{1350} value of 1.33 Pa·s.

Table 15.2. Viscosity-Temperature Data for AC2-15

T (°C)	η (Pa·s)	$10000/T$ (K ⁻¹)	$\ln[\eta(\text{Pa}\cdot\text{s})]$
1382	0.970	6.042	-0.030
1334	1.554	6.223	0.441
1284	2.550	6.423	0.936
1333	1.580	6.227	0.457
1382	1.001	6.042	0.001
1432	0.598	5.865	-0.514
1482	0.335	5.698	-1.093
1384	0.985	6.035	-0.015

Figure 15.1. Measured $\ln$ Viscosity vs. $10000/T$ for AC2-15

16.0 AC2-16

Glass AC2-16 is an extreme vertex on the inner level of glass compositions. This glass targets 58.4 mass percent Ln_2O_3 . The ratios of Al_2O_3 and SrO to other frit components are higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are low, high, low, and high, respectively, in comparison to the standard frit composition.

16.1 Liquidus Temperature

Table 16.1 summarizes the final heat treatments performed on this glass in Furnace #3. The T_L of this glass was estimated at 1298°C. The sample heat-treated at 1299°C appeared to be crystal free, and the sample at 1295°C formed a few Al_2O_3 crystals. Aluminum oxide crystals formed in samples of this glass heat treated at temperatures below T_L . The crystals were identified by the characteristic morphologies of the crystals formed below T_L and match those observed in samples of AC2-02, AC2-11, AC2-14, AC2-16, and AC2-21.

Six preliminary heat treatments were conducted from 1201°C to 1303°C with a combination of RES and Al_2O_3 crystals filling the sample at 1201°C. Al_2O_3 crystals were only in the three samples between 1273°C and 1286°C. Additional samples, heat treated in Furnace #8 between 1291°C and 1306°C, appeared crystal free; while samples heat treated in Furnaces #8 and #12 between 1275°C and 1286°C contained crystals.

Table 16.1. Heat-Treatment Summary for Determination of T_L for AC2-16

Crucible	Furnace	Time/Date IN	T (°C)	Time/Date OUT	Observations	Comments
16	12	3:00pm 06/18	1275	1:20pm 06/19	Crystals throughout	different furnace
16	8	5:00pm 06/24	1286	3:00pm 06/25	Crystals	different furnace, Buds
16	3	2:56pm 06/27	1295	1:00pm 06/28	Few crystals	T_C
16	8	11:00am 06/29	1306	9:20am 06/30	No crystals	different furnace
16	3	3:20pm 07/13	1299	2:45pm 07/14	No crystals	T_A
16	8	4:06pm 07/17	1297	3:21pm 07/18	No crystals	different furnace, Buds
16	8	2:45pm 07/23	1291	12:45pm 07/24	No crystals	different furnace, Buds

16.2 Viscosity

Table 16.2 lists the measured η -T values for AC2-16 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 16.1. Four data points from Table 16.2 were taken at T_s below T_L , at 1284°C, 1264°C, 1244°C, and 1225°C, and were excluded from calculations. The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -17.013 and 27690 K, respectively. The data appear to be nonlinear. Therefore, a η_{1350} value of

1.09 Pa·s was obtained by interpolation between the data points at 1344°C and 1363°C using Equation (2).

Table 16.2. Viscosity-Temperature Data for AC2-16

T (°C)	η (Pa·s)	10000/T (K ⁻¹)	ln[η (Pa·s)]
1382	0.759	6.042	-0.276
1403	0.579	5.967	-0.547
1383	0.756	6.039	-0.280
1363	0.948	6.112	-0.054
1344	1.165	6.184	0.153
1324	1.444	6.262	0.368
1304	1.605	6.341	0.473
1284	2.089	6.423	0.737
1264	2.846	6.506	1.046
1244	3.965	6.592	1.377
1225	5.875	6.676	1.771
1383	0.741	6.039	-0.299

Figure 16.1. Measured ln Viscosity vs. 10000/T for AC2-16

17.0 AC2-17

Glass AC2-17 is a centroid of the inner level of glass compositions. This glass targets 56.6 mass percent Ln_2O_3 . The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

17.1 Liquidus Temperature

Table 17.1 summarizes the final heat treatments performed on this glass. The T_L of this glass was estimated using Furnace #3 at 1273°C since a sample heat treated at 1275°C appeared to be crystal free, and a sample heat treated at 1265°C contained many crystals. This indicated that gross crystallization occurred at temperatures slightly below T_L . RES crystals formed in samples of this glass heat-treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals.

Five preliminary heat treatments were performed on samples from 1223°C to 1295°C with RES observed in the three samples below T_L , and no crystals were observed in samples at 1283°C and 1295°C. An additional sample, heat treated in Furnace #12 at 1275, appeared to be crystal free.

Table 17.1. Heat-Treatment Summary for Determination of T_L for AC2-17

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
17	12	3:00pm 06/18	1275	1:20pm 06/19	No crystals	different furnace
17	3	3:00pm 06/25	1265	1:20pm 06/26	Crystals throughout	T_C
17	3	1:30pm 07/09	1275	1:00pm 07/10	No crystals	T_A

17.2 Viscosity

Table 17.2 lists the measured η -T values for AC2-17 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 17.1. Three data points from Table 17.2 were excluded from calculations. One of these points, at 1433°C, had a measured η value below the instrument's range while two data points, at 1266°C and 1246°C, were taken at T_s below T_L . The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, shown as solid circles, to yield E and F values of -15.154 and 24804 K, respectively. This fit, which accurately portrays the data, was then used to calculate a η_{1350} value of 1.14 Pa·s.

Table 17.2. Viscosity-Temperature Data for AC2-17

T (°C)	η (Pa.s)	10000/T (K ⁻¹)	ln[η (Pa.s)]
1383	0.817	6.039	-0.202
1335	1.338	6.219	0.291
1285	2.108	6.418	0.746
1334	1.357	6.223	0.305
1384	0.840	6.035	-0.175
1433	0.471	5.862	-0.754
1384	0.827	6.035	-0.190
1266	2.837	6.498	1.043
1246	5.625	6.583	1.727

Figure 17.1. Measured ln Viscosity vs. 10000/T for AC2-17

18.0 AC2-18

Glass AC2-18 is an extreme vertex on the outer level of glass compositions. This glass targets 63.5 mass percent Ln_2O_3 . The ratio of SrO to other frit components is higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are same, same, same, and high, respectively, in comparison to the standard frit composition.

18.1 Liquidus Temperature

Table 18.1 summarizes the final heat treatments performed on this glass. The T_L of this glass was estimated using Furnace #8 at 1366°C since four samples were crystal free from 1374°C to 1401°C. The sample heat treated at 1364°C contained only a few small crystals on the bottom, and a sample heat treated at 1354°C was loaded with crystals. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies and by XRD. Figure 18.2 shows an XRD scan from a sample heat treated at 1303°C; the characteristic pattern obtained from RES crystals is apparent.

Nine preliminary heat treatments were performed on AC2-18 starting at 1215°C up to 1400°C. Many heat treatments were needed on this glass since it was at a higher T_L than most other Phase II glasses. An additional sample, heat treated at 1344°C in Furnace #5, was nearly completely crystallized, indicating that gross crystallization occurred at temperatures slightly below T_L .

Table 18.1. Heat-Treatment Summary for Determination of T_L for AC2-18

Crucible	Furnace	Time/Date IN	T (°C)	Time/Date OUT	Observations	Comments
L	8	2:00pm 05/10	1303	1:20pm 05/11	Crystals	Preliminary/XRD
18	8	9:25am 06/30	1401	9:25am 07/01	No crystals	
18	8	4:02pm 07/11	1393	2:45pm 07/12	No crystals	
18	8	3:20pm 07/13	1384	2:45pm 07/14	No crystals	
18	8	4:30pm 07/15	1374	2:40pm 07/16	No crystals	T_A
18	8	3:32pm 07/16	1364	2:57pm 07/17	Crystals	T_C
18	5	5:10pm 07/17	1344	3:19pm 07/18	Full of crystals	different furnace
18	8	4:30pm 07/22	1354	2:35pm 07/23	Full of crystals

Figure 18.1. Optical Micrograph of RES Crystals in AC2-18 (1344°C) at 100×

Figure 18.2. XRD scan of AC2-18 (1303°C)

18.2 Viscosity

Table 18.2 lists the measured η -T values for AC2-18 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 18.3. Three data points from Table 18.2 had measured η values below the instrument's range, at 1384°C, 1395°C, and 1385°C, and were excluded from calculations. The excluded data are plotted as open circles and listed in strike-through font. With only one valid data point for calculations, Equation (2) was not fitted for this glass.

Table 18.2. Viscosity-Temperature Data for AC2-18

T (°C)	η (Pa·s)	$10000/T$ (K ⁻¹)	$\ln[\eta(\text{Pa}\cdot\text{s})]$
1384	0.469	6.035	-0.757
1380	0.640	6.050	-0.446
1395	0.427	5.995	-0.851
1385	0.504	6.031	-0.684

Figure 18.3. Measured $\ln$ Viscosity vs. $10000/T$ for AC2-18

19.0 AC2-19

Glass AC2-19 is an extreme vertex on the outer level of glass compositions. This glass targets 53.9 mass percent Ln_2O_3 . The ratios of SiO_2 , B_2O_3 , and SrO to other frit components are higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are high, low, high, and high, respectively, in comparison to the standard frit composition.

19.1 Liquidus Temperature

Table 19.1 summarizes the heat treatments performed on this glass in Furnace #3. The T_L of this glass was estimated at 1249°C since a sample appeared crystal free after heat treatment at 1252°C, and a sample grossly crystallized during a heat treatment at 1242°C. This indicated that gross crystallization occurred at temperatures slightly below T_L . RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals.

Preliminary heat treatments were performed on five samples between 1215°C and 1261°C with RESs observed at 1215°C, 1240°C, and 1248°C, and no crystals were observed at 1257°C and 1261°C.

Table 19.1. Heat-Treatment Summary for Determination of T_L for AC2-19

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
19	3	4:45pm 06/08	1242	2:45pm 06/09	Crystals throughout	T_C
19	3	4:30pm 06/10	1252	2:30pm 06/11	No crystals	T_A

19.2 Viscosity

Table 19.2 lists the measured η -T values for AC2-19 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 19.1. A datum from Table 19.2, at 1235°C, was taken at a T below T_L and was excluded from calculations. The excluded point was plotted as an open circle and listed in strike-through font. Equation (2) was fitted to the remaining data, shown as solid circles, to yield E and F values of -14.837 and 24619 K, respectively. This fit, which accurately portrays the data, was then used to calculate a η_{1350} value of 1.39 Pa.s.

Table 19.2. Viscosity-Temperature Data for AC2-19

T (°C)	η (Pa·s)	$10000/T$ (K ⁻¹)	$\ln[\eta(\text{Pa}\cdot\text{s})]$
1383	1.011	6.039	0.011
1334	1.605	6.223	0.473
1284	2.653	6.423	0.976
1334	1.597	6.223	0.468
1383	1.052	6.039	0.051
1433	0.653	5.862	-0.427
1383	1.048	6.039	0.047
1235	5.786	6.631	1.755
1383	1.049	6.039	0.048

Figure 19.1. Measured $\ln$ Viscosity vs. $10000/T$ for AC2-19

20.0 AC2-20

Glass AC2-20 is an extreme vertex on the outer level of glass compositions. This glass targets 64.1 mass percent Ln_2O_3 . The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

20.1 Liquidus Temperature

Table 20.1 summarizes the heat treatments performed on this glass using Furnace #8. The T_L of this glass was estimated at 1343°C since a sample appeared to be crystal free after a heat treatment at 1349°C, and a sample formed crystals after a heat treatment at 1336°C. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals.

Preliminary heat treatments were performed on eight samples between 1215°C and 1400°C.

Table 20.1. Heat-Treatment Summary for Determination of T_L for AC2-20

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
20	8	1:30pm 07/09	1349	1:00pm 07/10	No crystals	T_A
20	8	3:00pm 07/14	1336	3:05pm 07/15	Crystals	T_C , Buds

20.2 Viscosity

Table 20.2 lists the measured η -T values for AC2-20 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 20.1. Two data points from Table 20.2 had measured η values below the instrument's range, at 1384°C and 1385°C, and were excluded from calculations. The excluded data are plotted as open circles and listed in strike-through font. With only one valid data point for calculations, Equation (2) was not fitted for this glass.

Table 20.2. Viscosity-Temperature Data for AC2-20

T (°C)	η (Pa·s)	10000/T (K ⁻¹)	$\ln[\eta$ (Pa·s)]
1384	0.4416	6.035	-0.817
1365	0.6254	6.105	-0.469
1385	0.4787	6.031	-0.737

Figure 20.1. Measured $\ln$ Viscosity vs. $10000/T$ for AC2-20

21.0 AC2-21

Glass AC2-21 is an extreme vertex on the outer level of glass compositions. This glass targets 55.6 mass percent Ln_2O_3 . The ratios of Al_2O_3 , B_2O_3 , and SrO to other frit components are higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are low, high, high, and high, respectively, in comparison to the standard frit composition.

21.1 Liquidus Temperature

Table 21.1 summarizes the heat treatments performed on this glass. The T_L was estimated using Furnace #8 at 1242°C. Aluminum oxide crystals formed in samples of this glass heat treated at temperatures below T_L . The crystals were identified by the characteristic morphologies of the crystals formed below T_L . The crystal morphologies matched those observed in samples of AC2-02, AC2-11, AC2-14, and AC2-16. In addition, EDS spectra of a preliminary glass sample suggested that the crystal contains Al and O alone (see Figure 21.1, Figure 21.2, and Figure 21.3).

Four preliminary heat treatments were conducted from 1212°C to 1275°C. Samples at 1220°C and 1229°C both were analyzed by SEM/EDS. Additional samples were heat treated in Furnaces #10, #3, and #12. One small crystal was identified in the 1243°C heat-treated sample.

Table 21.1. Heat-Treatment Summary for Determination of T_L for AC2-21

Crucible	Furnace	Time/Date IN	T (°C)	Time/Date OUT	Observations	Comments
11	10	5:51pm 06/07	1229	4:28pm 06/08	Large plate-like crystals	different furnace, SEM
L	8	3:00pm 06/13	1220	3:30pm 06/14	Lots of crystals	Preliminary, SEM
21	3	4:30pm 06/09	1243	3:00pm 06/10	One little crystal	different furnace
21	12	2:17pm 06/20	1259	1:00pm 06/21	No crystals	different furnace
21	8	2:00pm 06/22	1249	12:35pm 06/23	No crystals	T_A
21	12	8:45am 07/04	1255	8:44am 07/05	No crystals	different furnace
21	8	4:29pm 07/18	1240	2:43pm 07/19	Few crystals	T_C

21.2 Viscosity

Table 21.2 lists the measured η -T values for AC2-21 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 21.4. Three data points from Table 21.2 were excluded from calculations. One of these points, at 1404°C, had a measured η value below the instrument's range while two data points, at 1236°C and 1186°C, were taken at T_s below T_L . The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, shown as solid circles, to yield E and F values of -14.596 and 23443 K, respectively. This fit, which accurately portrays the data, was then used to calculate a η_{1350} value of 0.86 Pa.s.

Table 21.2. Viscosity-Temperature Data for AC2-21

T (°C)	η (Pa.s)	10000/T (K ⁻¹)	$\ln[\eta(\text{Pa.s})]$
1384	0.637	6.035	-0.452
1335	0.991	6.219	-0.009
1285	1.554	6.418	0.441
1334	1.004	6.223	0.004
1384	0.641	6.035	-0.444
1404	0.493	5.963	-0.707
1384	0.632	6.035	-0.459
1236	2.721	6.627	1.001
1186	6.995	6.854	1.945

Figure 21.4. Measured $\ln$ Viscosity vs. $10000/T$ for AC2-21

22.0 AC2-22

Glass AC2-22 is an extreme vertex on the outer level of glass compositions. This glass targets 53.9 mass percent Ln_2O_3 . The ratios of SiO_2 , B_2O_3 , and SrO to other frit components are higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are high, low, high, and high, respectively, in comparison to the standard frit composition.

22.1 Liquidus Temperature

In the final T_L measurements, four heat treatments were performed in Furnace #3 between 1265°C and 1287°C (Table 22.1). The sample heat treated at 1265°C was filled with crystals, indicating that gross crystallization occurred at temperatures slightly below T_L . At 1275°C, only a few clusters remained on the bottom of the sample; at 1283°C, only one crystal was observed in a corner; and at 1287°C, the heat-treated sample appeared to be crystal free by OM. The T_L was estimated to be 1284°C with RES as the primary phase. These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals.

Four preliminary heat treatments were performed between 1222°C and 1275°C at approximately 20°C intervals. Abundant crystals were observed at 1251°C and 1263°C, which matches the observations in the final heat treatments. The sample at 1275°C appeared to be crystal free by OM of the preliminary sample. An additional sample was heat treated at 1275°C in Furnace #12. This sample contained a few crystals, validating the T_L measurement.

Table 22.1. Heat-Treatment Summary for Determination of T_L for AC2-22

Crucible	Furnace	Time/Date IN	T (°C)	Time/Date OUT	Observations	Comments
22	12	3:00pm 06/18	1275	1:20pm 06/19	Few crystals	different furnace
22	3	3:00pm 06/25	1265	1:20pm 06/26	Crystals throughout	
22	3	1:30pm 07/09	1275	1:00pm 07/10	Crystals on bottom	
22	3	4:06pm 07/17	1283	3:22pm 07/18	One crystal	T_C
22	3	4:30pm 07/22	1287	2:35pm 07/23	No crystals	T_A

22.2 Viscosity

Table 22.2 lists the measured η -T values for AC2-22 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 22.1. A datum from Table 22.2 had a measured η value below the instrument's range, at 1483°C, which was excluded from calculations. The excluded point is shown as an open circle and listed in strike-through font. Equation (2) was fitted to the remaining data, shown as solid circles, to yield E and F values of –

15.045 and 24893 K, respectively. This fit, which accurately portrays the data, was then used to calculate a η_{1350} value of 1.34 Pa·s.

Table 22.2. Viscosity-Temperature Data for AC2-22

T (°C)	η (Pa·s)	10000/T (K ⁻¹)	ln[η (Pa·s)]
1383	0.994	6.039	-0.006
1335	1.534	6.219	0.428
1286	2.492	6.414	0.913
1334	1.573	6.223	0.453
1383	1.004	6.039	0.004
1433	0.624	5.862	-0.472
1483	0.345	5.695	-1.064
1385	0.970	6.031	-0.030

Figure 22.1. Measured ln Viscosity vs. 10000/T for AC2-22

23.0 AC2-23

Glass AC2-23 is an extreme vertex on the outer level of glass compositions. This glass targets 58.4 mass percent Ln_2O_3 . The ratios of B_2O_3 and SrO to other frit components are higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are low, low, high, and high, respectively, in comparison to the standard frit composition.

23.1 Liquidus Temperature

Table 23.1 summarizes the heat treatments performed on this glass using Furnace #3. The T_L of this glass was estimated at 1282°C since samples were crystal free after heat treatments at 1284°C and 1292°C , and a sample heat-treated at 1278°C contained crystals throughout. This indicated that gross crystallization occurred at temperatures slightly below T_L . Aluminum oxide crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals (Figure 23.1) were identified by EDS analysis of a crystal observed in the 1278°C heat-treated sample as shown in Figure 23.2. The crystal appears to be composed of Al and O alone.

Three preliminary heat treatments were performed between 1223°C and 1288°C at approximately 20°C intervals. Abundant crystals were observed at 1223°C , which matches the observations in the final heat treatments. The sample at 1288°C appeared to be crystal free by OM.

Table 23.1. Heat-Treatment Summary for Determination of T_L for AC2-23

Crucible	Furnace	Date/Time IN	T ($^\circ\text{C}$)	Date/Time OUT	Observations	Comments
23	3	3:00pm 6/13	1278	3:30pm 6/14	Crystals throughout	T_C , SEM
23	3	5:00pm 6/15	1292	3:20pm 6/16	No crystals	
23	3	5:00pm 6/16	1284	3:00pm 6/17	No crystals	T_A , Buds

Figure 23.1. SEM Micrograph of AC2-23 (1278°C)

Figure 23.2. EDS Spectrum of an Al_2O_3 Crystal in AC2-23 (1278°C)

23.2 Viscosity

Table 23.2 lists the measured η -T values for AC2-23 in chronological order. These data are shown on an Arrhenius plot ($\ln \eta$ vs. $1/T$) in Figure 23.3. Two data points from were excluded from calculations. One of these points, at 1434°C, had a measured η value below the instrument's range while the other, at 1266°C, was taken at a T below T_L . The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, shown as solid circles, to yield E and F values of -14.288 and 23182 K, respectively. This fit, which accurately portrays the data, was then used to calculate a η_{1350} value of 1.00 Pa.s.

Table 23.2. Viscosity-Temperature Data for AC2-23

T (°C)	η (Pa·s)	10000/T (K ⁻¹)	ln[η (Pa·s)]
1383	0.712	6.039	-0.339
1334	1.172	6.223	0.158
1285	1.763	6.418	0.567
1334	1.181	6.223	0.167
1384	0.753	6.035	-0.284
1434	0.430	5.858	-0.843
1385	0.743	6.031	-0.297
1266	3.043	6.498	1.113

Figure 23.3. Measured ln Viscosity vs. 10000/T for AC2-23

24.0 AC2-24

Glass AC2-24 is an extreme vertex on the outer level of glass compositions. This glass targets 61.3 mass percent Ln_2O_3 . The ratio of B_2O_3 to other frit components is higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are low, low, high, and low, respectively, in comparison to the standard frit composition.

24.1 Liquidus Temperature

Table 24.1 details the final heat treatments near T_L . A T_L of 1319°C was estimated using Furnace #3. A sample heat treated at 1321°C appeared to be crystal free by OM, and a sample heat treated at 1313°C was full of crystals. This indicated that gross crystallization occurred at temperatures slightly below T_L . The primary phase was identified by the characteristic morphologies of the crystals formed below T_L . The morphology matches that observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals. The sample heat treated at 1313°C contained dendritic ends on the RES crystals similar the observations in AC2-44 (Figure 4.4 in the main report). An EDS spectrum taken on one of these dendritic ends was indistinguishable from those measured on other RES crystals.

Six preliminary samples were heat treated between 1222°C and 1339°C at approximately 50°C intervals with samples full of crystals from 1222°C to 1292°C. Additional samples, heat treated at 1320°C and 1327°C in Furnaces #12 and #8, did not contain crystals.

Table 24.1. Heat-Treatment Summary for Determination of T_L for AC2-24

Crucible	Furnace	Time/Date IN	T (°C)	Time/Date OUT	Observations	Comments
24	3	3:00pm 06/18	1313	1:30pm 06/19	Very crystalline	T_C
24	8	4:30pm 06/23	1327	3:00pm 06/24	No crystals	different furnace
24	12	5:00pm 06/24	1320	3:00pm 06/25	No crystals	different furnace
24	3	3:00pm 07/14	1321	3:05pm 07/15	No crystals	T_A

24.2 Viscosity

Table 24.2 lists the measured η -T values for AC2-24 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 24.1. Three data points from Table 24.2 had measured η values below the instrument's range, at 1383°C, 1363°C, and 1382°C, and were excluded from calculations. The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining two data, plotted as filled circles, to yield E and F values of -19.831 and 31376 K, respectively. This fit, based on only two valid data points, was then used to calculate a η_{1350} value of 0.61 Pa·s.

Table 24.2. Viscosity-Temperature Data for AC2-24

T (°C)	η (Pa·s)	$10000/T$ (K ⁻¹)	$\ln[\eta(\text{Pa}\cdot\text{s})]$
1383	0.367	6.039	-1.001
1363	0.501	6.112	-0.691
1344	0.653	6.184	-0.427
1324	0.832	6.262	-0.184
1382	0.387	6.042	-0.950

Figure 24.1. Measured $\ln$ Viscosity vs. $10000/T$ for AC2-24

25.0 AC2-25

Glass AC2-25 is an extreme vertex on the outer level of glass compositions. This glass targets 58.4 mass percent Ln_2O_3 . The ratios of Al_2O_3 and SrO to other frit components are higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are low, high, low, and high, respectively, in comparison to the standard frit composition.

25.1 Liquidus Temperature

Four final heat treatments were performed in Furnace #3 between 1278°C and 1314°C. They are summarized in Table 25.1. The T_L of AC2-25 was estimated at 1305°C. Aluminum oxide crystals formed in samples of this glass heat treated at temperatures below T_L . The crystals were identified by the characteristic morphologies of the crystals formed below T_L and match those observed in samples of AC2-02, AC2-11, AC2-14, AC2-16, and AC2-21. Aluminum oxide crystals were observed by OM at 1278°C, 1292°C, and 1299°C, with no crystals observed at 1314°C.

Five preliminary heat treatments were conducted from 1222°C to 1292°C. An additional sample, heat treated in Furnace #8 at 1305°C, did not contain crystals.

Table 25.1. Heat-Treatment Summary for Determination of T_L for AC2-25

Crucible	Furnace	Time/Date IN	T (°C)	Time/Date OUT	Observations	Comments
25	3	3:00pm 06/13	1278	3:30pm 06/14	Crystals throughout	
25	3	5:00pm 06/15	1292	3:20pm 06/16	Crystals throughout	
25	8	11:00am 06/29	1305	9:20am 06/30	No crystals	different furnace
25	3	11:00am 06/29	1314	9:20am 06/30	No crystals	T_A
25	3	3:20pm 07/13	1299	2:45pm 07/14	Surface crystals	T_C

25.2 Viscosity

Table 25.2 lists the measured η -T values for AC2-25 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 25.1. Four data points from Table 25.2 were excluded from calculations. One of these points, at 1432°C, had measured η values below the instrument's range while three data points, at 1285°C, 1265°C, and 1245°C, were taken at T s below T_L . The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -15.537 and 25492 K, respectively. This fit, based on five data points at effectively two temperatures (1383°C and $1333.5 \pm 0.5^\circ\text{C}$), was then used to calculate a η_{1350} value of 1.18 Pa·s.

Table 25.2. Viscosity-Temperature Data for AC2-25

T (°C)	η (Pa·s)	10000/T (K ⁻¹)	ln[η (Pa·s)]
1383	0.866	6.039	-0.144
1334	1.386	6.223	0.327
1285	2.121	6.418	0.752
1333	1.397	6.227	0.335
1383	0.878	6.039	-0.130
1432	0.504	5.865	-0.684
1383	0.856	6.039	-0.156
1265	2.979	6.502	1.091
1245	4.255	6.588	1.448

Figure 25.1. Measured ln Viscosity vs. 10000/T for AC2-25

26.0 AC2-26

Glass AC2-26 is an extreme vertex on the outer level of glass compositions. This glass targets 52.2 mass percent Ln_2O_3 . The ratios of SiO_2 and Al_2O_3 to other frit components are higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are high, high, low, and low respectively, in comparison to the standard frit composition.

26.1 Liquidus Temperature

Table 26.1 summarizes the heat treatments performed on this glass. The T_L of this glass was estimated using Furnace #8 at 1208°C. The sample heat treated at 1210°C did not contain crystals, and clusters of needle-like crystals were observed on the bottom of the sample heat treated at 1204°C. The crystal number density increased when the sample was heat treated at 1196°C, and at 1191°C, there was a thin layer of crystals on the bottom. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals.

Five preliminary heat treatments were performed starting at 1149°C up to 1222°C with three of the samples above T_L , one at 1198°C containing a few crystals, and one loaded with RES crystals at 1149°C. An additional sample, heat treated at 1192°C in Furnace #12, contained hair-like crystals.

Table 26.1. Heat-Treatment Summary for Determination of T_L for AC2-26

Crucible	Furnace	Time/Date IN	T (°C)	Time/Date OUT	Observations	Comments
26	12	4:15pm 06/08	1192	2:45pm 06/09	hair-like crystals	different furnace
26	8	9:20am 06/28	1204	11:00am 06/29	Few crystals on the bottom	T_C
26	8	11:00am 07/02	1196	11:10am 07/03	Few crystals	
26	8	2:30pm 07/07	1191	1:10pm 07/08	Crystals throughout	
26	8	3:30pm 07/21	1210	4:10pm 07/22	No crystals	T_A

26.2 Viscosity

Table 26.2 lists the measured η -T values for AC2-26 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 26.1. A datum from Table 26.2, at 1185°C, was taken at T_s below T_L and was excluded from calculations. The excluded point was plotted as an open circle and listed in strike-through font. Equation (2) was fitted to the remaining data and plotted as filled circles to yield E and F values of -18.735 and 31846 K, respectively. The data appear to be nonlinear. Therefore, a η_{1350} value of 2.17 Pa·s was obtained by interpolation between the data points at 1334°C and 1383°C using Equation (2).

Table 26.2. Viscosity-Temperature Data for AC2-26

T (°C)	η (Pa·s)	$10000/T$ (K ⁻¹)	$\ln[\eta(\text{Pa}\cdot\text{s})]$
1384	1.451	6.035	0.372
1334	2.647	6.223	0.973
1285	5.577	6.418	1.719
1334	2.666	6.223	0.981
1383	1.476	6.039	0.390
1433	1.072	5.862	0.069
1482	0.680	5.698	-0.385
1383	1.425	6.039	0.354
1235	13.426	6.631	2.597
1185	34.791	6.859	3.549

Figure 26.1. Measured $\ln$ Viscosity vs. $10000/T$ for AC2-26

27.0 AC2-27

Glass AC2-27 is an extreme vertex on the outer level of glass compositions. This glass targets 52.9 mass percent Ln_2O_3 . The ratios of SiO_2 and Al_2O_3 to other frit components are higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are high, high, low, and low respectively, in comparison to the standard frit composition.

27.1 Liquidus Temperature

Table 27.1 summarizes the final heat treatments performed on samples of AC2-27 in Furnace #3. The T_L of this glass was estimated at 1242°C since only three small crystal clusters formed during the 1241°C heat treatment, and no crystals were observed in samples heat treated at 1240°C and 1242°C. This could have been caused by small composition fluctuations in the glass. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by their characteristic morphologies. The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals.

Three preliminary heat treatments were performed starting at 1222°C up to 1240°C. The sample at 1240°C appeared to be crystal free by OM.

Table 27.1. Heat-Treatment Summary for Determination of T_L for AC2-27

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
27	3	4:00pm 05/28	1241	2:00pm 05/29	Three small clusters	T_C
27	3	4:00pm 06/01	1233	2:30pm 06/02	Crystals throughout	XRD
27	3	3:30pm 06/02	1240	2:30pm 06/03	No crystals	T_A
27	3	4:45pm 06/08	1242	2:45pm 06/09	No crystals	

27.2 Viscosity

Table 27.2 lists the measured η -T values for AC2-27 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 27.1. Three data points from Table 27.2 were excluded from calculations. One of these points, at 1484°C, had a measured η value below the instrument's range while two data points, at 1237°C and 1187°C, were taken at T_s below T_L . The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -16.069 and 27223 K, respectively. The data appear to be nonlinear. Therefore, a η_{1350} value of 1.93 Pa·s was obtained by interpolation between the data points at 1335°C and 1385°C using Equation (2).

Table 27.2. Viscosity-Temperature Data for AC2-27

T (°C)	η (Pa·s)	10000/T (K ⁻¹)	ln[η (Pa·s)]
1385	1.436	6.031	0.362
1335	2.198	6.219	0.788
1286	4.400	6.414	1.482
1335	2.192	6.219	0.785
1385	1.438	6.031	0.363
1435	0.911	5.855	-0.094
1484	0.542	5.692	-0.613
1386	1.401	6.028	0.337
1237	9.832	6.623	2.286
1187	27.600	6.849	3.318

Figure 27.1. Measured ln Viscosity vs. 10000/T for AC2-27

28.0 AC2-28

Glass AC2-28 is an extreme vertex on the outer level of glass compositions. This glass targets 55.6 mass percent Ln_2O_3 . The ratios of Al_2O_3 , B_2O_3 , and SrO to other frit components are higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are low, high, high, and high, respectively, in comparison to the standard frit composition.

28.1 Liquidus Temperature

Table 28.1 summarizes the final heat treatments performed on AC2-28. The T_L was estimated using Furnace #8 at 1260°C. Aluminum oxide crystals formed in samples of this glass heat-treated at temperatures below T_L . The crystals were identified by the characteristic morphologies of the crystals formed below T_L and match those observed in samples of AC2-02, AC2-11, AC2-14, AC2-16, and AC2-21. Aluminum oxide crystals were observed by OM at 1249°C with no crystals observed at 1264°C. A sample treated at 1202°C was scanned under XRD (Figure 28.1), which showed that RES crystals formed at T significantly under T_L .

Six preliminary heat treatments were conducted from 1202°C to 1261°C with Al_2O_3 observed in all samples, except the sample heat treated at 1202°C in which a white, opaque layer was observed on the sample surface. Additional heat treatments were performed at 1242°C (Furnace #3), 1255°C (Furnace #12), and 1259°C (Furnace #12). All three of these samples contained crystals.

Table 28.1. Heat-Treatment Summary for Determination of T_L for AC2-28

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
M	8	3:00pm 05/17	1202	3:15pm 05/18	Many crystals	Preliminary, XRD
28	3	4:45pm 06/08	1242	2:45pm 06/09	Crystals throughout	different furnace
28	12	2:17pm 06/20	1259	1:00pm 06/21	One crystal	different furnace
28	8	2:00pm 06/22	1249	12:35pm 06/23	Crystals	T_C
28	12	8:45am 07/04	1255	8:44am 07/05	Crystals on bottom	different furnace
28	8	4:00pm 07/12	1264	2:45pm 07/13	No crystals	T_A

Figure 28.1. XRD Scan of AC2-28 (1202°C)

28.2 Viscosity

Table 28.2 lists the measured η -T values for AC2-28 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 28.2. Five data points from Table 28.2 were excluded from calculations. One of these points, at 1404°C, had a measured η value below the instrument's range while four data points, at 1247°C, 1227°C, 1206°C, and 1187°C, were taken at T_s below T_L . The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -16.096 and 25843 K, respectively. The data appear to be nonlinear. Therefore, a η_{1350} value of 0.86 Pa·s was obtained by interpolation between the data points at 1335°C and 1383°C using Equation (2).

Table 28.2. Viscosity-Temperature Data for AC2-28

T (°C)	η (Pa·s)	10000/T (K ⁻¹)	ln[η (Pa·s)]
1383	0.593	6.039	-0.522
1335	1.028	6.219	0.028
1286	1.512	6.414	0.413
1334	1.040	6.223	0.039
1384	0.601	6.035	-0.509
1404	0.487	5.963	-0.720
1384	0.601	6.035	-0.509
1266	2.034	6.498	0.710
1247	2.788	6.579	1.025
1227	3.876	6.667	1.355
1206	5.391	6.761	1.685
1187	7.793	6.849	2.053

Figure 28.2. Measured ln Viscosity vs. 10000/T for AC2-28

29.0 AC2-29

Glass AC2-29 is an extreme vertex on the outer level of glass compositions. This glass targets 61.3 mass percent Ln_2O_3 . The ratio of B_2O_3 to other frit components is higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are low, low, high, and low, respectively, in comparison to the standard frit composition.

29.1 Liquidus Temperature

Table 29.1 summarizes the final heat treatments performed on this glass. The T_L of this glass was estimated using Furnace #12 at 1300°C . One sample formed RES crystals in the corners of the crucible after a heat treatment at 1296°C while other samples, heat treated at 1306°C and above, did not contain crystals. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals. During the 1296°C and 1287°C heat treatments, small nodules or buds of glass formed at the upper edge of the Pt/Rh box folds.

Five preliminary heat treatments were performed between 1222°C and 1349°C at approximately 50°C intervals. Abundant crystals were observed at 1222°C and 1276°C , which matches the observations from final heat treatments. A sample heat treated at 1322°C also contained crystals, but this did not match any of the other observations for this glass. Samples heat treated at 1327°C and 1349°C appeared to be crystal free by OM. An additional sample, heat treated at 1289°C in Furnace #3, contained crystals.

Table 29.1. Heat-Treatment Summary for Determination of T_L for AC2-29

Crucible	Furnace	Date/Time IN	T ($^\circ\text{C}$)	Date/Time OUT	Observations	Comments
29	12	6:40pm 06/06	1338	3:30pm 06/07	No crystals	
29	12	1:35pm 06/19	1320	2:09pm 06/20	No crystals	
29	12	3:40pm 06/21	1312	3:30pm 06/22	No crystals	
29	12	3:00pm 06/25	1296	1:20pm 06/26	crystals in corner	T_C , Buds
29	12	2:00pm 06/23	1306	3:00pm 06/24	No crystals	T_A
29	3	9:30am 07/01	1289	10:45am 07/02	Crystals	different furnace
29	12	4:03pm 07/11	1288	2:15pm 07/12	Crystals	Buds

29.2 Viscosity

Table 29.2 lists the measured η -T values for AC2-29 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 29.3. Five data points from Table 29.2 were excluded from calculations. Four of these points, at 1383°C, 1403°C, and 1384°C, had measured η values below the instrument's range while the other, at 1285°C, was taken at a T below T_L . The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, shown as solid circles, to yield E and F values of -16.871 and 26717 K, respectively. This fit, which accurately portrays the data, was then used to calculate a η_{1350} value of 0.66 Pa.s.

Table 29.2. Viscosity-Temperature Data for AC2-29

T (°C)	η (Pa.s)	10000/T (K ⁻¹)	$\ln[\eta$ (Pa.s)]
1383	0.459	6.039	-0.778
1403	0.337	5.967	-1.088
1384	0.424	6.035	-0.858
1364	0.571	6.109	-0.561
1345	0.711	6.180	-0.341
1325	0.856	6.258	-0.156
1305	1.059	6.337	0.057
1285	1.602	6.418	0.471
1384	0.413	6.035	-0.885

Figure 29.3. Measured $\ln$ Viscosity vs. $10000/T$ for AC2-29

30.0 AC2-30

Glass AC2-30 is an extreme vertex on the outer level of glass compositions. This glass targets 49.4 mass percent Ln_2O_3 . The ratio of SrO to other frit components is lower in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are same, same, same, and low, respectively, in comparison to the standard frit composition.

30.1 Liquidus Temperature

Table 30.1 summarizes the final heat treatments performed on this glass. The T_L of this glass was estimated using Furnace #3 at 1227°C since only a few crystals formed in a sample heat treated at 1225°C while another sample formed no crystals after heat treatment at 1236°C. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals.

Six preliminary heat treatments were accomplished on this glass between 1201°C and 1274°C. RES crystals were observed at 1201°C and 1222°C with no crystals observed in the four samples heat treated above T_L : 1229°C, 1240°C, 1251°C, and 1274°C. Two additional samples, heat treated in Furnace #12 at 1227°C and 1225°C, did not contain crystals.

Table 30.1. Heat-Treatment Summary for Determination of T_L for AC2-30

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
30	3	2:30pm 06/12	1215	3:00pm 06/13	Very crystalline	
30	3	4:30pm 06/14	1236	3:40pm 06/15	No crystals	T_A
30	12	5:00pm 06/15	1227	3:20pm 06/16	No crystals	different furnace
30	12	5:00pm 06/16	1225	3:00pm 06/17	No crystals	different furnace
30	3	8:48am 07/05	1225	8:49am 07/06	Few crystals	T_C

30.2 Viscosity

Table 30.2 lists the measured η -T values for AC2-30 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 30.1. Two data points from Table 30.2 were excluded from calculations. One of these points, at 1482°C, had a measured η value below the instrument's range while the other, at 1186°C, was taken at a T below T_L . The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -16.765 and 27877 K, respectively. The data appear to be nonlinear. Therefore, a η_{1350} value of 1.35 Pa·s was obtained by interpolation between the data points at 1334°C and 1383°C using Equation (2).

Table 30.2. Viscosity-Temperature Data for AC2-30

T (°C)	η (Pa.s)	10000/T (K ⁻¹)	ln[η (Pa.s)]
1383	1.111	6.039	0.105
1334	1.489	6.223	0.398
1286	2.953	6.414	1.083
1333	1.531	6.227	0.426
1382	1.122	6.042	0.115
1432	0.741	5.865	-0.299
1482	0.445	5.698	-0.810
1383	1.107	6.039	0.102
1236	6.528	6.627	1.876
1186	15.316	6.854	2.729

Figure 30.1. Measured ln Viscosity vs. 10000/T for AC2-30

31.0 AC2-31

Glass AC2-31 is an extreme vertex on the outer level of glass compositions. This glass targets 56.7 mass percent Ln_2O_3 . The ratios of SiO_2 and SrO to other frit components are higher in this glass than in the baseline frit. In other words, the relative concentrations of SiO_2 , Al_2O_3 , B_2O_3 , and SrO are high, low, low, and high, respectively, in comparison to the standard frit composition.

31.1 Liquidus Temperature

Table 31.1 summarizes the heat treatments performed on this glass. The T_L of this glass was estimated using Furnace #12 at 1305°C. Gross crystallinity was observed in samples heat-treated at 1296°C and 1301°C while a sample heat treated at 1306°C did not contain crystals. This indicated that gross crystallization occurred at temperatures slightly below T_L . RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals.

Six preliminary heat treatments were performed on this glass between 1222°C and 1322°C in 50°C intervals, followed by three heat treatments near T_L at 1295°C, 1302°C, and 1312°C. An additional sample, heat treated in Furnace #3 at 1313°C, did not contain crystals.

Table 31.1. Heat-Treatment Summary for Determination of T_L for AC2-31

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
31	3	3:00pm 06/18	1313	1:30pm 06/19	No crystals	different furnace
31	12	3:40pm 06/21	1312	3:30pm 06/22	No crystals	
31	12	2:00pm 06/23	1306	3:00pm 06/24	No crystals	T_A
31	12	3:00pm 06/25	1296	1:20pm 06/26	Very crystalline	
31	12	8:52am 07/06	1301	10:50am 07/07	Lots of crystals	T_C

31.2 Viscosity

Table 31.2 lists the measured η -T values for AC2-31 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 31.1. A datum from Table 31.2, at 1483°C, had a measured η value below the instrument's range and was excluded from calculations. The excluded point is plotted as an open circle and listed in strike-through font. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -13.314 and 22414 K, respectively. The data appear to be nonlinear. Therefore, a η_{1350} value of 1.49 Pa·s was obtained by interpolation between the data points at 1345°C and 1384°C using Equation (2).

Table 31.2. Viscosity-Temperature Data for AC2-31

T (°C)	η (Pa·s)	$10000/T$ (K ⁻¹)	$\ln[\eta(\text{Pa}\cdot\text{s})]$
1384	1.264	6.035	0.234
1434	0.825	5.858	-0.192
1483	0.498	5.695	-0.697
1385	1.262	6.031	0.233
1345	1.521	6.180	0.420
1325	1.953	6.258	0.670
1305	2.647	6.337	0.973
1384	1.280	6.035	0.247

Figure 31.1. Measured $\ln$ Viscosity vs. $10000/T$ for AC2-31

Glass AC2-32 is a centroid of the outer level of glass compositions. This glass targets 56.6 mass percent Ln_2O_3 . The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

32.1 Liquidus Temperature

Table 32.1 summarizes the heat treatments performed on this glass. The T_L of this glass was estimated using Furnace #12 at 1264°C. A sample heat treated at 1260°C contained crystals throughout while a sample heat treated at 1265°C appeared crystal free. This indicated that gross crystallization occurred at temperatures slightly below T_L . RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals. During the 1265°C and 1273°C heat treatments, small nodules or buds of glass formed at the upper edge of the Pt/Rh box folds.

Six preliminary heat treatments were performed between 1222°C and 1274°C, with four of the heat treatments within 10°C of T_L .

Table 32.1. Heat-Treatment Summary for Determination of T_L for AC2-32

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
32	12	5:00pm 06/17	1260	3:00pm 06/18	Crystals throughout	T_C
32	8	3:00pm 06/25	1273	1:25pm 06/26	No crystals	different furnace, Buds
32	12	2:55pm 06/27	1265	1:00pm 06/28	No crystals	T_A , Buds

32.2 Viscosity

Table 32.2 lists the measured η -T values for AC2-32 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 32.1. A datum from Table 32.2, at 1246°C, was taken at a T below T_L and was excluded from calculations. The excluded point was plotted as an open circle and listed in strike-through font. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -15.834 and 25929 K, respectively. The data appear to be nonlinear. Therefore, a η_{1350} value of 1.18 Pa·s was obtained by interpolation between the data points at 1345°C and 1383°C using Equation (2).

Table 32.2. Viscosity-Temperature Data for AC2-32

T (°C)	η (Pa·s)	$10000/T$ (K ⁻¹)	$\ln[\eta(\text{Pa}\cdot\text{s})]$
1383	0.790	6.039	-0.236
1403	0.700	5.967	-0.357
1424	0.554	5.893	-0.590
1385	0.859	6.031	-0.152
1345	1.256	6.180	0.228
1325	1.521	6.258	0.420
1305	1.660	6.337	0.507
1285	2.150	6.418	0.766
1266	2.930	6.498	1.075
1246	4.311	6.583	1.461
1384	0.878	6.035	-0.130

Figure 32.1. Measured $\ln$ Viscosity vs. $10000/T$ for AC2-32

33.0 AC2-33

Glass AC2-33 is an interior point of the designed glass-composition region. This glass targets 56.4 mass percent Ln_2O_3 . The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

33.1 Liquidus Temperature

Table 33.1 summarizes the heat treatments performed on this glass. The T_L of this glass was estimated at 1271°C using Furnaces #8 and #12. In Furnace #12, a sample heat treated at 1265°C contained crystals throughout while a sample heat treated at 1274°C did not contain crystals. In Furnace #8, a sample heat treated at 1264°C contained crystals while a sample heat treated at 1273°C did not. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals. During the heat treatments in Furnace #8, small nodules or buds of glass formed at the upper edge of the Pt/Rh box folds.

Six preliminary heat treatments were done between 1215°C and 1303°C with four of the heat treatments near T_L .

Table 33.1. Heat-Treatment Summary for Determination of T_L for AC2-33

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
33	12	5:00pm 06/17	1260	3:00pm 06/18	Crystals throughout	
33	8	3:00pm 06/25	1273	1:25pm 06/26	No crystals	T_A - 2, Buds
33	12	2:55pm 06/27	1265	1:00pm 06/28	Crystals throughout	T_C - 1
33	12	8:50am 07/05	1274	8:51am 07/06	No crystals	T_A - 1
33	8	8:52am 07/05	1264	8:50am 07/06	Some crystals	T_C - 2, Buds

33.2 Viscosity

Table 33.2 lists the measured η -T values for AC2-33 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 33.1. Two data points from Table 33.2 were excluded from calculations. One of these points, at 1433°C, had measured η value below the instrument's range while the other, at 1237°C, was taken at a T below T_L . The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, shown as solid circles, to yield E and F values of -14.740 and 24182 K, respectively. This fit, which accurately portrays the data, was then used to calculate a η_{1350} value of 1.17 Pa·s.

Table 33.2. Viscosity-Temperature Data for AC2-33

T (°C)	η (Pa·s)	10000/T (K ⁻¹)	ln[η (Pa·s)]
1384	0.866	6.035	-0.144
1334	1.396	6.223	0.333
1285	2.128	6.418	0.755
1334	1.402	6.223	0.338
1383	0.875	6.039	-0.133
1433	0.542	5.862	-0.613
1384	0.837	6.035	-0.179
1237	7.027	6.623	1.950

Figure 33.1. Measured ln Viscosity vs. 10000/T for AC2-33

34.0 AC2-34

Glass AC2-34 is an interior point of the designed glass-composition region. This glass targets 57.2 mass percent Ln_2O_3 . The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

34.1 Liquidus Temperature

Four final heat treatments of AC2-34 glass were performed in Furnace #3 between 1215°C and 1236°C (see Table 34.1). The sample at 1225°C had needle-like crystals present, while the sample at 1235°C appeared to be crystal free by OM; therefore, T_L was estimated at 1232°C. A sample heat treated at 1236°C had a few dissolving, rounded RES crystals at some of the corners. This sample did not fit with the other heat-treatment measurements performed near T_L and was probably affected by inhomogeneity in the glass, volatilization, or impurities from another source. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals.

Six preliminary heat treatments were performed at 1198°C, 1212°C, 1220°C, 1222°C, 1223°C, and 1233°C. Additional heat treatments were performed in Furnace #12 at 1225°C and 1227°C; neither sample contained crystals.

Table 34.1. Heat-Treatment Summary for Determination of T_L for AC2-34

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
34	3	2:30pm 06/12	1215	3:00pm 06/13	very crystalline	
34	3	4:30pm 06/14	1236	3:40pm 06/15	few crystals	
34	12	5:00pm 06/15	1227	3:20pm 06/16	no crystals	different furnace
34	12	5:00pm 06/16	1225	3:00pm 06/17	no crystals	different furnace
34	3	8:48am 07/05	1225	8:49am 07/06	crystals throughout	T_C
34	3	4:17pm 07/19	1235	3:10pm 07/20	no crystals	T_A

34.2 Viscosity

Table 34.2 lists the measured η -T values for AC2-34 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 34.1. With a T_L of 1232°C, all the data in Table 34.2 are valid for calculations. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -14.092 and 23418 K, respectively. The data appear to be nonlinear. Therefore, a η_{1350} value of 1.44 Pa·s was obtained by interpolation between the data points at 1345°C and 1365°C using Equation (2).

Table 34.2. Viscosity-Temperature Data for AC2-34

T (°C)	η (Pa·s)	$10000/T$ (K ⁻¹)	$\ln[\eta$ (Pa·s)]
1384	1.061	6.035	0.059
1434	0.679	5.858	-0.388
1385	1.046	6.031	0.045
1365	1.286	6.105	0.252
1345	1.494	6.180	0.402
1325	1.547	6.258	0.437
1305	2.044	6.337	0.715
1285	2.759	6.418	1.015
1384	1.041	6.035	0.040

Figure 34.1. Measured $\ln$ Viscosity vs. $10000/T$ for AC2-34

35.0 AC2-35

Glass AC2-35 is an interior point of the designed glass-composition region. This glass targets 57.9 mass percent Ln_2O_3 . The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

35.1 Liquidus Temperature

Table 35.1 summarizes the heat treatments performed on this glass in Furnace #3. The T_L of this glass was estimated at 1287°C since one sample appeared to be crystal free after a heat treatment at 1289°C, and a second sample formed crystals during a heat treatment at 1284°C. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphology of the crystal as seen in Figure 35.1 and by SEM/EDS. Figure 35.2 shows the EDS spectrum of a crystal observed in the 1284°C heat-treated sample. The crystal appears to be composed of Ln_2O_3 and SiO_2 .

Three preliminary heat treatments were performed at 1223°C, 1278°C, and 1288°C.

Table 35.1. Heat-Treatment Summary for Determination of T_L for AC2-35

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
35	3	3:00pm 06/13	1278	3:30pm 06/14	Crystals	
35	3	5:00pm 06/15	1292	3:20pm 06/16	No crystals	
35	3	5:00pm 06/16	1284	3:00pm 06/17	Crystals	T_C , SEM
35	3	9:30am 07/01	1289	10:45am 07/02	No crystals	T_A

99S169-1(AC2-35;1284 oC)

Figure 35.1. SEM Micrograph of AC2-35 (1284°C)

BATTELLE-PNNL PSL/SEM THU 08-JUL-99 10:52
Cursor:10.230KeV = 98 ROI (1) 0.000: 0.000

Figure 35.2. EDS Spectrum of a RES Crystal in AC2-35 (1284°C)

35.2 Viscosity

Table 35.2 lists the measured η -T values for AC2-35 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 35.3. Two data points from Table 35.2 were excluded from calculations. One of these points, at 1432°C, had measured η values below the instrument's range while the other, at 1285°C, was taken at a T below T_L . The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -15.684 and 25573 K, respectively. This fit, based on five data points at effectively two temperatures ($1382.5 \pm 0.5^\circ\text{C}$ and 1333°C), was then used to calculate a η_{1350} value of 1.08 Pa-s.

Table 35.2. Viscosity-Temperature Data for AC2-35

T (°C)	η (Pa-s)	10000/T (K ⁻¹)	$\ln[\eta$ (Pa-s)]
1382	0.777	6.042	-0.252
1333	1.275	6.227	0.243
1285	1.915	6.418	0.650
1333	1.267	6.227	0.237
1383	0.791	6.039	-0.234
1432	0.459	5.865	-0.778
1383	0.796	6.039	-0.228

Figure 35.3. Measured $\ln$ Viscosity vs. $10000/T$ for AC2-35

36.0 AC2-36

Glass AC2-36 is an interior point of the designed glass-composition region. This glass targets 58.7 mass percent Ln_2O_3 . The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

36.1 Liquidus Temperature

Table 36.1 summarizes the final heat treatments performed on this glass. The T_L of this glass was estimated using Furnace #8 at 1294°C. A sample heat treated at 1289°C contained crystals while a sample heat treated at 1295°C did not. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by SEM/EDS analyses performed on a sample heat treated at 1278°C (Figure 36.1 and Figure 36.2) and by the characteristic morphologies of the crystals formed below T_L (Figure 36.3). The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals. During the 1286°C heat treatment, small nodules or buds of glass formed at the upper edge of the Pt/Rh box folds.

Eight preliminary heat treatments were accomplished at temperatures between 1223°C and 1349°C, and most of the samples measured near T_L . Two samples, heat treated in Furnace #12 above T_L , did not form crystals.

Table 36.1. Heat-Treatment Summary for Determination of T_L for AC2-36

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
36	12	4:40pm 06/05	1349	5:55pm 06/06	No crystals	different furnace
36	12	6:40pm 06/06	1338	3:30pm 06/07	No crystals	different furnace
36	3	5:00pm 06/17	1302	3:00pm 06/18	No crystals	
36	8	5:00pm 06/24	1286	3:00pm 06/25	Crystals	different furnace, Buds
36	3	2:56pm 06/27	1295	1:00pm 06/28	No crystals	T_A
36	3	9:30am 07/01	1289	10:45am 07/02	Crystals	T_C

36.2 Viscosity

Table 36.2 lists the measured η -T values for AC2-36 in chronological order. These data are shown on an Arrhenius plot ($\ln \eta$ vs. $1/T$) in Figure 36.4. Two data points from Table 36.2 were excluded from calculations. One of these points, at 1433°C, had measured η value below

the instrument's range while the other, at 1286°C, was taken at a T below T_L . The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, shown as solid circles, to yield E and F values of -15.187 and 24702 K, respectively. This fit, which accurately portrays the data, was then used to calculate a η_{1350} value of 1.03 Pa·s.

Table 36.2. Viscosity-Temperature Data for AC2-36

T (°C)	η (Pa·s)	10000/T (K ⁻¹)	ln[η (Pa·s)]
1384	0.738	6.035	-0.304
1335	1.189	6.219	0.174
1286	1.947	6.414	0.666
1384	0.761	6.035	-0.273
1433	0.467	5.862	-0.761
1384	0.769	6.035	-0.263
1325	1.333	6.258	0.287
1305	1.580	6.337	0.457

Figure 36.4. Measured ln Viscosity vs. 10000/T for AC2-36

37.0 AC2-37

Glass AC2-37 is an interior point of the designed glass-composition region. This glass targets 59.4 mass percent Ln_2O_3 . The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

37.1 Liquidus Temperature

Table 37.1 summarizes the final heat treatments performed on this glass. The T_L of this glass was estimated at 1305°C using Furnace #3. One sample appeared to be crystal free after a heat treatment at 1307°C, and a second sample formed crystals during a heat treatment at 1302°C. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were of a similar morphology to those seen in previous glasses; however, the ends of the crystals were “frizzed” as seen in Figure 37.1 and Figure 37.3. Figure 37.2 and Figure 37.4 confirm, by EDS spectra, that the crystals observed in the 1292°C and 1302°C heat-treated samples were RES.

Six preliminary heat treatments were performed at temperatures between 1223°C and 1339°C. The sample heat treated in Furnace #8 at 1292°C was used for SEM/EDS analysis since it had some unusual crystalline characteristics (heavily crystallized, very large needle-shaped crystals, and voids in some of the crystals). Two samples were heat treated in Furnace #12 at 1306°C and 1312°C; both were free of crystals.

Table 37.1. Heat-Treatment Summary for Determination of T_L for AC2-37

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
37	3	5:00pm 06/17	1302	3:00pm 06/18	Crystals throughout	T_C , SEM
37	12	3:40pm 06/21	1312	3:30pm 06/22	No crystals	different furnace
37	12	2:00pm 06/23	1306	3:00pm 06/24	No crystals	different furnace
37	3	9:30am 07/01	1289	10:45am 07/02	Very crystalline	
A	8	2:30pm 05/27	1292	2:40pm 05/28	Very crystalline	different furnace, SEM
37	3	1:15pm 07/08	1307	11:15am 07/09	No crystals	T_A

99S170-1(AC2-37:1292 oC)

Figure 37.1. SEM Micrograph of AC2-37 (1292C)

BATTELLE-PNNL PSL/SEM THU 08-JUL-99 13:13
Cursor:110.230keV = 106 ROI (1) 0.000 0.000

Figure 37.2. EDS Spectrum of a RES Crystal in AC2-37 (1292C)

99S158-1(AC2-37:1302 oC)

Figure 37.3. SEM Micrograph of AC2-37 (1302C)

BATTELLE-PNNL PSL/SEM MON 28-JUN-99 14:55
Cursor:110.230keV = 103

Figure 37.4. EDS Spectrum of a RES Crystal in AC2-37 (1302C)

37.2 Viscosity

Table 37.2 lists the measured η -T values for AC2-37 in chronological order. These data are shown on an Arrhenius plot ($\ln \eta$ vs. $1/T$) in Figure 37.5. A datum from Table 37.2, at

1404°C, had a measured η value below the instrument's range and was excluded from calculations. The excluded point is shown as an open circle and listed in strike-through font. Equation (2) was fitted to the remaining data, shown as solid circles, to yield E and F values of -17.594 and 28495 K, respectively. This fit, which accurately portrays the data, was then used to calculate a η_{1350} value of 0.96 Pa.s.

Table 37.2. Viscosity-Temperature Data for AC2-37

T (°C)	η (Pa.s)	10000/T (K ⁻¹)	ln[η (Pa.s)]
1383	0.670	6.039	-0.400
1335	1.140	6.219	0.131
1383	0.677	6.039	-0.390
1404	0.540	5.963	-0.616
1384	0.683	6.035	-0.381
1325	1.265	6.258	0.235
1305	1.591	6.337	0.464

Figure 37.5. Measured ln Viscosity vs. 10000/T for AC2-37

38.0 AC2-38

Glass AC2-38 is an interior point of the designed glass-composition region. This glass targets 60.2 mass percent Ln_2O_3 . The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

38.1 Liquidus Temperature

Table 38.1 summarizes the final heat treatments performed on this glass. The T_L of this glass was estimated at 1308°C using Furnace #3. Crystals formed in samples heat treated at 1307°C and 1302°C while the sample heat treated at 1313°C remained crystal free. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals.

Six preliminary heat treatments were performed at temperatures between 1251°C and 1339°C. Two additional samples were heat treated in Furnace #12 at 1312°C and 1306°C; both were crystal free.

Table 38.1. Heat-Treatment Summary for Determination of T_L for AC2-38

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
38	3	5:00pm 06/17	1302	3:00pm 06/18	Many surface crystals	
38	12	3:40pm 06/21	1312	3:30pm 06/22	No crystals	different furnace
38	12	2:00pm 06/23	1306	3:00pm 06/24	No crystals	different furnace
38	3	1:15pm 07/08	1307	11:15am 07/09	Crystals throughout	T_C
38	3	3:10pm 7/21	1313	4:10pm 7/22	No crystals	T_A

38.2 Viscosity

Table 38.2 lists the measured η -T values for AC2-38 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 38.1. Two data points from Table 38.2 were excluded from calculations. One of these points, at 1424°C, had measured η value below the instrument's range while the other, at 1305°C, was taken at a T below T_L . The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, shown as solid circles, to yield E and F values of -16.272 and 26319 K, respectively. This fit, which accurately portrays the data, was then used to calculate a η_{1350} value of 0.95 Pa·s.

Table 38.2. Viscosity-Temperature Data for AC2-38

T (°C)	η (Pa·s)	$10000/T$ (K ⁻¹)	$\ln[\eta(\text{Pa}\cdot\text{s})]$
1384	0.669	6.035	-0.402
1424	0.414	5.893	-0.881
1385	0.670	6.031	-0.400
1365	0.828	6.105	-0.188
1345	0.991	6.180	-0.009
1325	1.215	6.258	0.195
1305	1.523	6.337	0.421
1384	0.682	6.035	-0.383

Figure 38.1. Measured $\ln$ Viscosity vs. $10000/T$ for AC2-38

39.0 AC2-39

Glass AC2-39 is an interior point of the designed glass-composition region. This glass targets 60.9 mass percent Ln_2O_3 . The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

39.1 Liquidus Temperature

Four final heat treatments of AC2-39 glass were performed in Furnace #3 between 1302°C and 1321°C (see Table 39.1). The sample heat treated at 1314°C contained plate-like crystals, while the sample heat treated at 1321°C appeared to be crystal free by OM; therefore, T_L was estimated at 1318°C. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals.

Six preliminary heat treatments were performed at identical temperatures as those measured in AC2-38. Two additional samples were heat treated in Furnace #12 at 1312°C and 1306°C; both contained crystals.

Table 39.1. Heat-Treatment Summary for Determination of T_L for AC2-39

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
39	3	5:00pm 06/17	1302	3:00pm 06/18	many crystals	XRD
39	12	3:40pm 06/21	1312	3:30pm 06/22	one large cluster	different furnace
39	12	2:00pm 06/23	1306	3:00pm 06/24	crystals	different furnace
39	3	11:00am 06/29	1314	9:20am 06/30	plate-like crystals	T_C
39	3	1:15pm 07/08	1307	11:15am 07/09	lots of crystals	
39	3	3:00pm 07/14	1321	3:05pm 07/15	no crystals	T_A

39.2 Viscosity

Table 39.2 lists the measured η -T values for AC2-39 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 39.1. Two data points from Table 39.2 were excluded from calculations. One of these points, at 1424°C, had measured η value below the instrument's range while the other, at 1306°C, was taken at a T below T_L . The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, shown as solid circles, to yield E and F values of -16.986 and 27416 K, respectively. This fit, which accurately portrays the data, was then used to calculate a η_{1350} value of 0.91 Pa-s.

Table 39.2. Viscosity-Temperature Data for AC2-39

T (°C)	η (Pa.s)	10000/T (K ⁻¹)	ln[η (Pa.s)]
1385	0.619	6.031	-0.480
1424	0.380	5.893	-0.967
1385	0.654	6.031	-0.424
1365	0.787	6.105	-0.240
1345	0.964	6.180	-0.037
1325	1.180	6.258	0.165
1306	1.610	6.333	0.476
1384	0.641	6.035	-0.444

Figure 39.1. Measured ln Viscosity vs. 10000/T for AC2-39

40.0 AC2-40

Glass AC2-40 is an interior point of the designed glass-composition region. This glass targets 61.7 mass percent Ln_2O_3 . The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

40.1 Liquidus Temperature

Table 40.1 summarizes the final heat treatments performed on this glass. The T_L of this glass was estimated at 1325°C using Furnace #3. Crystals formed in samples heat treated at 1321°C and 1313°C while the sample heat treated at 1328°C did not contain crystals. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals.

Six preliminary heat treatments were performed at temperatures between 1251°C and 1339°C. Two additional heat treatments were performed at 1327°C (Furnace #8) and 1320°C (Furnace #12); neither appeared to contain crystals.

Table 40.1. Heat-Treatment Summary for Determination of T_L for AC2-40

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
40	3	3:00pm 06/18	1313	1:30pm 06/19	Many crystals	
40	8	4:30pm 06/23	1327	3:00pm 06/24	No crystals	different furnace
40	12	5:00pm 06/24	1320	3:00pm 06/25	No crystals	different furnace
40	3	3:00pm 07/14	1321	3:05pm 07/15	Weird crystals	T_C
40	3	3:34pm 07/16	1328	3:44pm 07/17	No crystals	T_A

40.2 Viscosity

Table 40.2 lists the measured η -T values for AC2-40 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 40.1. Two data points from Table 40.2 were excluded from calculations. One of these points, at 1422°C, had measured η values below the instrument's range while the other, at 1324°C, was taken at a T below T_L . The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -18.659 and 30139 K, respectively. This fit, based on four data points at effectively two temperatures ($1382.5 \pm 0.5^\circ\text{C}$ and 1344°C), was then used to calculate a η_{1350} value of 0.91 Pa·s.

Table 40.2. Viscosity-Temperature Data for AC2-40

T (°C)	η (Pa·s)	10000/T (K ⁻¹)	ln[η (Pa·s)]
1382	0.645	6.042	-0.439
1422	0.377	5.900	-0.975
1383	0.635	6.039	-0.454
1344	0.980	6.184	-0.020
1324	1.170	6.262	0.157
1383	0.624	6.039	-0.472

Figure 40.1. Measured ln Viscosity vs. 10000/T for AC2-40

41.0 AC2-41

Glass AC2-41 is an interior point of the designed glass-composition region. This glass targets 62.4 mass percent Ln_2O_3 . The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

41.1 Liquidus Temperature

Table 41.1 summarizes the heat treatments performed on this glass. The T_L of this glass was estimated at 1324°C using Furnace #8. A sample heat treated at 1320°C contained crystals while a sample heat treated at 1327°C did not. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals. During the 1320°C heat treatments, small nodules or buds of glass formed at the upper edge of the Pt/Rh box folds.

Seven preliminary heat treatments were performed between 1198°C and 1339°C at approximately 50°C intervals. Abundant crystals were observed at 1274°C and 1292°C, which matches the observations in the final heat treatments. Samples heat treated at 1326°C and 1339°C appeared to be crystal free by OM. Additional samples were heat treated at 1313°C (Furnace #3) and 1320°C (Furnace #12). The 1320°C sample contained crystals.

Table 41.1. Heat-Treatment Summary for Determination of T_L for AC2-41

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
41	3	3:00pm 06/18	1313	1:30pm 06/19	Many crystals	different furnace
41	8	4:30pm 06/23	1327	3:00pm 06/24	No crystals	T_A
41	12	5:00pm 06/24	1320	3:00pm 06/25	Few crystals	different furnace
41	8	1:15pm 07/08	1320	11:15am 07/09	Crystals	T_C , Buds

41.2 Viscosity

Table 41.2 lists the measured η -T values for AC2-41 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 41.1. With a T_L of 1324°C, all the data in Table 41.2 are valid for calculations. Equation (2) was fitted to the data to yield E and F values of -17.554 and 28261 K, respectively. This fit, which accurately portrays the data, was then used to calculate a η_{1350} value of 0.87 Pa·s.

Table 41.2. Viscosity-Temperature Data for AC2-41

T (°C)	η (Pa·s)	$10000/T$ (K ⁻¹)	$\ln[\eta(\text{Pa}\cdot\text{s})]$
1384	0.590	6.035	-0.528
1364	0.754	6.109	-0.282
1345	0.914	6.180	-0.090
1326	1.127	6.254	0.119
1385	0.616	6.031	-0.485

Figure 41.1. Measured $\ln$ Viscosity vs. $10000/T$ for AC2-41

42.0 AC2-42

Glass AC2-42 is an interior point of the designed glass-composition region. This glass targets 49.0 mass percent Ln_2O_3 . The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

42.1 Liquidus Temperature

Table 42.1 summarizes the heat treatments performed on this glass in Furnace #3. The T_L of this glass was estimated at 1180°C since a sample heat treated at 1179°C contained crystals while a sample heat treated at 1191°C did not. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals.

Seven preliminary samples were heat treated at temperatures ranging from 1135°C to 1223°C.

Table 42.1. Heat-Treatment Summary for Determination of T_L for AC2-42

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
42	3	9:25am 06/30	1179	9:25am 07/01	Few crystals	T_C
42	3	2:00pm 07/02	1164	1:55pm 07/03	Many crystals	
42	3	2:00pm 07/03	1172	8:42am 07/04	One crystals	T_A
42	3	3:30pm 07/12	1191	2:45pm 07/13	No crystals	

42.2 Viscosity

Table 42.2 lists the measured η -T values for AC2-42 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 42.1. With a T_L of 1180°C, all the data in Table 42.2 are valid for calculations. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -18.192 and 30646 K, respectively. The data appear to be nonlinear. Therefore, a η_{1350} value of 1.75 Pa·s was obtained by interpolation between the data points at 1333°C and 1382°C using Equation (2).

Table 42.2. Viscosity-Temperature Data for AC2-42

T (°C)	η (Pa·s)	10000/T (K ⁻¹)	ln[η (Pa·s)]
1382	1.381	6.042	0.323
1333	1.992	6.227	0.689
1284	4.005	6.423	1.388
1332	2.008	6.231	0.697
1382	1.391	6.042	0.330
1432	0.899	5.865	-0.106
1481	0.569	5.701	-0.564
1383	1.373	6.039	0.317
1235	8.558	6.631	2.147
1186	20.731	6.854	3.032

Figure 42.1. Measured ln Viscosity vs. 10000/T for AC2-42

43.0 AC2-43

Glass AC2-43 is an interior point of the designed glass-composition region. This glass targets 52.7 mass percent Ln_2O_3 . The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

43.1 Liquidus Temperature

Table 43.1 summarizes the heat treatments performed on this glass. The T_L of this glass was estimated at 1227°C using Furnace #3. Crystals formed in a sample heat treated at 1225°C while a sample heat treated at 1236°C did not contain crystals. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals.

Three preliminary heat treatments were performed at 1226°C, 1227°C, and 1233°C. This minute range of 7°C narrowed the T_L for final testing (Table 43.1) where five measurements were used to complete the testing on this glass. This is very uncommon, although nice. Additional heat treatments at 1225°C and 1227°C (in Furnace #12) produced samples that did not contain crystals.

Table 43.1. Heat-Treatment Summary for Determination of T_L for AC2-43

Crucible	Furnace	Time/Date IN	T (°C)	Time/Date OUT	Observations	Comments
43	3	2:30pm 06/12	1215	3:00pm 06/13	Very crystalline	
43	3	4:30pm 06/14	1236	3:40pm 06/15	No crystals	T_A
43	12	5:00pm 06/15	1227	3:20pm 06/16	No crystals	different furnace
43	12	5:00pm 06/16	1225	3:00pm 06/17	No crystals	different furnace
43	3	8:48am 07/05	1225	8:49am 07/06	Few crystals	T_C

43.2 Viscosity

Table 43.2 lists the measured η -T values for AC2-43 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 43.1. Two data points from Table 43.2 were excluded from calculations. One of these points, at 1482°C, had a measured η value below the instrument's range while the other, at 1187°C, was taken at a T below T_L . The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -18.055 and 30044 K, respectively. The data appear to be nonlinear. Therefore, a η_{1350} value of 1.32 Pa·s was obtained by interpolation between the data points at 1333°C and 1382°C using Equation (2).

Table 43.2. Viscosity-Temperature Data for AC2-43

T (°C)	η (Pa.s)	10000/T (K ⁻¹)	ln[η (Pa.s)]
1382	1.112	6.042	0.106
1333	1.428	6.227	0.356
1284	2.933	6.423	1.076
1333	1.457	6.227	0.376
1382	1.114	6.042	0.108
1432	0.738	5.865	-0.304
1482	0.435	5.698	-0.832
1384	1.397	6.035	0.335
1237	8.800	6.623	2.175
1187	21.597	6.849	3.073

Figure 43.1. Measured ln Viscosity vs. 10000/T for AC2-43

44.0 AC2-44

Glass AC2-44 is an interior point of the designed glass-composition region. This glass targets 63.9 mass percent Ln_2O_3 . The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

44.1 Liquidus Temperature

Table 44.1 summarizes the heat treatments performed on this glass. The T_L of this glass was estimated at 1305°C using Furnace #3. A sample heat treated at 1302°C formed crystals while one heat treated at 1307°C did not. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L and confirmed by SEM with EDS as shown in Figure 44.1 and Figure 44.2. These crystals, like those observed in samples of AC2-37 and AC2-39, had “frizzed” ends. During the 1307°C heat treatment, small nodules or buds of glass formed at the upper edge of the Pt/Rh box folds.

Six preliminary heat treatments were performed between 1251°C and 1339°C at approximately 50°C intervals. Abundant crystals were observed at 1292°C. This indicated that gross crystallization occurred at temperatures slightly below T_L . Samples at 1318°C and 1339°C appeared to be crystal free by OM. Additional samples were heat treated at 1312°C and 1306°C in Furnace #12; neither sample contained crystals.

Table 44.1. Heat-Treatment Summary for Determination of T_L for AC2-44

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
44	3	5:00pm 06/17	1302	3:00pm 06/18	Crystals throughout	T_C , SEM
44	12	3:40pm 06/21	1312	3:30pm 06/22	No crystals	different furnace
44	12	2:00pm 06/23	1306	3:00pm 06/24	No crystals	different furnace
44	3	1:15pm 07/08	1307	11:15am 07/09	No crystals	T_A , Buds

44.2 Viscosity

Table 44.2 lists the measured η -T values for AC2-41 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 44.3. With a T_L of 1305°C, all the data in Table 44.2 are valid for calculations. Equation (2) was fitted to the data to yield E and F values of -15.956 and 25803 K, respectively. This fit, which accurately portrays the data, was then used to calculate a η_{1350} value of 0.94 Pa·s.

Table 44.2. Viscosity-Temperature Data for AC2-44

T (°C)	η (Pa·s)	10000/T (K ⁻¹)	$\ln[\eta(\text{Pa}\cdot\text{s})]$
1384	0.667	6.035	-0.405
1404	0.55	5.963	-0.598
1384	0.69	6.035	-0.371
1364	0.833	6.109	-0.183
1345	0.996	6.180	-0.004
1325	1.204	6.258	0.186
1305	1.473	6.337	0.387
1383	0.711	6.039	-0.341

Figure 44.3. Measured ln Viscosity vs. 10000/T for AC2-44

45.0 AC2-45

Glass AC2-45 is an interior point of the designed glass-composition region. This glass targets 56.4 mass percent Ln_2O_3 , with double the Ce_2O_3 from the normal feed. The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

45.1 Liquidus Temperature

Table 45.1 summarizes the heat treatments performed on this glass. The T_L of this glass was estimated at 1259°C using Furnace #12. Crystals formed in a sample heat treated at 1255°C while a sample heat treated at 1260°C did not contain crystals. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals.

Seven preliminary heat treatments were performed between 1198°C and 1274°C at approximately 50°C intervals. Abundant crystals were observed at 1248°C, indicating that gross crystallization occurred at temperatures slightly below T_L . Samples at 1263°C, 1270°C, 1274°C appeared to be crystal free by OM. An additional sample was heat treated at 1249°C in Furnace #8. This sample grossly crystallized.

Table 45.1. Heat-Treatment Summary for Determination of T_L for AC2-45

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
45	12	5:00pm 06/17	1260	3:00pm 06/18	No crystals	T_A
45	8	2:00pm 06/22	1249	12:35pm 06/23	Crystals throughout	different furnace
45	12	8:45am 07/04	1255	8:44am 07/05	Crystals	T_C

45.2 Viscosity

Table 45.2 lists the measured η -T values for AC2-45 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 45.1. A datum from Table 45.2, at 1236°C, was taken at a T below T_L and was excluded from calculations. The excluded point was plotted as an open circle and listed in strike-through font. Equation (2) was fitted to the remaining data, shown as solid circles, to yield E and F values of -14.669 and 23967 K, respectively. This fit, which accurately portrays the data, was then used to calculate a η_{1350} value of 1.10 Pa·s.

Table 45.2. Viscosity-Temperature Data for AC2-45

T (°C)	η (Pa·s)	10000/T (K ⁻¹)	ln[η (Pa·s)]
1384	0.803	6.035	-0.220
1336	1.306	6.215	0.267
1286	1.944	6.414	0.665
1335	1.315	6.219	0.274
1384	0.811	6.035	-0.210
1404	0.679	5.963	-0.388
1384	0.809	6.035	-0.212
1236	6.109	6.627	1.810

Figure 45.1. Measured ln Viscosity vs. 10000/T for AC2-45

46.0 AC2-46

Glass AC2-46 is an interior point of the designed glass-composition region. This glass targets 60.2 mass percent Ln_2O_3 . The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

46.1 Liquidus Temperature

Table 46.1 summarizes the heat treatments performed on this glass. The T_L of this glass was estimated at 1333°C using Furnace #3. A sample heat treated at 1328°C contained crystals while a sample heat treated at 1335°C did not. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals.

Five preliminary samples were heat treated at temperatures ranging from 1135°C to 1223°C. Two additional samples were heat treated at 1320°C (Furnace #12) and 1327°C (Furnace #8); both contained crystals.

Table 46.1. Heat-Treatment Summary for Determination of T_L for AC2-46

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
46	12	1:35pm 06/19	1320	2:09pm 06/20	Crystals throughout	different furnace
46	8	4:30pm 06/23	1327	3:00pm 06/24	Crystals	different furnace
46	3	1:30pm 06/26	1335	2:52pm 06/27	No crystals	T_A
46	3	3:00pm 07/14	1321	3:05pm 07/15	Weird crystals	
46	3	3:34pm 07/16	1328	3:44pm 07/17	Lots of crystals	T_C

46.2 Viscosity

Table 46.2 lists the measured η -T values for AC2-29 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 46.1. Four data points from Table 46.2 were excluded from calculations. Three of these points, two at 1384°C and one at 1404°C, had measured η values below the instrument's range while the other, at 1326°C, was taken at a T below T_L . The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, shown as solid circles, to yield E and F values of -16.753 and 26832 K, respectively. This fit, which accurately portrays the data, was then used to calculate a η_{1350} value of 0.80 Pa·s.

Table 46.2. Viscosity-Temperature Data for AC2-46

T (°C)	η (Pa.s)	10000/T (K ⁻¹)	ln[η (Pa.s)]
1384	0.533	6.035	-0.628
1404	0.421	5.963	-0.866
1384	0.540	6.035	-0.616
1366	0.685	6.101	-0.378
1346	0.833	6.177	-0.182
1326	1.414	6.254	0.346
1385	0.564	6.031	-0.572

Figure 46.1. Measured ln Viscosity vs. 10000/T for AC2-46

47.0 AC2-47

Glass AC2-47 is an interior point of the designed glass-composition region. This glass targets 56.4 mass percent Ln_2O_3 and is a duplicate of glass AC2-33. The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

47.1 Liquidus Temperature

Table 47.1 summarizes the heat treatments performed on this glass. The T_L of this glass was estimated at 1272°C using Furnace #12. A sample heat treated at 1265°C contained crystals while a sample heat treated at 1274°C did not. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals. During the 1273°C (Furnace #8) and 1274°C heat treatments, small nodules or buds of glass formed at the upper edge of the Pt/Rh box folds.

Six preliminary heat treatments were performed between 1212°C and 1278°C at approximately 20°C intervals. Abundant crystals were observed at 1240°C and 1258°C. Samples at 1275°C and 1278°C appeared to be crystal free by OM.

Table 47.1. Heat-Treatment Summary for Determination of T_L for AC2-47

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
47	12	5:00pm 06/17	1260	3:00pm 06/18	Crystals throughout	
47	8	3:00pm 06/25	1273	1:25pm 06/26	No crystals	different furnace, Buds
47	12	2:55pm 06/27	1265	1:00pm 06/28	Crystals throughout	T_C
47	12	8:50am 07/05	1274	8:51am 07/06	No crystals	T_A , Buds

47.2 Viscosity

Table 47.2 lists the measured η -T values for AC2-47 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 47.1. A datum from Table 47.2, at 1236°C, was taken at a T below T_L and was excluded from calculations. The excluded point was plotted as an open circle and listed in strike-through font. Equation (2) was fitted to the remaining data, shown as solid circles, to yield E and F values of -14.938 and 24506 K, respectively. This fit, which accurately portrays the data, was then used to calculate a η_{1350} value of 1.17 Pa·s.

Table 47.2. Viscosity-Temperature Data for AC2-47

T (°C)	η (Pa·s)	10000/T (K ⁻¹)	$\ln[\eta(\text{Pa}\cdot\text{s})]$
1382	0.877	6.042	-0.131
1334	1.38	6.223	0.322
1285	2.16	6.418	0.770
1333	1.399	6.227	0.336
1383	0.877	6.039	-0.131
1433	0.553	5.862	-0.592
1384	0.867	6.035	-0.143
1236	7.632	6.627	2.032

Figure 47.1. Measured $\ln$ Viscosity vs. 10000/T for AC2-47

48.0 AC2-48

Glass AC2-48 is an interior point of the designed glass-composition region. This glass targets 49.0 mass percent Ln_2O_3 and is a duplicate of glass AC2-42. The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

48.1 Liquidus Temperature

Table 48.1 summarizes the heat treatments performed on this glass. The T_L of this glass was estimated at 1175°C using Furnace #3. A sample heat treated at 1171°C contained crystals while a sample heat treated at 1179°C did not. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals.

Five preliminary heat treatments were performed between 1149°C and 1233°C at approximately 20°C intervals. Abundant crystals were observed at 1149°C. Samples at 1212°C and 1220°C appeared to be crystal free by OM. An additional sample, heat treated at 1192°C in Furnace #12, did not contain crystals.

Table 48.1. Heat-Treatment Summary for Determination of T_L for AC2-48

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
48	12	4:15pm 06/08	1192	2:45pm 06/09	No crystals	different furnace
48	3	1:05pm 06/28	1185	11:00am 06/29	No crystals	
48	3	9:25am 06/30	1179	9:25am 07/01	No crystals	T_A
48	3	1:15pm 07/10	1171	3:45pm 07/11	Crystals throughout	T_C

48.2 Viscosity

Table 48.2 lists the measured η -T values for AC2-48 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 48.1. A datum from Table 48.2, at 1482°C, had a measured η value below the instrument's range and was excluded from calculations. The excluded point is plotted as an open circle and listed in strike-through font. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -18.693 and 31393 K, respectively. The data appear to be nonlinear. Therefore, a η_{1350} value of 1.73 Pa·s was obtained by interpolation between the data points at 1334°C and 1382°C using Equation (2).

Table 48.2. Viscosity-Temperature Data for AC2-48

T (°C)	η (Pa·s)	10000/T (K ⁻¹)	$\ln[\eta(\text{Pa}\cdot\text{s})]$
1382	1.364	6.042	0.310
1334	1.960	6.223	0.673
1284	3.860	6.423	1.351
1334	1.957	6.223	0.671
1383	1.362	6.039	0.309
1433	0.888	5.862	-0.119
1482	0.540	5.698	-0.616
1383	1.357	6.039	0.305
1235	8.268	6.631	2.112
1186	20.143	6.854	3.003

Figure 48.1. Measured $\ln$ Viscosity vs. 10000/T for AC2-48

49.0 AC2-49

Glass AC2-49 is an interior point of the designed glass-composition region. This glass targets 52.7 mass percent Ln_2O_3 and is a duplicate of glass AC2-43. The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

49.1 Liquidus Temperature

Table 49.1 summarizes the heat treatments performed on this glass. The T_L of this glass was estimated at 1220°C using Furnace #12. A sample heat treated at 1219°C contained crystals while samples heat treated at 1226°C did not. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals.

Four preliminary samples were heat treated at temperatures ranging from 1198°C to 1251°C. An additional sample, heat treated in Furnace #3 at 1242°C, did not contain crystals.

Table 49.1. Heat-Treatment Summary for Determination of T_L for AC2-49

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
49	3	4:45pm 06/08	1242	2:45pm 06/09	No crystals	different furnace
49	12	1:30pm 06/26	1233	2:51pm 06/27	No crystals	
49	12	2:55pm 07/03	1226	8:41am 07/04	No crystals	T_A
49	12	1:15pm 07/10	1219	3:46pm 07/11	One crystal	T_C
49	12	1:45pm 07/14	1210	3:05pm 07/15	Crystals throughout	

49.2 Viscosity

Table 49.2 lists the measured η -T values for AC2-43 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 49.1. Two data points from Table 49.2 were excluded from calculations. One of these points, at 1483°C, had a measured η value below the instrument's range while the other, at 1186°C, was taken at a T below T_L . The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -16.762 and 27886 K, respectively. The data appear to be nonlinear. Therefore, a η_{1350} value of 1.46 Pa·s was obtained by interpolation between the data points at 1334°C and 1383°C using Equation (2).

Table 49.2. Viscosity-Temperature Data for AC2-49

T (°C)	η (Pa.s)	10000/T (K ⁻¹)	ln[η (Pa.s)]
1383	1.101	6.039	0.096
1334	1.541	6.223	0.432
1285	2.975	6.418	1.090
1334	1.528	6.223	0.424
1384	1.128	6.035	0.121
1433	0.732	5.862	-0.312
1483	0.455	5.695	-0.789
1384	1.106	6.035	0.100
1236	6.568	6.627	1.882
1186	15.533	6.854	2.743

Figure 49.1. Measured ln Viscosity vs. 10000/T for AC2-49

50.0 AC2-50

Glass AC2-50 is an interior point of the designed glass-composition region. This glass targets 56.4 mass percent Ln_2O_3 and is a duplicate of glass AC2-33 with a spike of minor components. The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

50.1 Liquidus Temperature

Table 50.1 summarizes the heat treatments performed on this glass. The T_L of this glass was estimated at 1272°C using Furnace #12. A sample heat treated at 1265°C contained crystals while samples heat treated at 1274°C did not. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals. During the 1265°C, 1273°C (Furnace #8), and 1274°C heat treatments, small nodules or buds of glass formed at the upper edge of the Pt/Rh box folds.

Five preliminary heat treatments were performed between 1227°C and 1275°C at approximately 20°C intervals. Abundant crystals were observed at 1227°C and 1233°C. The sample at 1275°C appeared to be crystal free by OM.

Table 50.1. Heat-Treatment Summary for Determination of T_L for AC2-50

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
50	12	5:00pm 06/17	1260	3:00pm 06/18	Crystals throughout	
50	8	3:00pm 06/25	1273	1:25pm 06/26	No crystals	different furnace, Buds
50	12	2:55pm 06/27	1265	1:00pm 06/28	Crystals throughout	T_C , Buds
50	12	8:50am 07/05	1274	8:51am 07/06	No crystals	T_A , Buds

50.2 Viscosity

Table 50.2 lists the measured η -T values for AC2-50 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 50.1. Two data points from Table 50.2 were excluded from calculations. One of these points, at 1434°C, had measured η value below the instrument's range while the other, at 1236°C, was taken at a T below T_L . The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, shown as solid circles, to yield E and F values of -14.338 and 23449 K, respectively. This fit, which accurately portrays the data, was then used to calculate a η_{1350} value of 1.12 Pa·s.

Table 50.2. Viscosity-Temperature Data for AC2-50

T (°C)	η (Pa.s)	10000/T (K ⁻¹)	ln[η (Pa.s)]
1385	0.819	6.031	-0.200
1434	0.461	5.858	-0.774
1385	0.827	6.031	-0.190
1365	0.962	6.105	-0.039
1335	1.326	6.219	0.283
1286	1.989	6.414	0.688
1236	5.230	6.627	1.654
1385	0.819	6.031	-0.200

Figure 50.1. Measured ln Viscosity vs. 10000/T for AC2-50

51.0 AC2-51

Glass AC2-51 is an interior point of the designed glass-composition region. This glass targets 60.2 mass percent Ln_2O_3 and is a duplicate of glass AC2-38 with a spike of minor components. The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

51.1 Liquidus Temperature

Table 51.1 summarizes the heat treatments performed on this glass. The T_L of this glass was estimated at 1305°C using Furnace #3. A sample heat treated at 1300°C contained crystals while a sample heat treated at 1307°C did not. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals.

Five preliminary heat treatments were performed between 1227°C and 1313°C at approximately 50°C intervals. Abundant crystals were observed at 1227°C and 1275°C. The sample at 1313°C appeared to be crystal free by OM. Additional samples, heat treated at 1306°C (Furnace #8) and 1312°C (Furnace #12), did not contain crystals.

Table 51.1. Heat-Treatment Summary for Determination of T_L for AC2-51

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
51	3	1:35pm 06/19	1300	2:09pm 06/20	Very crystalline	T_C
51	12	3:40pm 06/21	1312	3:30pm 06/22	No crystals	different furnace
51	8	11:00am 06/29	1306	9:20am 06/30	No crystals	different furnace
51	3	1:15pm 07/08	1307	11:15am 07/09	No crystals	T_A

51.2 Viscosity

Table 51.2 lists the measured η -T values for AC2-51 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 51.1. A datum from Table 51.2, at 1403°C, had a measured η value below the instrument's range and was excluded from calculations. The excluded point is shown as an open circle and listed in strike-through font. Equation (2) was fitted to the remaining data, shown as solid circles, to yield E and F values of –16.473 and 26651 K, respectively. This fit, which accurately portrays the data, was then used to calculate a η_{1350} value of 0.95 Pa·s.

Table 51.2. Viscosity-Temperature Data for AC2-51

T (°C)	η (Pa·s)	10000/T (K ⁻¹)	ln[η (Pa·s)]
1383	0.672	6.039	-0.397
1403	0.548	5.967	-0.601
1384	0.675	6.035	-0.393
1365	0.830	6.105	-0.186
1345	1.009	6.180	0.009
1325	1.218	6.258	0.198
1305	1.510	6.337	0.412
1384	0.680	6.035	-0.385

Figure 51.1. Measured ln Viscosity vs. 10000/T for AC2-51

52.0 AC2-52

Glass AC2-52 is an interior point of the designed glass-composition region. This glass targets 49.0 mass percent Ln_2O_3 and is a duplicate of glass AC2-42 with a spike of minor components. The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

52.1 Liquidus Temperature

Table 52.1 summarizes the heat treatments performed on this glass using Furnace #3. The T_L of this glass was estimated at 1153°C since a sample heat treated at 1150°C contained crystals while samples heat treated at 1158°C did not. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals.

Five preliminary samples were heat treated at temperatures ranging from 1111°C to 1227°C.

Table 52.1. Heat-Treatment Summary for Determination of T_L for AC2-52

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
52	3	2:18pm 06/20	1173	1:00pm 06/21	No crystals	
52	3	2:00pm 07/02	1164	1:55pm 07/03	No crystals	
52	3	2:30pm 07/07	1158	1:10pm 07/08	No crystals	T_A
52	3	4:30pm 07/15	1150	2:37pm 07/16	Very crystalline	T_C
52	3	2:45pm 07/23	1144	12:45pm 07/24	Few crystals	

52.2 Viscosity

Table 52.2 lists the measured η -T values for AC2-52 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 52.1. With a T_L of 1153°C, all the data in Table 52.2 are valid for calculations. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -17.425 and 29700 K, respectively. The data appear to be nonlinear. Therefore, a η_{1350} value of 2.17 Pa·s was obtained by interpolation between the data points at 1333°C and 1382°C using Equation (2).

Table 52.2. Viscosity-Temperature Data for AC2-52

T (°C)	η (Pa.s)	10000/T (K ⁻¹)	ln[η (Pa.s)]
1382	1.460	6.042	0.379
1333	2.669	6.227	0.982
1284	5.335	6.423	1.674
1333	2.653	6.227	0.976
1382	1.544	6.042	0.434
1432	1.138	5.865	0.129
1481	0.756	5.701	-0.280
1383	1.405	6.039	0.340
1236	11.717	6.627	2.461
1187	29.797	6.849	3.394

Figure 52.1. Measured ln Viscosity vs. 10000/T for AC2-52

53.0 AC2-53

Glass AC2-53 is an interior point of the designed glass-composition region. This glass targets 52.7 mass percent Ln_2O_3 and is a duplicate of glass AC2-43 with a spike of minor components. The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

53.1 Liquidus Temperature

Table 53.1 summarizes the heat treatments performed on this glass using Furnace #3. The T_L of this glass was estimated at 1192°C since a sample heat treated at 1191°C contained crystals while a sample heat treated at 1197°C did not. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals.

Five preliminary samples were heat treated at temperatures ranging from 1149°C to 1227°C.

Table 53.1. Heat-Treatment Summary for Determination of T_L for AC2-53

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
53	3	2:18pm 06/20	1173	1:00pm 06/21	Crystals throughout	
53	3	1:05pm 06/28	1185	11:00am 06/29	Few crystals	
53	3	9:25am 06/30	1179	9:25am 07/01	Crystals throughout	
53	3	3:30pm 07/12	1191	2:45pm 07/13	Few crystals	T_C
53	3	4:36pm 07/18	1197	2:42pm 07/19	No crystals	T_A

53.2 Viscosity

Table 53.2 lists the measured η -T values for AC2-53 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 53.1. A datum from Table 53.2, at 1187°C, was taken at a T below T_L and was excluded from calculations. The excluded point was plotted as an open circle and listed in strike-through font. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -16.316 and 27609 K, respectively. The data appear to be nonlinear. Therefore, a η_{1350} value of 1.76 Pa·s was obtained by interpolation between the data points at 1334°C and 1383°C using Equation (2).

Table 53.2. Viscosity-Temperature Data for AC2-53

T (°C)	η (Pa.s)	10000/T (K ⁻¹)	ln[η (Pa.s)]
1383	1.381	6.039	0.323
1334	1.973	6.223	0.679
1285	4.110	6.418	1.413
1334	1.976	6.223	0.681
1383	1.426	6.039	0.355
1433	0.933	5.862	-0.069
1482	0.637	5.698	-0.452
1384	1.397	6.035	0.335
1237	8.800	6.623	2.175
1187	21.597	6.849	3.073

Figure 53.1. Measured ln Viscosity vs. 10000/T for AC2-53

54.0 AC2-54

Glass AC2-54 is an interior point of the designed glass-composition region. This glass targets 63.9 mass percent Ln_2O_3 and is a duplicate of glass AC2-44 with a spike of minor components. The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

54.1 Liquidus Temperature

Table 54.1 summarizes the heat treatments performed on this glass using Furnace #3. The T_L of this glass was estimated at 1326°C since a sample heat treated at 1321°C contained crystals while a sample heat treated at 1328°C did not. RES crystals formed in samples of this glass heat treated at temperatures below T_L . The primary phase was identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals.

Five preliminary samples were heat treated at temperatures ranging from 1227°C to 1334°C.

Table 54.1. Heat-Treatment Summary for Determination of T_L for AC2-54

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
54	3	1:30pm 06/26	1335	2:52pm 06/27	No crystals	
54	3	11:00am 06/29	1314	9:20am 06/30	Very crystalline	
54	3	3:00pm 07/14	1321	3:05pm 07/15	Weird crystals	T_C , Buds
54	3	3:34pm 07/16	1328	3:44pm 07/17	No crystals	T_{A_2} , Buds

54.2 Viscosity

Table 54.2 lists the measured η -T values for AC2-54 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 54.1. A datum from Table 54.2, at 1404°C, had a measured η value below the instrument's range and was excluded from the calculations. The excluded point is shown as an open circle and listed in strike-through font. Equation (2) was fitted to the remaining data, shown as solid circles, to yield E and F values of -17.617 and 28349 K, respectively. This fit, which accurately portrays the data, was then used to calculate a η_{1350} value of 0.86 Pa·s.

Table 54.2. Viscosity-Temperature Data for AC2-54

T (°C)	η (Pa·s)	$10000/T$ (K ⁻¹)	$\ln[\eta(\text{Pa}\cdot\text{s})]$
1384	0.593	6.035	-0.522
1404	0.480	5.963	-0.733
1385	0.614	6.031	-0.488
1365	0.751	6.105	-0.286
1345	0.911	6.180	-0.094
1326	1.109	6.254	0.103
1385	0.575	6.031	-0.553

Figure 54.1. Measured $\ln$ Viscosity vs. $10000/T$ for AC2-54

55.0 AC2-55

Glass AC2-55 is an interior point of the designed glass-composition region. This glass targets 56.5 mass percent Ln_2O_3 , with double the Ce_2O_3 as compared to the normal feed. The Ce_2O_3 doubling was achieved by replacing Nd_2O_3 in the glass. The ratios of frit components, SiO_2 , Al_2O_3 , B_2O_3 , and SrO , are the same as those in the baseline frit.

55.1 Liquidus Temperature

Table 55.1 summarizes the heat treatments performed on this glass. The T_L of this glass was estimated at 1255°C using Furnace #8. A sample heat treated at 1249°C contained crystals while a sample heat treated at 1257°C did not. RES crystals formed in samples of this glass heat treated at temperatures below T_L . These crystals were identified by the characteristic morphologies of the crystals formed below T_L . The morphologies matched those observed in samples of AC2-03, AC2-05, and other glasses that were shown by SEM and/or XRD to be RES crystals.

Three preliminary samples were heat treated at temperatures ranging from 1226°C to 1251°C. An additional sample was heat treated at 1242°C in Furnace #3. This sample contained crystals.

Table 55.1. Heat-Treatment Summary for Determination of T_L for AC2-55

Crucible	Furnace	Date/Time IN	T (°C)	Date/Time OUT	Observations	Comments
55	3	4:45pm 06/08	1242	2:45pm 06/09	Lots of crystals	different furnace
55	8	2:00pm 06/22	1249	12:35pm 06/23	Lots of crystals	T_C
55	8	8:45am 07/04	1257	8:43am 07/05	No crystals	T_A , Buds
55	8	4:00pm 07/12	1264	2:45pm 07/13	No crystals	

55.2 Viscosity

Table 55.2 lists the measured η -T values for AC2-55 in chronological order. These data are shown on an Arrhenius plot ($\ln\eta$ vs. $1/T$) in Figure 55.1. Two data points from Table 55.2 were excluded from calculations. One of these points, at 1434°C, had measured η value below the instrument's range while the other, at 1237°C, was taken at a T below T_L . The excluded data are plotted as open circles and listed in strike-through font. Equation (2) was fitted to the remaining data, plotted as filled circles, to yield E and F values of -5.474 and 8887 K, respectively. The data appear to be highly nonlinear. Therefore, a η_{1350} value of 1.14 Pa·s was obtained by interpolation between the data points at 1335°C and 1384°C using Equation (2).

Table 55.2. Viscosity-Temperature Data for AC2-55

T (°C)	η (Pa.s)	10000/T (K ⁻¹)	ln[η (Pa.s)]
1384	0.819	6.035	-0.200
1335	1.315	6.219	0.274
1286	1.009	6.414	0.009
1335	1.322	6.219	0.279
1384	0.830	6.035	-0.186
1434	0.492	5.858	-0.710
1385	0.837	6.031	-0.179
1237	5.673	6.623	1.736

Figure 55.1. Measured ln Viscosity vs. 10000/T for AC2-55