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Sulfate Fining Chemistry in Oxidized and Reduced Soda-Lime-Silica Glasses (G Plus Project for Visteon Inc.)

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Sulfate Fining Chemistry in Oxidized and Reduced Soda-Lime-Silica Glasses (G Plus Project for Visteon Inc.)

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Abstract

Various reducing agents were used and their additions were varied to 1) increase glass quality through eliminating defects from silica scum, 2) decrease SO_x emissions through changing the kind and quantity of reducing agents, and 3) improve production efficiency through increased flexibility of glass redox control during continuous processing.

The work included measuring silica sand dissolution and sulfate decomposition in melts from glass batches. Glass batches were heated at a temperature-increase rate deemed similar to that experienced in the melting furnace. The sulfate decomposition kinetics was investigated with thermogravimetric analysis-differential thermal analysis and evolved gas analysis. Sulfur concentrations in glasses quenched at different temperatures were determined using X-ray fluorescence spectroscopy. The distribution of residual sand (that was not dissolved during the initial batch reactions) in the glass was obtained as a function of temperature with optical microscopy in thin-sections of melts. The fraction of undissolved sand was measured with X-ray diffraction.

The results of the present study helped Visteon Inc. reduce the energy consumption and establish the batch containing 0.118 mass% of graphite as the best candidate for Visteon glass production. The improved glass batch has a lower potential for silica scum formation and for brown fault occurrence in the final glass product. It was established that bubbles trapped in the melt even at 1450°C have a high probability to be refined when reaching the hot zone in the glass furnace. Furthermore, silica sand does not accumulate at the glass surface and dissolves faster in the batch with graphite than in the batch with carbocite.

Acronyms

DTA-TGA   Differential Thermal Analysis-Thermogravimetric Analysis

EGA   Evolved Gas Analysis

GC-MS   Gas chromatography-Mass spectrometry

XRF   X-Ray Fluorescence

XRD   X-Ray Diffraction

Contents

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1.0   Introduction

Sulfate is used in soda-lime-silica glasses as a fining agent. Its fining action is caused by the excess sulfate that decomposes, releasing SO_2 and O_2. The need to decrease the use of sulfate, thus minimizing SO_2 emissions, is especially important when mildly reduced soda-lime-silica glass is produced; in these glasses, the solubility of sulfur in glass is low, and thus more SO_2 can be evolved. Unfortunately, adding sulfide instead of sulfate for a better control of SO_2 emissions makes molten glass prone to form silica-sand scum. Silica scum is relatively stable and a source of defects in the glass product. The mechanism of its formation is largely unknown. In this work, we modified the kind and addition of reducing agents in a glass batch to eliminate silica scum formation while reducing SO_2 emissions and allowing a greater flexibility of glass redox control during continuous processing.

The experimental program included measuring silica sand dissolution and sulfate decomposition in melts from batches containing sulfate plus varied fractions of carbocite, graphite, or pyrite. Glass batches were heated at a temperature-increase rate deemed similar to that experienced in the melting furnace. Melts were terminated at different temperatures and quenched for further analyses.

Thermogravimetric analysis-differential thermal analysis (TGA-DTA) with evolved gas analysis (EGA) was used to determine sulfate decomposition kinetics. Optical microscopy and quantitative X-ray diffraction (XRD) were used to determine the distribution and fraction of residual sand (that was not dissolved during the initial batch reactions) in glass as a function of temperature.

The outcome of the study helped to 1) increase glass product quality through eliminating defects from silica scum, and 2) decrease SO_2 emissions through modifying the kind and quantity of reducing agents. Glass production efficiency was improved through an increased flexibility of glass redox control.

2.0   Experimental Methods

This section describes crucible melts, TGA-DTA, hot stage microscopy, EGA, X-ray fluorescence (XRF), and XRD.

2.1   Crucible melts

Oxidized and reduced glass batches were prepared in various amounts following Visteon batch recipes. Each batch was prepared separately. The correct amounts of reagents were added and thoroughly mixed, first by hand in a plastic bag (roll and shake), and then in an agate disc mill for 2 min to ensure batch homogeneity.

Approximately 100-g glass batches were heated up in silica crucibles at 5°C/min, starting from 300°C. At different temperatures (1450°C and 1480°C), silica crucibles were transferred from high-temperature furnace into a preheated oven for annealing. Glass was annealed at 520°C for 2 h. Silica crucibles were cut in half, thin sectioned, and polished for optical microscopy observations.

Approximately 25-g glass batches were heated up in platinum crucibles at 5°C/min from 300°C to temperatures between 1350°C and 1560°C. At different temperatures (1450°C and 1480°C), Pt-crucibles were transferred from high-temperature furnace into a preheated oven for annealing. Glass was annealed at 520°C for 2 h, removed from the Pt-crucible, cut, thin-sectioned, and polished for optical microscopy observations. The baseline reduced batch with graphite accommodated 75% graphite by weight, 0.089 mass%, compared to carbocite[a], 0.118 mass%, in a reduced batch containing carbocite. The amount of graphite in 25-g reduced batches was gradually increased to 0.118 mass%, 0.177 mass%, 0.294 mass%, 0.587 mass%, and 1.167 mass%.

2.2   TGA-DTA

The TGA and DTA (SDT 2960 Simultaneous TGA-DTA) was performed on an oxidized batch with and without adding rouge, a reduced batch with rouge and carbocite, and a reduced batch with pyrite. The ambient flow rate of CO_2 was kept at 25 ml/min. Each glass batch was prepared separately to make sure the correct amounts of reagents were added to each melt. Approximately 140-mg samples were heated at 5°C/min from room temperature to 1450°C.

2.3   Hot Stage Microscopy

About 0.2-g reduced batch containing either carbocite or graphite was heated up in a Pt-crucible as fast as possible from room temperature to desired temperatures with a 10-min hold at each temperature. The progress of batch melting was recorded using a digital camera.

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[a] Carbocite is a trademark of Shamokin Filler Co. in Shamokin, PA for their anthracite coal product.

2.4   EGA

Approximately 25-g of oxidized batch with rouge was heated at 5°C/min in a tall quartz crucible, starting from 300˚C and ending at 1500˚C. The off-gas carried in a 50-ml/min stream of pure He was analyzed quantitatively by gas chromatography-mass spectrometry (GC-MS) (Hewlett Packard 5890A GC with 5971A MS).

2.5   XRF

Approximately 25-g reduced glass batches doped with carbocite or graphite were heated up in Pt-crucibles at 5°C/min and quenched at temperatures from 1000°C to 1500°C by 100°C increments. Glass samples were removed from Pt-crucibles and milled for 4 min in a tungsten carbide mill. Approximately 10-g of samples were placed in a tungsten carbide grinding vessel and ground twice for 30 sec using a Herzog HSM-100/H Semi-Automatic Pulverizer. After the second grinding, 1 gram of Chemplex SpectroBlend Briquetting additive was added to the grinding vessel and the glass was ground for 10 sec. Briquetting additive aids in the grinding and briquetting process by reducing particles to a uniform size and distribution forming a homogeneous sample blend for XRF analysis. Two 38-mm XRF pellet caps were prepared for each glass sample. Each pellet cap was placed into a pellet die and compressed to 35,000 psi using a Carver Press and held for 3 min. Sulfur retention in the glass was measured with an S4 Pioneer wave length dispersive XRF instrument from Bruker AXS (Madison WI) calibrated with glass of known composition.

2.6   XRD

Approximately 25-g reduced glass batches containing either carbocite or graphite were heated up in Pt-crucibles at 5°C/min starting from 300°C. Heat-treatments were terminated at temperatures from 1000°C to 1500°C by 100°C increments, and samples were quenched in air, ground, and mixed with 5 mass% internal standard (CaF_2) using a tungsten carbide mill. Undissolved sand was identified and the mass fraction in each sample was determined with an XRD (Scintag PAD-V) equipped with Peltier detector and Cu target. The scan parameters were step size 0.04°, dwell time 4 sec, and scan range from 5 to 70 2θ. Jade software was used to analyze the scans, and the RIQAS 3.1 program was used to determine mass fractions of undissolved quartz in glass samples.

3.0 Description of Visteon Glass Batches

Soda-lime-silica glasses have been prepared from silica sand, soda ash, dolomite, limestone, nepheline syenite, Brickox, salt cake, rouge, pyrite, graphite, and carbocite provided by Visteon Inc. Table 3.1 summarizes the compositions of oxidized and reduced glass batches used in melting experiments. Figure 2.6.1 shows the silica sand particle size distribution obtained with an image analysis.

Table 3.1. Compositions of Oxidized and Reduced Glass Batches

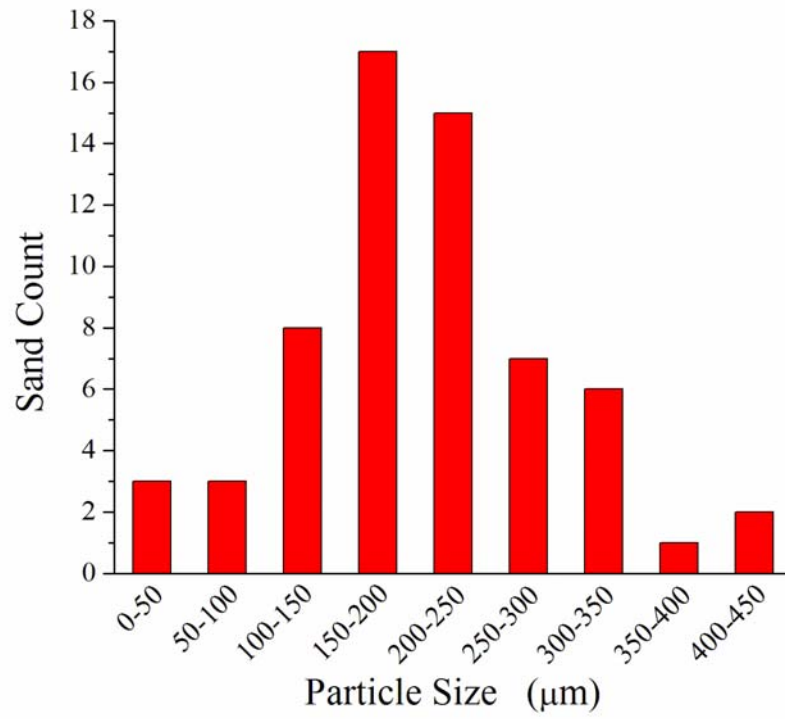|	Oxidized		Reduced		
	with rouge	without rouge	with rouge, carbocite	with pyrite	with rouge, graphite
	Mass%		Mass%		
sand	58.92	59.05	58.95	58.56	58.97
soda ash	19.50	19.54	19.51	19.38	19.52
dolomite	15.59	15.63	15.60	15.50	15.60
limestone	4.67	4.68	4.68	4.65	4.68
nepheline syenite	0.47	0.47	0.47	0.47	0.47
Brickox	0.16	0.16	0.16	0.16	0.16
salt cake	0.46	0.47	0.29	0.79	0.29
rouge (~99.0% Fe_2O_3)	0.22	0	0.22	0	0.22
carbocite	0	0	0.12	0	0
graphite	0	0	0	0	0.09
pyrite	0	0	0	0.49	0
total	100.00	100.00	100.00	100.00	100.00

Figure 2.6.1. Silica Sand Particle Size Distribution

4.0   Results and Discussion

4.1   Crucible Melts

Figure 4.1.1 and Figure 4.1.2 display thin sections of oxidized batch with rouge, reduced batch with rouge and carbocite, reduced batch with pyrite, and oxidized batch with rouge not containing salt cake. A batch without salt cake makes a clear silica scum at 1480°C; see Figure 4.1.2. Silica grain agglomerates in an oxidized batch with rouge at 1450°C are regularly distributed within the melt, and they gradually dissolve with increasing temperature. In a reduced batch with rouge and carbocite, the silica grain agglomerates tend to accumulate below the glass surface. This indicates a possibility of a silica scum formation below 1450°C. There are only a few grains left at 1480°C, and a lot of bubbles gathered up on glass surface next to silica crucible walls. The higher concentration of salt cake in a reduced batch with pyrite than in other batches causes higher foaming at 1480°C but helps disperse the silica grain agglomerates and accelerates the dissolution of silica grains residues. Figure 4.1.3 shows the maximum foam height in oxidized batch with rouge, reduced batch with rouge and carbocite, reduced batch with pyrite, and oxidized batch without salt cake addition. Reduced batch with pyrite is sensitive to foaming at temperatures between 1450°C and 1480°C.

Thin sections of reduced batch with varying addition of graphite are shown in Figure 4.1.4. Due to increasing graphite addition, the glass color changed from yellowish green (0.089 mass% of graphite) to green (0.118 mass% of graphite), dark green with a few brown spots (0.177 mass% of graphite), dark green with a brown-colored flow pattern (0.294 mass% of graphite), brown (0.587 mass% of graphite), and finally to amber coloration (1.167 mass% of graphite). The Amber coloration was produced by the combination of sulfur and iron oxide in a soda-lime-silica glass melted under strongly reducing conditions, which was achieved by appropriate additions of graphite to the glass batch. The sulfide-sulfate redox couples shifted progressively in favor of the sulfide as the carbon level in the glass batch increased. The amber color became more intense as the amount of sulfide was increased.

At 1350°C, large undissolved silica agglomerates and tiny bubbles were present throughout the glasses containing from 0.089 mass% to 0.177 mass% of graphite. Large bubbles accumulated at the glass surface in glasses containing higher concentrations of graphite. The reboil tendency of Visteon glasses was increased with increased addition of graphite.

At 1450°C, the number of tiny bubbles and small silica agglomerates observed close to the glass surface significantly decreased in glasses containing from 0.089 mass % to 0.167 mass% graphite. As the temperature increased, the large silica agglomerates dissolved and dispersed, and bubbles refined.

Large bubbles and a change in melt surface tension due to vaporization in the meniscus caused enough convection to facilitate silica sand dissolution and disrupt silica scum formation.

Traces of unreacted graphite were found in glasses containing 0.294 mass% and 0.587 mass% of graphite and larger amounts in glasses containing 1.167 mass% of graphite.

Figure 4.1.5 through Figure 4.1.8 display in more detail fining bubbles, silica agglomerates, individual silica grains, and brown coloration presented in reduced batches doped with graphite for different melting temperatures and additions of graphite.

A reduced batch with 0.118 mass % graphite seems to be the best candidate for Visteon glass production. The glass has low potential both for silica scum formation and for brown fault in the final product. Bubbles trapped in the melt even at 1450°C are likely to be refined after reaching the hot zone in the glass furnace. Figure 4.1.9 compares oxidized glass and reduced glass doped with carbocite melted at different temperatures, and Figure 4.1.10 compares reduced batches doped with the same concentration of either carbocite or graphite. Silica sand in a reduced batch doped with graphite does not tend to accumulate at the glass surface and dissolves faster than in a reduced batch doped with carbocite. Silica scum formation in glass without salt cake is shown in Figure 4.1.11.

Oxidized batch with rouge	Reduced batch with rouge and carbocite	Reduced batch with pyrite
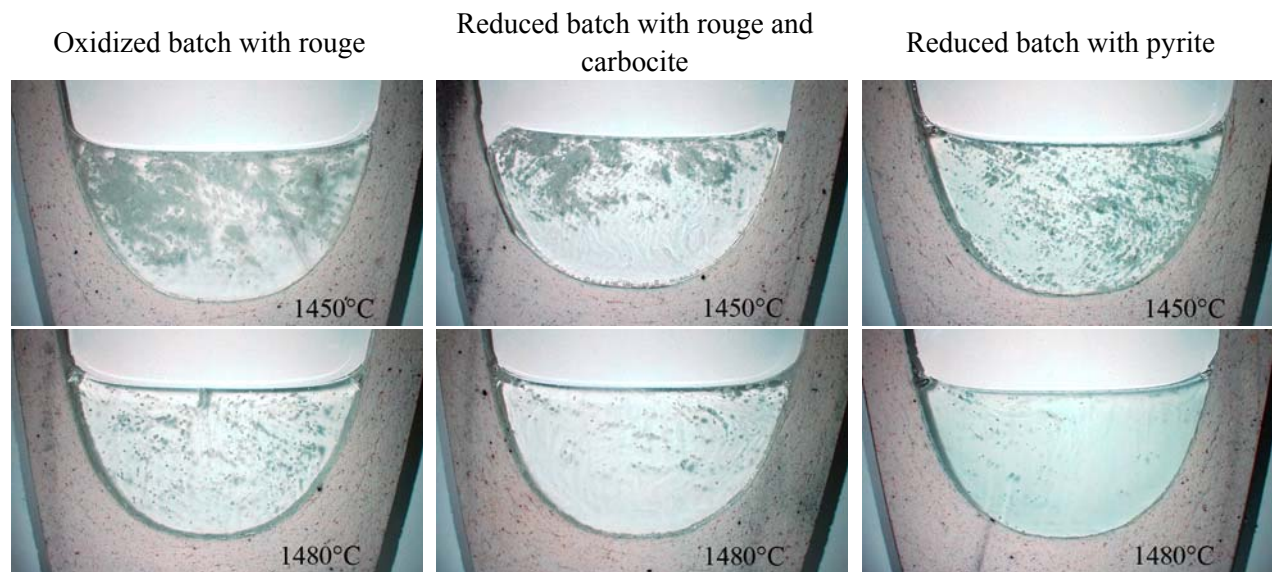
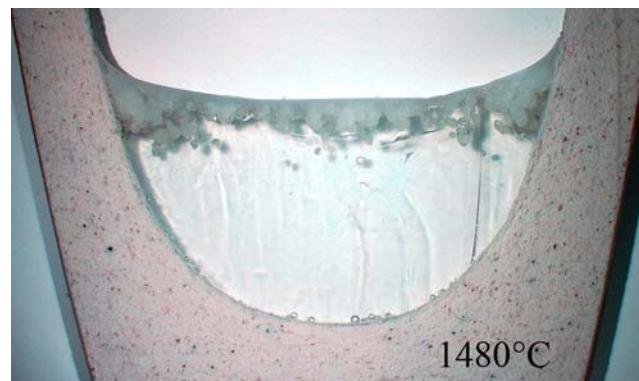**Figure 4.1.1. Oxidized and Reduced Batches Melted at 1450°C and 1480°C**

Figure 4.1.2. Oxidized Batch Without Salt Cake Melted at 1480°C
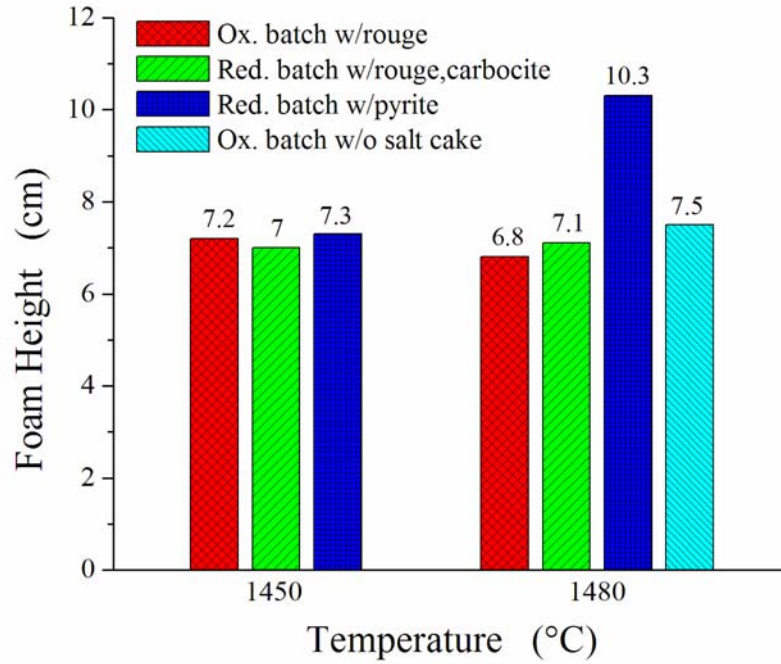
Figure 4.1.3. Measured Foam Height of Oxidized Batch with Rouge and Salt Cake, Oxidized Batch with Rouge and without Salt Cake Addition, Reduced Batch with Rouge and Carbocite, and Reduced Batch with Pyrite
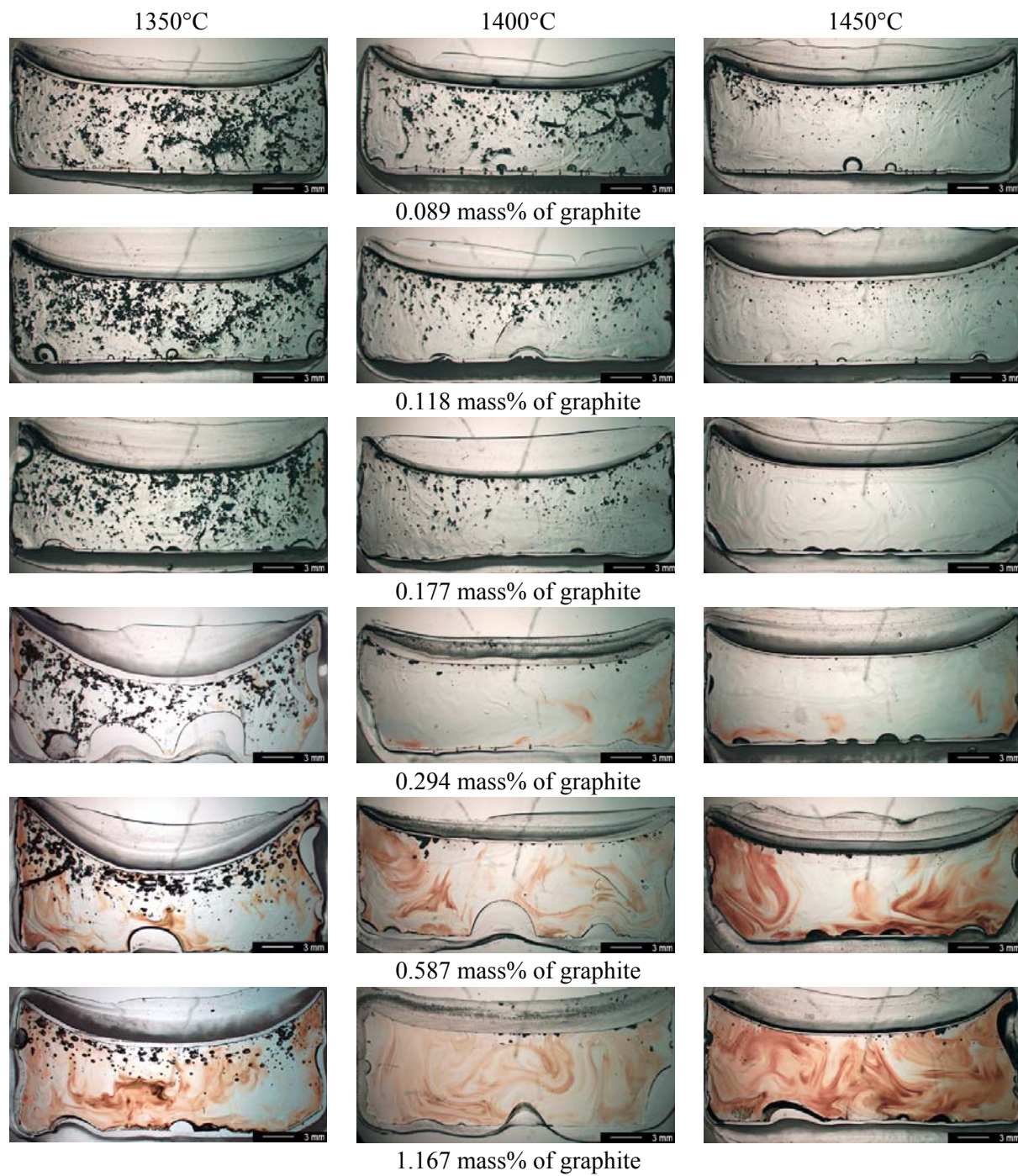
1350°C   1400°C   1450°C

0.089 mass% of graphite

0.118 mass% of graphite

0.177 mass% of graphite

0.294 mass% of graphite

0.587 mass% of graphite

1.167 mass% of graphite

Figure 4.1.4. Reduced Glasses Doped with Different Concentrations of Graphite Quenched at 1350°C, 1400°C, and 1450°C

4.4
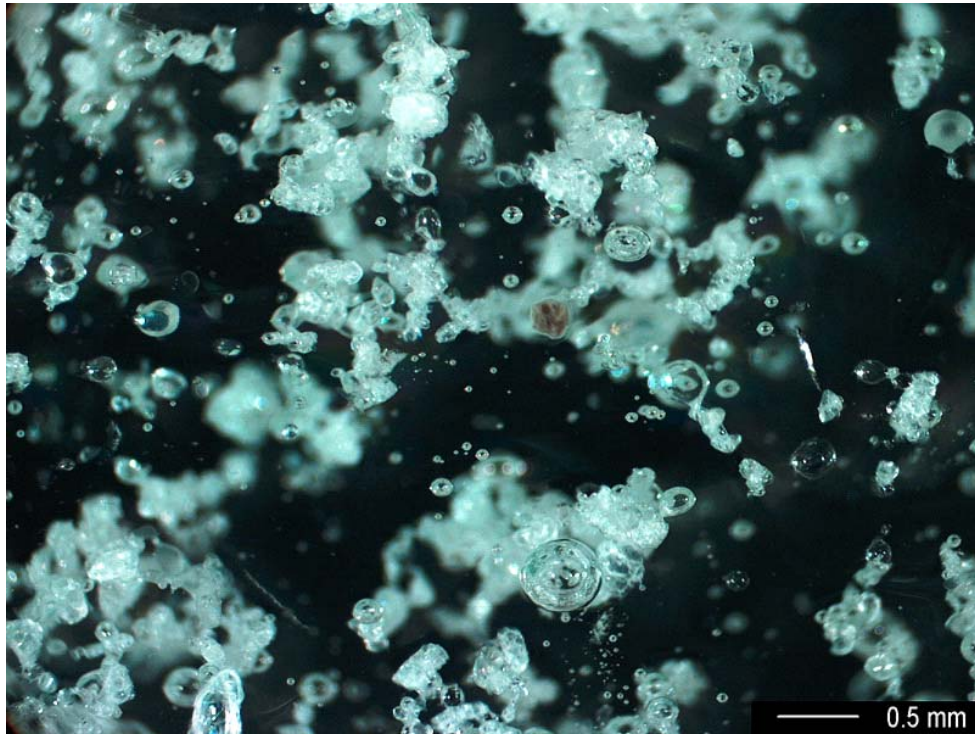
Figure 4.1.5. Reduced Glass Containing 0.177 mass% of Graphite Quenched at 1350°C
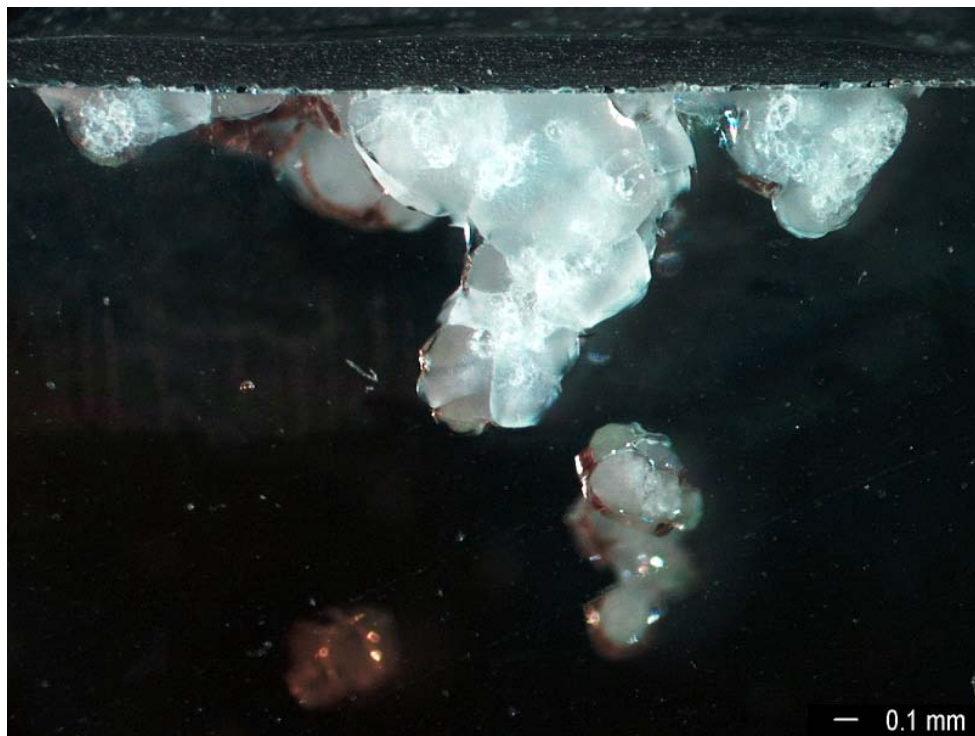
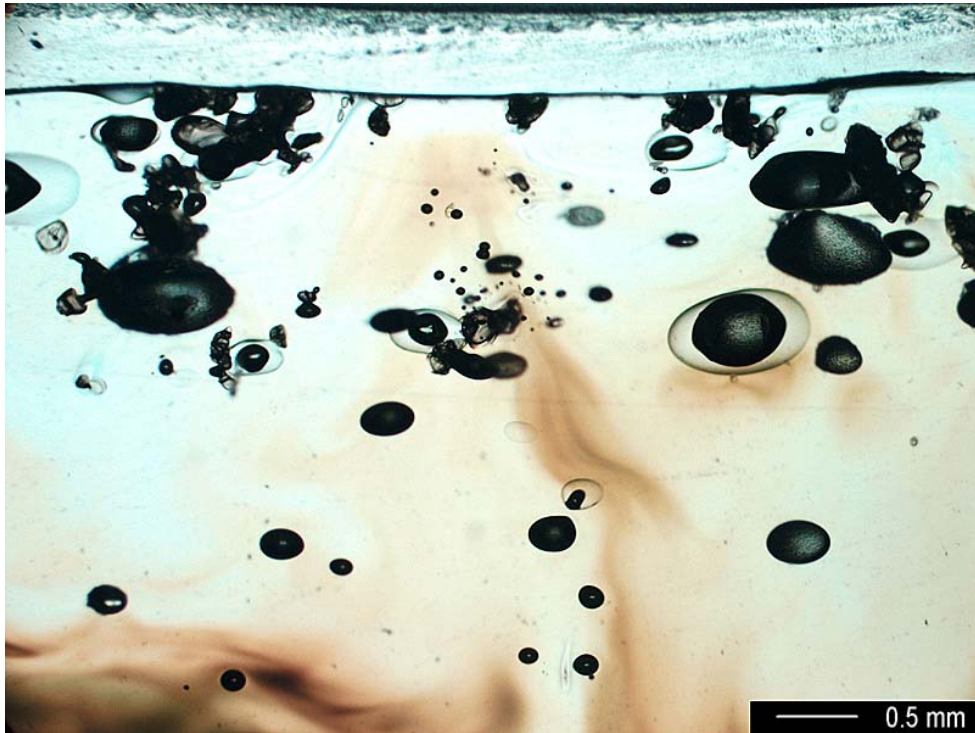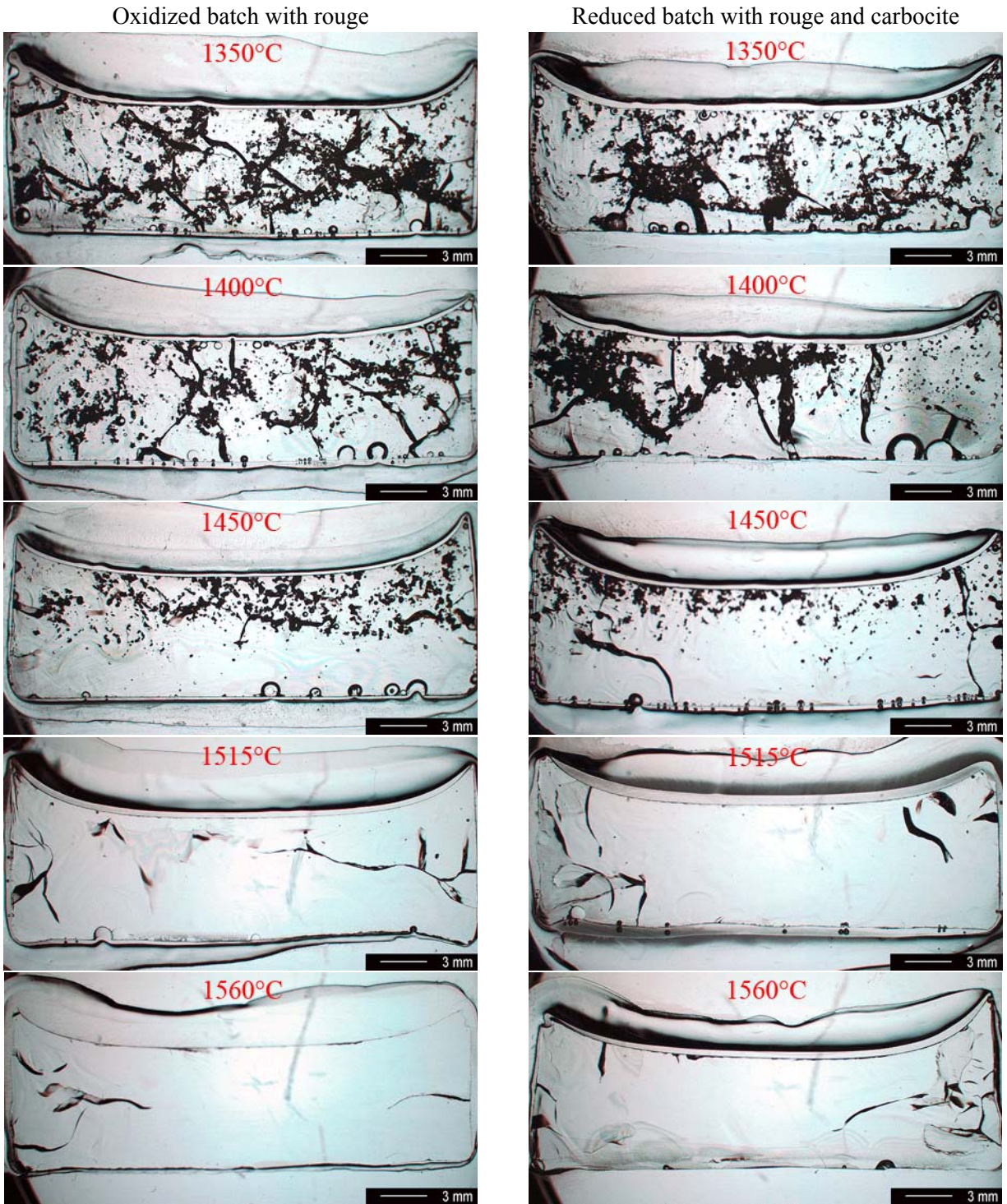**Figure 4.1.6. Reduced Glass Containing 0.587 mass% of Graphite Quenched at 1400°C**
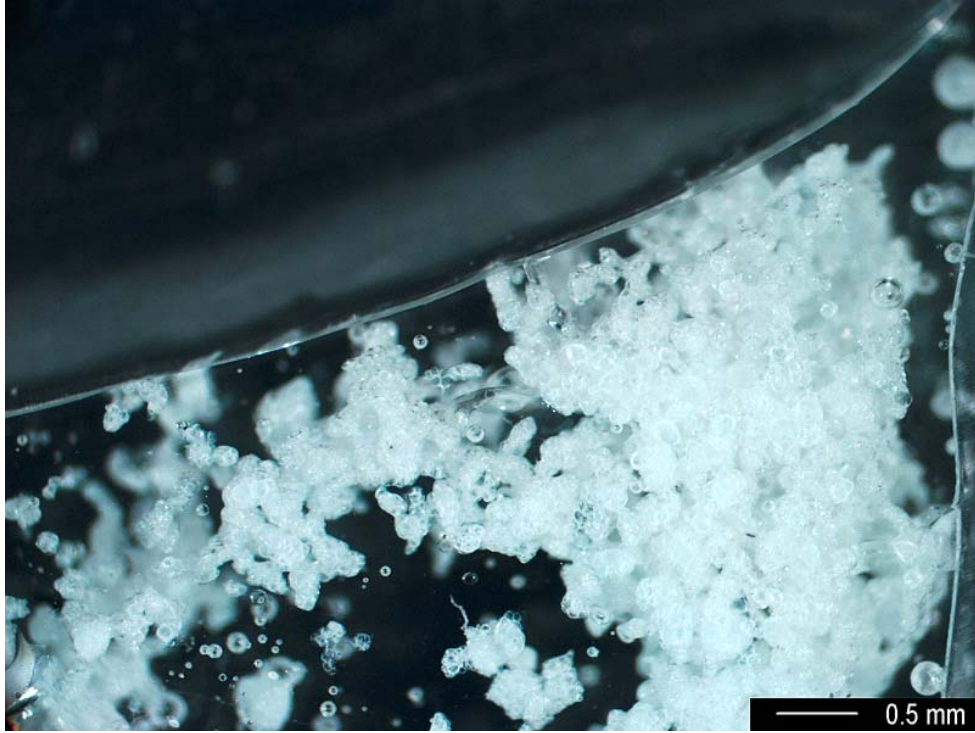
Figure 4.1.7. Reduced Glass Containing 0.089 mass% of Graphite Quenched at 1400°C

Figure 4.1.8. Reduced Glass Containing 1.167 mass% of Graphite Quenched at 1350°C

Oxidized batch with rouge

Reduced batch with rouge and carbocite

**Figure 4.1.9. Oxidized Glass and Reduced Glass Containing Carbocite
Quenched at 1350°C, 1400°C, 1450°C, 1515°C, and 1560°C**

4.7

Reduced batch with carbocite   Reduced batch with graphite
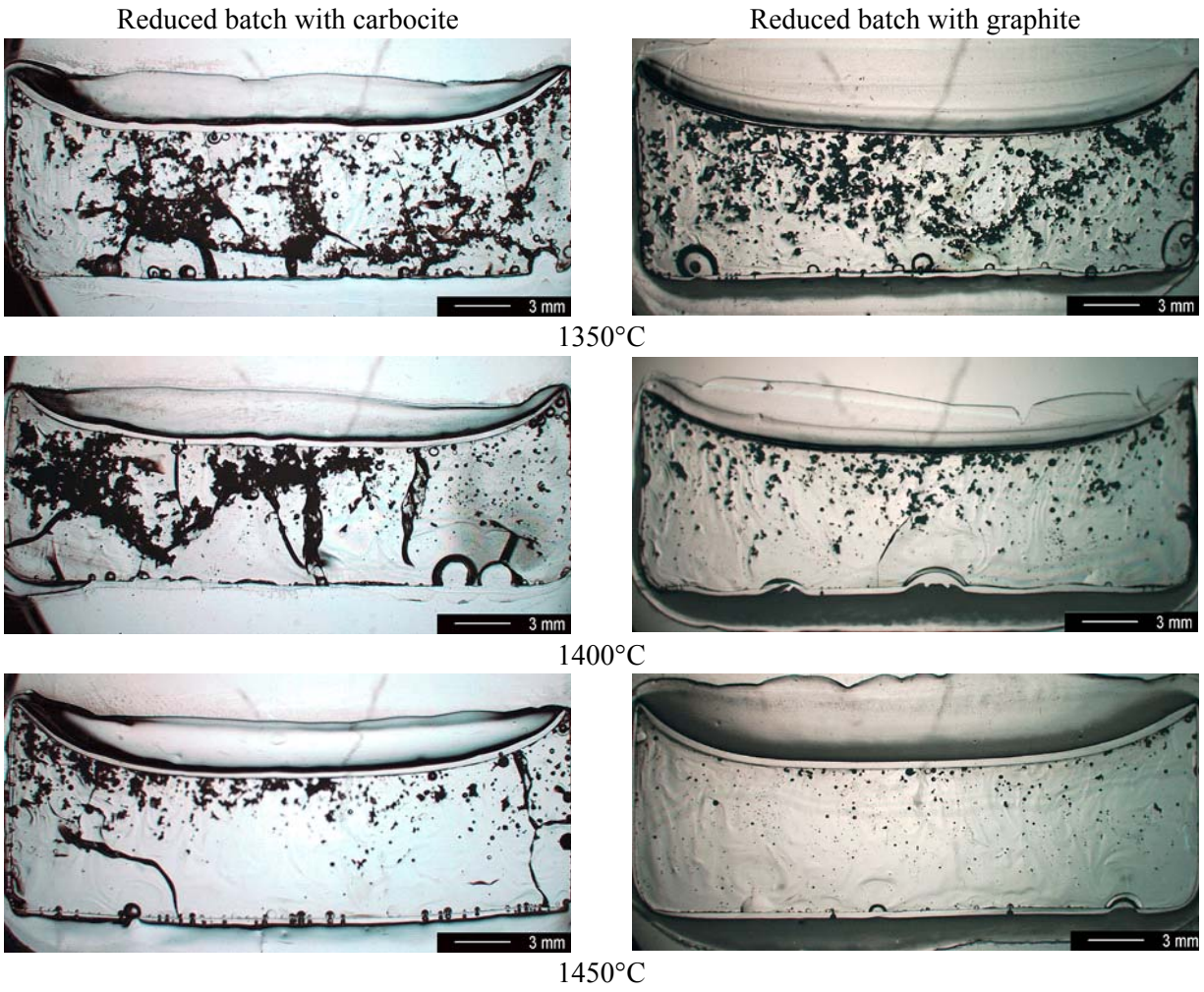
1350°C

1400°C

1450°C

**Figure 4.1.10. Reduced Glass Containing Carbocite or Graphite
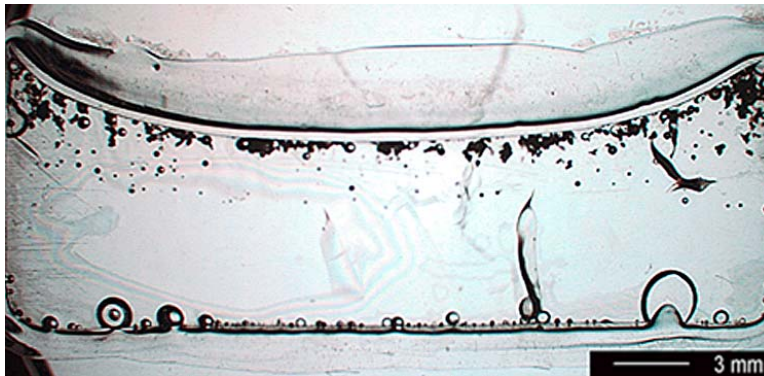Quenched at 1350°C, 1400°C, and 1450°C**

Figure 4.1.11. Oxidized Glass Without Salt Cake Addition Quenched at 1450°C

4.2 TGA-DTA

The results of simultaneous TGA-DTA for oxidized batch with rouge, oxidized batch without rouge, reduced batch with rouge and carbocite, and reduced batch with pyrite are shown in Figure 4.2.1 through Figure 4.2.4.

The salt cake decomposition temperature in Visteon batches was not determined because only a tiny amount of salt cake was added. The endothermic peak at 565°C is associated with the decomposition of $MgCO_3$. Two endothermic peaks at temperatures of 860°C and 877°C are associated with the decomposition of dolomite. Dolomite decomposes to MgO and $CaCO_3$ at 860°C, releasing CO_2, and $CaCO_3$ produced from dolomite decomposes at 877°C, releasing CO_2. Mass loss continues to ~1090°C in an oxidized batch with rouge and a reduced batch with rouge and carbocite and to ~1160°C in a reduced batch with pyrite because of the continuing gas-releasing reactions of soda and limestone.
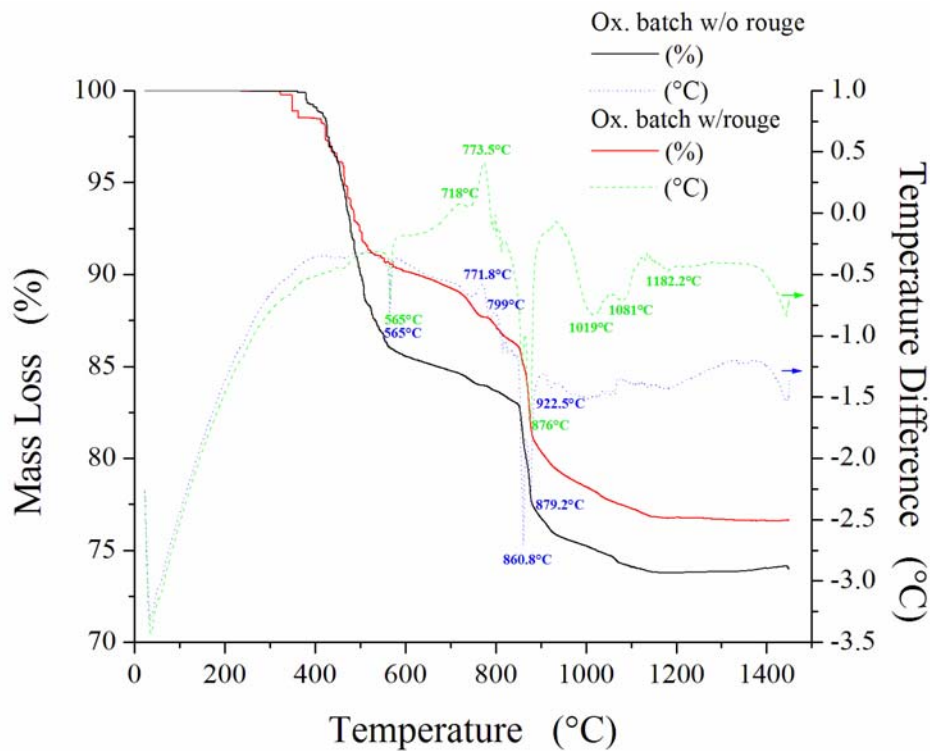

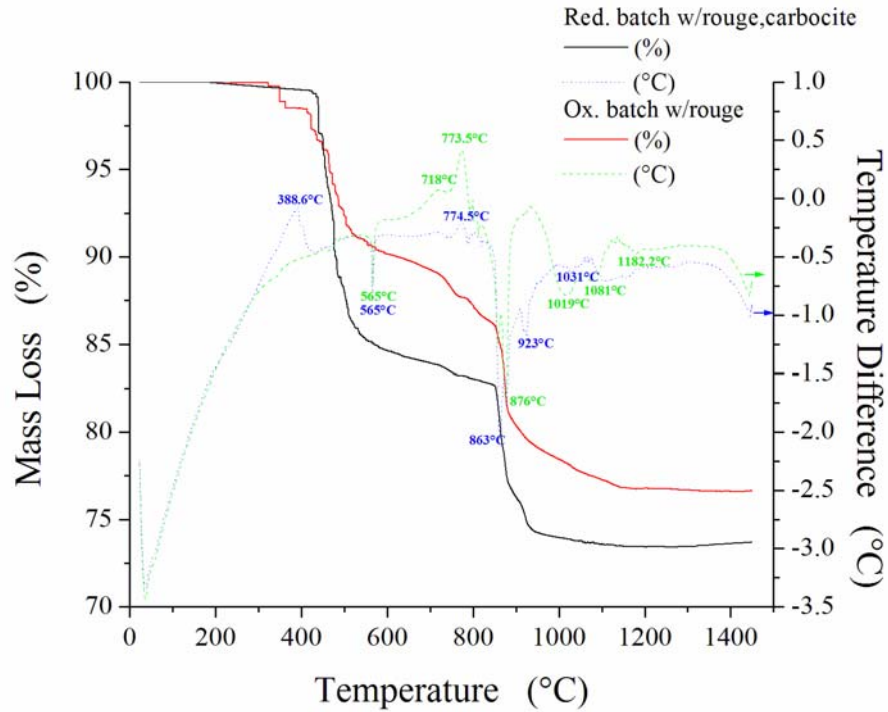**Figure 4.2.1 TGA-DTA Curves of Oxidized Batch with and without Rouge Addition**

Figure 4.2.2. TGA-DTA Curves of Reduced Batch with Rouge and Carbocite and Oxidized Batch with Rouge
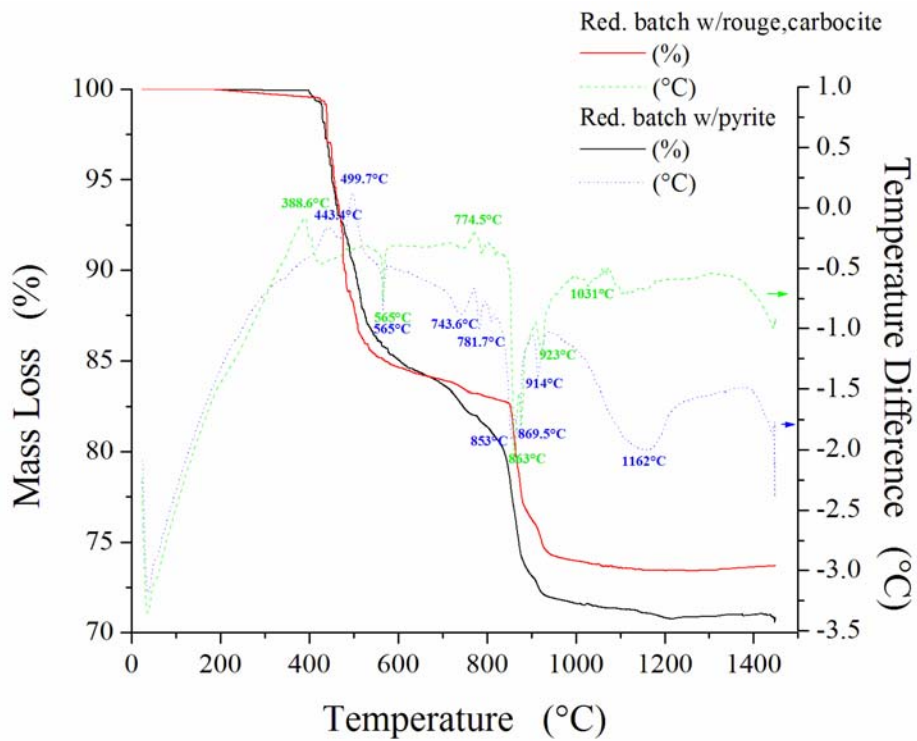

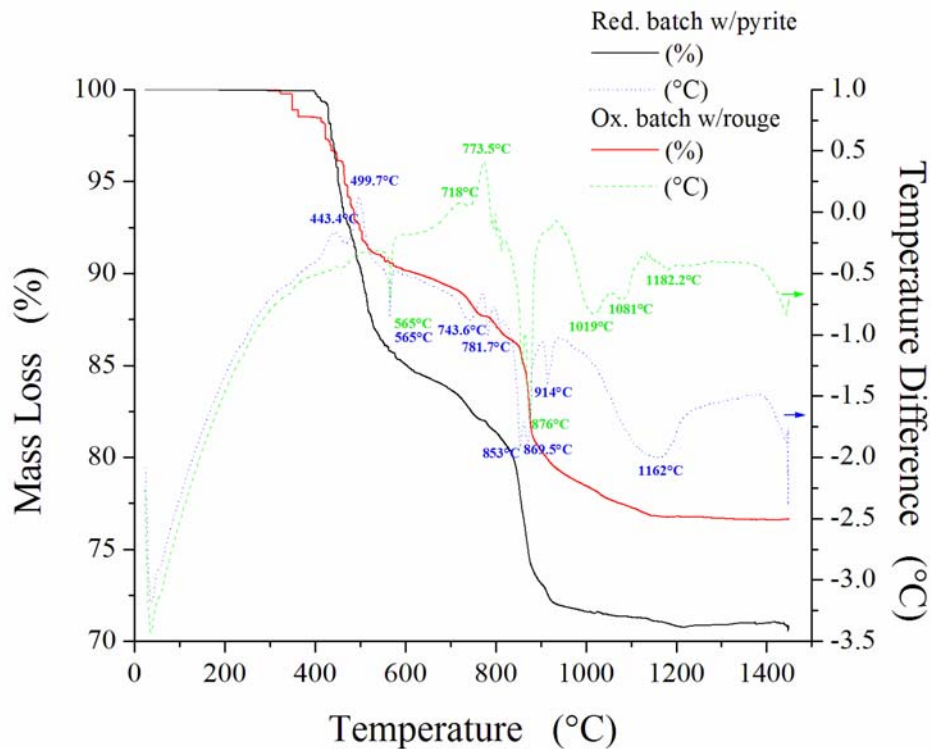**Figure 4.2.3. TGA-DTA Curves of Reduced Batch with Rouge and Carbocite, and Reduced Batch with Pyrite**

Figure 4.2.4. TGA-DTA Curves of Reduced Batch with Pyrite and Oxidized Batch with Rouge

4.3 Hot Stage Microscopy

Sample temperature history is given in Figure 4.3.1. Figure 4.3.2 displays the progress of melting of a reduced batch containing 0.089 mass% of graphite. The grains of dolomite, limestone, and soda ash jiggle slightly after increasing the temperature from 400°C to 500°C and vigorously between 500°C and 700°C. The silica sand dissolution, melt forming, and bubbling become perceptible at 1000°C. A reduced batch containing graphite starts to melt faster, and a larger amount of melt is formed at 1100°C, i.e., at a temperature lower (1200°C), than in a reduced batch with carbocite. However, undissolved sand grains and trapped bubbles formed a silica scum, as seen in Figure 4.3.3.

Figure 4.3.4 displays the progress of melting of a reduced batch containing 0.118 mass% of carbocite. The grains of dolomite, limestone, and soda ash jiggle slightly after a temperature increase from 400°C to 500°C and vigorously between 500°C and 700°C. The silica sand dissolution, melt forming, and bubbling become perceptible at 1100°C. Undissolved small size sand grains and bubbles formed a silica scum that was pushed to the crucible sides by bubble motion and by a surface tension gradient; see Figure 4.3.5.
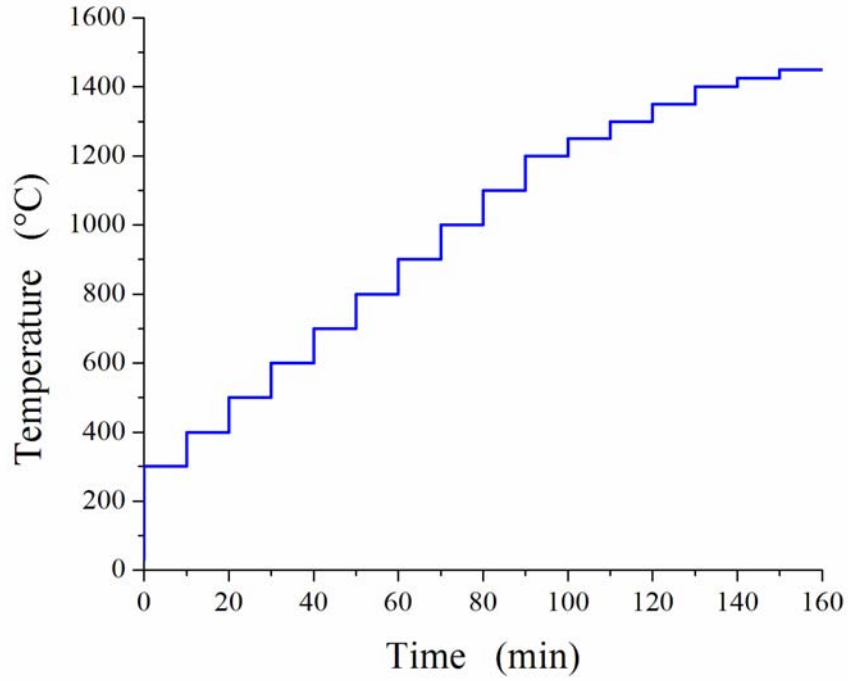
Figure 4.3.1. Temperature History of Reduced Batch Containing Graphite or Carbocite
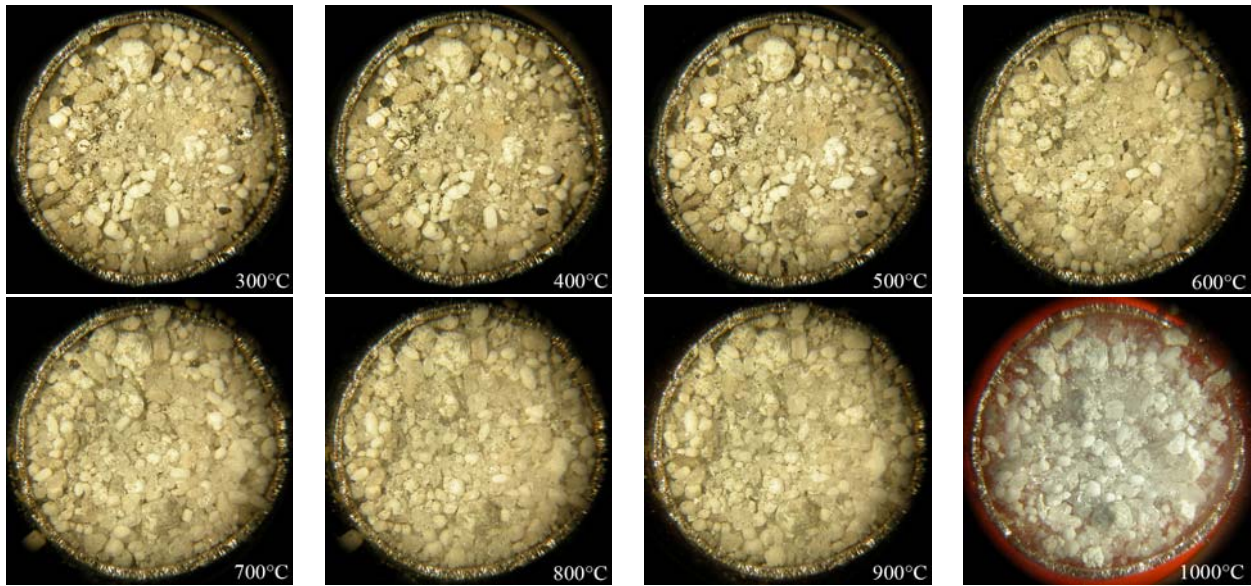

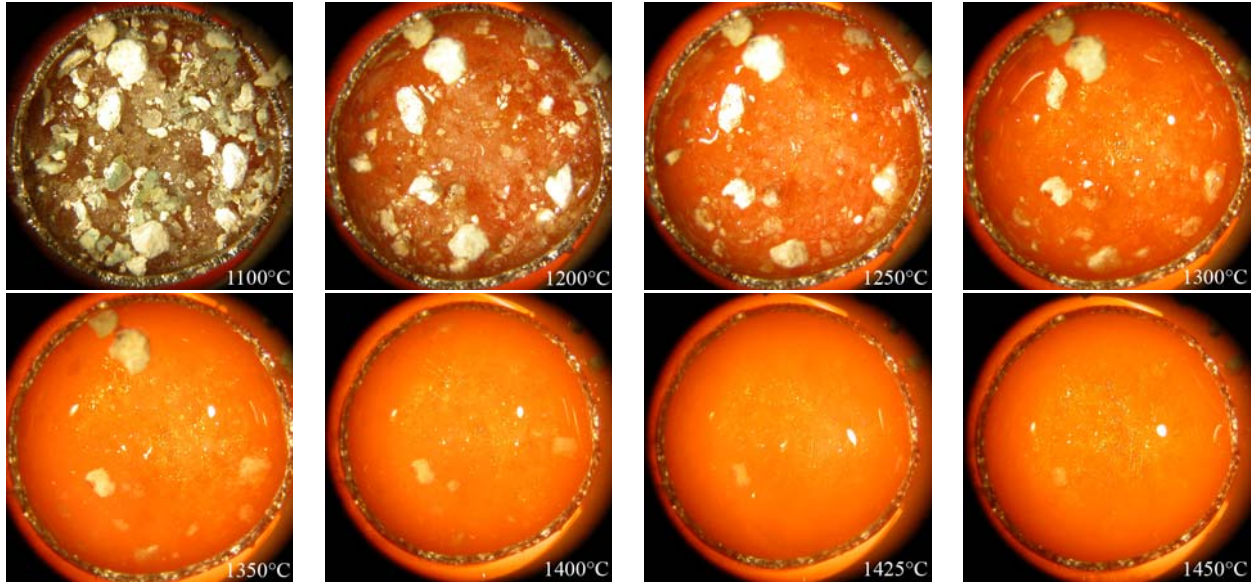**Figure 4.3.2. Melting of Reduced Batch Containing 0.089 Mass% of Graphite**
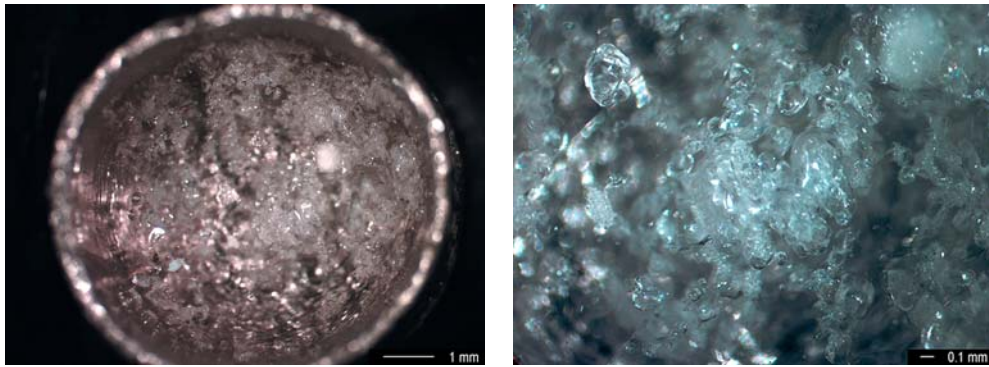
Figure 4.3.2 (contd)

Figure 4.3.3. Undissolved Silica Grains in Reduced Batch Containing 0.089 Mass% of Graphite after Melting to 1450°C
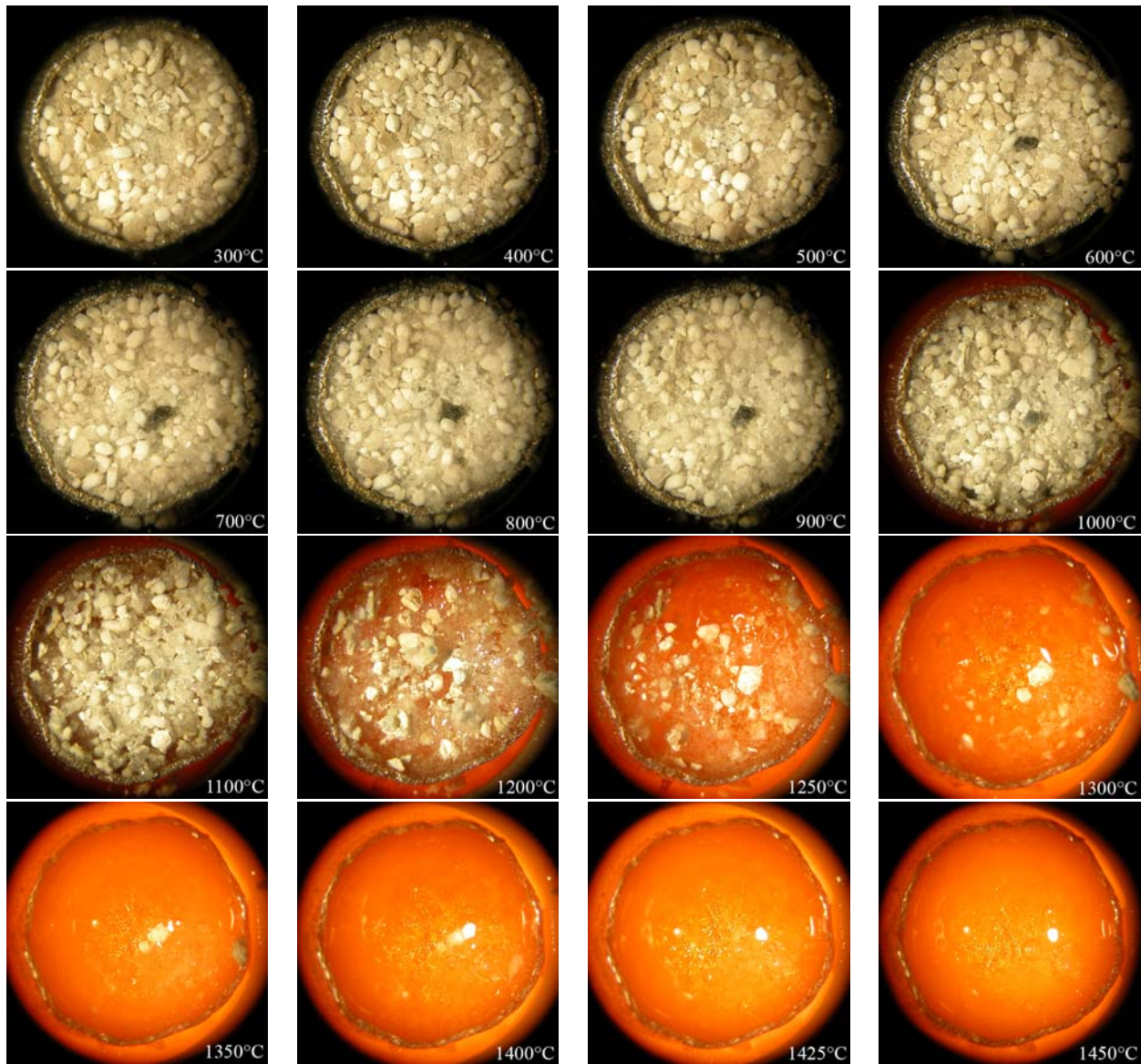
4.13

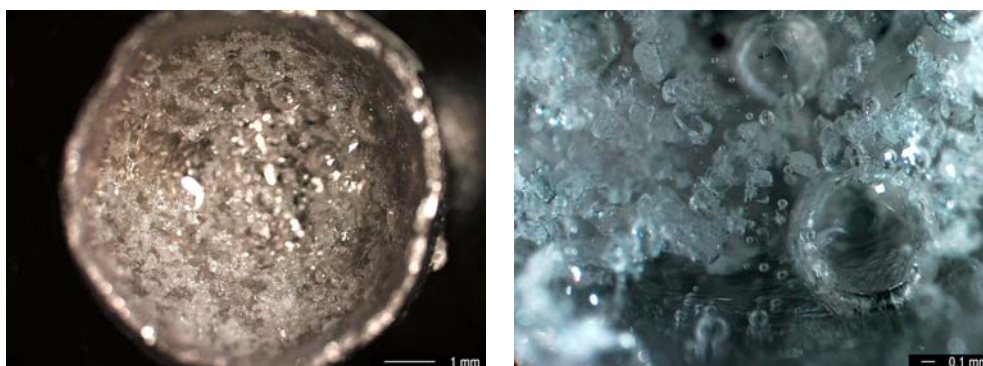**Figure 4.3.4. Melting of Reduced Batch Containing 0.118 Mass% of Carbocite**

Figure 4.3.5. Bubbles and Undissolved Silica Grains in Reduced Batch Containing 0.118 Mass% of Carbocite

4.4   EGA

Figure 4.4.1 shows that CO_2 was the only gas evolved from 25-g oxidized batch with rouge. Before the oxidized batch with rouge second run, the mass spectrometer was adjusted to detect SO_2 only, the 100-g batch was premelted at 1000°C to get rid off most of carbonates, and the precaution was made to prevent SO_2 absorption on any tiny drop of humidity and an SO_2 reaction with stainless steel transfer lines. The stainless steel lines were replaced by Teflon transfer lines heated to 120°C.

In spite of these precautions, we were unable to determine the salt cake decomposition temperature. The SO_2 was not detected, not so much because of insufficient sensitivity of the instrument, but most likely because of SO_2 retention in glass.
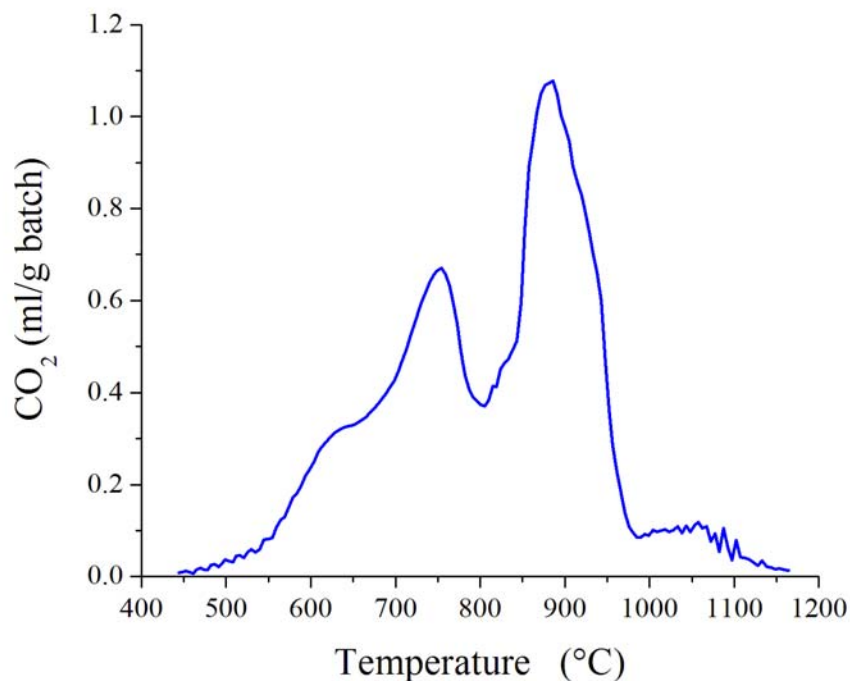

Figure 4.4.1. CO_2 Evolved from 25-g Oxidized Batch with Rouge During Melting

4.5 XRF

Figure 4.5.1 shows sulfur concentration in glasses as a function of temperature for reduced batch containing 0.12 mass% of carbocite and for reduced batch containing 0.089 mass% of graphite. Reduced glass doped with graphite has a higher Fe(II)/Fe(total) ratio and a lower sulfur retention compare to reduced glass doped with carbocite.
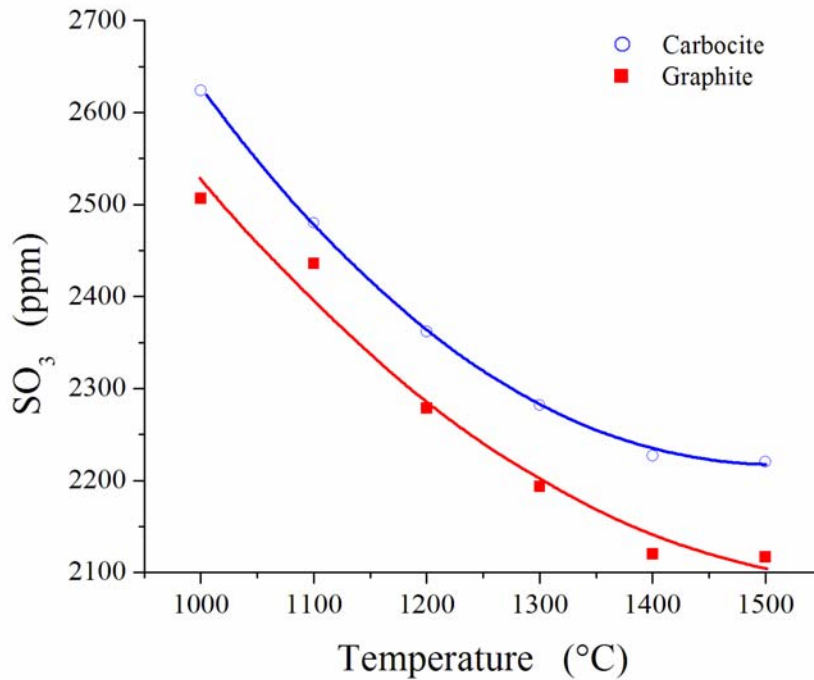

Figure 4.5.1. Changes in SO₃ Concentration During Melting of Reduced Batches Containing 0.118 Mass% of Carbocite or 0.089 Mass% of Graphite

4.6 XRD

Figure 4.6.1 shows undissolved sand (quartz plus cristobalite) as a function of temperature in reduced batch with 0.118 mass% of carbocite and reduced batch with 0.089 mass % of graphite. Figure 4.6.2 and Figure 4.6.3 shows XRD quantitative analysis of crystalline phases identified in reduced batch with 0.118 mass% of carbocite and reduced batch with 0.089 mass % of graphite.
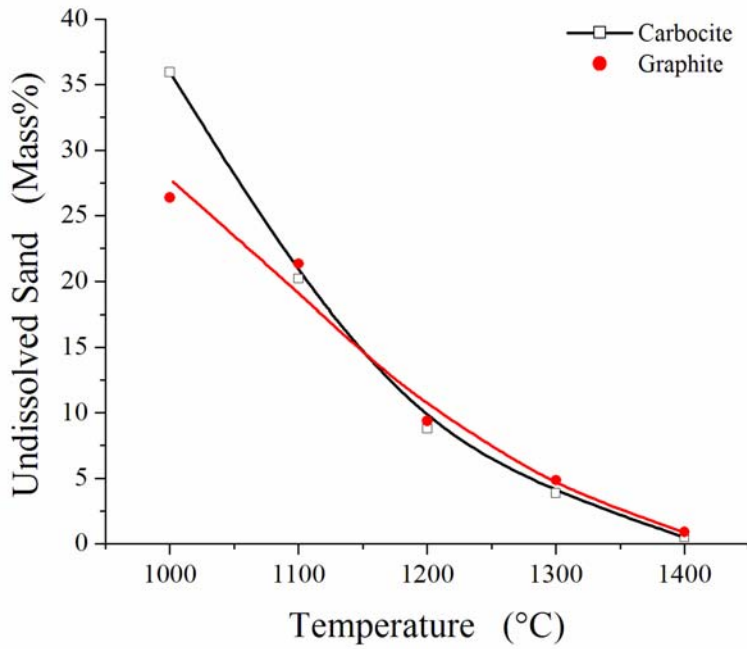
Figure 4.6.1. XRD Quantitative Analysis of Reduced Batch with 0.118 Mass% of Carbocite and Reduced Batch with 0.089 Mass % of Graphite
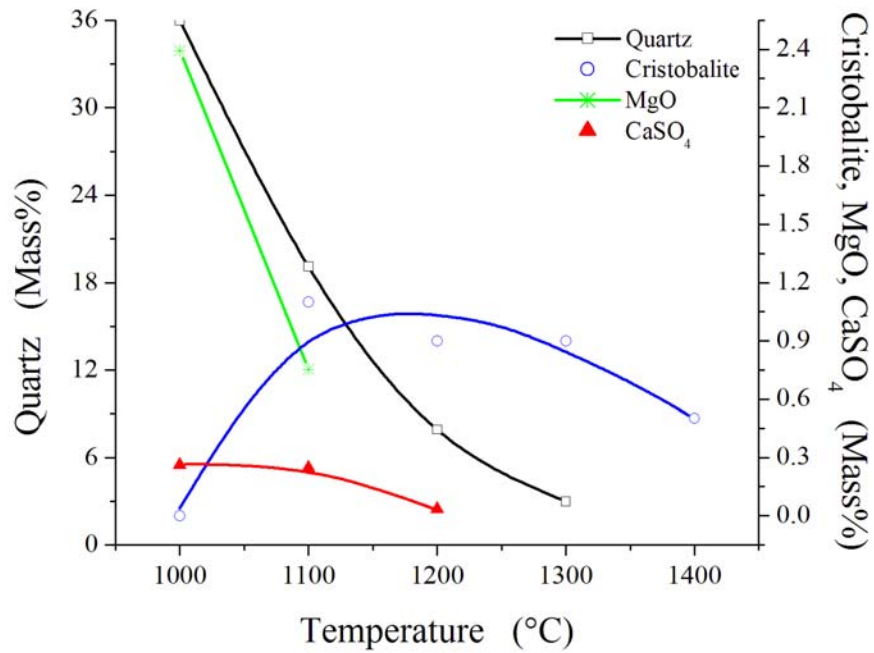

Figure 4.6.2. XRD Quantitative Analysis of Reduced Batch with 0.118 Mass% of Carbocite Quenched at Different Temperatures
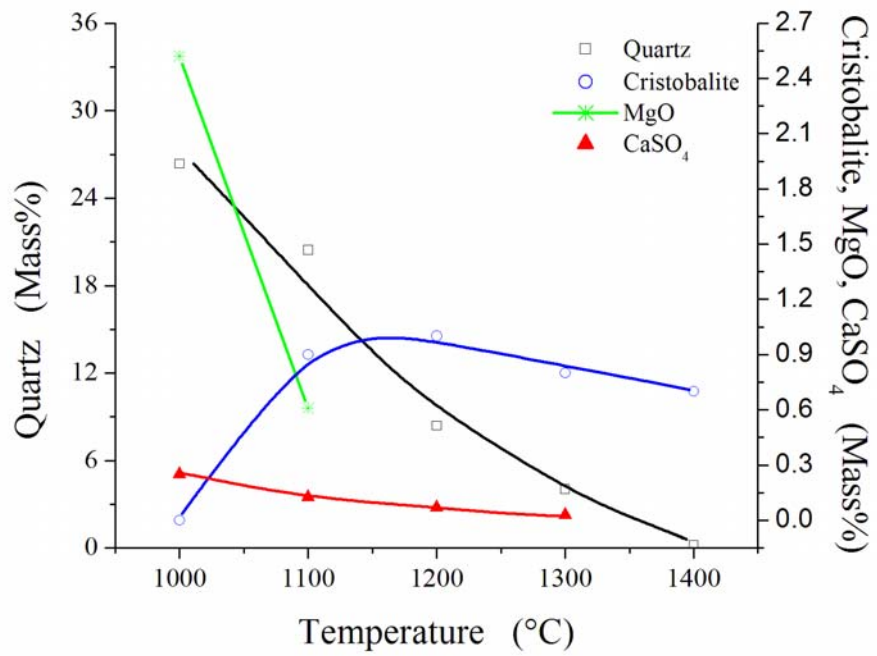
4.17

Figure 4.6.3. XRD Quantitative Analysis of Reduced Batch with 0.089 Mass% of Graphite Quenched at Different Temperatures

5.0   Discussion

The addition of elementary carbon to the glass batch reduces the partial pressure of oxygen in the glass melt. The temperature of decomposition of sulfate, and thus the vigorous generation of bubbles, decreases as the partial pressure of oxygen in the melt decreases. Bubbles nucleate on the residues of silica grains, causing their rapid dissolution. Consequently, residual silica grains dissolve at a lower temperature when carbon is added.

As Figure 5.1. and Figure 5.2. show, silica does not fully dissolve even at 1480°C in a batch that does not contain sulfate. Moreover, in the absence of sulfate, undissolved silica tends to accumulate on the top surface creating silica scum. Addition of sulfate helps disperse the silica scum and accelerates the dissolution of silica grains residues. The temperature at which sulfate decomposes is decreased by adding carbon, but not all carbon added to the batch is effective. A part of the carbon burns before the glass-forming melt gets connected. In the melt, the carbon reacts with Fe_2O_3, increasing the redox ratio, hence decreasing sulfate decomposition temperature. As a result, residual silica disappears from the melt at a lower temperature.

Oxidized batch without salt cake   Oxidized batch with rouge   Reduced batch with rouge and carbocite

Figure 5.1. Oxidized Batches and Reduced Batch with Rouge and Carbocite Melted in Silica Crucibles at 1480°C

The effect of carbon addition is shown in Figure 5.2.: the fraction of silica undissolved at 1450°C decreases in the order zero carbon addition—0.118 mass% carbocite addition—0.118 mass% graphite addition. Figure 5.1. indicates that sulfate decomposes around 1480°C when carbocite is added. This temperature is lowered by 20–30°C when graphite is used instead of carbocite. The different impact of identical addition of carbon in the form of carbocite and graphite can be rationalized by assuming that a larger portion of carbocite is oxidized during early stages of melting. As Figure 4.1.4 shows, an increasing fraction of carbon (graphite) decreases the amount of silica present in the melt at a given temperature. Excessive graphite (>0.2 mass%) remains in the melt at high temperatures, causing brown streaks.
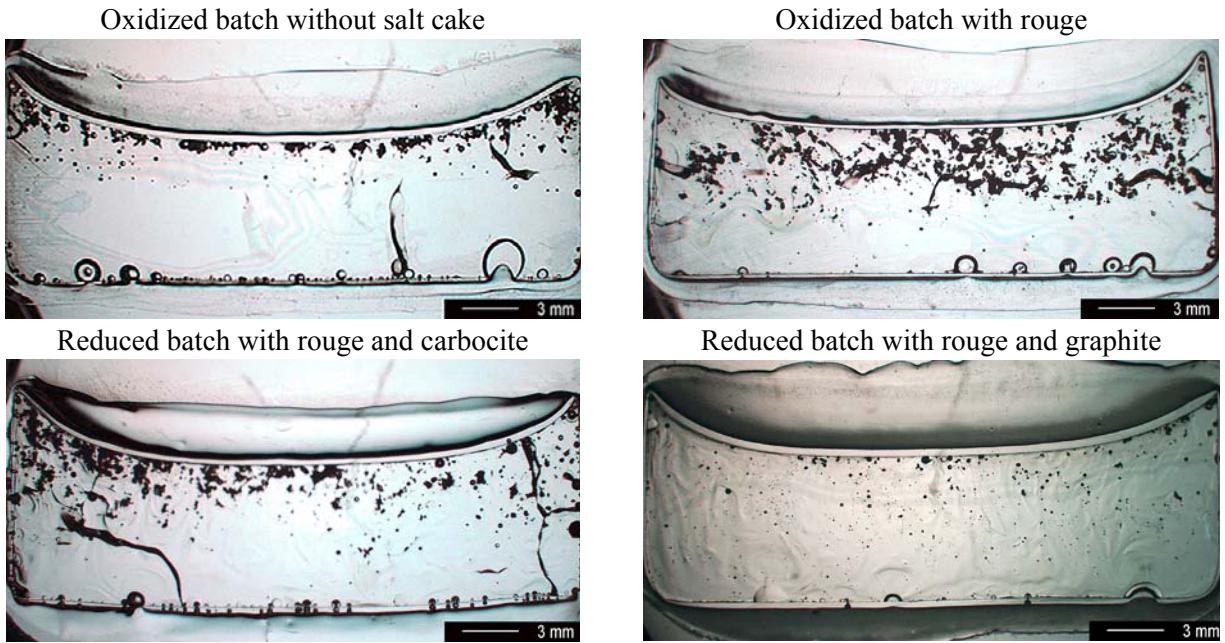
Oxidized batch without salt cake

Oxidized batch with rouge

Reduced batch with rouge and carbocite

Reduced batch with rouge and graphite

Figure 5.2. Oxidized and Reduced Batches Melted in Pt-crucibles at 1450°C

6.0   Conclusions and Recommendations

The results of the present study helped Visteon Inc. reduce the glass production energy consumption and established the batch containing 0.118 mass% of graphite as the best candidate for Visteon glass production. The improved glass batch has a lower potential for silica scum formation and for brown fault occurrence in the final glass product. It was established that bubbles trapped in the melt even at 1450°C have a high probability to be refined when reaching the hot zone in the glass furnace. Furthermore, silica sand does not accumulate at the glass surface and dissolves faster in the batch with graphite than in the batch with carbocite.

Appendix A

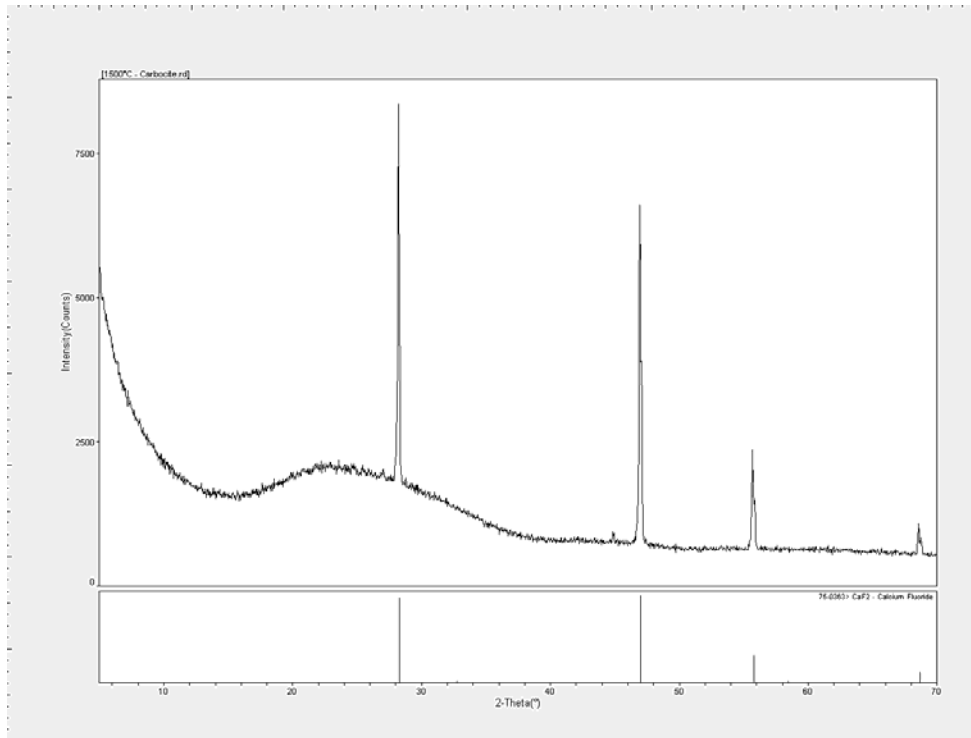# XRD Patterns

Appendix A: XRD Patterns

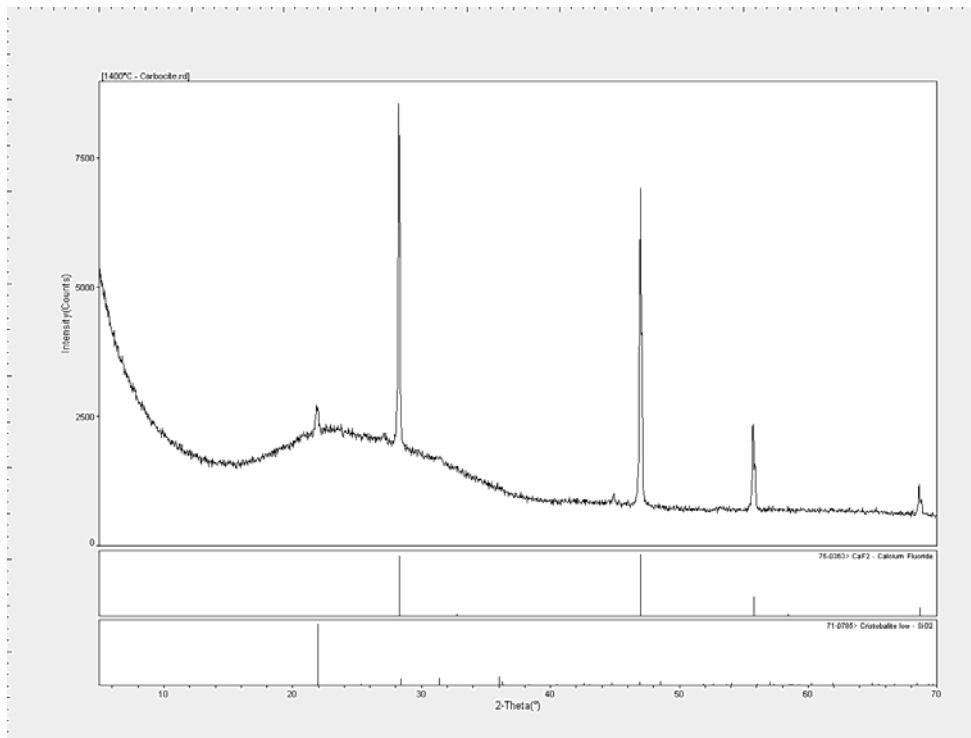**Figure A.1. XRD Pattern of Reduced Glass with 0.118 Mass% of Carbocite Quenched at 1500°C**

Figure A.2. XRD Pattern of Reduced Glass with 0.118 Mass% of Carbocite Quenched at 1400°C
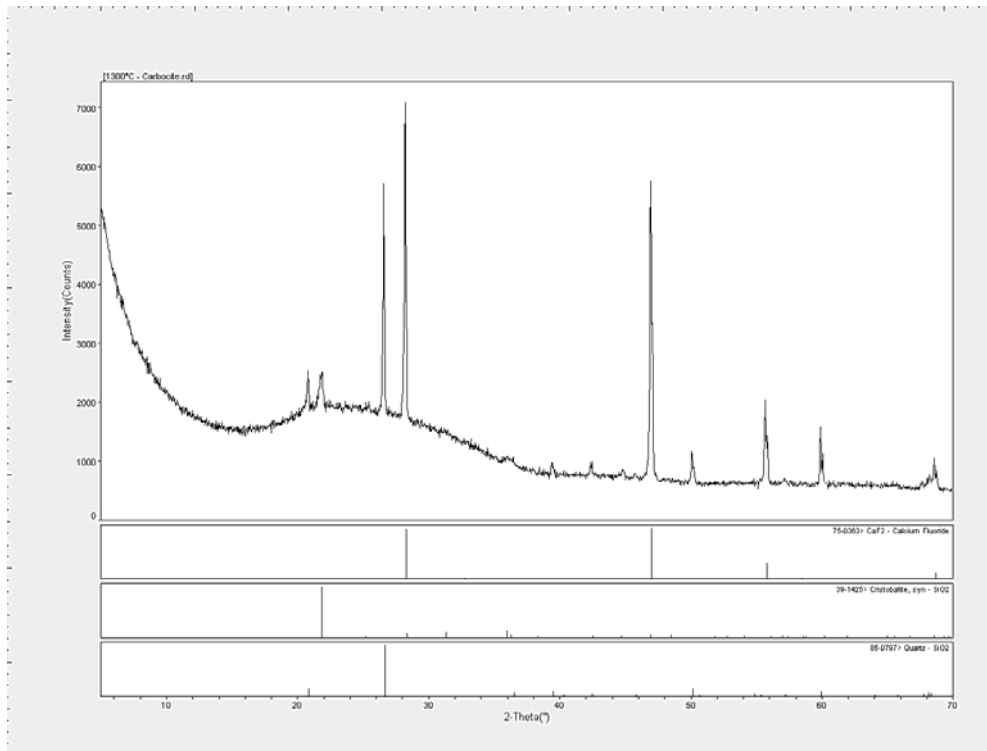
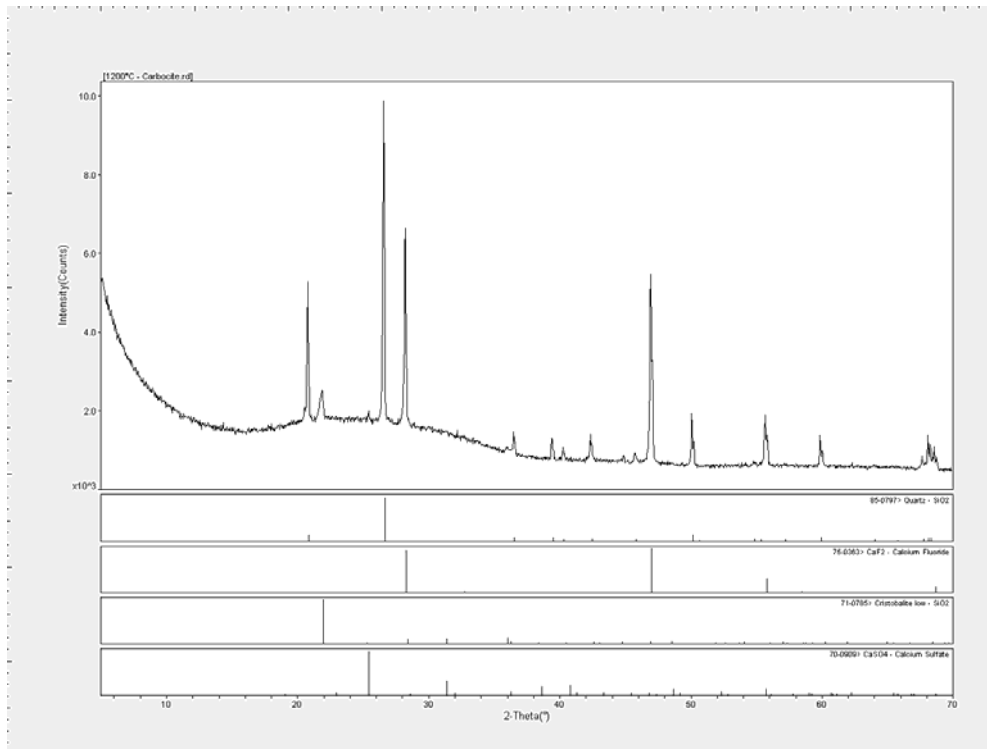**Figure A.3. XRD Pattern of Reduced Glass with 0.118 Mass% of Carbocite Quenched at 1300°C**

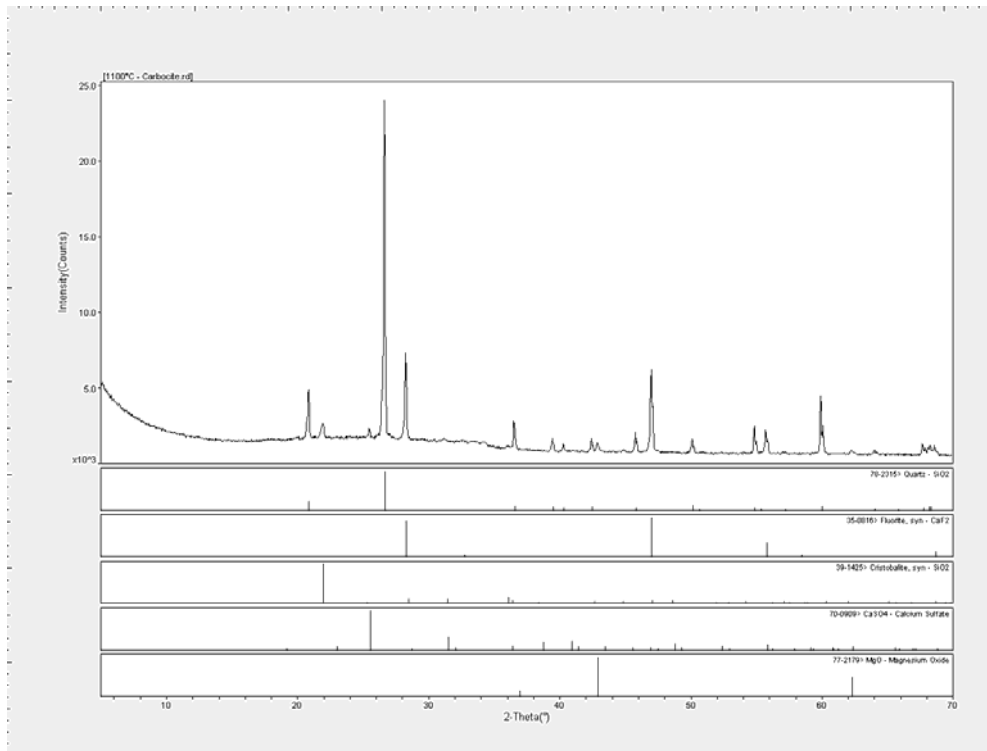**Figure A.4. XRD Pattern of Reduced Glass with 0.118 Mass% of Carbocite Quenched at 1200°C**

Figure A.5. XRD Pattern of Reduced Glass with 0.118 Mass% of Carbocite Quenched at 1100°C
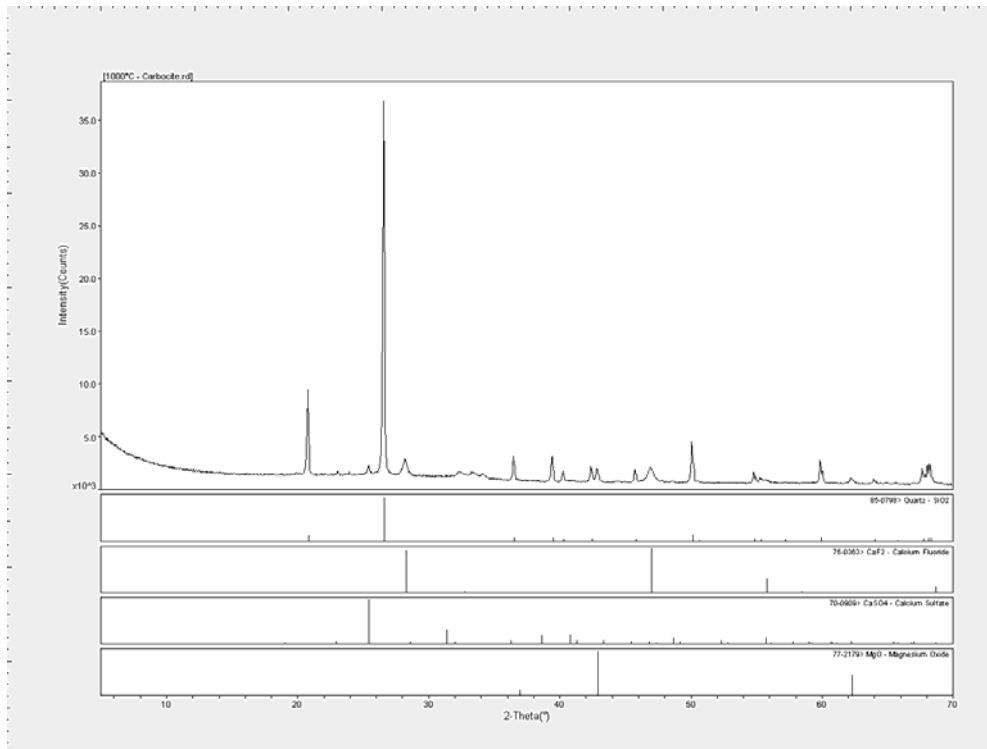

Figure A.6. XRD Pattern of Reduced Glass with 0.118 Mass% of Carbocite Quenched at 1000°C
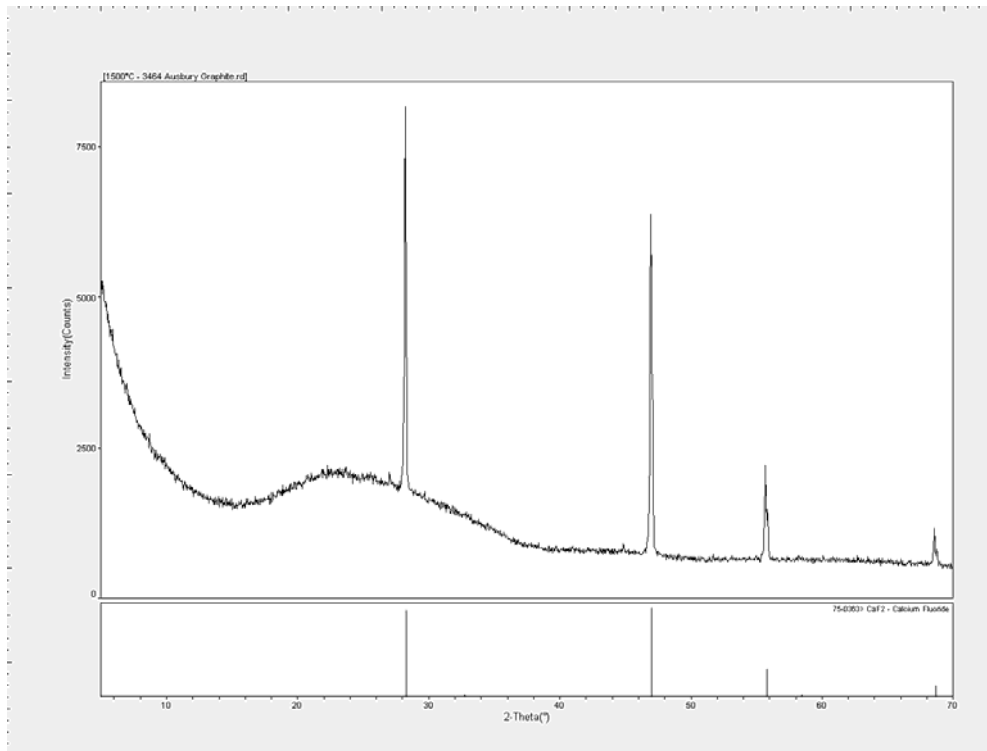
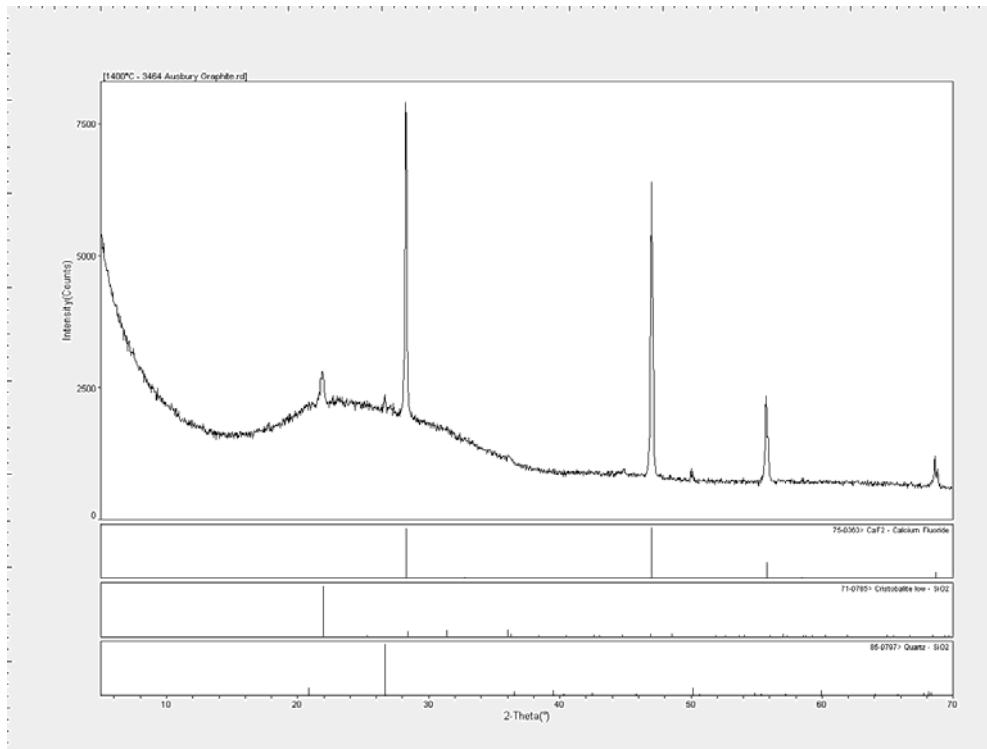**Figure A.7. XRD Pattern of Reduced Glass with 0.089 Mass% of Graphite Quenched at 1500°C**

Figure A.8. XRD Pattern of Reduced Glass with 0.089 Mass% of Graphite Quenched at 1400°C

Figure A.9. XRD Pattern of Reduced Glass with 0.089 Mass% of Graphite Quenched at 1300°C

Figure A.10. XRD Pattern of Reduced Glass with 0.089 Mass% of Graphite Quenched at 1200°C

Figure A.11. XRD Pattern of Reduced Glass with 0.089 Mass% of Graphite Quenched at 1100°C

Figure A.12. XRD Pattern of Reduced Glass with 0.089 Mass% of Graphite Quenched at 1000°C
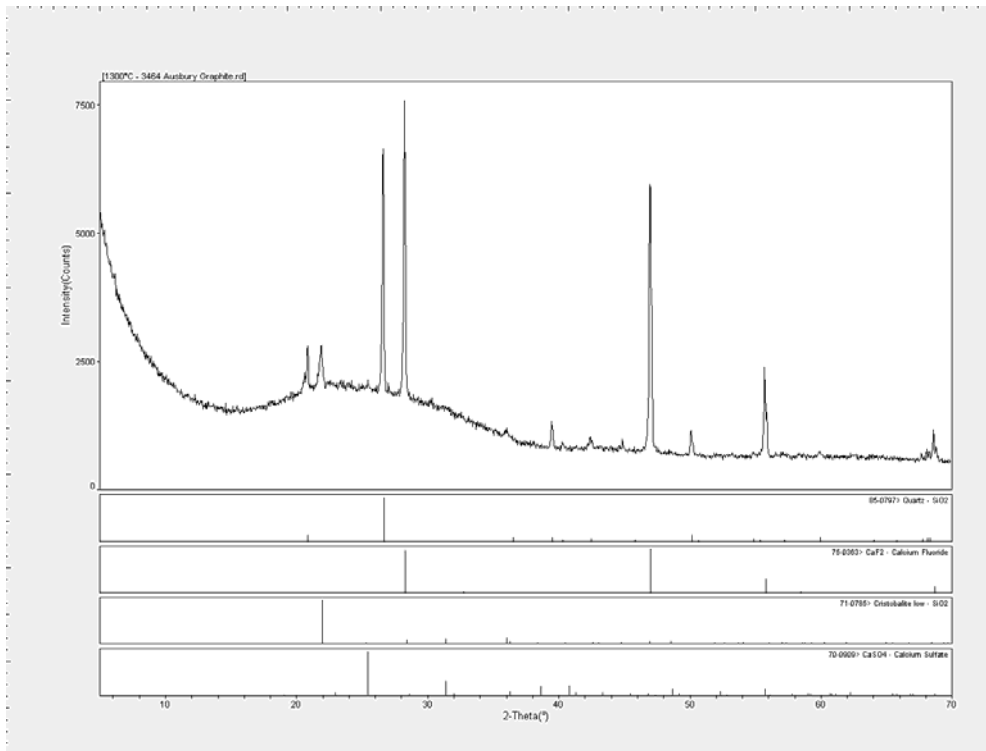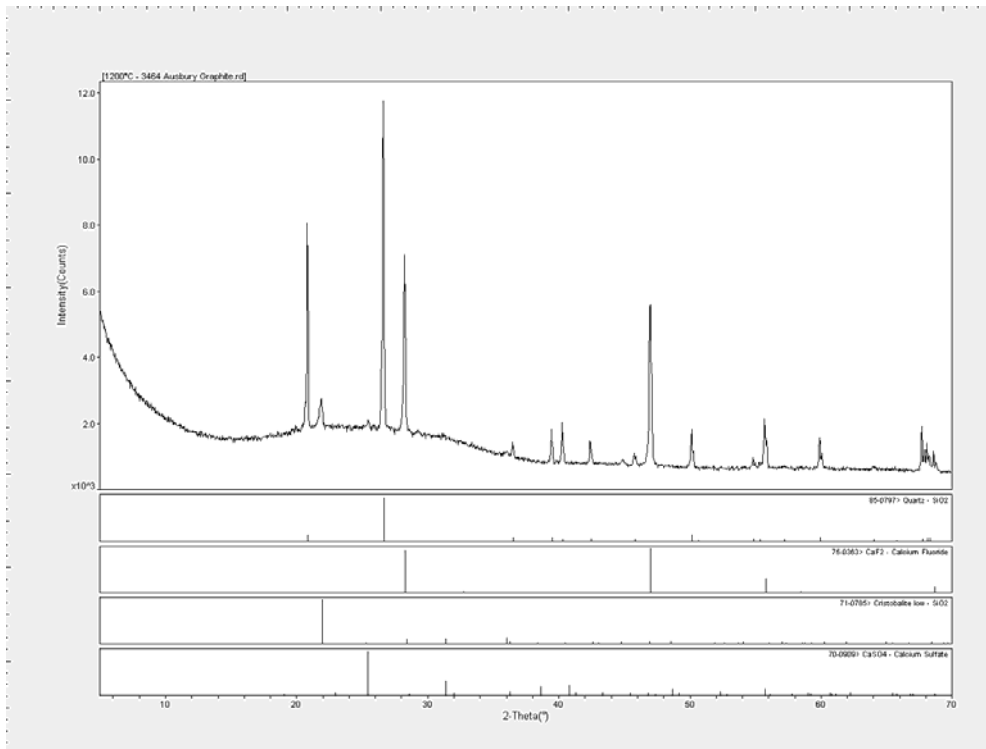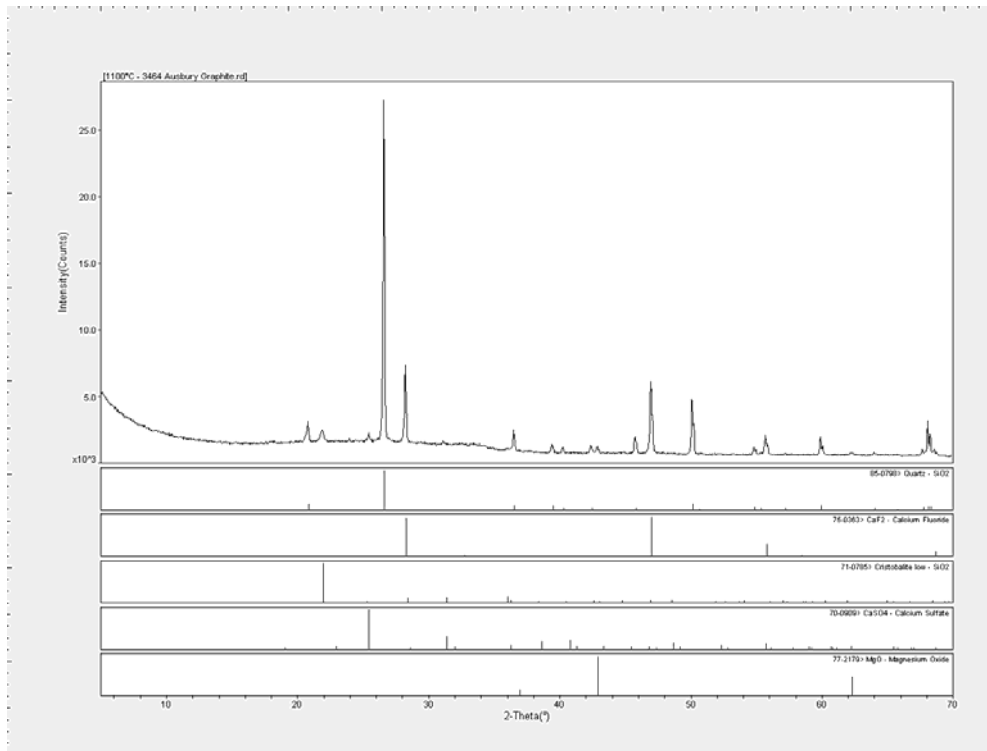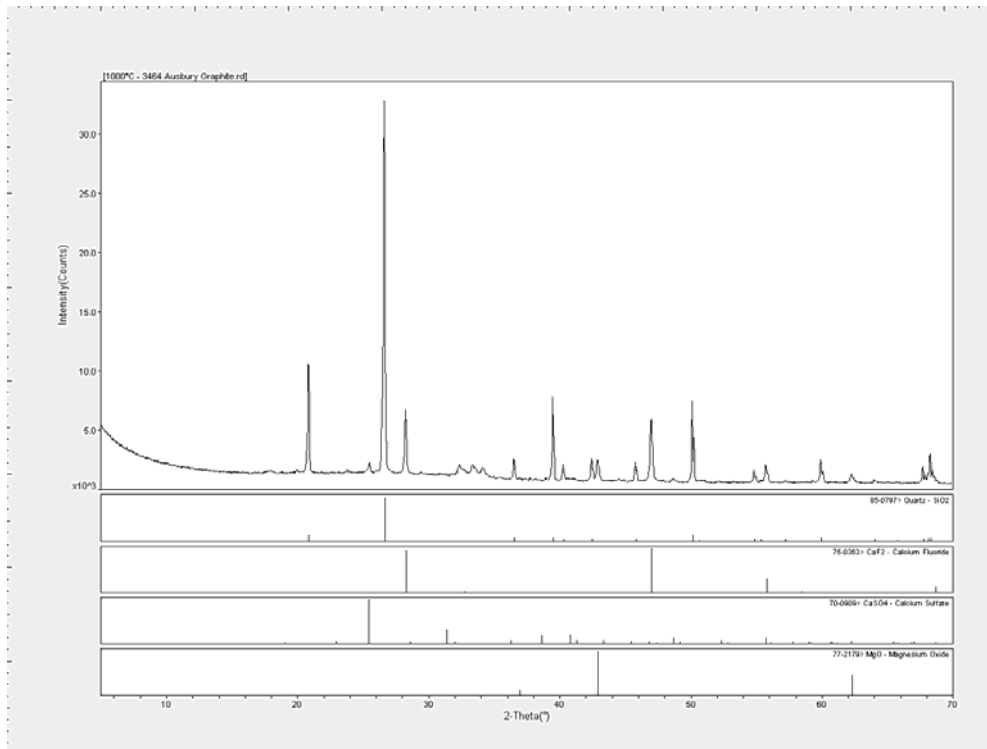
Appendix B

XRF Raw Data

Appendix B: XRF Raw Data

Table B.1. XRF Analysis Data for Reduced Batch Containing 0.118 Mass% of Carbocite

Temperature (°C)	SO3	Na2O	MgO	Al2O3	SiO2	K2O	CaO	TiO2	MnO	Fe2O3
	(mass %)									
1000	0.262	14.975	4.550	0.340	69.835	0.058	9.560	0.016	0.121	0.291
1100	0.248	15.015	4.215	0.295	70.295	0.054	9.455	0.014	0.124	0.277
1200	0.236	15.040	3.865	0.235	70.870	0.047	9.290	0.012	0.127	0.266
1300	0.228	15.100	3.790	0.230	70.945	0.047	9.245	0.011	0.128	0.269
1400	0.223	15.205	3.730	0.225	70.930	0.045	9.230	0.010	0.130	0.263
1500	0.222	15.180	3.735	0.220	70.965	0.044	9.230	0.010	0.129	0.260

Table B.2. XRF Analysis Data for Reduced Batch Containing 0.089 Mass% of Graphite

Temperature (°C)	SO3	Na2O	MgO	Al2O3	SiO2	K2O	CaO	TiO2	MnO	Fe2O3
	mass %									
1000	0.251	15.165	4.475	0.220	69.825	0.044	9.500	0.011	0.121	0.247
1100	0.244	15.115	4.175	0.215	70.280	0.043	9.390	0.011	0.124	0.257
1200	0.228	14.965	3.865	0.215	70.925	0.043	9.345	0.011	0.127	0.264
1300	0.220	15.065	3.800	0.215	70.950	0.043	9.290	0.010	0.128	0.263
1400	0.212	15.200	3.725	0.210	70.965	0.044	9.230	0.009	0.129	0.263
1500	0.212	15.155	3.705	0.205	71.010	0.043	9.245	0.010	0.129	0.273