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Analysis of Natural Graphite, Synthetic Graphite, and Thermosetting Resin Candidates for Use in Fuel Compact Matrix

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Executive Summary

This report is a revision to a previous report issued in 2009 by Peter J. Pappano entitled "Report on Analysis of Synthetic Graphite and Thermosetting Resin Candidates for Use in Fuel Compact Matrix," ORNL/TM-2009/315. Revisions to the previous report are documented in Appendix 2. This revised report addresses Statement of Work 4516 Rev 12, Section 1.2.7.1, which included a task to evaluate natural flake graphites from at least two suppliers. In satisfying this task, seven natural graphites were analyzed, two from Timcal, one from Superior Graphite Co., three from Asbury Graphite Mills, and one from GrafTech International. These natural graphites were analyzed for impurities by GDMS, surface area by BET, and tap density for Carr compressibility index and powder flowability. This information is included in the corresponding analysis sections of this report.

Statement of Work 4516 Rev 9, Section 1.2.7 “Support of compact process scale up and development” included a task to evaluate resin and graphite purity. Specifically, the task called for the analysis of up to five resins and at least six synthetic graphites. Following analysis, a recommendation for a suitable synthetic graphite was requested. This report summarizes the analyses that were performed on thermosetting resins and synthetic graphites and provides a recommendation for suitable candidates that could potentially be used in a scaled up compacting process. Six resins were analyzed, three from Plenco, one from Georgia Pacific, and two from Hexion. The resins were charred to 950°C in flowing helium and then analyzed for impurities by glow discharge mass spectrometry (GDMS). Three selected resins were also subjected to rheological testing in order to determine flow and curing characteristics. Twelve synthetic graphites were analyzed, four from Asbury Graphite Mills, four from Timcal, and four from Graftech International. These graphites were also analyzed by GDMS for impurities, surface area by BET, morphology by scanning electron microscopy, and tap density for Carr compressibility index and powder flowability.

It was found that Hexion resins AD-5614 and SD-1708 may be suitable replacements for Hexion SC-1008, in terms of similar levels of impurities. However, AD-5614, as well as all of the other resins tested here, contained high levels of elements that are not called out in the AGR-2 compact impurities specification.

A synthetic graphite most likely to replace KRB2000 was a specially developed material from Graftech International called GTI-D (unofficially named because, at this point, it is an experimental graphite designed specifically for this project). Of the twelve (12) graphites analyzed, only GTI-D was similar to KRB2000 in low levels of impurities. Also, the BET surface area and tap density testing suggest KRB2000 is denser than any of the graphites tested here. GTI-D was closest in terms of tap density, but still not as dense as KRB2000.

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1.0 Introduction

The AGR-1 and AGR-2 compacting process involved overcoating TRISO particles and compacting them in a steel die. The overcoating step is the process of applying matrix to the OPyC layer of TRISO particles in a rotating drum in order to build up an overcoat layer of desired thickness. The matrix used in overcoating is a mixture of natural graphite, synthetic graphite, and thermosetting resin in the ratio, by weight, of 64:16:20. A wet mixing process was used for AGR-1 and AGR-2, in that the graphites and resin were mixed in the presence of ethyl alcohol. The goal of the wet mixing process was to “resinate” the graphite particles, or coat each individual graphite particle with a thin layer of resin. This matrix production process was similar to the German, Chinese, Japanese, and South African methods, which also use various amount of solvent during mixing. See Appendix 1 for information on these countries matrix production techniques.

The resin used for AGR-1 and AGR-2 was provided by Hexion, specifically Hexion grade Durite SC1008. Durite SC1008 is a solvated (liquid) resole phenolic resin. A resole resin does not typically have a hardening agent added. The major constituent of SC1008 is phenol, with minor amounts of formaldehyde. Durite SC1008 is high viscosity, so additional ethyl alcohol was added during matrix production in order to reduce its viscosity and enhance graphite particle resination.

The current compacting scale up plan departs from a wet mixing process. The matrix production method specified in the scale up plan is a co-grinding jet mill process where powdered phenolic resin and graphite are all fed into a jet mill at the same time. Because of the change in matrix production style, SC1008 cannot be used in the jet milling process because it is a liquid. The jet milling/mixing process requires that a suite of solid or powdered resins be investigated.

The synthetic graphite used in AGR-1 and AGR-2 was provided by SGL Carbon, grade KRB2000. KRB2000 is a graphitized petroleum coke. The availability of KRB2000 is perhaps in question, so a replacement synthetic graphite may need to be identified. This report presents data on potential replacements for KRB2000.

2.0 Experimental

The following techniques were used to characterize the resins and graphites: glow discharge mass spectrometry (GDMS) for impurities, BET for surface area, scanning electron microscopy, and tap density. The resins were characterized only by GDMS while each technique was performed on the graphites.

2.1 Glow Discharge Mass Spectrometry

GDMS was performed by Evans Analytical Group in Syracuse NY (formerly Shiva Technologies). GDMS involves using the sample, in this case graphite or charred resin in powder form, as a cathode. The cathode is placed in a low pressure environment with argon discharge gas. The argon gas is excited to a plasma, and positive ions are directed at the surface of the cathode (i.e. sample). The impaction of the positive argon ions on the sample's surface causes sputtering of surface atoms. Once in the argon plasma, the sputtered sample atoms are ionized. The excited sample atoms lose energy via atomic emission, which is evidenced by the generation of specific wavelength light. The atom from the sample is identified by the wavelength of light emitted during atomic emission. The concentration of the atoms in the sample is measured by the intensity of the light emitted.

2.2 Rheology

The resins were subjected to rheology testing using a Rheometric Scientific ARES-M unit. The powdered resins were first pressed into approximately 3 mm thick discs using a Carver hydraulic press. The discs were then mounted on the bottom circular plate of the ARES-M unit. The run conditions were then entered. This unit has both isotherm and temperature ramp capability; isotherm data was collected for this report. The unit measures the viscosity of the material as a function of temperature by rotating the resin disk against a fixed plate. The torque needed to turn the bottom plate (the top plate is fixed) is translated into a viscosity, and this is reported over a given time period in the isotherm mode.

2.3 BET surface area

The ASTM method D 6556-04 covers the standard procedure for measuring total and external surface area of carbon black by nitrogen adsorption. Another ASTM method (D 4780-95 covers determination of surface area using krypton adsorption and is applicable to materials with low surface area (between 0.05 and ~10 m^2/g). Both procedures have been used. Measurements using the N_2 adsorption method were made at Quantachrome Instruments, and measurements using Kr adsorption were made at ORNL. Both methods provide determination of two properties, the total surface area and the external surface area. The total surface area, sometimes referred to as nitrogen surface area (NSA), is calculated from multipoint adsorption data based on

BET theory and includes all internal and external surfaces (including the internal surface of micropores, which are pores with widths less than 2 nm). The external surface area, also known as statistical thickness surface area, is calculated based on the statistical thickness method (also known as “t-method”) and is defined as the specific surface area minus the micropore internal surface.

A commercial gas adsorption apparatus, Autosorb-1C from Quantachrome Instruments, was used for characterization of BET surface area by gas adsorption (N_2 or Kr). This is a high sensitivity instrument for measuring N_2 or Kr adsorption isotherms over a pressure range from 10^{-5} Pa to 0.1 MPa and liquid nitrogen temperature. The instrument is used for surface area and porosity distribution characterization of porous or powdered materials, including several modifications of carbon. As a part of standard laboratory practice, the correct operation of the instrument is checked monthly by measuring the surface area of a silica-alumina standard (32.16 ± 0.31 m^2/g) supplied by Quantachrome. The operation is considered correct unless the test value differs from the expected result by more than the 95% reproducibility limits (± 2.03 m^2/g for multi-point BET).

The sample preparation procedure recommended by ASTM D 6556-04 and D 4780-95 was followed. Before measurements, graphite samples (usually 0.3 – 0.7 grams) were outgased in situ at 300 ^oC for at least 3-5 hours. The multipoint BET method was used, based on collection of 7 – 11 data points covering the range of reduced pressures $P/P_o < 0.3$, where P_o is the saturation vapor pressure for the adsorbed gas at the liquid nitrogen temperature (77.3 K). The results were calculated using the commercial software developed by Quantachrome Instruments and distributed with the instrument

2.4 Scanning Electron Microscopy

SEM was performed on a JEOL 6500 unit. The graphites were mounted on two sided conducting tape and inserted into the unit’s sample port. A vacuum was pulled in the sample port and SEM investigation was initiated. Magnifications of 1000x to 20,000x were used to visualize the shape and overall morphology of the graphite particles.

2.5 Tap density

Tap density was performed on a Quantachrome Dual Tapper unit. A quantity of graphite powder was poured into the graduated cylinder and an initial volume was recorded. The cylinder was then tapped 2000 times and a final volume was recorded. The tap density was found by dividing the mass of the powder by its final volume. The difference in initial volume (V_i) and final volume (V_f) was used to calculate Carr’s compressibility index (C), which was found by using equation:

$$C = 100 \times ((V_i-V_f)/V_i)$$

3.0 Results and Discussion

Table 1 lists the resins and graphites that were analyzed for this report. Plenco 11956 and Georgia Pacific 445D05 are solvated resole resins, but were included because of previous research experience in related projects on-going at ORNL. Plenco 12114 and 14043 are both powders, but 12114 is a resole resin and 14043 is a novolac resin. Hexion AD 5614 is a powdered novolac resin and SD 1708 is a high purity version of AD 5614.

Graftech produced four specialty synthetic graphites for this research, hence the labels A, B, C, and D. There are currently no commercial grade or ID numbers for these graphites and they cannot be purchased off the shelf. Asbury Graphite Mills (AGM) supplied four graphite samples with the following identification and source material: A99 (conventional electrographite powder), 4421 (mag purified version of A99), TC303 (graphitized needle coke), and 7105 (a purified graphitized isotropic coke). Timcal provided four graphites with the following identification: KS15, KS44, T44, and SFG44.

Table 1: List of resins and graphites analyzed in this report

Material	Manufacturer and ID
Resin	Plenco 12114
Resin	Plenco 11956
Resin	Plenco 14043
Resin	Georgia Pacific 445D05
Resin	Hexion AD-5614
Resin	Hexion SD-1708
Synthetic Graphite	Graftech-A
Synthetic Graphite	Graftech-B
Synthetic Graphite	Graftech-C
Synthetic Graphite	Graftech-D
Synthetic Graphite	AGM-A99
Synthetic Graphite	AGM-4421
Synthetic Graphite	AGM-TC301
Synthetic Graphite	AGM-7105
Synthetic Graphite	Timcal-KS15
Synthetic Graphite	Timcal-KS44
Synthetic Graphite	Timcal-T44
Synthetic Graphite	Timcal-SFG44
Natural Graphite	Timcal-PG06
Natural Graphite	Timcal-Micro 890
Natural Graphite	Superior Graphite-Thermopure
Natural Graphite	Asbury Graphite Mills-3482
Natural Graphite	Asbury Graphite Mills-RD13371
Natural Graphite	Asbury Graphite Mills-RD13382
Natural Graphite	Graftech-30 micron

3.1 GDMS results

GDMS was performed on all of the samples, both resins and graphites, in order to determine the level of impurities. Tables 2 through 8 provide the full scan GDMS results for resins; Table 8 shows data for Hexion SC1008, which was used in making AGR-1 and AGR-2 compacts, as reference. The elements that were called out in the AGR-2 compact specification are shown in bold in these tables.

Figure 1 shows the levels of AGR-2 specified elements in each of the resins tested. The sum, in ppm, of the specified elements is listed at the top of the columns for each resin. The Plenco resin, 14043, had the highest total impurities, at 571.47 ppm. The bulk of this value is the result of a high calcium concentration, at 470 ppm. The other two Plenco resins, 12114 and 11956, were similar in concentration of AGR-2 specified impurities; they were 70.55 and 72.72 ppm, respectively. The 11956 resin had a low iron value, under 2 ppm, which would make it an attractive candidate for matrix production. Iron perhaps has the greatest potential for detrimental effects on a TRISO particle because of its ability to migrate through graphite and attack the SiC layer of the particle. The 12114 resin has an iron concentration of 19, which is close to the desired upper limit for a matrix constituent. From AGR-1 and AGR-2 experience, an initial iron value of less than 20 ppm was targeted; the SC1008 resin's iron value was 1.1 ppm.

Although not called out in the AGR-2 compact impurity level specification, resin 12114 has high concentrations of fluorine (~1000 ppm), sodium (43 ppm), magnesium (33 ppm), and silicon (42 ppm). Resin 11956 also has some high concentrations of unspecified elements, namely sodium (20 ppm), silicon (80 ppm), and chlorine (200 ppm). The impact of these elements on TRISO particle and compact integrity during irradiation will need to be investigated if these resins are selected.

The total impurities concentration for the Hexion resins, AD5614 and SC1008, was 20.19 and 11.44 ppm, respectively. The AD5614 resin had an iron concentration of 2.3 ppm, which would make it a better candidate for a matrix that could be used to overcoat particles and form a compact whose final impurities were within specification. Titanium and vanadium have sometimes been near the upper limit of the compact specification, so Ti and V values of 2 and 0.02 ppm are a positive.

Again, however, the AD5614 resin has some high concentrations of elements that are not called out in the compact specification. These elements are fluorine (270 ppm), silicon (29 ppm), and tungsten (45 ppm); a sodium concentration of 16 ppm may also be higher than desired.

The Hexion SC1008 resin also has unspecified elements present in high concentrations. Some of these elements are sodium (25 ppm), silicon (60 ppm), and sulfur (40 ppm). This resin was used in making the AGR-1 compacts, which were recently removed from ATR having exhibited no apparent particle failures or unexpected gaseous releases. The PIE of the compacts will elucidate many properties of the compacts, including, perhaps, the effect of these elements on compact properties after irradiation.

The Hexion AD-5614 and SD-1708 resins, as well as Plenco 14043, were selected for char yield testing. The mass of the resin was recorded before and after carbonization to 950°C. The 5614 and 14043 resins had basically the same char yields, less than 60%. This would be slightly lower than that of SC1008. The SD-1708 resin had a char yield of 41%.

Based on the GDMS data to date, the Hexion AD5614 resin would be a suitable replacement for SC1008, with the caveat that some unspecified elements may be higher than acceptable (but not yet defined) limits. How a given resin performs in the matrix fabrication process must also be investigated, whether it is a wet mixing slurry type process, or a co-grinding jet mill technique.

The synthetic graphites were also tested for impurities by GDMS and the full scan results are shown in Tables 10 through 22; Table 22 shows the impurity data for KRB2000, which was used in making the AGR-1 and AGR-2 compacts, as a reference.

In terms of low levels of specified impurities, only the GTI-D graphite tested here compares to SGL graphite KRB2000. Figures 2 through 4 highlight the AGR-2 specified impurities as compared to KRB2000. The total concentration of elements for KRB2000 is 4.53 ppm. The closest graphite in terms of lowest elemental concentration is GTI-D with total impurities of 8.1 ppm (which is “low” and definitely acceptable for use in compacting, but still nearly double the impurities of KRB2000). Asbury Graphite Mills (AGM) grade 7105 was the next closest graphite in terms of low levels of impurities, with 63.30 ppm. However, 50 of the total 63.30 ppm concentration is attributed to chromium. The AGM 7105 iron value of 4 ppm is acceptable and should be able to produce a matrix that is within specification, but it is still nearly four times higher than KRB2000’s iron concentration of 1.4 ppm.

In terms of high concentrations of elements with no specified upper limit, AGM 7105 has magnesium (100 ppm), silicon (40 ppm), phosphorus (15 ppm), and chlorine (50 ppm). Conversely, KRB2000 has no elemental concentration greater than 9 ppm (sulfur).

At this point in time, based on impurities data by GDMS, there is only one potential replacement for KRB2000, which is GTI-D. This material had to be specially made for the AGR program. Should a need for multi-ton quantities arise, GTI-D could be re-made and given an official grade name.

Tables 23 through 29 provide impurity data by GDMS for the selected natural graphites listed in Table 1. Figure 5 highlights impurity levels (ppm) of elements specified for AGR-2 in each of the natural graphites. The Timcal natural graphites PG09 and Micro 890 are very high in overall impurities and would not be considered a good candidate for matrix production. In Figure 6, the Timcal graphites have been removed and the figure re-scaled to provide better resolution for the other graphites. Asbury Graphite

Mills RD 13371 was chosen for the AGR-1 and AGR-2 compacting campaigns based on several characteristics including low impurities. It is included here as a reference. Looking at the results, RD13371 has the lowest overall impurity levels at 36.55 (ppm) and remains the most likely candidate for fuel compacting. All impurities fall below the specified levels before heat treatment. Asbury Graphite Mills grade 13382 and 3482 have relatively low overall impurity levels, 57.6 and 79.3 (ppm) respectively, but are both high in aluminum (Al) and iron (Fe). Graftech GTI-30 Micron has an overall impurity level of 66.96 (ppm) but has a high concentration of titanium (Ti). Similarly, Thermopure at 64.67 (ppm) is high in titanium (Ti) and iron (Fe).

Table 2. GDMS results for Plenco 12114 carbonized at 950°C. Elements in bold are included in AGR-2 compact specification.

Element	[ppm wt]	Element	[ppm wt]
Li	0.76	Pd	< 0.1
Be	< 0.01	Ag	< 0.1
B	0.05	Cd	< 0.1
C	Matrix	In	Binder
N	-	Sn	< 0.5
O	-	Sb	< 0.5
F	~ 1000	Te	< 0.1
Na	43	I	< 20
Mg	33	Cs	< 0.1
Al	**0.48**	Ba	0.45
Si	42	La	=< 5
P	0.68	Ce	< 0.5
S	1.5	Pr	=< 0.15
Cl	2.9	Nd	< 0.05
K	0.44	Sm	< 0.05
Ca	**44**	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	**0.06**	Tb	< 0.05
V	**< 0.01**	Dy	< 0.05
Cr	**< 0.5**	Ho	< 0.05
Mn	**0.45**	Er	< 0.05
Fe	**19**	Tm	< 0.05
Co	**< 0.05**	Yb	< 0.05
Ni	**6**	Lu	< 0.05
Cu	8	Hf	< 0.05
Zn	< 0.1	Ta	< 5
Ga	< 0.1	W	1
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	0.12	Hg	< 0.5
Y	< 0.05	Tl	< 0.1
Zr	< 0.05	Pb	< 0.5
Nb	< 0.1	Bi	< 0.1
Mo	< 0.05	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		< 0.1

Table 3. GDMS results for Plenco 11956 carbonized at 950°C. Elements in bold are included in AGR-2 compact specification.

Element	[ppm wt]	Element	[ppm wt]
Li	0.1	Pd	< 0.1
Be	< 0.01	Ag	< 0.1
B	1.5	Cd	< 0.1
C	Matrix	In	Secondary Cathode
N	-	Sn	< 0.5
O	-	Sb	< 0.5
F	< 1	Te	< 0.1
Na	20	I	< 0.5
Mg	< 0.5	Cs	< 0.5
Al	**50**	Ba	5
Si	80	La	< 0.5
P	< 0.1	Ce	< 0.05
S	20	Pr	< 0.05
Cl	200	Nd	< 0.05
K	< 1	Sm	< 0.05
Ca	**20**	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	**0.85**	Tb	< 0.05
V	**0.03**	Dy	< 0.05
Cr	**< 0.5**	Ho	< 0.05
Mn	**0.09**	Er	< 0.05
Fe	**1.5**	Tm	< 0.05
Co	**< 0.05**	Yb	< 0.05
Ni	**0.25**	Lu	< 0.05
Cu	< 0.1	Hf	< 0.05
Zn	< 0.1	Ta	< 5
Ga	< 0.1	W	5
Ge	< 0.1	Re	< 0.05
As	1.5	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	0.2	Au	< 0.1
Sr	0.2	Hg	< 0.5
Y	4	Tl	< 0.1
Zr	40	Pb	< 0.5
Nb	< 0.1	Bi	< 0.1
Mo	< 0.05	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		

Table 4. GDMS results for Plenco 14043 carbonized at 950°C. Elements in bold are included in AGR-2 compact specification.

Element	[ppm wt]	Element	[ppm wt]
Li	0.42	Pd	< 0.1
Be	< 0.01	Ag	< 0.1
B	0.71	Cd	< 0.1
C	Matrix	In	Binder
N	-	Sn	< 0.5
O	-	Sb	< 0.5
F	~ 300	Te	< 0.1
Na	18	I	< 20
Mg	28	Cs	< 0.1
Al	**85**	Ba	0.95
Si	270	La	=< 2
P	1.9	Ce	< 0.5
S	370	Pr	< 0.05
Cl	11	Nd	< 0.05
K	3.1	Sm	< 0.05
Ca	**470**	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	**4.1**	Tb	< 0.05
V	**0.07**	Dy	< 0.05
Cr	**0.75**	Ho	< 0.05
Mn	**0.25**	Er	< 0.05
Fe	**11**	Tm	< 0.05
Co	**< 0.05**	Yb	< 0.05
Ni	**0.25**	Lu	< 0.05
Cu	1.3	Hf	< 0.05
Zn	< 0.1	Ta	< 5
Ga	< 0.1	W	< 0.05
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	0.29	Hg	< 0.5
Y	< 0.05	Tl	< 0.1
Zr	< 0.05	Pb	< 0.5
Nb	< 0.1	Bi	< 0.1
Mo	0.07	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		

Table 5. GDMS results for Georgia Pacific 445D05 carbonized at 950°C. Elements in bold are included in AGR-2 compact specification.

Element	[ppm wt]	Element	[ppm wt]
Li	0.15	Pd	< 0.1
Be	0.02	Ag	< 0.1
B	3	Cd	< 0.1
C	Matrix	In	Binder
N	-	Sn	< 0.5
O	-	Sb	< 0.5
F	< 5	Te	< 0.1
Na	150	I	< 0.5
Mg	20	Cs	< 0.5
Al	**100**	Ba	< 0.1
Si	150	La	< 0.5
P	1	Ce	< 0.05
S	50	Pr	< 0.05
Cl	10	Nd	< 0.05
K	1	Sm	< 0.05
Ca	**20**	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	**2**	Tb	< 0.05
V	**0.1**	Dy	< 0.05
Cr	**< 0.5**	Ho	< 0.05
Mn	**0.5**	Er	< 0.05
Fe	**20**	Tm	< 0.05
Co	**< 0.05**	Yb	< 0.05
Ni	**2.5**	Lu	< 0.05
Cu	< 0.5	Hf	< 0.05
Zn	< 0.5	Ta	< 5
Ga	< 0.1	W	3
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	0.1	Hg	< 0.5
Y	< 0.05	Tl	< 0.1
Zr	0.5	Pb	< 0.5
Nb	< 0.1	Bi	< 0.1
Mo	1	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		

Table 6. GDMS results for Hexion AD-5614 carbonized at 950°C. Elements in bold are included in AGR-2 compact specification.

Element	[ppm wt]	Element	[ppm wt]
Li	0.35	Pd	< 0.1
Be	< 0.01	Ag	< 0.1
B	0.09	Cd	< 0.1
C	Matrix	In	Binder
N	-	Sn	< 0.5
O	-	Sb	< 0.5
F	~ 270	Te	< 0.1
Na	16	I	< 20
Mg	2	Cs	< 0.1
Al	**0.32**	Ba	< 0.1
Si	29	La	< 0.5
P	0.73	Ce	< 0.5
S	6.5	Pr	< 0.05
Cl	1.5	Nd	< 0.05
K	< 0.1	Sm	< 0.05
Ca	**9.5**	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	**2**	Tb	< 0.05
V	**0.02**	Dy	< 0.05
Cr	**5**	Ho	< 0.05
Mn	**0.1**	Er	< 0.05
Fe	**2.3**	Tm	< 0.05
Co	**< 0.05**	Yb	< 0.05
Ni	**0.9**	Lu	< 0.05
Cu	0.2	Hf	< 0.05
Zn	< 0.1	Ta	< 5
Ga	< 0.1	W	45
Ge	< 0.1	Re	< 0.05
As	0.22	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	< 0.05	Hg	< 0.5
Y	< 0.05	Tl	< 0.1
Zr	< 0.05	Pb	< 0.5
Nb	< 0.1	Bi	< 0.1
Mo	0.35	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		

Table 7. GDMS results for Hexion AD-5614 carbonized at 950°C. Elements in bold are included in AGR-2 compact specification.

Element	[ppm wt]	Element	[ppm wt]
Li	0.13	Pd	< 0.1
Be	< 0.01	Ag	< 0.1
B	0.15	Cd	< 0.1
C	Matrix	In	Binder
N	-	Sn	< 0.5
O	-	Sb	< 0.5
F	~ 200	Te	< 0.1
Na	2.8	I	=< 100
Mg	< 0.5	Cs	< 0.1
Al	**0.12**	Ba	< 0.1
Si	34	La	< 0.5
P	0.31	Ce	< 0.5
S	17	Pr	< 0.05
Cl	7.7	Nd	< 0.05
K	0.29	Sm	< 0.05
Ca	**=< 0.5**	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	**0.02**	Tb	< 0.05
V	**0.01**	Dy	< 0.05
Cr	**1.6**	Ho	< 0.05
Mn	**< 0.05**	Er	< 0.05
Fe	**5.3**	Tm	< 0.05
Co	**< 0.05**	Yb	< 0.05
Ni	**1.7**	Lu	< 0.05
Cu	< 0.1	Hf	< 0.05
Zn	< 0.1	Ta	< 5
Ga	< 0.1	W	< 0.05
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	< 0.05	Hg	< 0.5
Y	< 0.05	Tl	< 0.1
Zr	< 0.05	Pb	< 0.5
Nb	< 0.1	Bi	< 0.1
Mo	0.42	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		

Table 8. GDMS results for Hexion SC-1008 carbonized at 950°C. Elements in bold are included in AGR-2 compact specification. This resin was used for fabrication of the AGR-1 and AGR-2 compacts.

Element	[ppm wt]	Element	[ppm wt]
Li	0.5	Pd	< 0.1
Be	< 0.01	Ag	< 0.1
B	2.3	Cd	< 0.1
C	Matrix	In	Secondary Cathode
N	-	Sn	< 0.5
O	-	Sb	< 0.5
F	< 1	Te	< 0.1
Na	25	I	< 0.5
Mg	< 0.5	Cs	< 0.5
Al	**50**	Ba	< 0.1
Si	60	La	< 0.5
P	< 0.1	Ce	< 0.05
S	40	Pr	< 0.05
Cl	1.1	Nd	< 0.05
K	< 1	Sm	< 0.05
Ca	**1**	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	**0.35**	Tb	< 0.05
V	**0.02**	Dy	< 0.05
Cr	**< 0.5**	Ho	< 0.05
Mn	**0.1**	Er	< 0.05
Fe	**2.5**	Tm	< 0.05
Co	**0.25**	Yb	< 0.05
Ni	**0.25**	Lu	< 0.05
Cu	< 0.1	Hf	< 0.05
Zn	< 0.1	Ta	< 5
Ga	< 0.1	W	15
Ge	< 0.1	Re	< 0.05
As	0.2	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	0.9	Au	< 0.1
Sr	< 0.05	Hg	< 0.5
Y	< 0.05	Tl	< 0.1
Zr	0.1	Pb	< 0.5
Nb	< 0.1	Bi	< 0.1
Mo	< 0.05	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		

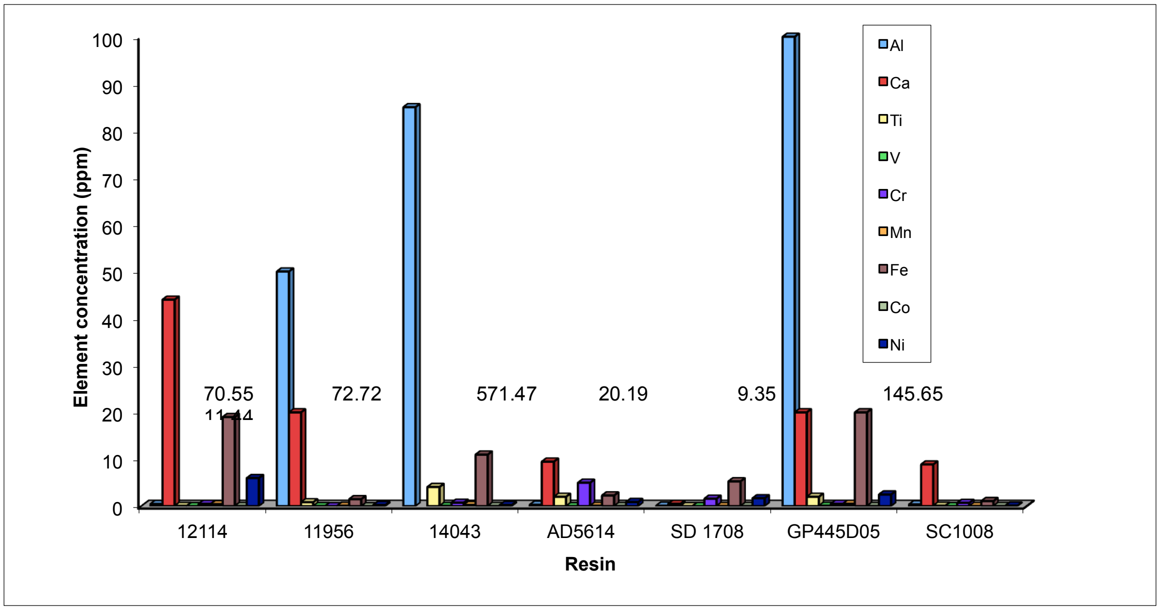

Figure 1: Summary of AGR-2 specified element levels for resins, SC1008 (used in fabrication of the AGR-2 compacts) is shown as a reference. The sum (in ppm) of all specified elements is shown above each resin. Note, Ca for 14043 was removed for visualization purposes; it was 470 ppm.

Table 9. Char yield of selected resins

Resin	Initial mass (g)	Final mass (g)	Char yield (%)
AD-5614	2.7913	1.6207	58
14043	2.2868	1.3011	57
SD-1708	4.5373	1.8491	41

Table 10. GDMS results for GTI experimental grade-A. Elements in bold are included in AGR-2 compact specification.

Element	[ppm wt]	Element	[ppm wt]
Li	0.08	Pd	< 0.1
Be	< 0.01	Ag	< 0.1
B	1.7	Cd	< 0.1
C	Matrix	In	Binder
N	-	Sn	< 0.5
O	-	Sb	< 0.5
F	~ 0.15 wt%	Te	< 0.1
Na	20	I	< 20
Mg	21	Cs	< 0.1
Al	**35**	Ba	1.2
Si	60	La	=< 2
P	2.9	Ce	< 0.5
S	800	Pr	=< 0.25
Cl	18	Nd	0.65
K	< 0.1	Sm	< 0.05
Ca	**150**	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	**9.7**	Tb	< 0.05
V	**780**	Dy	< 0.05
Cr	**9.7**	Ho	< 0.05
Mn	**3.5**	Er	< 0.05
Fe	**450**	Tm	< 0.05
Co	**10**	Yb	< 0.05
Ni	**500**	Lu	< 0.05
Cu	6.5	Hf	< 0.05
Zn	0.26	Ta	< 5
Ga	0.35	W	1
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	0.7	Hg	< 0.5
Y	1.1	Tl	< 0.1
Zr	0.59	Pb	< 0.5
Nb	< 0.1	Bi	< 0.1
Mo	8	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		

Table 11. GDMS results for GTI experimental grade-B. Elements in bold are included in AGR-2 compact specification.

Element	[ppm wt]	Element	[ppm wt]
Li	< 0.01	Pd	< 0.1
Be	< 0.01	Ag	< 0.1
B	0.8	Cd	< 0.1
C	Matrix	In	Binder
N	-	Sn	< 0.5
O	-	Sb	< 0.5
F	~ 150	Te	< 0.1
Na	15	I	< 20
Mg	3.5	Cs	< 0.1
Al	**36**	Ba	0.4
Si	40	La	=< 5
P	0.82	Ce	< 0.5
S	310	Pr	=< 0.7
Cl	5	Nd	< 0.05
K	< 0.1	Sm	< 0.05
Ca	**8.5**	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	**2.5**	Tb	< 0.05
V	**12**	Dy	< 0.05
Cr	**5.7**	Ho	< 0.05
Mn	**1.1**	Er	< 0.05
Fe	**65**	Tm	< 0.05
Co	**0.61**	Yb	< 0.05
Ni	**15**	Lu	< 0.05
Cu	0.48	Hf	< 0.05
Zn	0.58	Ta	< 5
Ga	0.57	W	< 0.05
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	0.16	Hg	< 0.5
Y	< 0.05	Tl	< 0.1
Zr	< 0.05	Pb	< 0.5
Nb	< 0.1	Bi	< 0.1
Mo	0.25	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		

Table 12. GDMS results for GTI experimental grade-C. Elements in bold are included in AGR-2 compact specification.

Element	[ppm wt]	Element	[ppm wt]
Li	0.07	Pd	< 0.1
Be	< 0.01	Ag	< 0.1
B	0.45	Cd	< 0.1
C	Matrix	In	Binder
N	-	Sn	< 0.5
O	-	Sb	< 0.5
F	~ 0.65 wt%	Te	< 0.1
Na	7.5	I	< 20
Mg	3.2	Cs	< 0.1
Al	**50**	Ba	1
Si	49	La	=< 5
P	3.2	Ce	< 0.5
S	350	Pr	=< 1
Cl	8.7	Nd	1.5
K	0.85	Sm	< 0.05
Ca	**18**	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	**3.5**	Tb	< 0.05
V	**21**	Dy	< 0.05
Cr	**12**	Ho	< 0.05
Mn	**1**	Er	< 0.05
Fe	**110**	Tm	< 0.05
Co	**0.57**	Yb	< 0.05
Ni	**26**	Lu	< 0.05
Cu	0.65	Hf	< 0.05
Zn	0.3	Ta	< 5
Ga	0.4	W	< 0.05
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	0.22	Hg	< 0.5
Y	< 0.05	Tl	< 0.1
Zr	0.12	Pb	< 0.5
Nb	< 0.1	Bi	< 0.1
Mo	0.3	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		

Table 13. GDMS results for GTI experimental grade-D. Elements in bold are included in AGR-2 compact specification.

Element	[ppm wt]	Element	[ppm wt]
Li	< 0.01	Pd	< 0.1
Be	< 0.01	Ag	< 0.1
B	0.55	Cd	< 0.1
C	Matrix	In	Binder
N	-	Sn	< 0.5
O	-	Sb	< 0.5
F	< 5	Te	< 0.1
Na	0.92	I	=< 100
Mg	< 0.5	Cs	< 0.1
Al	**< 0.05**	Ba	< 0.1
Si	6.8	La	=< 10
P	0.61	Ce	< 0.5
S	7.2	Pr	< 0.05
Cl	8.4	Nd	< 0.05
K	< 0.1	Sm	< 0.05
Ca	**=< 0.5**	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	**1.9**	Tb	< 0.05
V	**4.7**	Dy	< 0.05
Cr	**< 0.5**	Ho	< 0.05
Mn	**< 0.05**	Er	< 0.05
Fe	**0.25**	Tm	< 0.05
Co	**< 0.05**	Yb	< 0.05
Ni	**< 0.1**	Lu	< 0.05
Cu	< 0.1	Hf	< 0.05
Zn	< 0.1	Ta	< 5
Ga	< 0.1	W	< 0.05
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	< 0.05	Hg	< 0.5
Y	< 0.05	Tl	< 0.1
Zr	0.06	Pb	< 0.5
Nb	< 0.1	Bi	< 0.1
Mo	0.12	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		

Table 14. GDMS results for AGM grade A99. Elements in bold are included in AGR-2 compact specification.

Element	[ppm wt]	Element	[ppm wt]
Li	0.32	Pd	< 0.1
Be	< 0.01	Ag	< 0.1
B	2.8	Cd	< 0.1
C	Matrix	In	Binder
N	-	Sn	< 0.5
O	-	Sb	< 0.5
F	~ 15	Te	< 0.1
Na	4.5	I	< 20
Mg	48	Cs	< 0.1
Al	**180**	Ba	< 0.1
Si	980	La	< 0.5
P	5	Ce	< 0.5
S	210	Pr	< 0.05
Cl	4.4	Nd	< 0.05
K	24	Sm	< 0.05
Ca	**170**	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	**14**	Tb	< 0.05
V	**6.7**	Dy	< 0.05
Cr	**2.1**	Ho	< 0.05
Mn	**5**	Er	< 0.05
Fe	**950**	Tm	< 0.05
Co	**0.31**	Yb	< 0.05
Ni	**8.5**	Lu	< 0.05
Cu	22	Hf	< 0.05
Zn	27	Ta	< 5
Ga	< 0.1	W	0.17
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	0.89	Hg	< 0.5
Y	0.35	Tl	< 0.1
Zr	4.5	Pb	0.7
Nb	0.28	Bi	< 0.1
Mo	1.6	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		

Table 15. GDMS results for AGM grade 4421. Elements in bold are included in AGR-2 compact specification.

Element	[ppm wt]	Element	[ppm wt]
Li	0.24	Pd	< 0.1
Be	< 0.01	Ag	< 0.1
B	1.3	Cd	< 0.1
C	Matrix	In	Binder
N	-	Sn	< 0.5
O	-	Sb	< 0.5
F	< 5	Te	< 0.1
Na	41	I	< 20
Mg	4.5	Cs	< 0.1
Al	**26**	Ba	2
Si	180	La	< 0.5
P	1.2	Ce	< 0.5
S	180	Pr	< 0.05
Cl	38	Nd	< 0.05
K	2	Sm	< 0.05
Ca	**110**	Eu	< 0.05
Sc	0.09	Gd	< 0.05
Ti	**19**	Tb	< 0.05
V	**9**	Dy	< 0.05
Cr	**12**	Ho	< 0.05
Mn	**2**	Er	< 0.05
Fe	**~ 0.15 wt%**	Tm	< 0.05
Co	**7**	Yb	< 0.05
Ni	**10**	Lu	< 0.05
Cu	6	Hf	< 0.05
Zn	0.8	Ta	< 5
Ga	< 0.1	W	1.7
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	1.5	Hg	< 0.5
Y	0.41	Tl	< 0.1
Zr	0.83	Pb	< 0.5
Nb	0.64	Bi	< 0.1
Mo	15	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		

Table 16. GDMS results for AGM grade TC301. Elements in bold are included in AGR-2 compact specification.

Element	[ppm wt]	Element	[ppm wt]
Li	< 0.01	Pd	< 0.1
Be	< 0.01	Ag	< 0.1
B	0.56	Cd	< 0.1
C	Matrix	In	Binder
N	-	Sn	< 0.5
O	-	Sb	< 0.5
F	< 5	Te	< 0.1
Na	2	I	< 20
Mg	0.77	Cs	< 0.1
Al	**7.5**	Ba	< 0.1
Si	6.9	La	< 0.5
P	0.14	Ce	< 0.5
S	71	Pr	< 0.05
Cl	3.2	Nd	< 0.05
K	0.52	Sm	< 0.05
Ca	**14**	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	**0.55**	Tb	< 0.05
V	**0.6**	Dy	< 0.05
Cr	**4.3**	Ho	< 0.05
Mn	**0.13**	Er	< 0.05
Fe	**47**	Tm	< 0.05
Co	**< 0.05**	Yb	< 0.05
Ni	**3**	Lu	< 0.05
Cu	16	Hf	< 0.05
Zn	< 0.1	Ta	< 5
Ga	< 0.1	W	< 0.05
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	< 0.05	Hg	< 0.5
Y	< 0.05	Tl	< 0.1
Zr	< 0.05	Pb	< 0.5
Nb	< 0.1	Bi	< 0.1
Mo	< 0.05	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		

Table 17. GDMS results for AGM grade 7105. Elements in bold are included in AGR-2 compact specification.

Element	[ppm wt]	Element	[ppm wt]
Li	4	Pd	0.1
Be	0.01	Ag	0.1
B	2	Cd	0.1
C	0	In	Secondary Cathode
N	5	Sn	0.5
O	8	Sb	0.1
F	5	Te	0.5
Na	7	I	0.5
Mg	100	Cs	0.7
Al	**1.5**	Ba	0.5
Si	40	La	0.05
P	15	Ce	0.05
S	10	Pr	0.05
Cl	50	Nd	0.05
K	0.05	Sm	0.05
Ca	**3.5**	Eu	0.05
Sc	6	Gd	0.05
Ti	**0.5**	Tb	0.05
V	**3.5**	Dy	0.05
Cr	**50**	Ho	0.05
Mn	**0.1**	Er	0.05
Fe	**4**	Tm	0.05
Co	**0.1**	Yb	0.05
Ni	**0.1**	Lu	0.05
Cu	0.1	Hf	5
Zn	0.1	Ta	1
Ga	0.15	W	0.05
Ge	0.1	Re	0.05
As	0.1	Os	0.05
Se	0.05	Ir	0.05
Br	0.7	Pt	0.1
Rb	0.06	Au	0.5
Sr	1	Hg	0.1
Y	0.2	Tl	0.5
Zr	0.3	Pb	0.1
Nb	0.1	Bi	0.05
Mo	0.1	Th	0.05
Ru	4	U	0.1
Rh	0.01		

Table 18. GDMS results for Timcal grade KS-15. Elements in bold are included in AGR-2 compact specification.

Element	[ppm wt]	Element	[ppm wt]
Li	5	Pd	< 0.05
Be	< 0.01	Ag	< 0.1
B	2.9	Cd	< 0.1
C	Matrix	In	Secondary Cathode
N	-	Sn	< 0.5
O	-	Sb	< 0.1
F	< 1	Te	< 0.1
Na	3.5	I	< 0.1
Mg	< 0.5	Cs	< 0.1
Al	**2.3**	Ba	1.5
Si	100	La	< 0.5
P	1.5	Ce	0.09
S	45	Pr	< 0.05
Cl	3.1	Nd	< 0.05
K	4.5	Sm	< 0.05
Ca	**50**	Eu	< 0.05
Sc	< 0.01	Gd	< 0.05
Ti	**5.5**	Tb	< 0.05
V	**3.5**	Dy	< 0.05
Cr	**< 0.5**	Ho	< 0.05
Mn	**6.7**	Er	< 0.05
Fe	**55**	Tm	< 0.05
Co	**< 0.05**	Yb	< 0.05
Ni	**2.5**	Lu	< 0.05
Cu	< 0.5	Hf	< 0.05
Zn	< 0.1	Ta	< 75
Ga	< 0.1	W	50
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	< 0.01	Au	< 0.05
Sr	0.85	Hg	< 0.5
Y	0.07	Tl	< 0.1
Zr	3.5	Pb	< 0.5
Nb	0.1	Bi	< 0.1
Mo	0.35	Th	< 0.05
Ru	< 0.05	U	< 0.05
Rh	< 0.05		

Table 19. GDMS results for Timcal grade KS-44. Elements in bold are included in AGR-2 compact specification.

Element	[ppm wt]	Element	[ppm wt]
Li	5.1	Pd	< 0.1
Be	< 0.01	Ag	< 0.1
B	1.4	Cd	< 0.1
C	Matrix	In	Binder
N	-	Sn	< 0.5
O	-	Sb	< 0.5
F	~ 450	Te	< 0.1
Na	6.5	I	< 20
Mg	3.3	Cs	< 0.1
Al	**1.5**	Ba	0.85
Si	56	La	< 0.5
P	1.6	Ce	< 0.5
S	38	Pr	< 0.05
Cl	4.5	Nd	< 0.05
K	4.2	Sm	< 0.05
Ca	**30**	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	**22**	Tb	< 0.05
V	**17**	Dy	< 0.05
Cr	**1.5**	Ho	< 0.05
Mn	**6.5**	Er	< 0.05
Fe	**32**	Tm	< 0.05
Co	**0.08**	Yb	< 0.05
Ni	2.7	Lu	< 0.05
Cu	1.1	Hf	< 0.05
Zn	1.2	Ta	< 5
Ga	0.15	W	< 0.05
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	0.46	Hg	< 0.5
Y	0.09	Tl	< 0.1
Zr	5.8	Pb	< 0.5
Nb	< 0.1	Bi	< 0.1
Mo	1.2	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		

Table 20. GDMS results for Timcal grade T-44. Elements in bold are included in AGR-2 compact specification.

Element	[ppm wt]	Element	[ppm wt]
Li	3.1	Pd	< 0.1
Be	< 0.01	Ag	0.4
B	1.9	Cd	< 0.1
C	Matrix	In	Binder
N	-	Sn	< 0.5
O	-	Sb	4
F	~ 0.22 wt%	Te	< 0.1
Na	6.9	I	< 20
Mg	2	Cs	< 0.1
Al	**3**	Ba	1
Si	190	La	=< 1
P	2.8	Ce	=< 1.5
S	31	Pr	=< 0.15
Cl	19	Nd	0.55
K	1.6	Sm	< 0.05
Ca	**35**	Eu	< 0.05
Sc	0.56	Gd	< 0.05
Ti	**53**	Tb	< 0.05
V	**8.5**	Dy	< 0.05
Cr	**1**	Ho	< 0.05
Mn	**4.3**	Er	< 0.05
Fe	**44**	Tm	< 0.05
Co	**0.2**	Yb	< 0.05
Ni	**1.4**	Lu	< 0.05
Cu	0.65	Hf	< 0.05
Zn	0.5	Ta	< 5
Ga	< 0.1	W	0.6
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	0.71	Hg	< 0.5
Y	2	Tl	< 0.1
Zr	7.3	Pb	< 0.5
Nb	1	Bi	< 0.1
Mo	1	Th	0.25
Ru	< 0.1	U	0.19
Rh	< 0.1		

Table 21. GDMS results for Timcal grade SFG-44. Elements in bold are included in AGR-2 compact specification.

Element	[ppm wt]	Element	[ppm wt]
Li	8.2	Pd	< 0.1
Be	< 0.01	Ag	< 0.1
B	1.4	Cd	< 0.1
C	Matrix	In	Binder
N	-	Sn	< 0.5
O	-	Sb	< 0.5
F	~ 0.85 wt%	Te	< 0.1
Na	12	I	< 20
Mg	1.3	Cs	< 0.1
Al	**3.5**	Ba	1
Si	54	La	< 0.5
P	3	Ce	< 0.5
S	58	Pr	< 0.05
Cl	8.5	Nd	< 0.05
K	0.62	Sm	< 0.05
Ca	**36**	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	**15**	Tb	< 0.05
V	**6**	Dy	< 0.05
Cr	**2.4**	Ho	< 0.05
Mn	**0.28**	Er	< 0.05
Fe	**34**	Tm	< 0.05
Co	**0.09**	Yb	< 0.05
Ni	3.8	Lu	< 0.05
Cu	2.2	Hf	< 0.05
Zn	< 0.1	Ta	< 5
Ga	< 0.1	W	< 0.05
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	0.49	Hg	< 0.5
Y	0.24	Tl	< 0.1
Zr	5.5	Pb	< 0.5
Nb	< 0.1	Bi	< 0.1
Mo	0.45	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		

Table 22. GDMS results for SGL grade KRB-2000. Elements in bold are included in AGR-2 compact specification. This synthetic graphite was used for fabrication of the AGR-1 and AGR-2 compacts.

Element	[ppm wt]	Element	[ppm wt]
Li	< 0.01	Pd	< 0.1
Be	< 0.01	Ag	< 0.1
B	2.1	Cd	< 0.1
C	Matrix	In	Binder
N	-	Sn	< 0.5
O	-	Sb	< 0.5
F	< 5	Te	< 0.1
Na	0.45	I	< 0.5
Mg	0.2	Cs	< 0.5
Al	**0.35**	Ba	< 0.1
Si	3.1	La	< 0.5
P	0.11	Ce	< 0.05
S	9	Pr	< 0.05
Cl	3.2	Nd	< 0.05
K	0.45	Sm	< 0.05
Ca	**0.7**	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	**0.06**	Tb	< 0.05
V	**0.02**	Dy	< 0.05
Cr	**< 0.5**	Ho	< 0.05
Mn	**< 0.05**	Er	< 0.05
Fe	**1.4**	Tm	< 0.05
Co	**0.25**	Yb	< 0.05
Ni	**1.2**	Lu	< 0.05
Cu	< 0.5	Hf	< 0.05
Zn	< 0.5	Ta	< 5
Ga	< 0.1	W	2.7
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	< 0.05	Hg	< 0.5
Y	< 0.05	Tl	< 0.1
Zr	< 0.05	Pb	< 0.5
Nb	< 0.1	Bi	< 0.1
Mo	< 0.05	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		

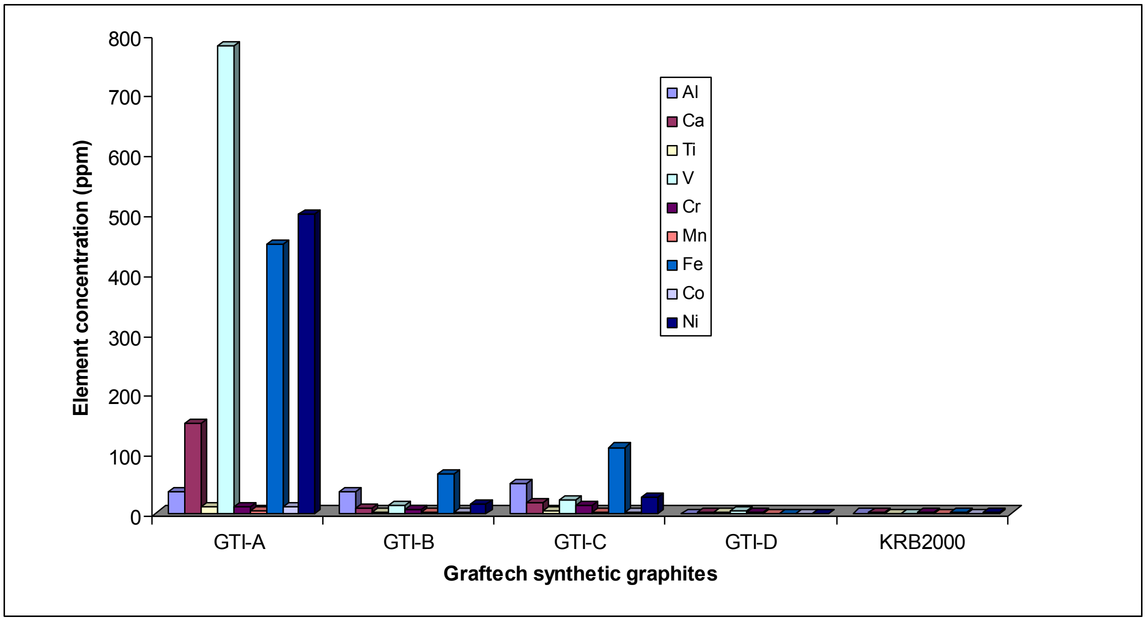

Figure 2. Summary of AGR-2 specified element levels for Graftech experimental grades, KRB-2000 (used in fabrication of the AGR-2 compacts) is shown as a reference.

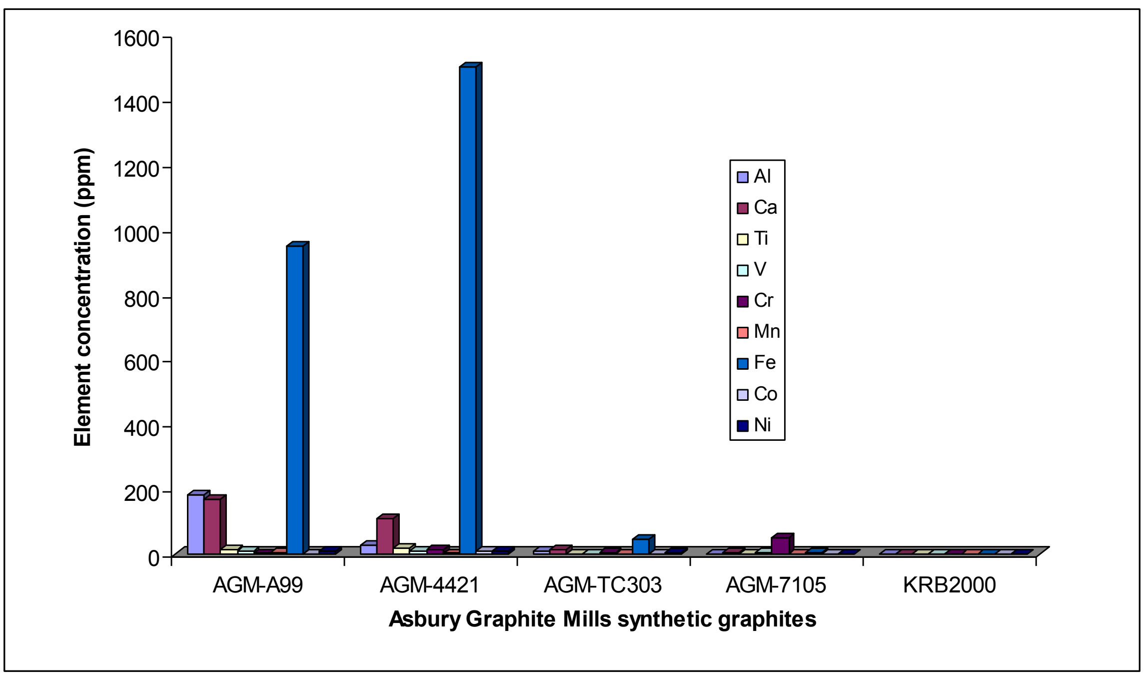

Figure 3. Summary of AGR-2 specified element levels for Asbury Graphite Mills grades, KRB-2000 (used in fabrication of the AGR-2 compacts) is shown as a reference.

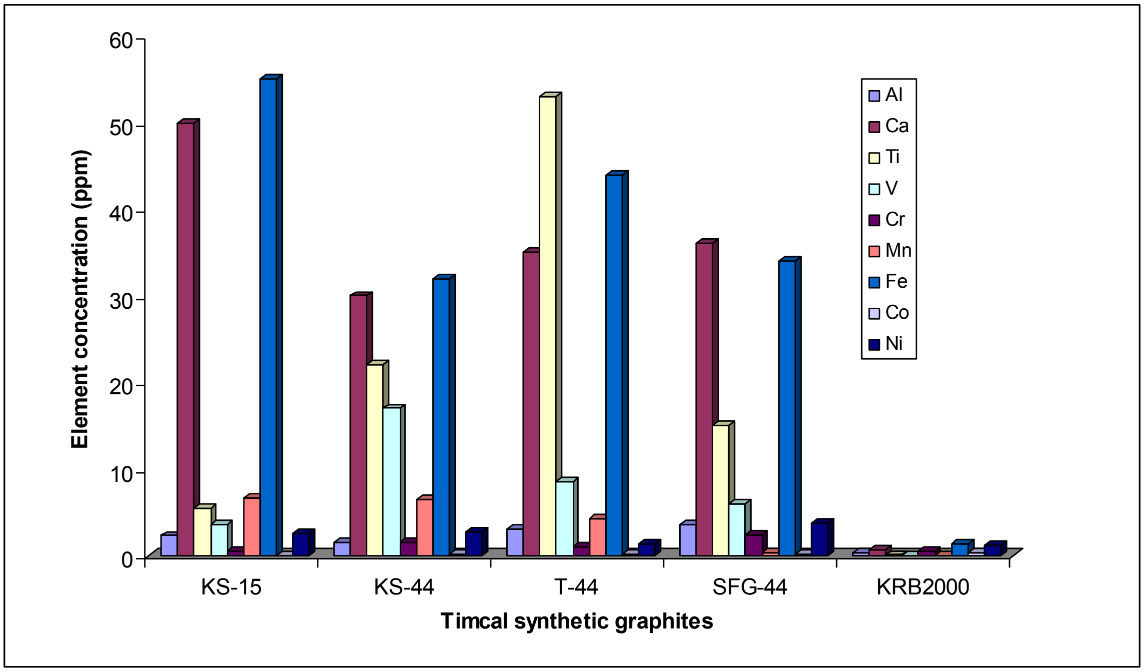

Figure 4. Summary of AGR-2 specified element levels for Timcal grades, KRB-2000 (used in fabrication of the AGR-2 compacts) is shown as a reference.

Table 23. GDMS results for Timcal grade PG06. Elements in bold are included in AGR-2 compact specification.

Element	[ppm wt]	Element	[ppm wt]
Li	< 0.01	Pd	< 0.1
Be	< 0.01	Ag	< 0.1
B	2.1	Cd	< 0.1
C	Matrix	In	Binder
N	-	Sn	< 0.5
O	-	Sb	< 0.5
F	~ 110	Te	< 0.1
Na	21	I	< 20
Mg	150	Cs	< 0.1
Al	**95**	Ba	0.19
Si	0.22 wt%	La	< 0.5
P	10	Ce	< 0.5
S	~ 0.25 wt%	Pr	< 0.05
Cl	24	Nd	< 0.05
K	10	Sm	< 0.05
Ca	**430**	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	**5.4**	Tb	< 0.05
V	**0.29**	Dy	< 0.05
Cr	**4.4**	Ho	< 0.05
Mn	**23**	Er	< 0.05
Fe	**~ 0.37 wt%**	Tm	< 0.05
Co	**4.3**	Yb	< 0.05
Ni	**47**	Lu	< 0.05
Cu	160	Hf	< 0.05
Zn	3.9	Ta	< 5
Ga	< 0.1	W	0.82
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	0.47	Hg	< 0.5
Y	0.08	Tl	< 0.1
Zr	0.2	Pb	2.3
Nb	< 0.1	Bi	1.2
Mo	1.9	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		

Table 24. GDMS results for Timcal grade Micro 890. Elements in bold are included in AGR-2 compact specification.

Element	[ppm wt]	Element	[ppm wt]
Li	0.39	Pd	< 0.1
Be	< 0.01	Ag	0.5
B	2.7	Cd	< 0.1
C	Matrix	In	Binder
N	-	Sn	< 0.5
O	-	Sb	< 0.5
F	~ 200	Te	< 0.1
Na	120	I	< 20
Mg	750	Cs	< 0.1
Al	**880**	Ba	7.5
Si	~ 0.2 wt%	La	=< 5
P	7.5	Ce	3.9
S	960	Pr	0.25
Cl	60	Nd	0.45
K	85	Sm	0.12
Ca	**600**	Eu	< 0.05
Sc	< 0.05	Gd	0.06
Ti	**33**	Tb	< 0.05
V	**15**	Dy	< 0.05
Cr	**1.4**	Ho	< 0.05
Mn	**6.8**	Er	< 0.05
Fe	**910**	Tm	< 0.05
Co	**0.95**	Yb	< 0.05
Ni	**23**	Lu	< 0.05
Cu	8.8	Hf	< 0.05
Zn	3.1	Ta	< 5
Ga	< 0.1	W	0.88
Ge	< 0.1	Re	< 0.05
As	0.92	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	15	Pt	< 0.05
Rb	0.35	Au	< 0.1
Sr	3.5	Hg	< 0.5
Y	1.3	Tl	< 0.1
Zr	3.2	Pb	3.1
Nb	< 0.1	Bi	< 0.1
Mo	40	Th	0.15
Ru	< 0.1	U	< 0.05
Rh	< 0.1		

Table 25. GDMS results for Superior Graphite Co. grade Thermopure # 2939APH BT51516. Elements in bold are included in AGR-2 compact specification.

Element	[ppm wt]	Element	[ppm wt]
Li	< 0.01	Pd	< 0.1
Be	< 0.01	Ag	< 0.1
B	1.9	Cd	< 0.1
C	Matrix	In	Binder
N	-	Sn	< 0.5
O	-	Sb	< 0.5
F	~ 250	Te	< 0.1
Na	19	I	< 20
Mg	1.6	Cs	< 0.1
Al	**2.6**	Ba	< 0.1
Si	38	La	< 0.5
P	0.22	Ce	< 0.5
S	25	Pr	< 0.05
Cl	11	Nd	< 0.05
K	0.28	Sm	< 0.05
Ca	**6.8**	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	**25**	Tb	< 0.05
V	**4.1**	Dy	< 0.05
Cr	**< 0.5**	Ho	< 0.05
Mn	**< 0.05**	Er	< 0.05
Fe	**25**	Tm	< 0.05
Co	**0.11**	Yb	< 0.05
Ni	**0.51**	Lu	< 0.05
Cu	< 0.1	Hf	< 0.05
Zn	< 0.1	Ta	< 5
Ga	< 0.1	W	< 0.05
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	0.19	Hg	< 0.5
Y	0.55	Tl	< 0.1
Zr	12	Pb	< 0.5
Nb	< 0.1	Bi	< 0.1
Mo	1.5	Th	0.15
Ru	< 0.1	U	0.11
Rh	< 0.1		

Table 26. GDMS results for Asbury Graphite Mills grade 3482. Elements in bold are included in AGR-2 compact specification.

Element	[ppm wt]	Element	[ppm wt]
Li	< 0.01	Pd	< 0.1
Be	< 0.01	Ag	< 0.1
B	0.24	Cd	< 0.1
C	Matrix	In	Binder
N	-	Sn	< 0.5
O	-	Sb	< 0.5
F	~ 20	Te	< 0.1
Na	1.5	I	< 20
Mg	80	Cs	< 0.1
Al	**29**	Ba	0.8
Si	710	La	< 0.5
P	0.82	Ce	< 0.5
S	25	Pr	< 0.05
Cl	2.5	Nd	< 0.05
K	0.2	Sm	< 0.05
Ca	**8.2**	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	**2.3**	Tb	< 0.05
V	**0.45**	Dy	< 0.05
Cr	**< 0.5**	Ho	< 0.05
Mn	**0.55**	Er	< 0.05
Fe	**38**	Tm	< 0.05
Co	**< 0.05**	Yb	< 0.05
Ni	**0.25**	Lu	< 0.05
Cu	2.5	Hf	< 0.05
Zn	0.4	Ta	< 5
Ga	< 0.1	W	0.15
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	0.41	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	0.08	Hg	< 0.5
Y	0.2	Tl	< 0.1
Zr	0.6	Pb	< 0.5
Nb	< 0.1	Bi	< 0.1
Mo	< 0.05	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		

Table 27. GDMS results for Asbury Graphite Mills grade RD13371. Elements in bold are included in AGR-2 compact specification.

Element	[ppm wt]	Element	[ppm wt]
Li	0.02	Pd	< 0.1
Be	< 0.01	Ag	< 0.1
B	0.48	Cd	< 0.1
C	Matrix	In	Binder
N	-	Sn	< 0.5
O	-	Sb	< 0.5
F	=< 400	Te	< 0.1
Na	1.7	I	< 20
Mg	4.8	Cs	< 0.1
Al	**8.3**	Ba	19
Si	260	La	< 0.5
P	0.22	Ce	< 0.5
S	60	Pr	< 0.05
Cl	1.5	Nd	< 0.05
K	0.68	Sm	< 0.05
Ca	**10**	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	**0.66**	Tb	< 0.05
V	**0.35**	Dy	< 0.05
Cr	**< 0.5**	Ho	< 0.05
Mn	**0.29**	Er	< 0.05
Fe	**13**	Tm	< 0.05
Co	**< 0.05**	Yb	< 0.05
Ni	**1.4**	Lu	< 0.05
Cu	3.8	Hf	< 0.05
Zn	0.85	Ta	< 5
Ga	< 0.1	W	0.45
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	0.66	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	0.14	Hg	< 0.5
Y	0.06	Tl	< 0.1
Zr	0.15	Pb	< 0.5
Nb	< 0.1	Bi	< 0.1
Mo	0.06	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		

Table 28. GDMS results for Asbury Graphite Mills grade RD13382. Elements in bold are included in AGR-2 compact specification.

Element	[ppm wt]	Element	[ppm wt]
Li	0.03	Pd	< 0.1
Be	< 0.01	Ag	< 0.1
B	0.27	Cd	< 0.1
C	Matrix	In	Binder
N	-	Sn	< 0.5
O	-	Sb	< 0.5
F	~ 800	Te	< 0.1
Na	5.5	I	< 20
Mg	7.2	Cs	< 0.1
Al	**20**	Ba	18
Si	330	La	=< 430
P	0.51	Ce	< 0.5
S	88	Pr	< 0.05
Cl	11	Nd	< 0.05
K	0.66	Sm	< 0.05
Ca	**16**	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	**0.42**	Tb	< 0.05
V	**0.05**	Dy	< 0.05
Cr	**< 0.5**	Ho	< 0.05
Mn	**0.38**	Er	< 0.05
Fe	**20**	Tm	< 0.05
Co	**< 0.05**	Yb	< 0.05
Ni	**0.21**	Lu	< 0.05
Cu	1.7	Hf	< 0.05
Zn	0.32	Ta	< 5
Ga	< 0.1	W	< 0.05
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	0.09	Hg	< 0.5
Y	0.09	Tl	< 0.1
Zr	0.45	Pb	< 0.5
Nb	< 0.1	Bi	< 0.1
Mo	< 0.05	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		

Table 29. GDMS results for GrafTech grade GTI 30 Micron. Elements in bold are included in AGR-2 compact specification.

Element	[ppm wt]	Element	[ppm wt]
Li	< 0.01	Pd	< 0.1
Be	< 0.01	Ag	< 0.1
B	0.87	Cd	< 0.1
C	Matrix	In	Binder
N	-	Sn	< 0.5
O	-	Sb	< 0.5
F	~ 90	Te	< 0.1
Na	1.5	I	< 20
Mg	0.55	Cs	< 0.1
Al	**1.6**	Ba	< 0.1
Si	25	La	< 0.5
P	0.29	Ce	< 0.5
S	14	Pr	< 0.05
Cl	10	Nd	< 0.05
K	< 0.1	Sm	< 0.05
Ca	**3.1**	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	**43**	Tb	< 0.05
V	**5.1**	Dy	< 0.05
Cr	**0.61**	Ho	< 0.05
Mn	**< 0.05**	Er	< 0.05
Fe	**13**	Tm	< 0.05
Co	**< 0.05**	Yb	< 0.05
Ni	**0.45**	Lu	< 0.05
Cu	< 0.1	Hf	< 0.05
Zn	< 0.1	Ta	< 5
Ga	< 0.1	W	0.79
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	Ir	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	0.46	Hg	< 0.5
Y	0.19	Tl	< 0.1
Zr	7.5	Pb	< 0.5
Nb	< 0.1	Bi	< 0.1
Mo	9.5	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		

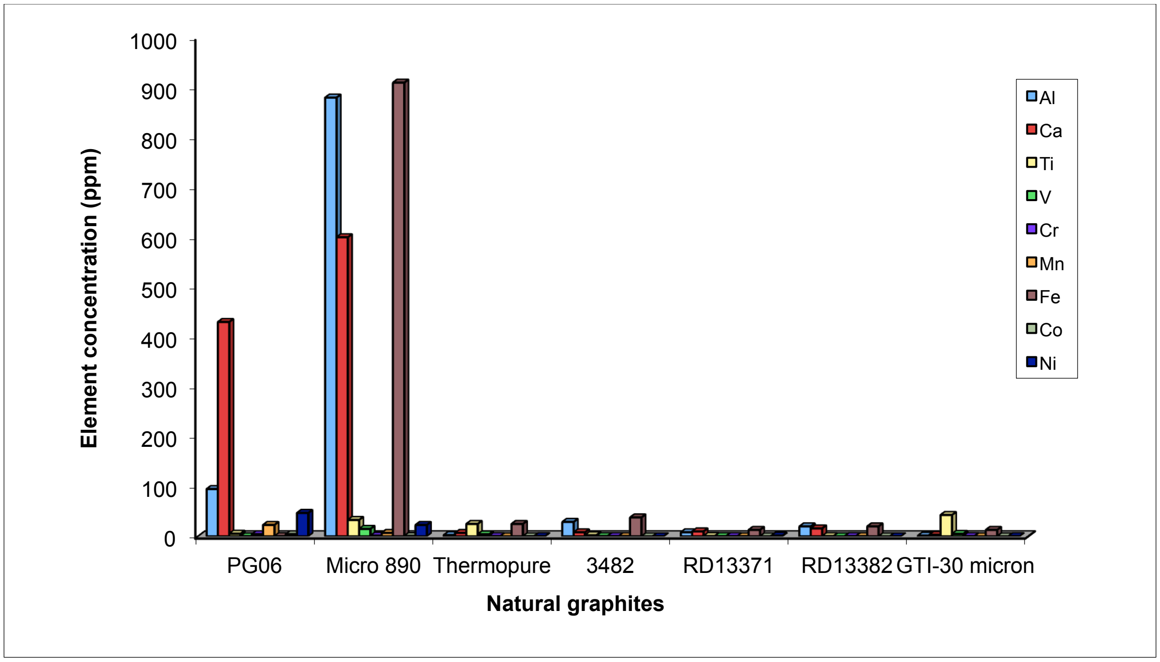

Figure 5. Summary of AGR-2 specified element levels for selected natural graphites, RD13371 (used in fabrication of the AGR-2 compacts) is shown as a reference

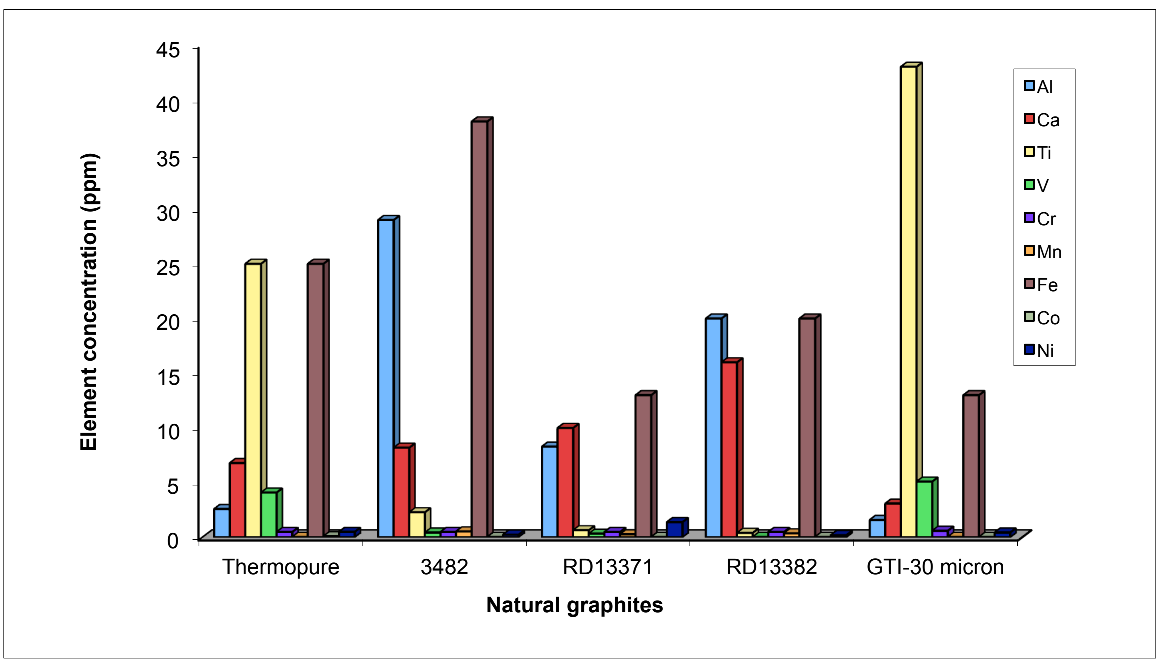

Figure 6. Summary of AGR-2 specified element levels for selected natural graphites, RD13371 (used in fabrication of the AGR-2 compacts) is shown as a reference

3.2 Resin rheology results

Rheology testing was performed on the Hexion AD-5614, SD-1708, and Plenco 14043 resins. Isotherms were taken from 100 to 150°C at 10°C intervals, except for the SD-1708 resin because of reasons that will be explained shortly.

Figure 7 shows the viscosity versus time data for resin AD-5614. An overall observation is that the viscosity drops with increasing temperature to about 140°C, and then increases sharply at higher temperatures.

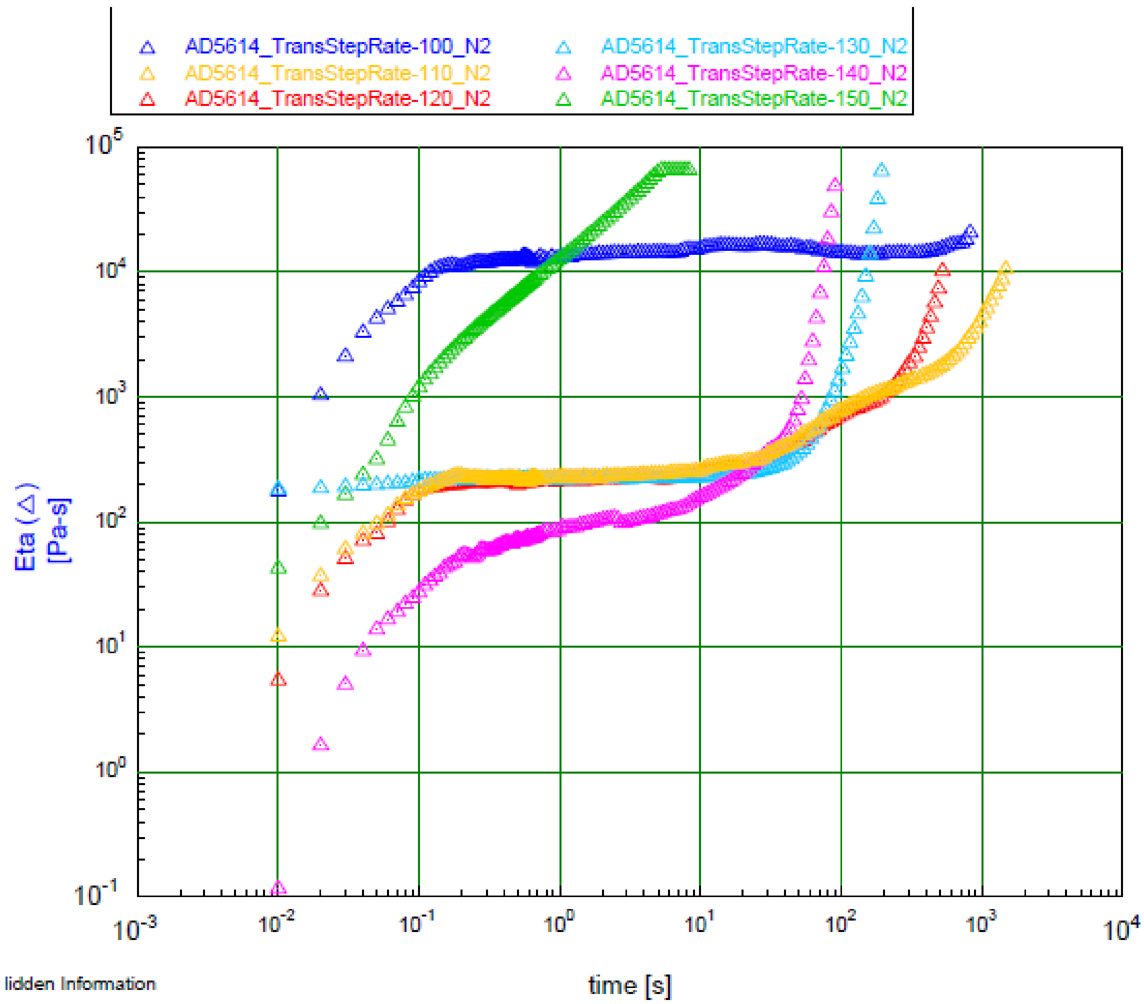

Figure 7. Rheology data, viscosity vs. time, for Hexion AD-5614 resin.

At 100°C the curve is flat, showing that the viscosity was unchanged over the length of the run. This was because the softening point of the resin was not reached and it did not deform or flow during the test. At 110°C the viscosity remains constant for about 20-30 seconds before the viscosity starts to increase. The reason for the increase in viscosity is that the resin begins to cure, or set up. The curing process involves the coalescence of aromatic molecules that comprise the resin. As these molecules combine, the aromaticity factor increases, causing an increase in viscosity. These condensation reactions occur until the resin is completely cured and a solid is formed. For the 110°C test, this occurred after about 1000 seconds, when the viscosity approached 10^4 Pa-s.

The 120°C test was similar to the 110°C test in terms of time until initiation of curing. However, at 120°C the sample was finished curing after about 400 seconds, as opposed to 1000 seconds.

Again, in the 130°C sample the time to curing initiation is about the same. However, the time from curing onset to curing commencement is reduced. The resin begins curing after about 20-30 seconds, but is completely cured after about 100 seconds. At 110 and 120°C, the curing also started after about 20-30 seconds, but time to curing commencement took ~1000 and ~400 seconds, respectively.

At 140 and 150°C, the resin cures more quickly. At 140°C, curing begins after about 10 seconds and a solid is formed after less than 100 seconds. At 150°C, the curing is basically instantaneous.

The isotherms for Plenco 14043 are shown in Figure 8. A similar series of curves were found. The viscosity drops from 100 to 130°C, and then increases for the 140 and 150°C tests. In general, the 14043 resin exhibited longer flow times prior to complete curing. As was the case with the AD-5614 resin at 100°C, the softening point was not achieved so the resin remained a solid during the entire run. The 110°C sample began curing at around 200 seconds and fully cured near 1000 seconds. The viscosity of the resin continued to decrease at 120°C, and the onset of curing took place after about 40 seconds. The resin finished curing after 400 seconds.

The lowest viscosity was observed during the 130°C isotherm test. Curing onset occurred shortly after 10 seconds and the sample reached total cure at about 100 seconds.

The viscosity of the resin then showed an initial higher viscosity value in the 140 and 150°C isotherms. In both of these samples, the onset of curing is nearly instantaneous, but complete curing did not occur until 50 seconds (for the 140°C isotherm) and 30 seconds (for the 150°C isotherm).

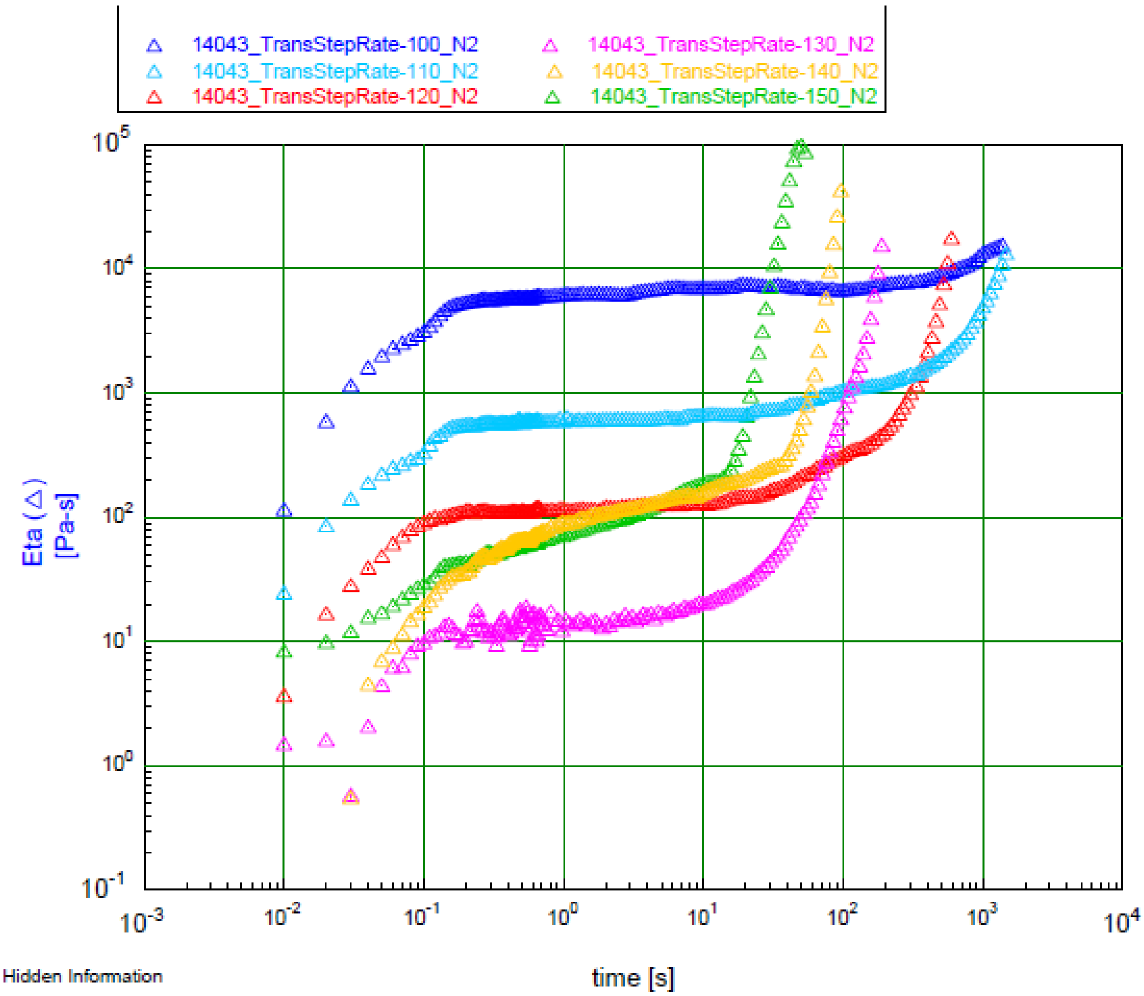

Figure 8. Rheology data, viscosity vs. time, for Plenco 14043 resin

The isotherm data for the Hexion SD-1708 resin is shown in Figure 9. An immediate difference from the AD-5614 and 14043 isotherms is that complete curing was not observed at the temperature tested here. This was due to the absence of a separate hardening agent. The AD-5614 and 14043 resins both contain a hardening agent called hexamine. The addition of this agent, thus making the resin a “two part” resin, decreases the time to complete cure by promoting condensation reactions and increasing molecular weight. The SD-1708 sample sent to ORNL for testing did not have this hardening agent added.

Note that the viscosity drops with increasing temperature, but no curing is observed, even after 1000 seconds. This resin isotherm behavior may be useful should a compacting approach that avoids curing in the die be adopted.

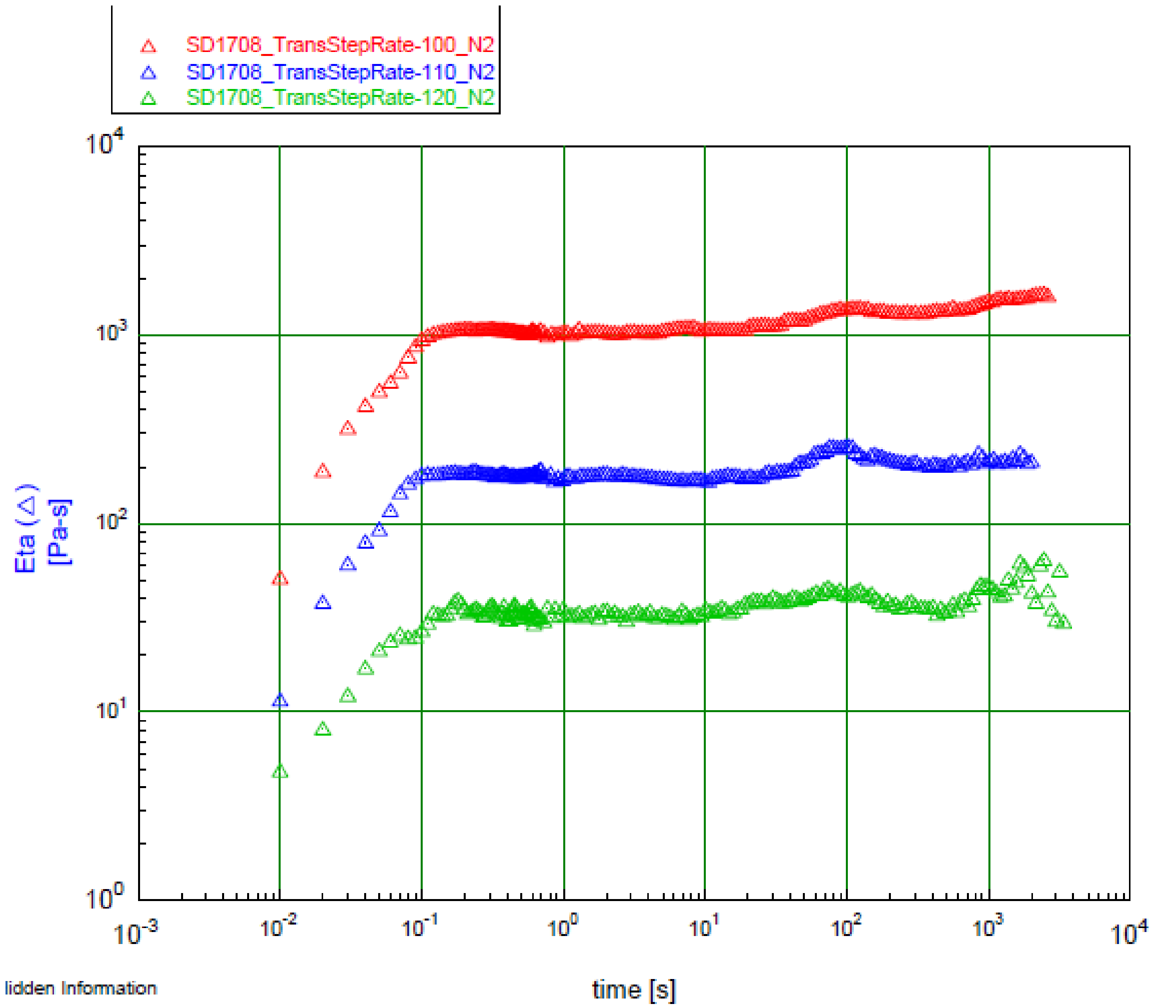

Figure 9. Rheology data, viscosity vs. time, for Hexion SD-1708 resin

3.3 BET surface area results

BET for surface area was performed on the synthetic graphite samples only, as the purpose of the resin is to coat graphite particles, acting as a bonding agent. Table 30 provides the BET surface areas for six synthetic graphites that are being considered as a replacement for KRB2000, which is also shown for reference.

Table 30 shows that KRB 2000 has the lowest surface area (1.33 m^2/g) of the graphites tested to date. The next closest graphite is GTI-D at 2.77 m^2/g. This low surface area suggests that KRB2000 may have been impregnated at least once in powder form. The likely impregnant would be petroleum pitch or coal-tar pitch.

Table 31 provides the BET surface area for the seven natural graphites tested. All values are relatively close with the exception of Asbury 3482. At 1.32 (m^2/g), it has a much lower surface area than any other sample. This suggests that Asbury 3482 is comprised of larger particles.

Table 30. BET surface area values for synthetic graphites

Synthetic Graphite	BET surface area (m^2/g)
Graftech-A	19.38
Graftech-B	15.02
Graftech-C	15.01
Graftech-D	2.77
AGM-A99	13.11
AGM-4421	12.61
AGM-TC301	15.46
AGM-7105	14.47
Timcal-KS15	17.9
Timcal-KS44	10.25
Timcal-T44	13.08
Timcal-SFG44	10.72
KRB2000	1.33

Table 31. BET surface area values for natural graphites

Natural Graphite	BET surface area (m^2/g)
PG06	7.41
Micro 890	7.56
Thermopure	7.29
Asbury 3482 lot 5675	1.32
Asbury RD13371	5.91
Asbury RD13382	5.64
GTI-30 micron	4.32

3.4 SEM investigation for morphology

SEM analysis was performed on the synthetic graphites in order to characterize, as best as possible, the morphology of particles. Not much obvious difference was observed in morphology between these samples. The Timcal grades had perhaps the most flake-like shape, while the GTI grades were more spherical. KRB2000 may be more spherical than flake in nature, but no striking differences were observed.

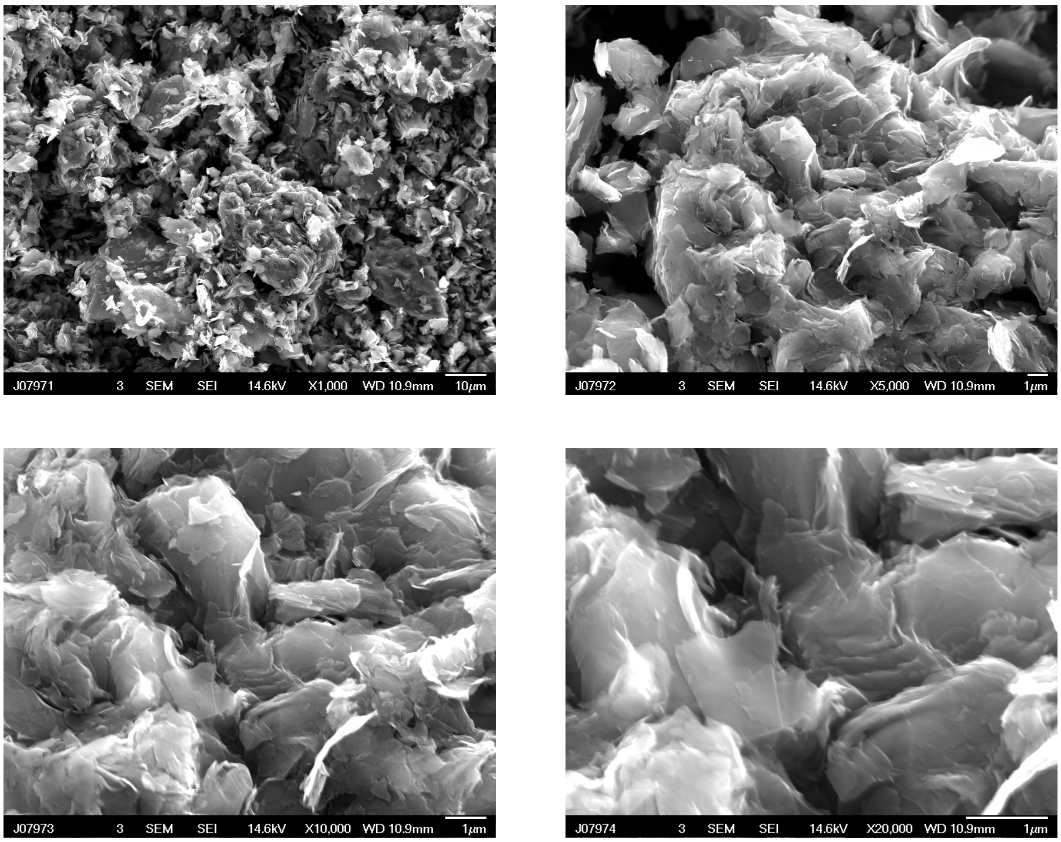

Figure 10. SEM images of GTI-A synthetic graphite

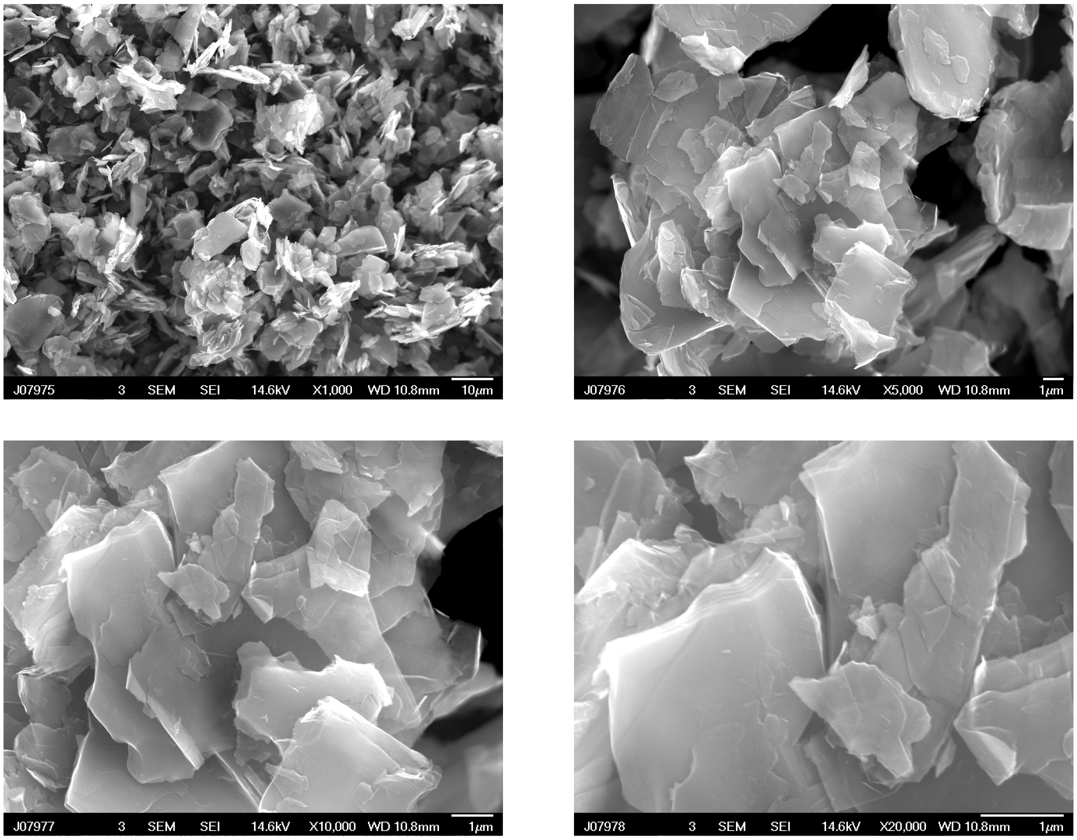

Figure 11. SEM images of GTI-B synthetic graphite

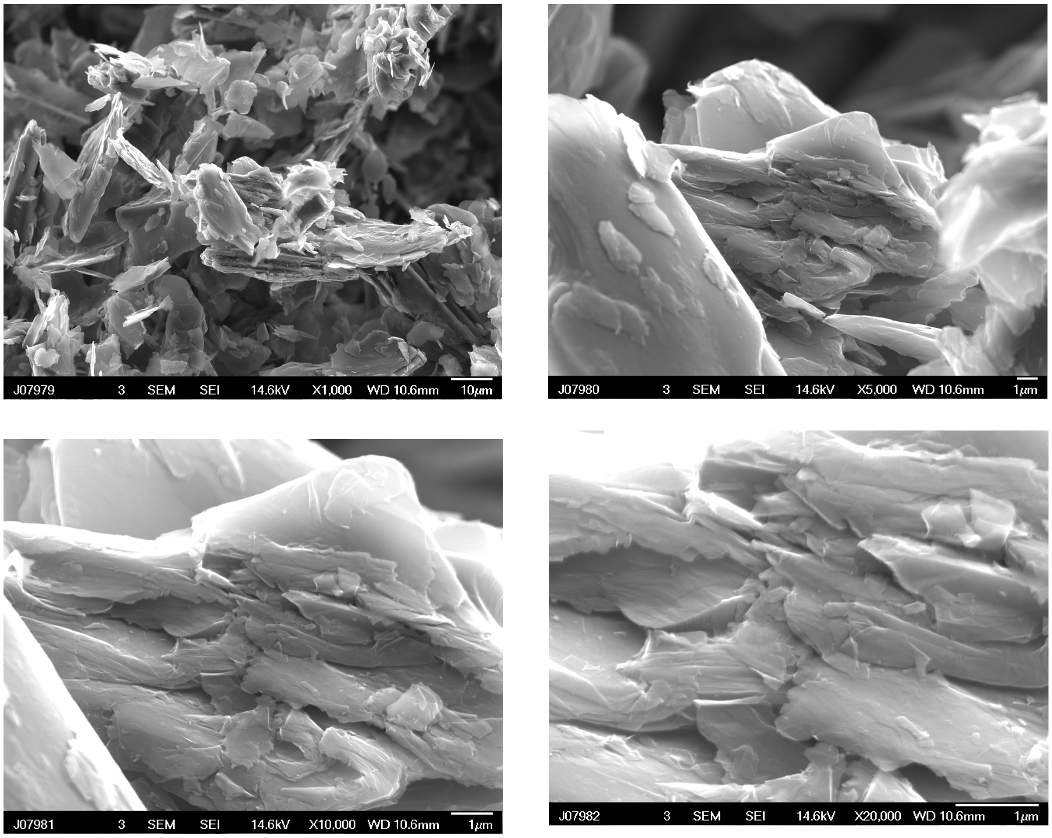

Figure 12. SEM images of GTI-C synthetic graphite

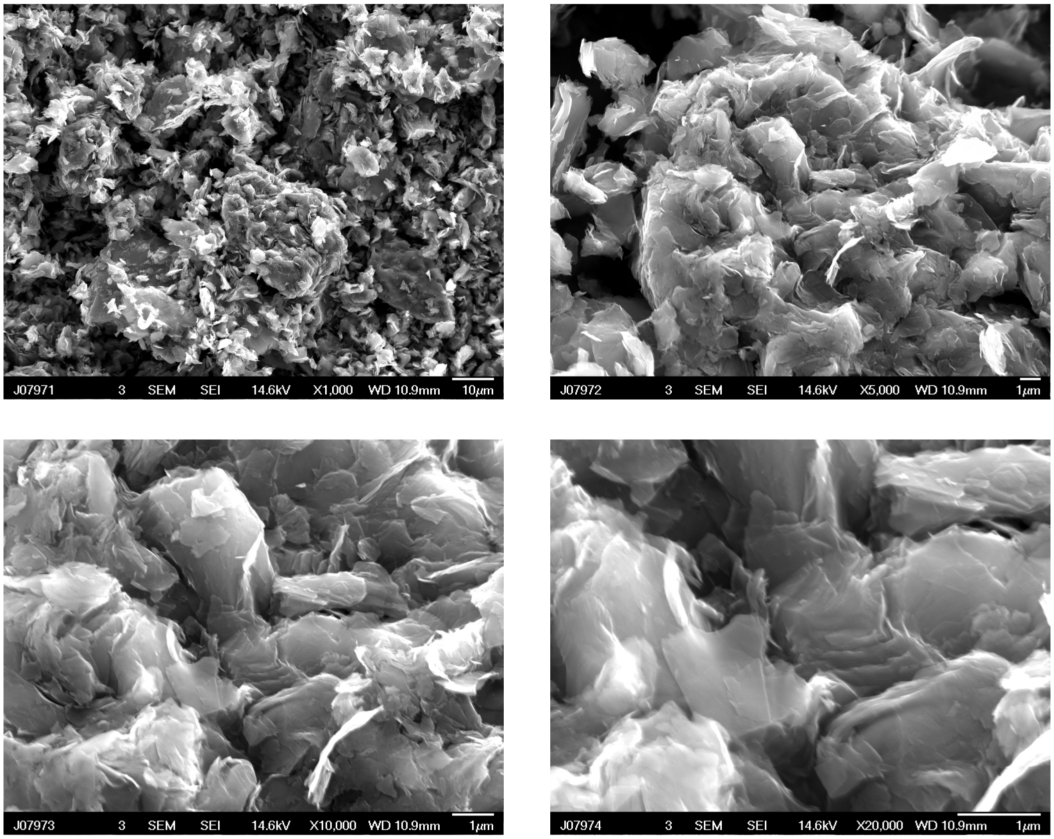

Figure 13. SEM images of AGM TC303 synthetic graphite

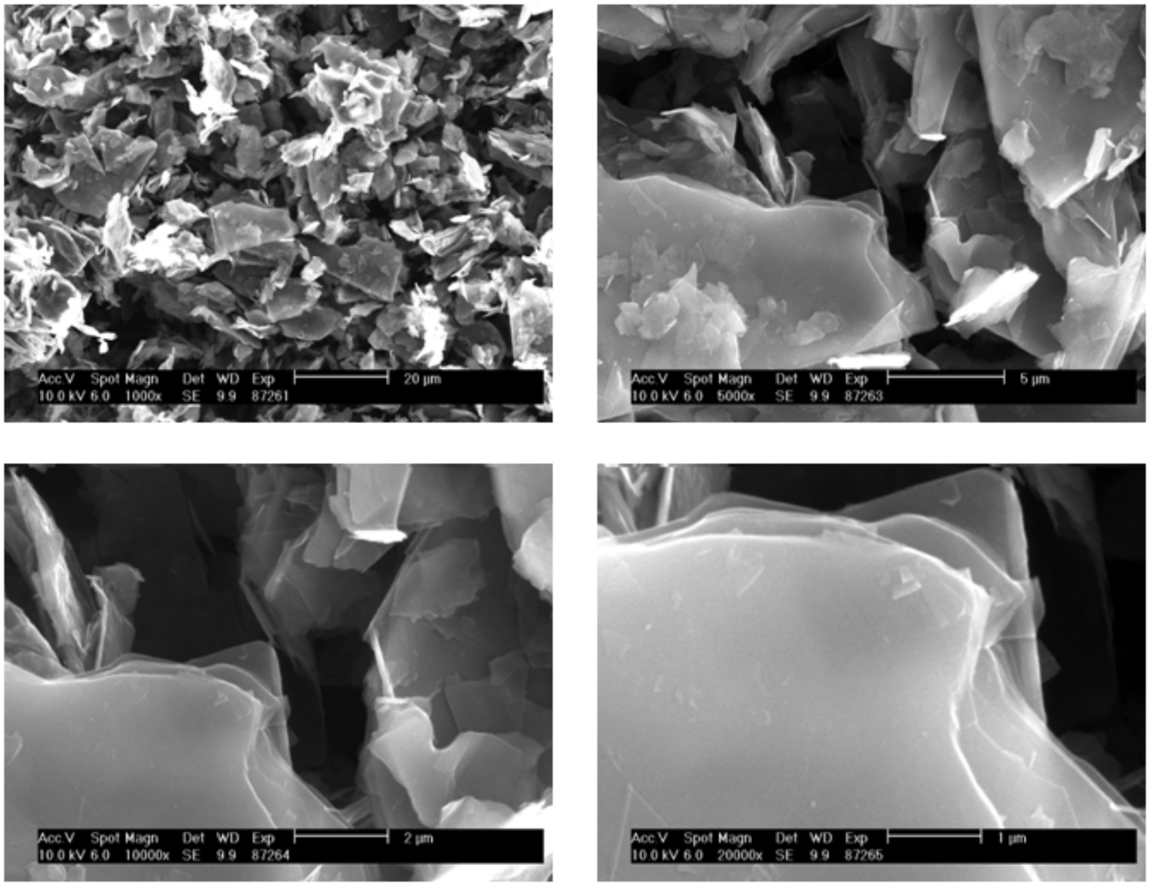

Figure 14. SEM images of Timcal KS-15

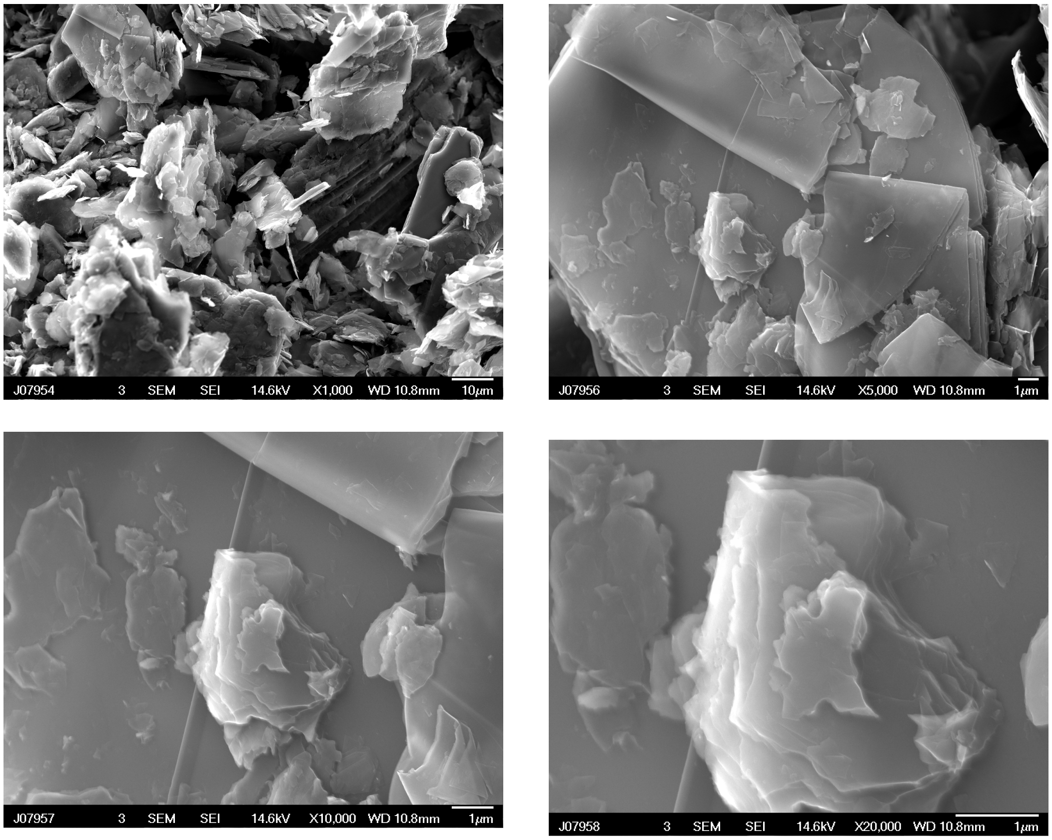

Figure 15. SEM images of Timcal KS44 synthetic graphites

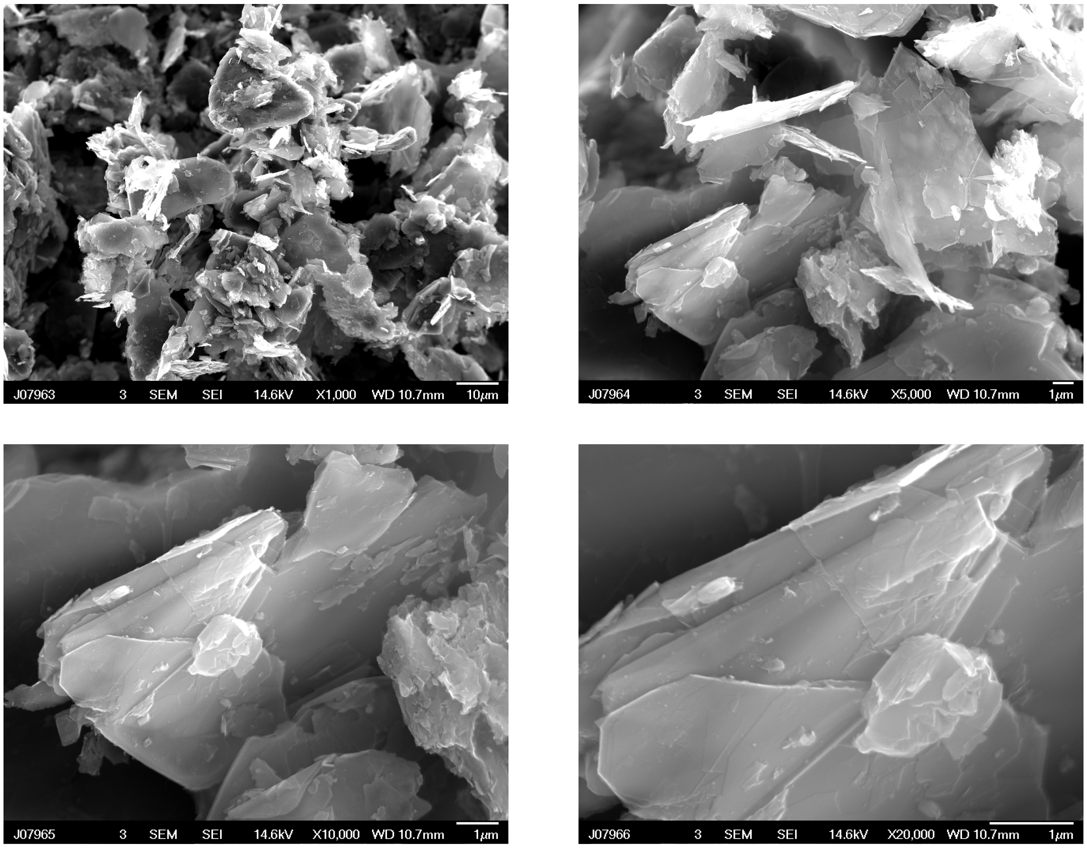

Figure 16. SEM images of Timcal T44 synthetic graphites

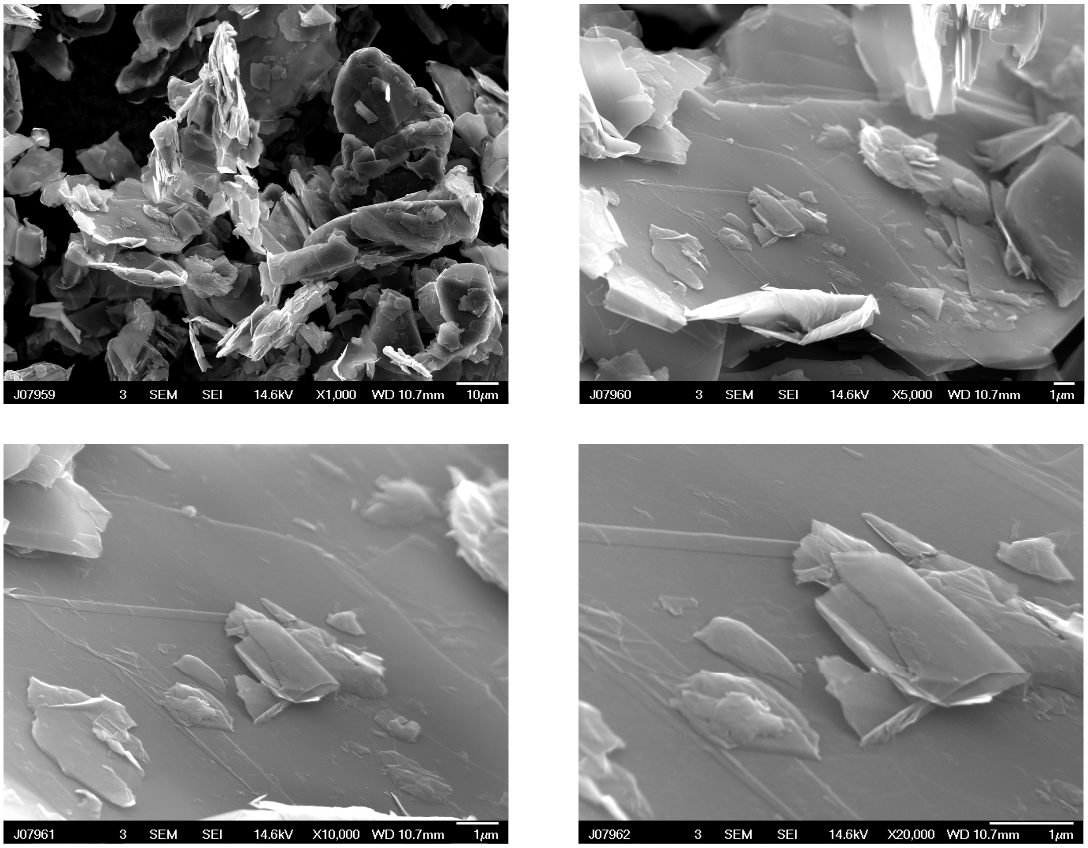

Figure 17. SEM images of Timcal SFG44 synthetic graphite

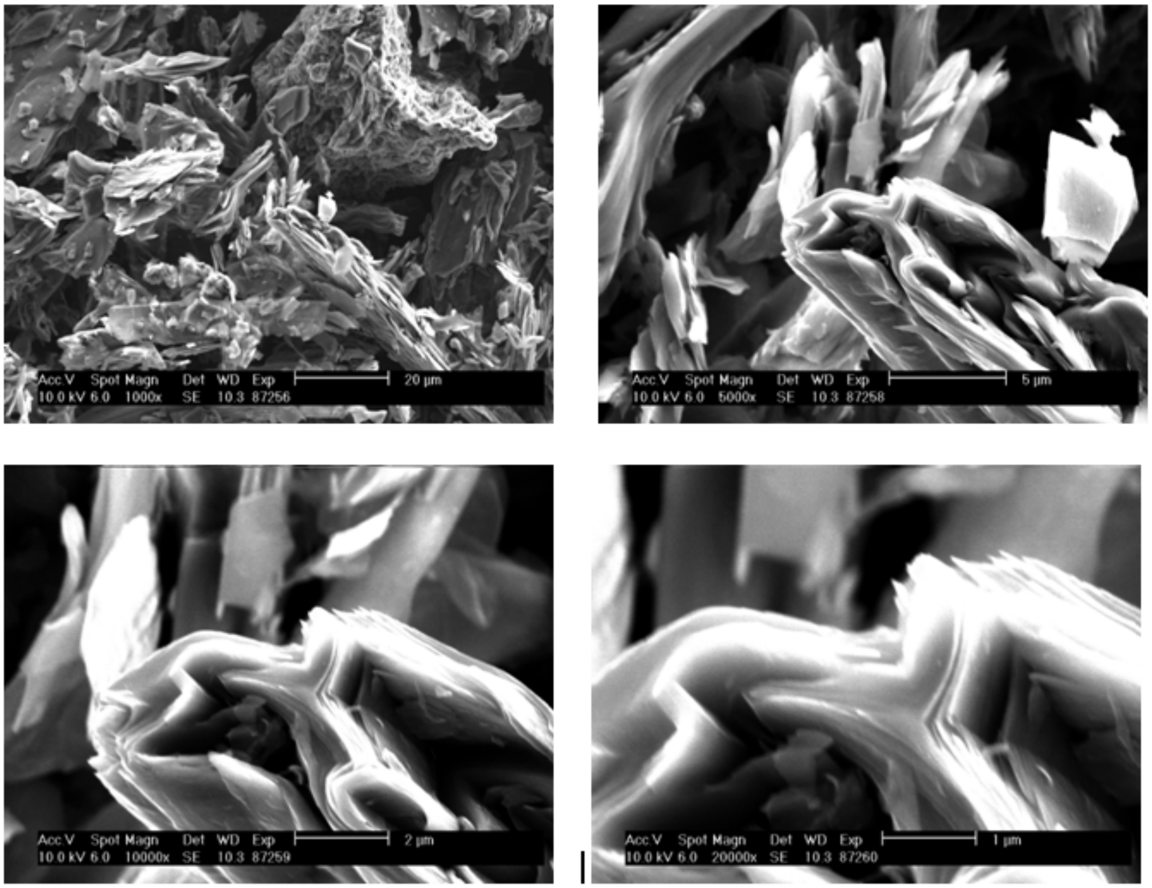

Figure 18. SEM images of SGL KRB2000 synthetic graphite

3.5 Tap Density and Compressibility Results

Tap density was performed on the natural and synthetic graphite in order to characterize the flowability and compressibility of each sample. Table 32 shows the tap densities and Carr compressibility values that were calculated for each synthetic graphite sample. KRB2000 had a tap density of nearly double each of the other graphite samples. However, the compressibility index was 26, which was about in the median for each of the samples.

Table 32. Tap density and Carr compressibility results for synthetic graphites

Synthetic Graphite	Mass (g)	V_i (mL)	V_f (mL)	Tap density (g/mL)	Carr compressibility
Graftech-A	33	138	107	0.31	22
Graftech-B	23	152	108	0.32	34
Graftech-C	28	133	88	0.21	29
Graftech-D	68	155	116	0.59	25
AGM-A99	41	155	109	0.51	33
AGM-4421	49	154	109	0.40	33
AGM-TC303	47	139	93	0.41	29
AGM-7105	38	150	118	0.44	31
Timcal-KS15	33	150	121	0.38	30
Timcal-KS44	44	143	99	0.45	29
Timcal-T44	36	126	89	0.28	19
Timcal-SFG44	38	141	94	0.32	21
KRB2000	110	150	111	0.99	26

The tap density and BET surface area of KRB2000 suggests that it is made up of denser particles than any of the other graphites. Again, this may mean that it was impregnated with pitch at some point, or made from a very dense petroleum or pitch-based coke. However, the properties of the GTI-D may be sufficient to make it a replacement. Further testing on the strength and toughness of a compact made using each material is required.

Table 33 shows the tap density and Carr compressibility values that were calculated for each natural graphite sample. These values provide information as a reference on a broad range of natural graphites. In order to better understand how these values correspond to individual physical properties in compacts, further testing is necessary.

Table 33. Tap density and Carr compressibility results for natural graphites

Natural Graphite	**Mass (g)**	**V_i (mL)**	**V_f (mL)**	**Tap density (g/mL)**	**Carr compressibility**
PG06	33	150	115	0.29	23
Micro 890	27	150	110	0.25	27
Thermopure	40	150	116	0.34	23
Asbury 3482	78	150	118	0.66	21
Asbury RD13371	44	140	94	0.47	33
Asbury RD13382	45	150	92	0.48	39
GTI-30 micron	37	150	92	0.40	39

4.0 Conclusions

GDMS was performed on all of the resin, natural graphite, and synthetic graphite samples. The synthetic and natural graphite samples were also characterized for surface area by BET and powder compressibility by tap density. In addition, the synthetic graphite samples were characterized for morphology by SEM. The following observations were made:

1. Hexion SC1008, which was used in making the AGR-1 and AGR-2 compacts, had the lowest level of impurities of the resins tested here.
2. Hexion AD-5614 or SD-1708 should be a suitable replacement for SC1008, in terms of elements that are called out in the AGR-2 compact specification. However, AD-5614 does contain high levels of elements that are not specified in the AGR-2 compact specification, namely fluorine, silicon, and tungsten. The behavior of this resin when used to fabricate matrix, either by a slurry or jet milling process, must be investigated further.
3. SGL KRB2000, which was used in making the AGR-1 and AGR-2 compacts, had the lowest level of impurities of the graphites tested here.
4. Graftech International grade GTI-D could be a suitable replacement for KRB2000. The total level of impurities was nearly double that of KRB2000, but still less than 10 ppm.
5. KRB2000 had the lowest BET surface area compared to the other graphites, suggesting little porosity. A lack of porosity may be the result of an impregnation step with petroleum or coal-tar pitch during formation. The low porosity may also be the result of the coke or pitch-coke used to make KRB2000.
6. The GTI-D sample was next closest in surface area and tap density. The impact of a high density synthetic graphite on the properties of a fuel compact needs to be investigated further, namely strength and toughness.
7. Tap density testing showed KRB2000 had a tap density nearly double that of the other graphites. However, the Carr compressibility index for KRB2000 was about in the middle to lower range of values for the samples
8. Asbury RD13371 remains the best natural graphite candidate based on results of GDMS analysis. Other candidate such as RD13382, Asbury 3482, and Thermopure may be suitable candidates provided impurity levels are below the specified limits after final heat treatment of the compacts. The natural graphite may also affect the mechanical properties of the compacts, so this factor must also be considered.
9. The SEM investigation for morphology showed little obvious difference in the graphites. Some may have been slightly more flake-like in nature, but no striking differences were seen.

Appendix 1: Additional Information on Matrix Production Methods of Foreign Countries Utilizing Overcoating and Compacting Process

From: Shohei UETA [mailto:ueta.shohei@jaea.go.jp]
Sent: Thursday, December 11, 2008 11:13 PM
To: Feltus, Madeline
Subject: Fwd: RE: Resination of Matrix in HTTR Process

Dear Feltus-san, I try to send to you again the following...

I am very sorry to delay may reply for your e-mail dated Dec 05 due to my participation for CRP6 last week. How are you?

For detail on fuel compact matrix materials, electro-graphite powder, natural graphite powder, and phenolic resin are mixed in the ratio of "weight" per sent 16%, 64%, 20%, respectively.
Alcohol is also added as same (or more) weight as these matrix powder to mix them homogeniously.
After that, they are dried and milled to make resinated graphite powder.
CFPs are overcoated by resinated graphite powder with alcohol.
Then, amount of alcohol to wet both particle and powder is not so much, which depends on season (humidity, temperature, etc.) .

Concerning reference 5.4, we have published only Japanese version up to now.

with best regards,

Shohei UETA of JAEA

From: 唐春和 [mailto:tangch@mail.tsinghua.edu.cn]
Sent: Monday, December 08, 2008 11:46 PM
To: Feltus, Madeline
Cc: Charles M Barnes; john.saurwein@gat.com; Pappano, Peter J.; David.Petti@inl.gov; uner; Uner COLAK
Subject: Re: RE: Resination of Matrix in HTTR Process

Dear Dr.Madeline,
Sorry to reply later due to IAEA CRP-6 meeting trip.
The following is our A3 matrix material preparation process:
1.The weight per cent of binder in the mixture of natural graphite powder, electro-graphite powder and binder is 20%.
2.At first mixing binder and alcohol. The weight per cent of alcohol is more than 50%.
3.Kneading natural graphite powder, electro-graphite powder and binder containing alcohol.
4.drying.
5.milling.

That is all.
With best regards,

Chunhe Tang
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Fax: +86-10-62771150
Email: tangch@mail.tsinghua.edu.cn

--Message from Danie Jacobs, PBMR pebble fabrication lead

Pete.

It is good to hear from you and compliments for the season.

We first mix the two graphites (electro plus natural), then (separately) dissolves the phenol binder in methanol, transfer the mixed graphites into the kneader, then add the phenol binder solvent into the kneader and then do kneading. Our phenol binder is a solidified droplet ~5mm in size. The reason for first mixing the two graphites is that in this way it is easier to achieve homogeneity between the two graphites and for pre-dissolving the phenol binder in the methanol is to more easily achieve covering each powder particle with the phenol binder. In our case, moving more towards a slurry causes the phenol binder to sag out before the pressing powder will be dry.

Are there any further news on burn leach results since we visited you last year? If it is possible for you, will you let me know.

Regards,
Danie.

From: Pappano, Peter J. [mailto:pappanopj@ornl.gov]
Sent: Tuesday, January 05, 2010 10:51 PM
To: Danie Jacobs
Subject: PBMR matrix preparation

Danie,

Although the New Year is quite new, I hope that it has been treating you well.

I was wondering if you could confirm in an email or via the presentation you showed during your visit to ORNL that the matrix production method you use involves alcohol to solvate the resin. If memory serves you combine the natural and synthetic graphite with powdered phenol binder and then add alcohol to form a “dough” which is kneaded and then dried. We use an excess of solvent and form a slurry.

Anyway, I was hoping you could confirm this for me.

Thanks
Pete

Appendix 2: Revision Log

This report is a revision to a previous report issued in 2009 by Peter J. Pappano entitled "Report on Analysis of Synthetic Graphite and Thermosetting Resin Candidates for Use in Fuel Compact Matrix," ORNL/TM-2009/315. Revisions to the previous report are documented in Appendix 2.

Rev.	Pages	Revision Description
0	3	1. Added first paragraph to describe revisions added to this report
	4-7	2. Revised Table of Contents and List of Tables and List of Figures as needed
	11	3. Added list of natural graphites from most recent study to Table 1
	13-14	4. Added paragraph at the end of section 3.1 to discuss impurity analysis of natural graphite
	38-44	5. Added Tables 23-29 containing GDMS impurity results for natural graphite
	45	6. Added Figures 5 and 6 to compare impurity results for natural graphite
	50	7. Added paragraph to the end of section 3.3 and added Table 31 to report BET analysis results for natural graphite
	60-61	8. Added paragraph to the end of section 3.5 and added Table 33 to report tap density results for natural graphite
	62	9. Revised first paragraph in Conclusion to include natural graphite and inserted conclusion #8
	66	10. Added this revision log