INL/RPT-24-77904
Revision 0

Elevated Temperature Graphite Mechanical Testing

April 2024

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Idaho National Laboratory

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http://www.art.inl.gov

Prepared for the
U.S. Department of Energy
Office of Nuclear Energy
Under DOE Idaho Operations Office
Contract DE-AC07-05ID14517

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INL ART Program

Elevated Temperature Graphite Mechanical Testing

INL/RPT-24-77904
Revision 0

April 2024

Technical Reviewer: (Confirmation of mathematical accuracy, and correctness of data and appropriateness of assumptions.)

for | 4/24/2024

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ABSTRACT

High-purity graphite will be used for core components within most high-temperature reactor (HTR) designs. Several "high-tech" industries currently utilize synthetic, high-purity, commercially available graphite components to fabricate photovoltaic cells, semiconductors, optical fibers, and other high-value electronic industry products. New advanced HTR designs are also interested in using these graphite grades for long-term, internal core component applications. Consequently, the United States (U.S.) Department of Energy (DOE) Advanced Reactor Technologies (ART) program has spent several years testing different high-purity graphite grades for potential use within these new HTR designs.

A significant part of that effort has been in the development and improvement of American Society for Testing and Materials (ASTM) test standards—specifically for nuclear graphite grades. Nearly all ASTM test standards have either been developed or improved by the ART program over the past 25 years, which continues to assist in the development of new ASTM test standards in support of the future commercial HTR fleet currently being designed and built in the U.S. The newest effort undertaken by ART is the development of high-temperature mechanical testing practices that may be acceptable for a future ASTM test standard (or guide) for this critical material property measurement.

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ACRONYMS

AG	against-grain
AGC	advanced graphite creep
ART	Advanced Reactor Technologies
ASTM	American Society for Testing and Materials
ATS	Applied Test Systems
DOE	U.S. Department of Energy
HTR	high-temperature reactor
R&D	research and development
U.S.	United States
UTS	ultimate tensile strength
WG	with-grain

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Elevated Temperature Graphite Mechanical Testing

1. INTRODUCTION

Graphite is known to have an increased mechanical strength at higher than ambient temperatures. Currently, there is no American Society for Testing and Materials (ASTM) guidance for conducting high-temperature strength testing, a key operational condition for high-temperature reactor (HTR) core components. The United States (U.S.) Department of Energy (DOE) Advanced Reactor Technologies (ART) Graphite research and development (R&D) program is currently exploring test methodologies for high-temperature strength testing at anticipated HTR operational temperatures. Several grades of nuclear graphite are to be tested at temperature with the results compared against ambient temperature test measurements conducted using ASTM-approved room-temperature mechanical test standards.

To date, compressive strength and split-disc mechanical tests have been conducted at elevated temperatures and compared to measured results at ambient temperatures [1,2]. The high-temperature testing procedures followed the ambient testing methodology as closely as possible. Thus, specimen sizes, specimen geometries, test fixtures, and loading rates are directly comparable to the established ambient test standard specifications to minimize the development process for these tests. By using established specimen geometries and testing equipment from the already approved ambient test standards, it is hoped that test reproducibility will be maximized, and the precision of the room-temperature test standard can be retained. Only the temperature directly surrounding the specimen was modified from the ambient test standards. Elevated testing results for IG-110, NBG-18, PCEA, and 2114 graphite grades were then compared to the measured results from the room-temperature testing standards. Initial results from this development effort are described and discussed in the sections that follow.

2. PROCEDURE

Both mechanical tests were conducted on an Instron Model 5982 load frame. An Applied Test Systems (ATS) tube furnace was mounted on the load frame to heat the samples and was controlled via a Eurotherm 2404 temperature controller. Special compression fittings were made to extend into the furnace and conduct the testing at the midline of the furnace. Figure 1 displays an overall view of the fittings installed with the furnace opened for display.

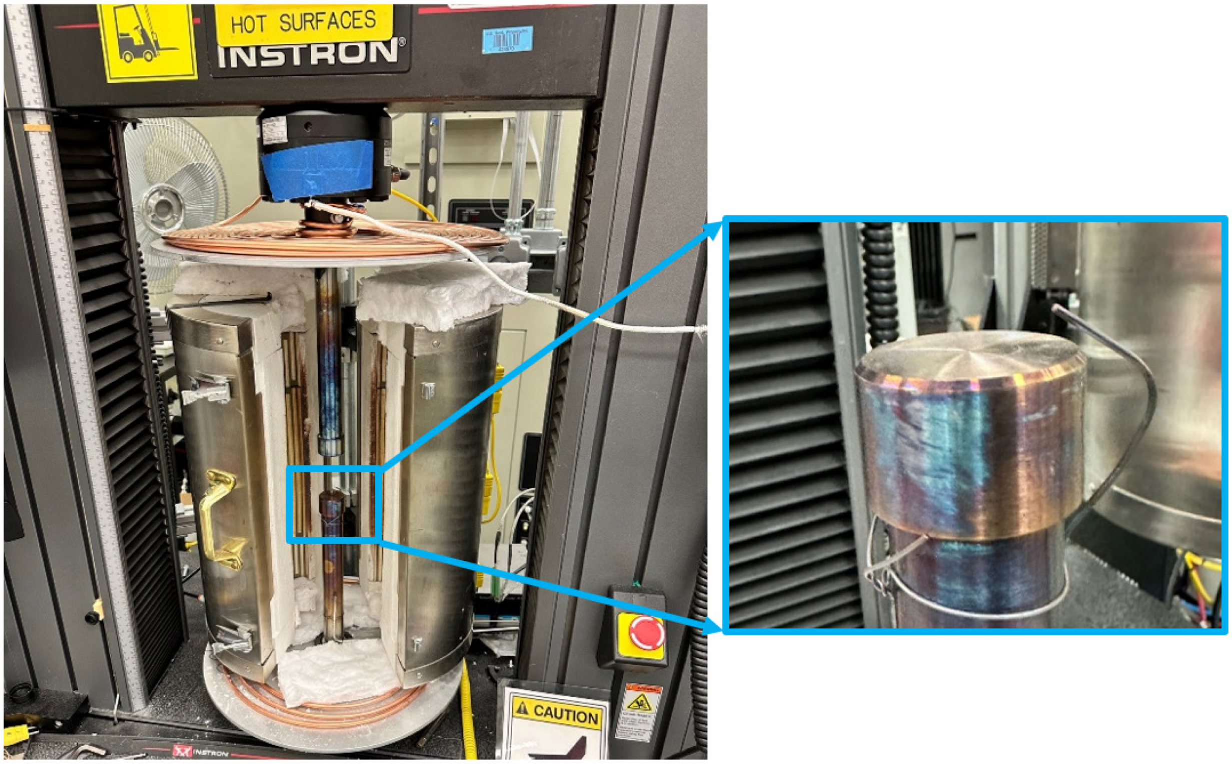

Figure 1. Overall view of experimental setup (left) and a close-up view (right).

Two aluminum heat shields were mounted above and below the furnace to protect the load frame components from the heat of the furnace. Copper coils were placed on the heat shields to remove heat during testing. Chilled cooling water was run through the copper coils from a closed-loop Neslab chiller to remove heat buildup on the heat shields. A layer of ceramic insulation was layered in between the furnace and the heat shield. Figure 2 shows the top cooling coils from the backside and the Neslab M25 chiller utilized in the testing.

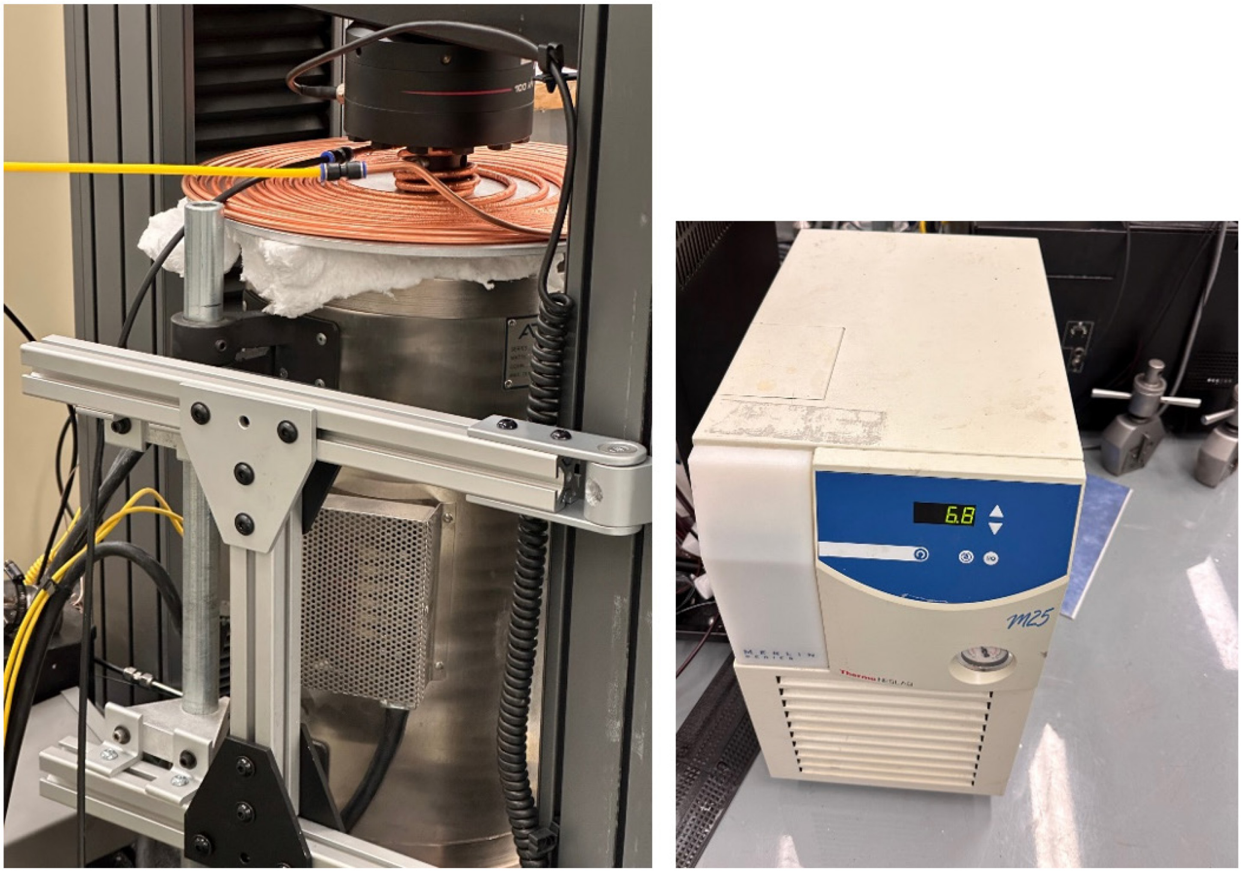

Figure 2. Neslab chiller (right) and the top cooling coils (left).

A thermocouple was placed at the midline of the furnace to monitor sample temperatures. This thermocouple is shown in the right image in Figure 3. The furnace controller setpoint was adjusted to generate the desired test temperature at the sample location. Test temperatures of 20°C, 500°C, and 800°C have been conducted so far, with more temperatures planned to be tested as well.

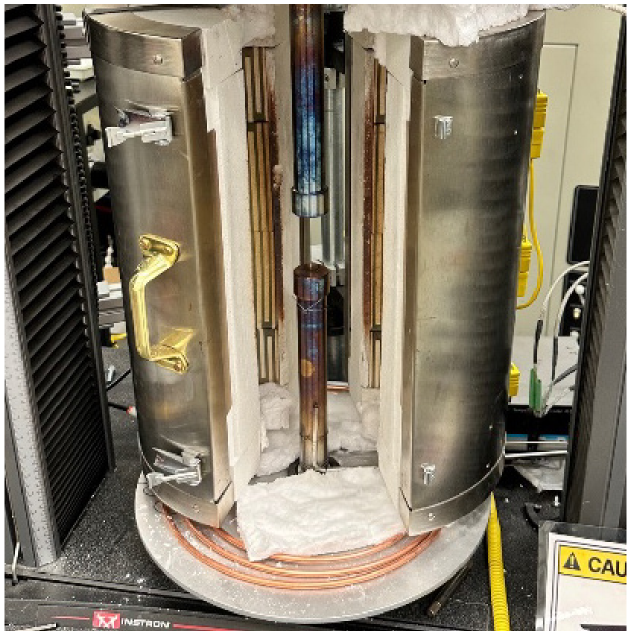

Figure 3. Position of thermocouple at midline to furnace.

A stainless-steel tubing line introduced nitrogen from the bottom of the furnace. Nitrogen was flowed at 20 liters per minute during the testing to mitigate oxidation of the graphite during the elevated testing. Figure 4 illustrates the stainless-steel tubing location on the bottom of the furnace and how it is secured to the lower test fitting. The thermocouple was secured on the opposite side from the nitrogen tubing. For this mechanical load frame, the bottom fitting stays in a fixed position, while the top platen moves downward to apply a mechanical load to the specimen. Figure 5 shows a surface thermocouple taped to the outer surface of the load frame load cell to ensure the manufacturer's load frame temperature limit is not exceeded.

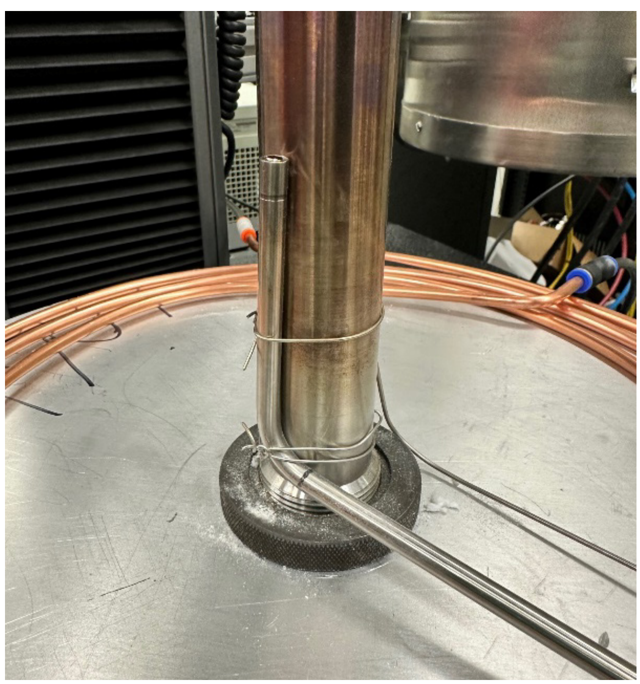

Figure 4. Nitrogen tubing on the front side and the thermocouple on the back.

Figure 5. Surface thermocouple taped (blue) to load cell.

Figure 6 shows the complete test equipment setup including the load frame, furnace, and external thermocouple readout and nitrogen gas controller. This same setup was utilized for both the compression strength testing and the split-disc testing.

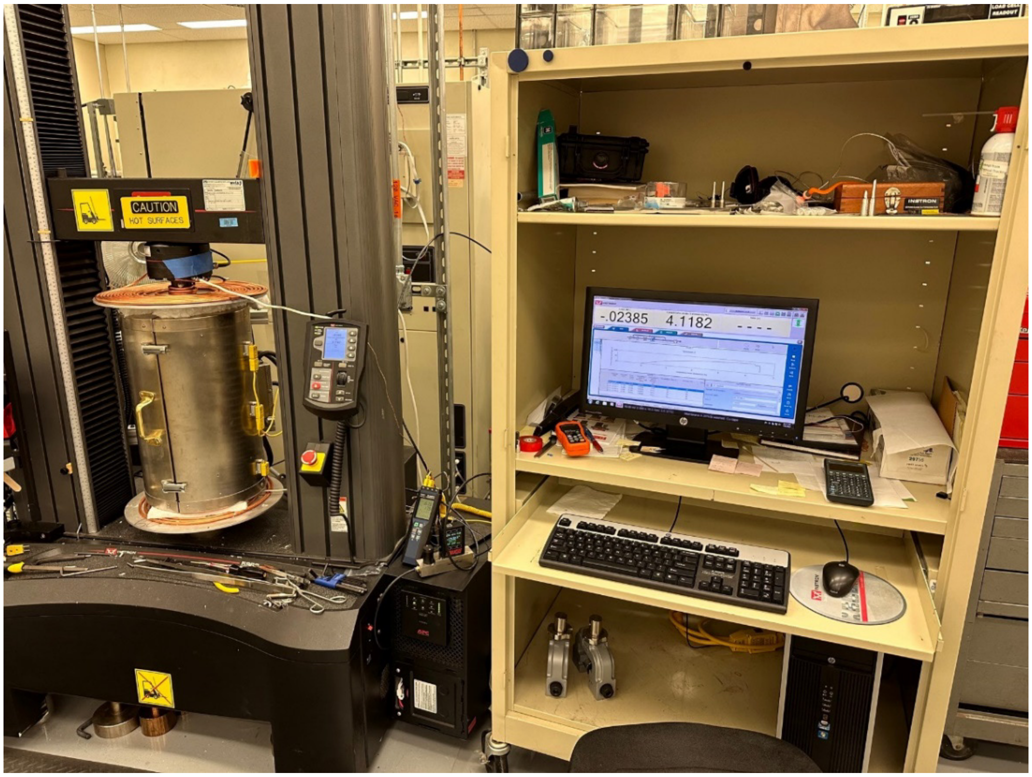

Figure 6. Overall complete test equipment setup.

2.1 Compressive Testing

The compressive strength testing at ambient temperature is governed by ASTM C695, "Standard Test Method for Compressive Strength of Carbon and Graphite" [1]. The same Instron Bluehill control program utilized for the ambient testing was also used for the high-temperature testing. However, several modifications to the controller were necessary. Since the furnace was not designed to be used with an extensometer, the crosshead extension was utilized in the strain calculations. This simplification to determine strain was deemed appropriate because ultimate tensile strength (UTS) was the primary measurement for these initial studies. Elevated-temperature strain outputs using this simplified method are acceptable for these initial comparisons to results from the ambient mechanical strength testing.

The graphite compressive strength samples are right cylinders measuring 12.5 mm in diameter and 25.4 mm in length in standard advanced graphite creep (AGC) geometries. Figure 7 shows an example of this compressive sample. Samples were machined from the following grades and orientations of graphite: IG-110, 2114, NBG-18 (WG), NBG-18 (AG), PCEA (WG), and PCEA (AG). The NBG-18 and PCEA had two sets of samples machined, one from the "with-grain" (WG) direction and one from the "against-grain" (AG) direction. Five replicates were tested from each grade/orientation at room-temperature (20°C) and at 500°C. A sixth replicate was available and tested for NGB-18 WG and NGB-18 AG.

A methodology for testing at elevated temperatures was developed by first bringing the empty furnace to the desired temperature. The furnace was minimally opened by a few inches and the sample placed in the center of the bottom compression fitting surface with a pair of tongs. The samples were soaked for 20 minutes after reaching the temperature setpoints, and read from the external thermocouple at the furnace centerline. After each test was completed, power was shut off from the furnace so it could be cracked open several inches. A pair of long tongs was used to remove the fragments of the tested sample from the furnace and to place a new sample on the lower fixture. Then, the furnace was closed, and the power turned back on. After the sample reached the temperature setpoint, it was soaked for an additional 20 minutes. Typically, the furnace only lost 60°C during a typical sample changeout.

Figure 7. Typical AGC compressive strength graphite sample.

2.2 Split-Disc Testing

The split-disc testing at room-temperature is guided by ASTM D8289, “Standard Test Method for Tensile Strength Estimate by Disc Compression of Manufactured Graphite” [2]. As for the compression testing, the same Instron Bluehill program was utilized for the elevated-temperature testing, with several modifications from the ambient temperature version. Since the furnace was not designed for use with an extensometer, the crosshead displacement was again utilized for any strain measurements. Because the splitting tensile strength is calculated from the maximum compressive load at break, these simplified strain measurements would not affect the results.

The same basic setup with the Instron load frame and furnace was utilized for the splitting tensile strength testing. The split-disc samples are button-shaped and measure 12.7 mm in diameter and 6.35 mm thick. They are placed in a Hastelloy anvil fitting that holds it in place during testing with the 12.7 mm diameter facing out. The load frame compresses the anvil until the sample fractures through the vertical axial centerline of the sample [2]. It should be noted that in some tests the centerline fracture can be driven all the way to an edge of the disc sample. Figure 8 displays the button sample and anvil fitting used in the testing.

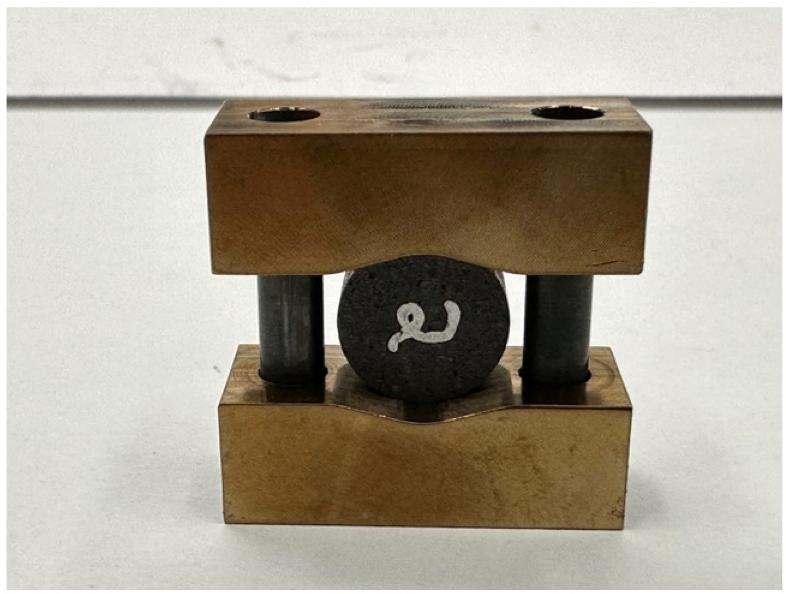

Figure 8. Two-piece anvil and sample (left) and in test configuration (right).

A fracture from a successful split-disc test is shown at 10× magnification in Figure 9. The initial split-disc testing procedure was identical to the compression testing procedure, where tests at 20°C were compared to 500°C and 800°C. The same graphite grades of 2114, IG-110, NBG-18, and PCEA were tested. Five replicates of each grade and orientation were tested, 2114, IG-110, NBG-18-WG, NBG-18-AG, PCEA-WG, and PCEA-AG.

For the elevated-temperature testing, the furnace was empty as it was first brought to temperature. Then the furnace was cracked open a few inches and the anvil with the sample was placed in the center of the bottom compression fitting surface with a pair of tongs. The samples were soaked for 20 minutes after reaching the setpoints, and read from the external thermocouple at the furnace centerline. After each test was completed, power was shut off from the furnace so it could be cracked open several inches. A pair of long tongs was used to remove the anvil of the tested sample and to place a new anvil and sample on the lower fixture. Then, the furnace was closed, and the power turned back on. After the sample reached the setpoint, it was soaked for an additional 20 minutes. Typically, the furnace only lost 60°C during the sample changeout. A set of three anvils was used during the testing: one heating inside the furnace, one cooling off outside the furnace, and one at room-temperature ready to be tested next.

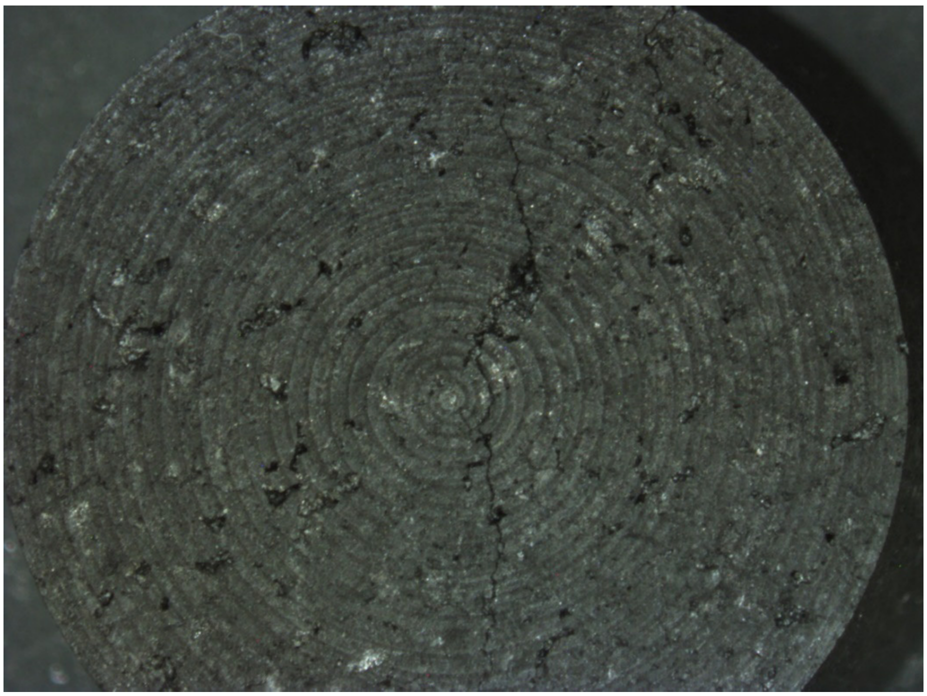

Figure 9. Vertical fracture from successful split-disc test.

Figure 10 shows a test anvil being placed into the furnace with the long pair of tongs. After several tests at 500°C, the anvils began to jam and not slide smoothly due to oxide buildup on the pins and holes.

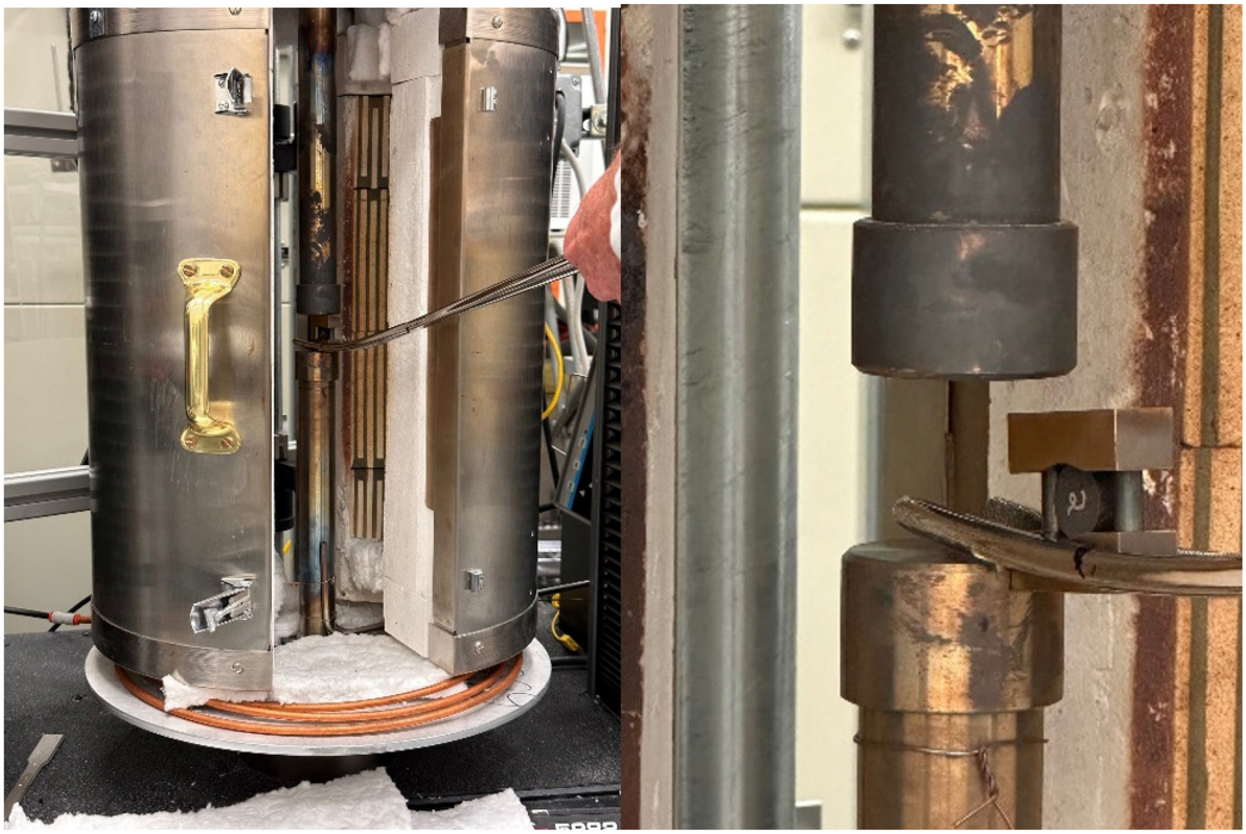

Figure 10. Split-disc anvil being placed into furnace.

The oxide was easily removed by using Scotch-Brite scrubbing pads on the pins, and a bore brush on the penetrations. Figure 11 shows the bore brush cleaning out the guide holes for the pins. The bore brush was mounted in a cordless drill and run for 3–4 seconds on each guide hole.

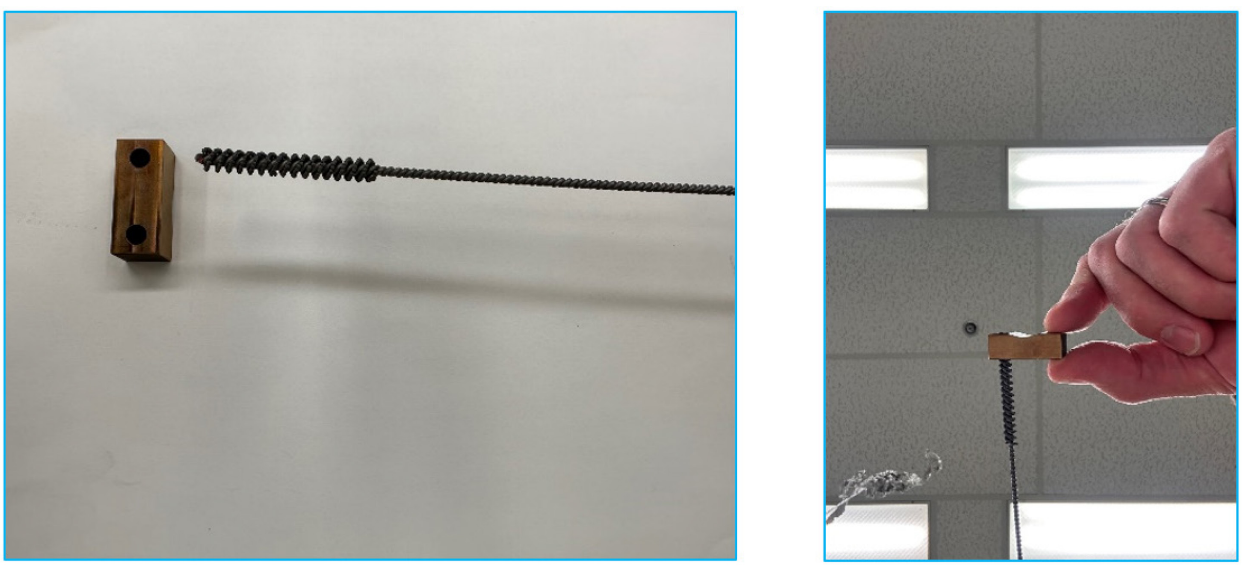

Figure 11. Bore brush for cleaning out the guide holes.

3. MEASUREMENTS AND RESULTS

The following plots present the compressive strength results from the first five samples tested of each of the examined graphite grades. Anisotropic grades were tested in both the WG and AG direction. Following are the split-disc tensile strength results.

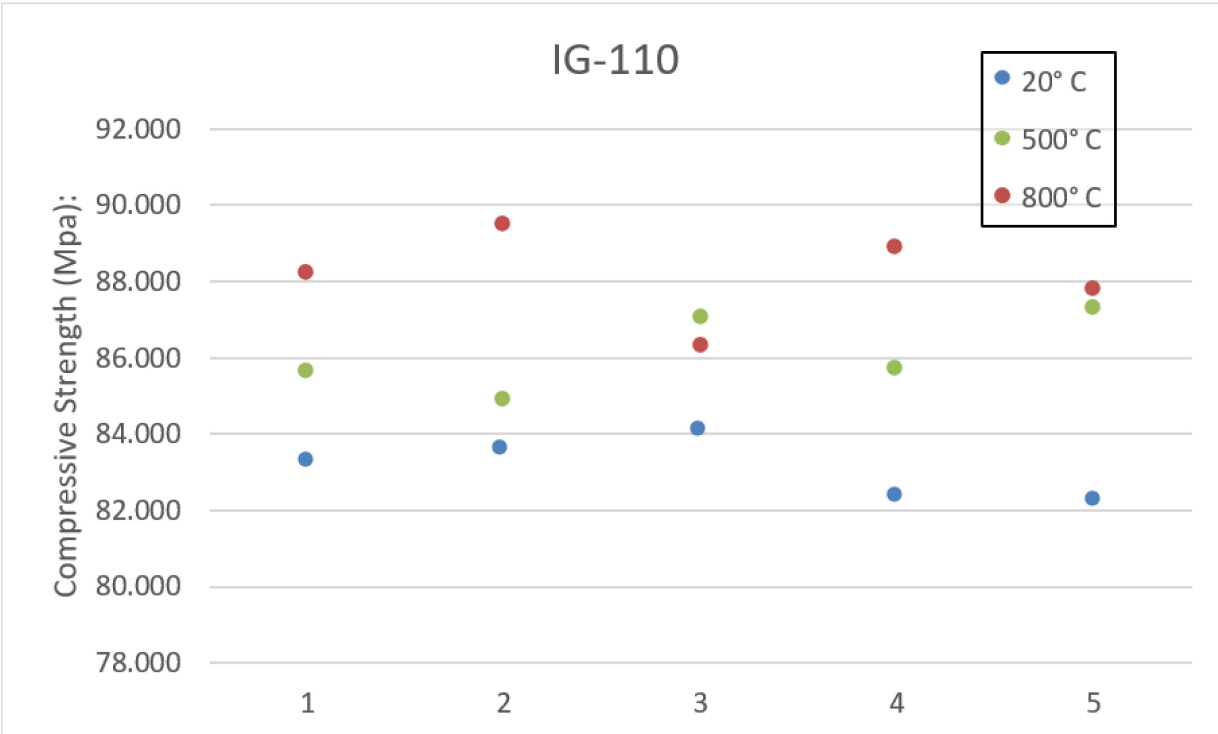

Figure 12. IG-110 compressive strength change with temperature.

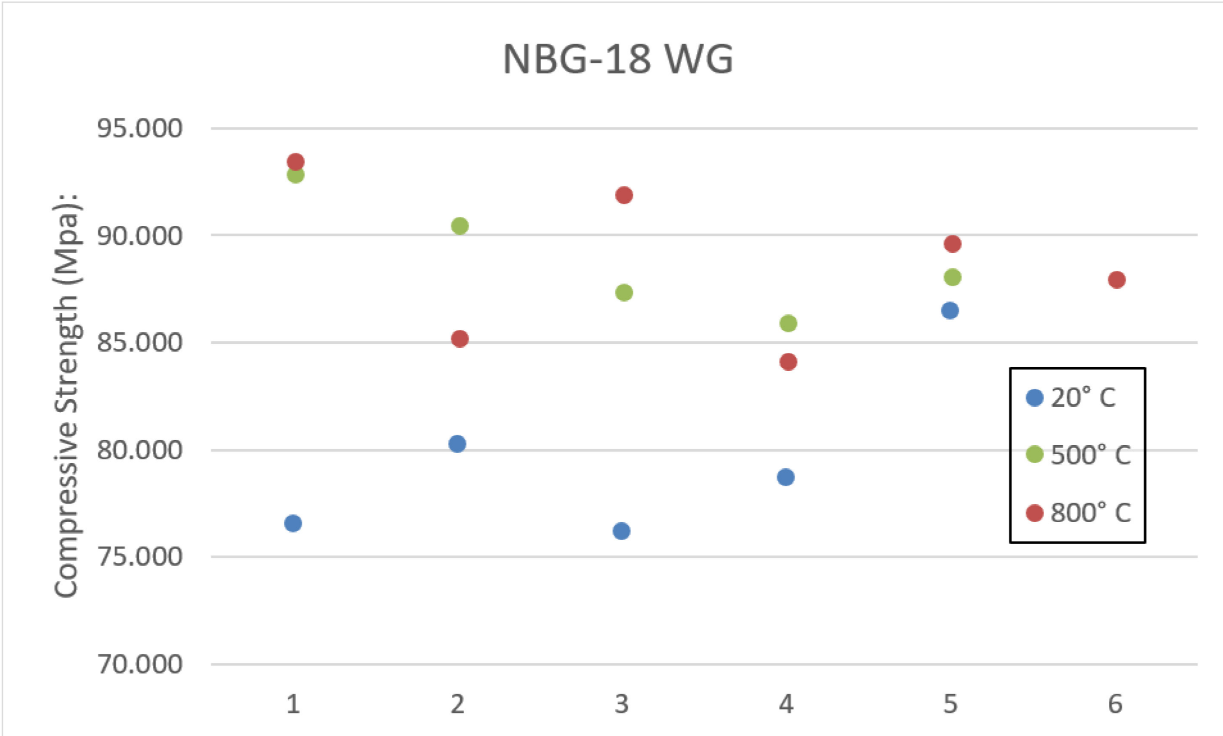

Figure 13. NBG-18 WG compressive strength change with temperature.

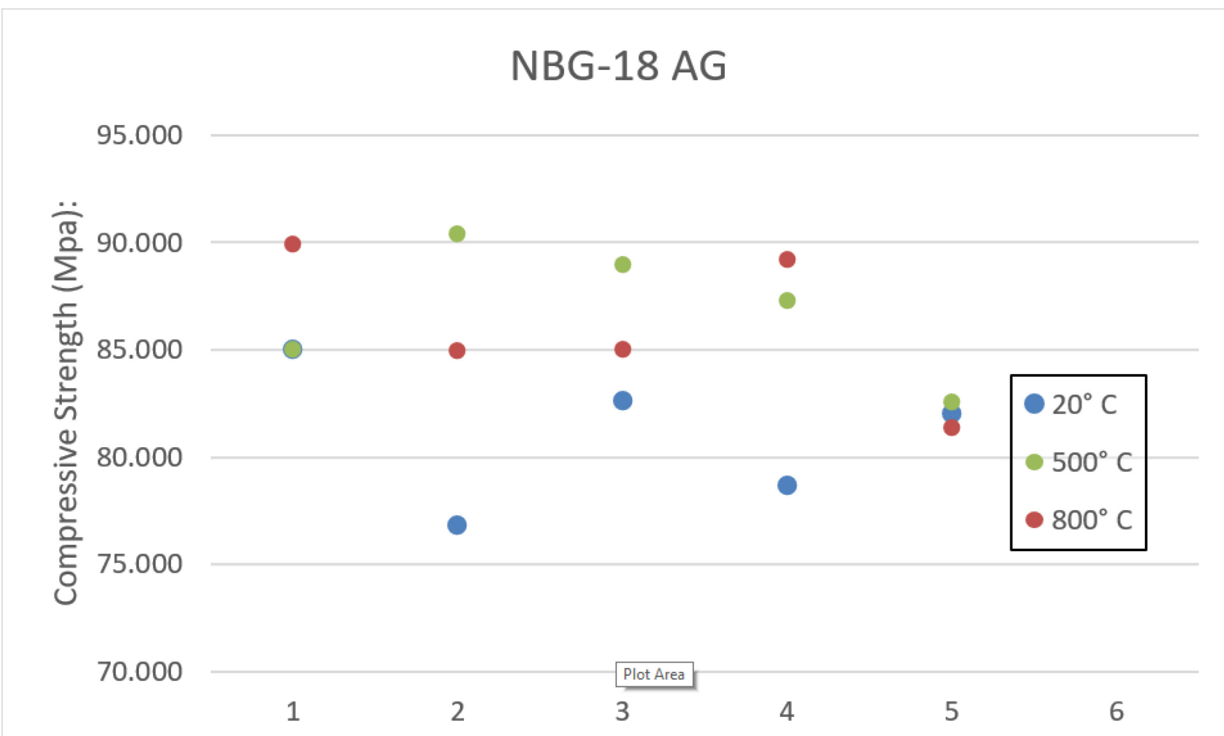

Figure 14. NBG-18 AG compressive strength change with temperature.

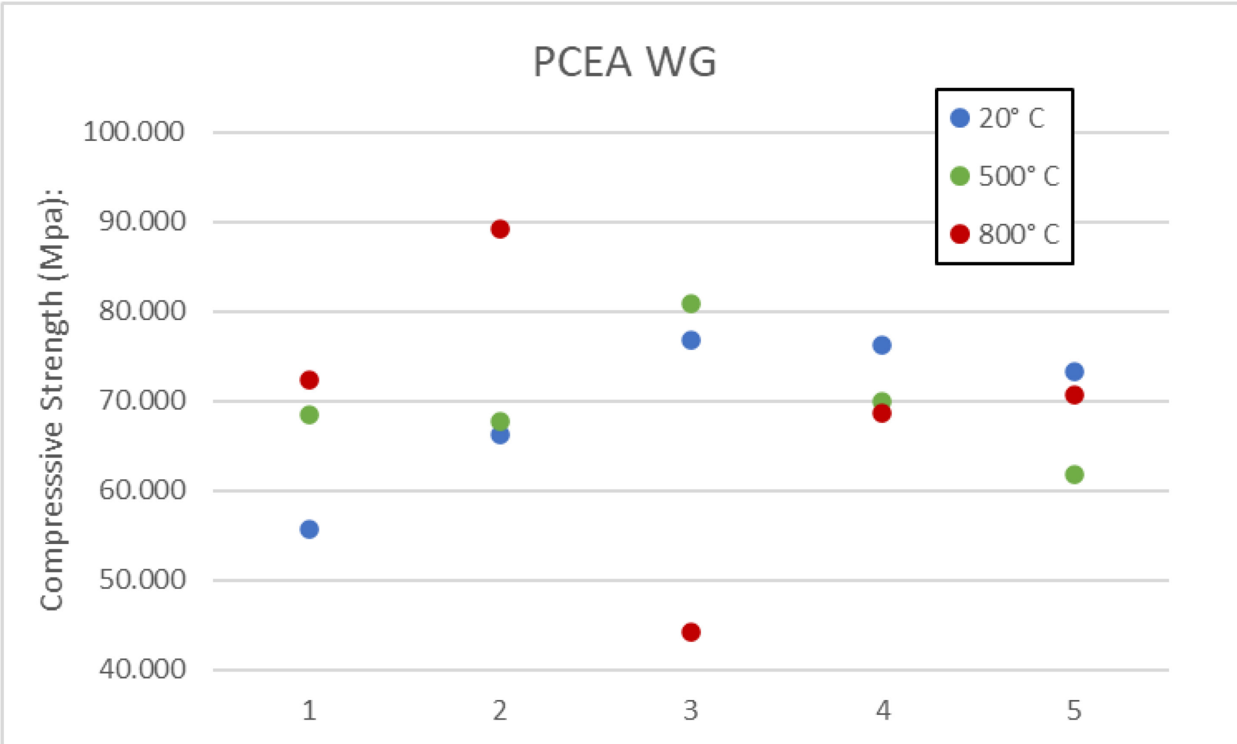

Figure 15. PCEA WG compressive strength change with temperature.

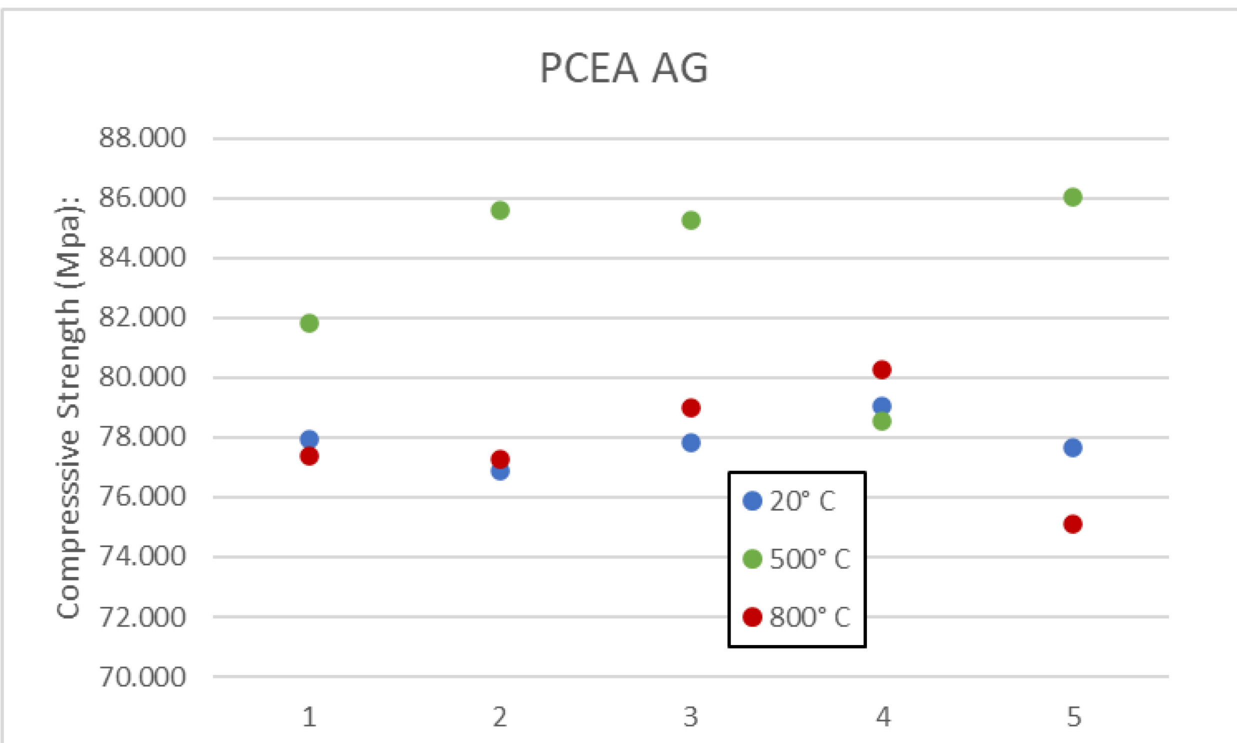

Figure 16. PCEA AG compressive strength change with temperature.

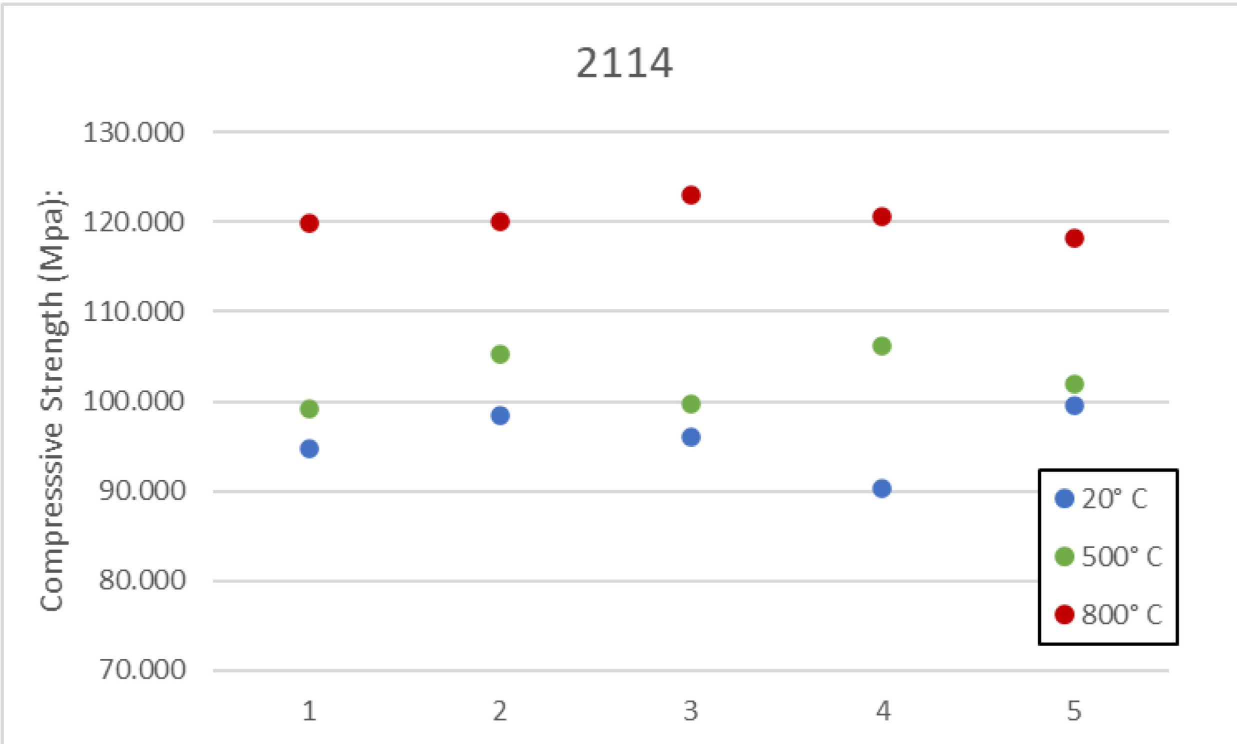

Figure 17. 2114 compressive strength change with temperature.

In all instances, excluding PCEA, the compressive strength increases with temperature. In the fine-grain graphite grades IG-110 and 2114, the strength continues to increase at higher temperatures, whereas the medium-grain NBG-18 does not appear to increase with temperature. Larger scatter within the PCEA and NBG-18 results may be attributed to their larger grain size since these sub-sized compression specimens are sensitive to the material grain size, as well as any inherent defects that can cause variability as well.

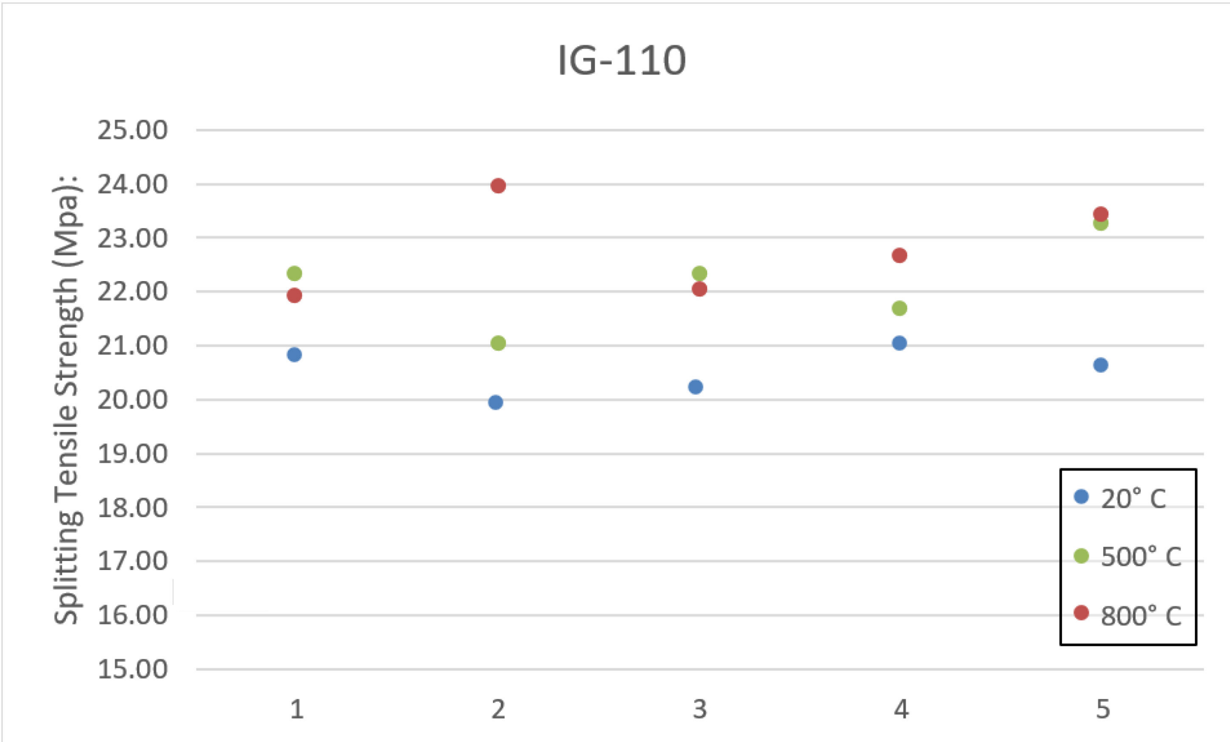

Figure 18. IG-110 split-disc tensile strength change with temperature.

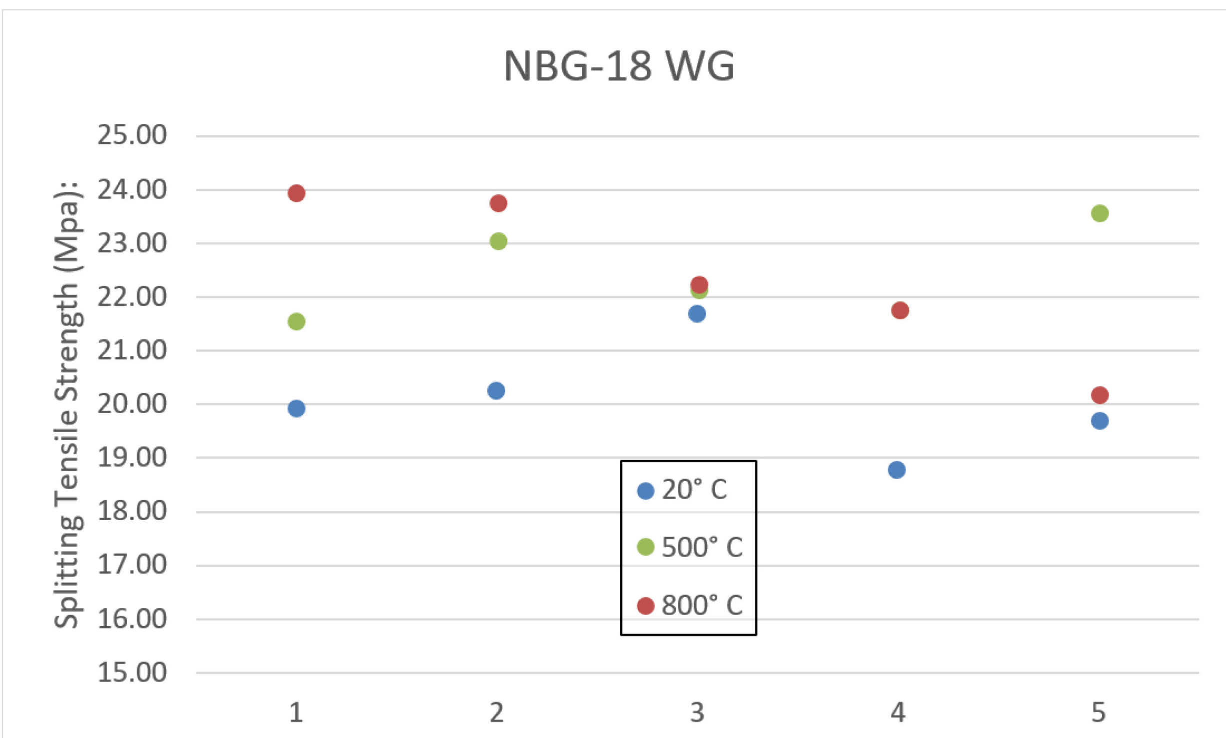

Figure 19. NBG-18 with-grain split-disc tensile strength change with temperature.

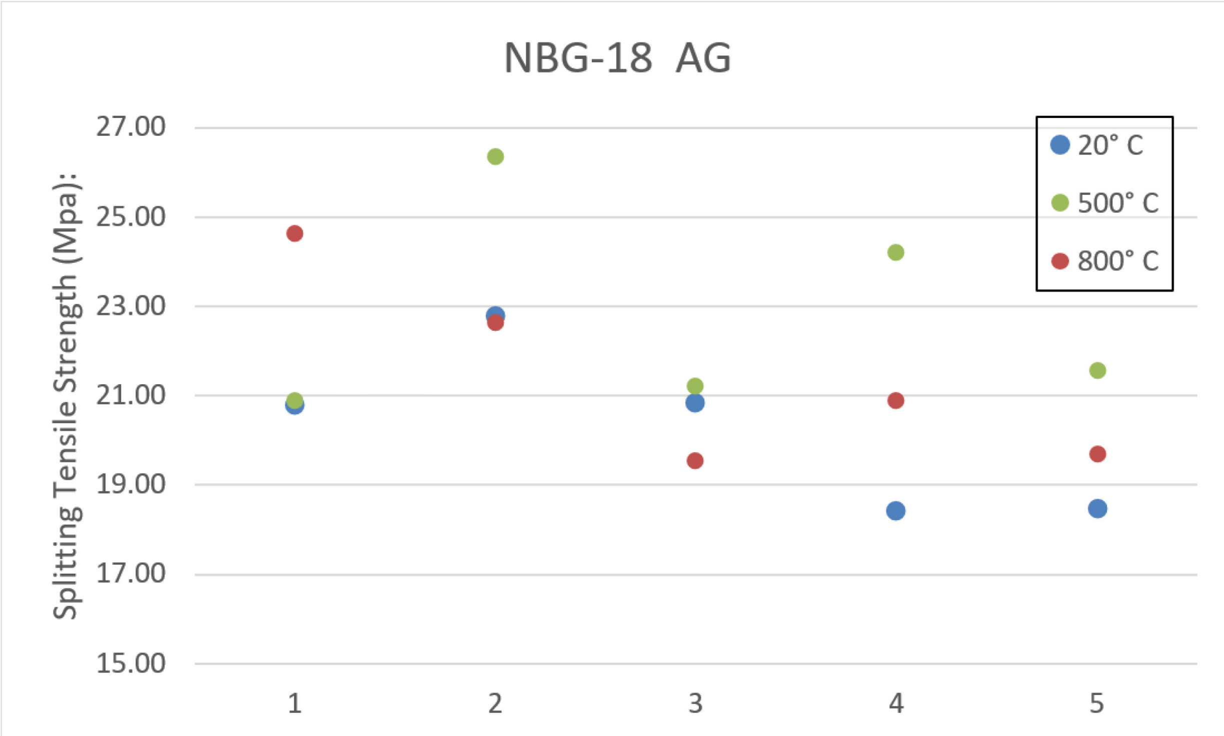

Figure 20. NBG-18 against-grain split-disc tensile strength change with temperature.

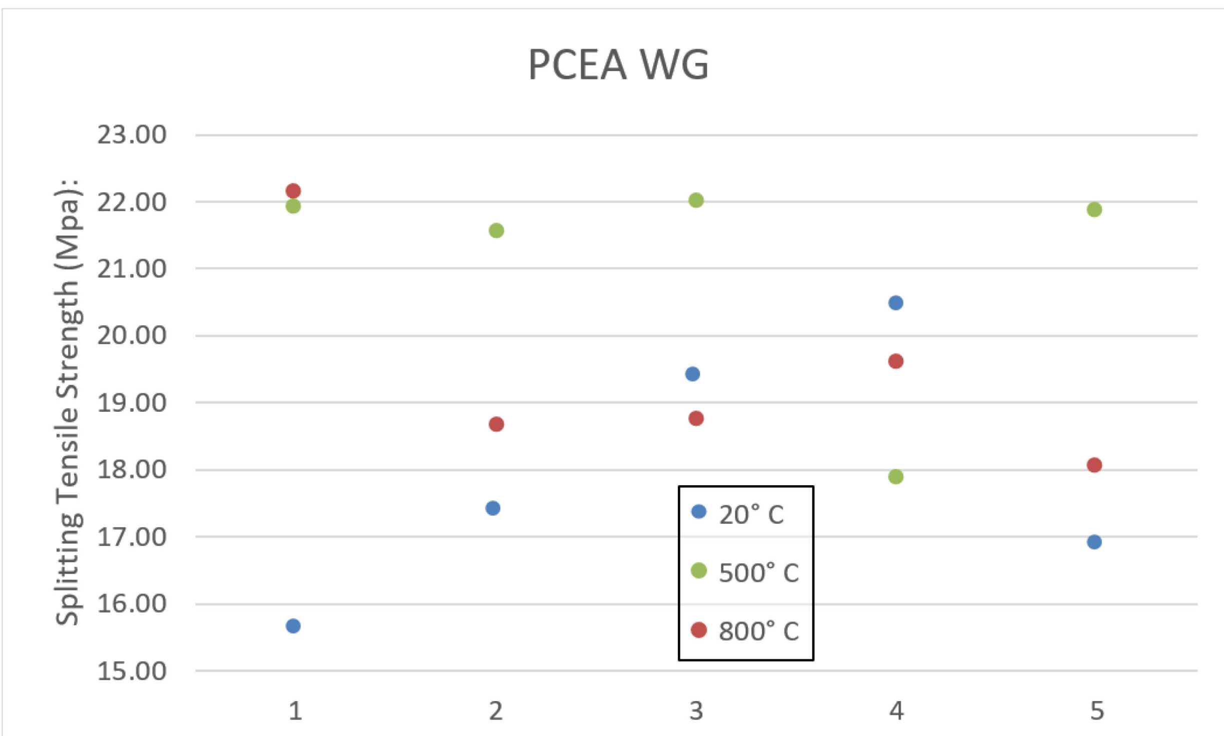

Figure 21. PCEA with-grain split-disc tensile strength change with temperature.

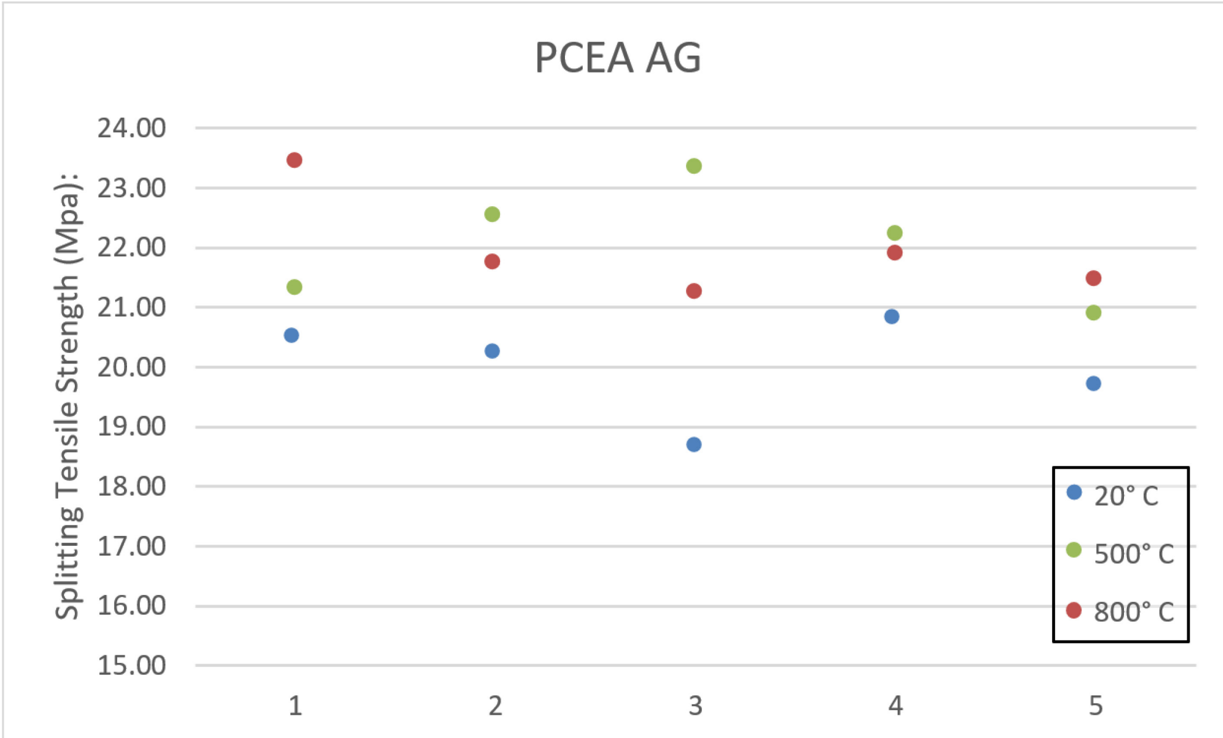

Figure 22. PCEA against-grain split-disc tensile strength change with temperature.

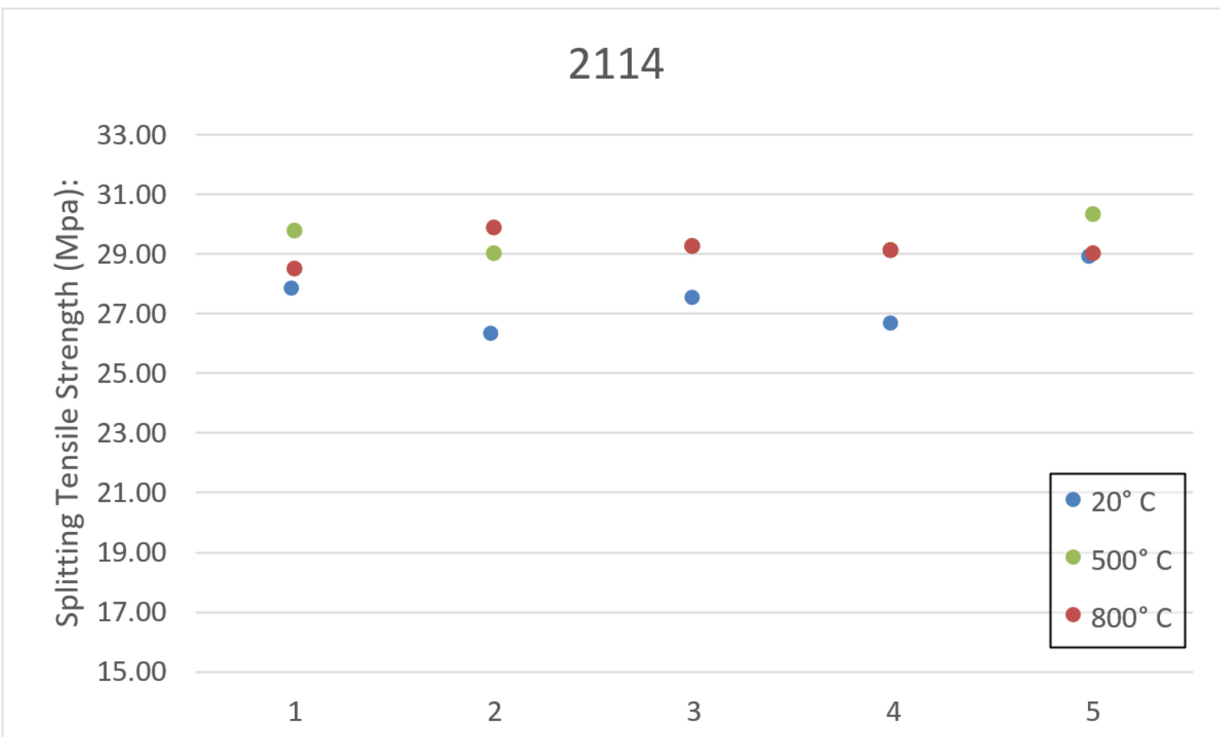

Figure 23. 2114 split-disc tensile strength change with temperature.

Again, accounting for material variability, the strength increases with temperature with the finer grain graphite grades display a more consistent increase with temperature. The average increase in strength for each case is tabulated in Table 1.

Table 1. Average strength increase for each graphite grade and temperature.

Compressive Strength		
	Temperature (°/C)	Average % Strength Increase
IG-110	500	3.6
	800	6.1
NBG-18 WG	500	11.7
	800	11.4
NBG-18 AG	500	7.2
	800	6.2
PCEA-WG	500	0.1
	800	-0.9
PCEA-AG	500	7.2
	800	-0.1
2114	500	7.0
	800	25.6
Splitting Tensile Strength		
	Temperature (°/C)	Average % Strength Increase
IG-110	500	7.9
	800	11.2
NBG-18 WG	500	11.7
	800	11.8
NBG-18 AG	500	12.7
	800	6.0
PCEA-WG	500	17.3
	800	8.4
PCEA-AG	500	10.4
	800	10.0
2114	500	7.8
	800	6.4

Table 1 more directly shows the inconsistencies with the larger grain samples with the PCEA grade appearing to weaken in compression at higher temperatures. As described previously, this may be due to the small specimen geometry and low sample numbers, resulting in statistical inconsistencies. More testing will be required with a larger number of samples and possible differing sample geometries.

4. REFERENCES

1. ASTM C695-21. 2021. “Standard Test Method for Compressive Strength of Carbon and Graphite,” American Society for Testing and Materials. West Conshohocken, PA. USA.
2. ASTM D8289-19. 2020, “Standard Test Method for Tensile Strength Estimate by Disc Compression of Manufactured Graphite.” American Society for Testing and Materials. West Conshohocken, PA. USA.