ORNL/TM-2020/1458

Mersen Grade 2114: A Comparison of Tensile Strength Data

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OAK RIDGE NATIONAL LABORATORY
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Materials Science and Technology Division

MERSEN GRADE 2114: A COMPARISON OF TENSILE STRENGTH DATA

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Prepared by
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managed by
UT-BATTELLE, LLC
for the
US DEPARTMENT OF ENERGY
under contract DE-AC05-00OR22725

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ABSTRACT

The ASTM Brazilian disc graphite strength (splitting tensile strength, σ_{sts}) test method (ASTM D8289) is of interest because the small-specimen geometry is compatible with that of environmental effects specimens, thus allowing for environmental effects such as irradiation, irradiation creep, or oxidation on tensile strength to be investigated. The Brazilian disc strength of Mersen grade 2114 graphite is reported and compared with strength data previously obtained using larger cylindrical ASTM dog-bone specimens.

1. INTRODUCTION

The tensile strength determined by the Brazilian disc[1] method for Mersen 2114 grade graphite is reported and compared with the tensile strength determined previously[2] from ASTM C749 dog-bone type samples.[3]

Recently, a small-specimen Brazilian disc method[1] was added to the ASTM standards and is available for use. The small size of the specimen makes it compatible with irradiation specimens. The method allows a small graphite disc, stressed in compression between curved anvils to reduce Hertzian contact stresses,[4] to fail under the induced vertical diametral tensile stress, thus yielding a tensile strength. One of the purposes of this work is to compare the tensile strength determined by the Brazilian disc method with that obtained from conventional ASTM C749 dog-bone specimens.

Although this specimen geometry is compatible with that of irradiation specimen geometries, it is possible that the shape of the irradiation sample will have changed as a result of irradiation and thus may require some machining before use if the ASTM standard test fixture is to be used.

In this work, two discs, 6 mm in diameter and 3 mm thick, were machined from the heads of 24 type 109 ASTM C749 (dog-bone) unirradiated broken tensile specimens, yielding a total of 48 disc specimens. Half (24) of the specimens were strength tested; the duplicate specimens will be used for future determinations of the effect of chronic oxidation on tensile strength. The C749 specimens were all from the center slab of a Mersen grade 2114 billet (No. 116310). Previous testing[2] established that grade 2114 graphite could be considered isotropic; thus, the perpendicular orientation (to the C749 fracture) of the discs expected fracture should not be significant.

2. EXPERIMENTAL

2.1 MATERIALS

The disc specimens were prepared from the heads of previously tested[2] ASTM C749 type 109 samples from a slab 5 (billet center) of a Mersen 2114 graphite (billet number 116310). The in-billet location of the ASTM C749 tensile specimens used for disc preparation is shown in Figure 1. Mersen grade 2114 is a fine-grain (13 µm) isotropic, isostatically molded graphite.

2.2 TEST METHOD

All disc testing was conducted in accordance with the ASTM standard.[1] The Brazilian disc geometry used is shown in Figure 2. An Instron 4465 test instrument was used at a loading rate of 0.4572 mm/min (0.018 in/min). The lower platen used on the test frame was fitted with a spherical seating as required by the ASTM disc standard[1] (Figure 3). The specimen was located between two radiused bearing blocks (Figure 4) to minimize the Hertzian contact stresses.[1,4] The Instron test machine had a 2 kN (450 lbf) capacity load cell (signal-to-noise ratio 73750). The test lab temperature was 21.6°C and the relative humidity was 22%. Splitting tensile strength, σ_{sts}, (MPa) was calculated from

$$\sigma_{sts} = 0.931\frac{P}{\pi LR},$$

where P = maximum applied load, N (lbf)
L = thickness of specimen, mm (in.)
R = specimen radius, mm (in.).

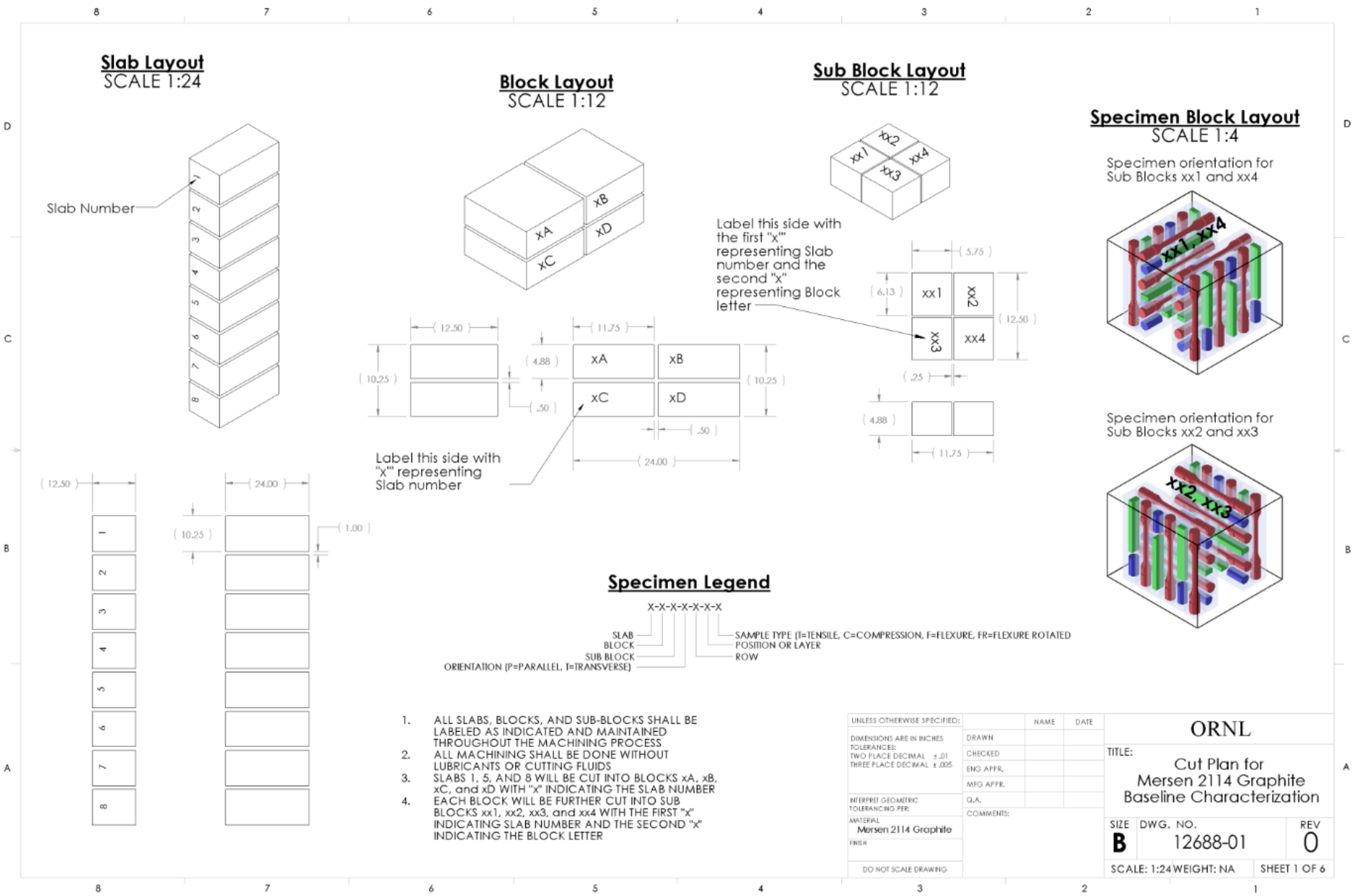

Figure 1. Mersen 2114 billet 116310.

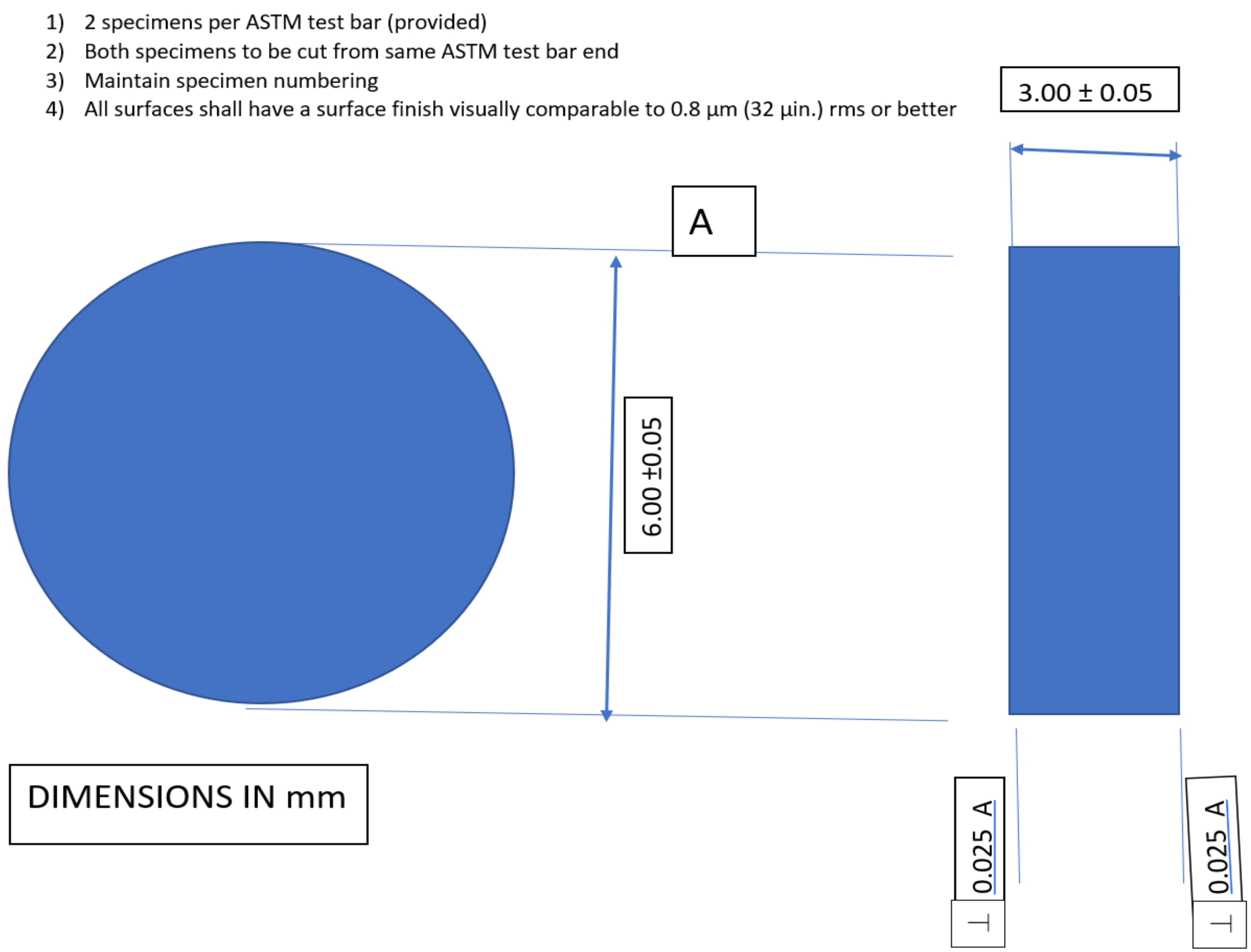

Figure 2. Brazilian disc specimen geometry used in this work.

Figure 3. Instron 4465 test frame showing spherical lower platen used for the testing.

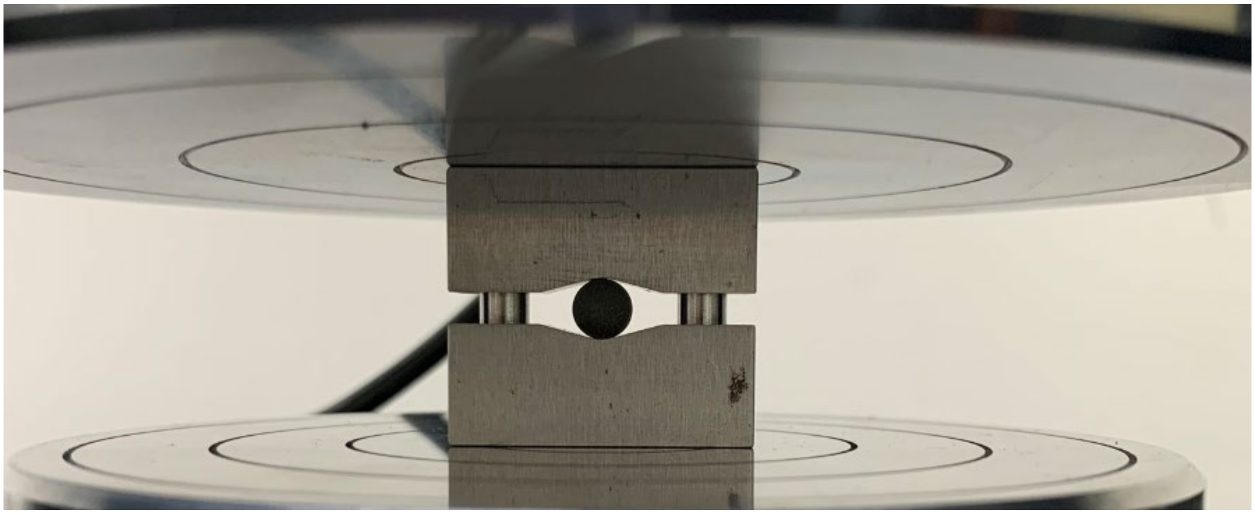

Figure 4. Tool steel blocks radiused to limit contact stress.[1,4]

3. RESULTS AND DISCUSSION

The ASTM disc standard[1] mandates that the sample diameter be at least twice the process zone (PZ) size as calculated from the ASTM D7779[5] value of K_{Ic}. The PZ is taken from [6]

$$Process\ Zone\ Size, \qquad r_c = \frac{1}{2\pi}\left[\frac{K_{Ic}}{\sigma_t}\right]^2,$$

where K_{Ic} is the critical stress intensity factor obtained in accordance with ASTM D7779, and σ_t is the tensile strength.

Taking K_{Ic} as 1.15 MPa√m and σ_t as 29 MPa gives a PZ size of 0.25 mm. Thus, an acceptable specimen should exceed 0.5 mm in diameter. The 6.00 mm diameter specimen used here was therefore D8289 compliant.

The results of disc testing are summarized in Table 1 and reported in full in (Appendix A). The data are plotted as a histogram in Figure 5 and show the remarkable consistency of the Brazilian disc tensile strength data for grade 2114.

Table 1. Summary of tensile strength data for Mersen 2114 graphite obtained from ASTM C749 specimens and ASTM D8289 disc specimens

Tensile strength ASTM method	Mean TS (MPa)	Standard deviation of TS (MPa)	Population (n)
C749	28.63	3.22	24
D8289 (disc)	29.17	0.82	24

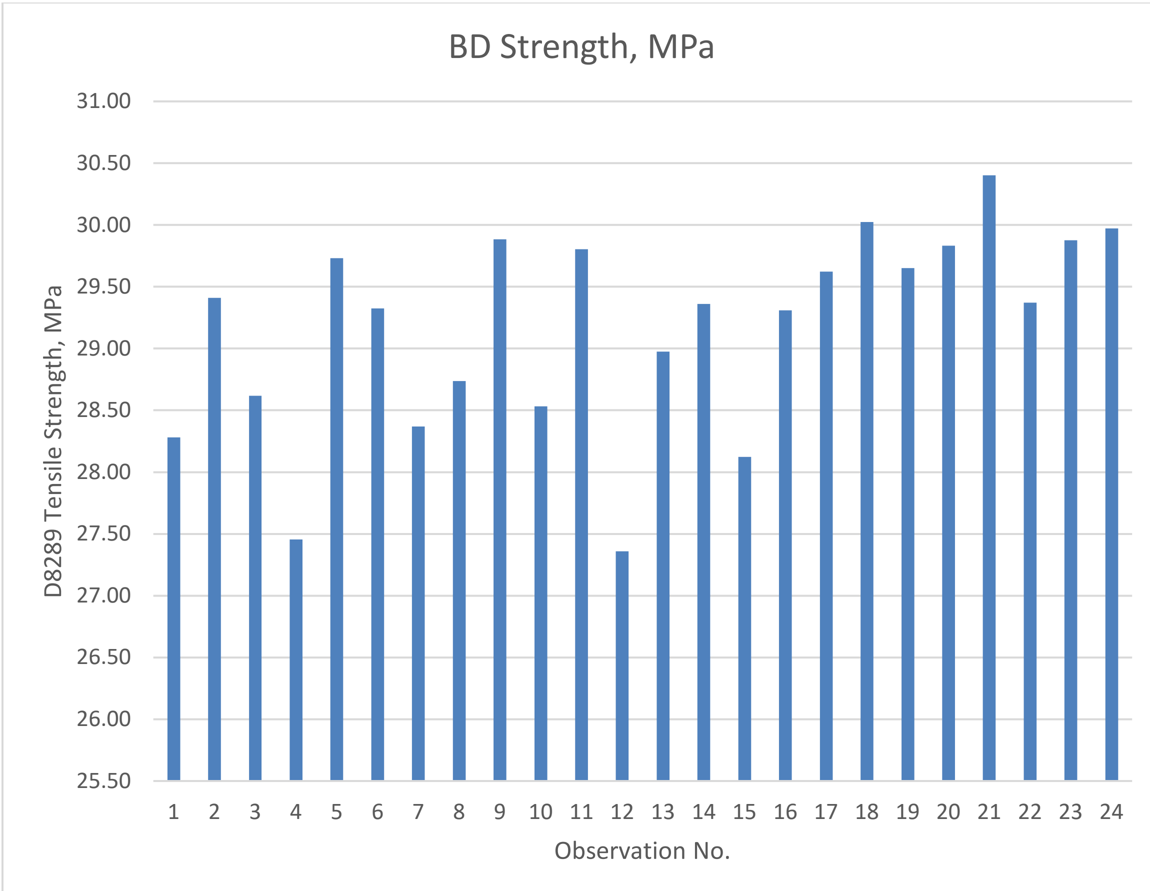

Figure 5. Brazilian disc tensile strengths.

Most of the fractures were vertical and initiated at the specimen center (Figure 6). A few of the cracks branched but still appeared to initiate at the specimen center, and the resulting disc strength appeared to be appropriate.

The mean disc tensile strength appeared to be slightly larger than the mean tensile strength previously obtained with C749 samples. This could be due to a Weibull stress volume effect (a smaller stressed volume causes higher strength because of the decreased likelihood of a critical flaw).

The difference between the two means (Table 1) was significance tested[7] to determine if the mean values were statistically different. The significance threshold was set at P=0.05. The outcome of the two-tailed significance test was P=0.43 (≥0.05 – the threshold value). Thus, the difference between the means was considered "not statistically significant."

For a fine-grain graphite such as Mersen 2114 (grain size = 13µm), the two methods of obtaining the tensile strength appear comparable. The disc method is particularly attractive because its small size is compatible with the irradiation specimen geometry. However, changes in specimen shape upon irradiation may mean specimen remachining is required. Moreover, the applicability of the Brazilian disc method to a larger-grain graphite should not be assumed, especially for a 6 mm specimen diameter.

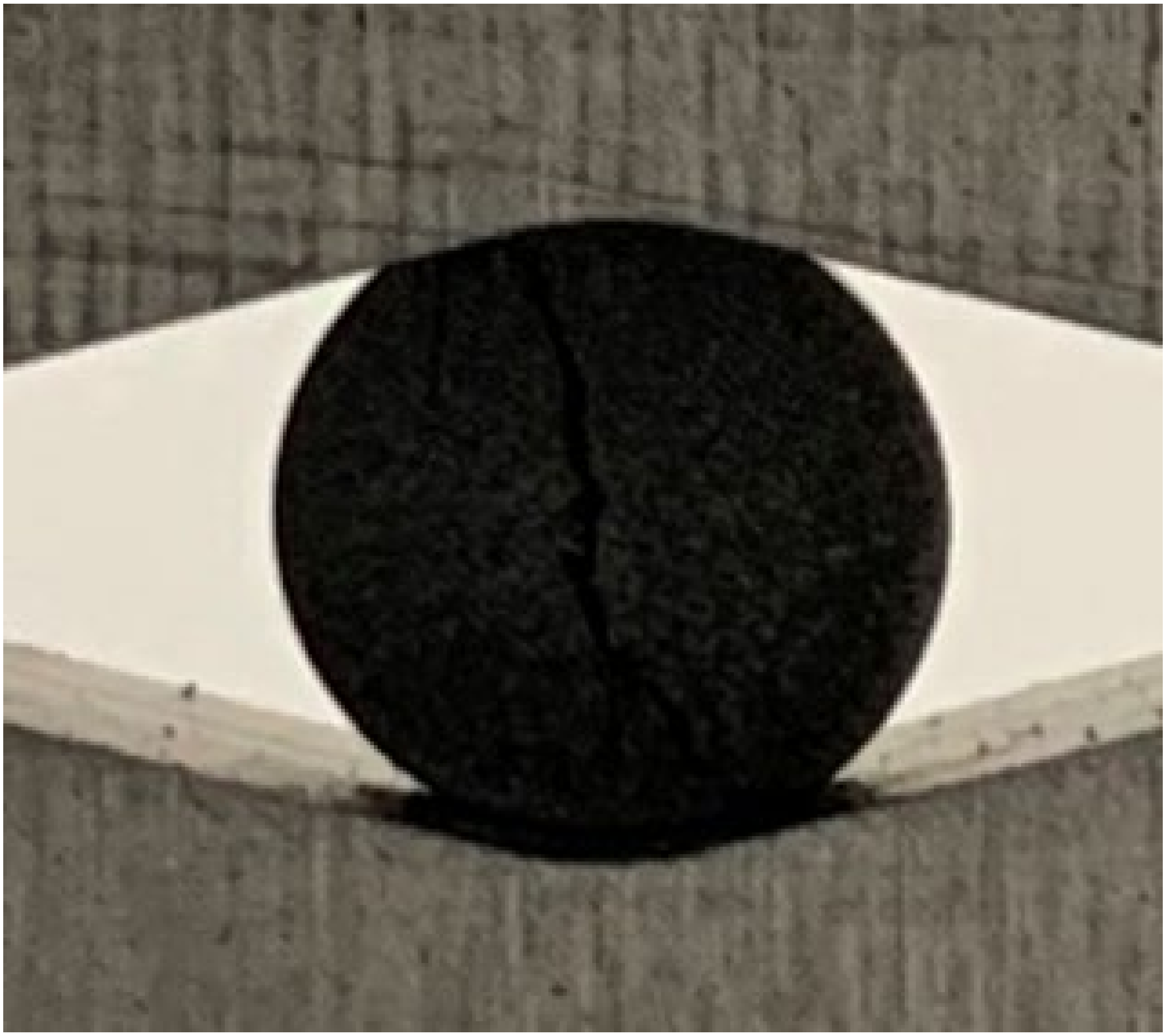

Figure 6. Tensile failure of specimen 5A1T2L1T showing vertically centred fracture.

4. CONCLUSIONS

The mean tensile strength for Mersen 2114 as determined from ASTM D8289 discs[1] was 29.17 MPa with a standard deviation of 0.82 MPa. The mean tensile strength as previously determined from ASTM C749 type 109 samples[2,1] was 28.63 MPa with a standard deviation of 3.22 MPa. The 6 mm diameter disc was found to be a valid test method for this grade of fine-grain graphite.

5. QUALITY ASSURANCE

The described technical work scope and related activities were conducted in accordance with the applicable requirements of the ASME NQA-1-2008 standard (including the 1a 2009 Addendum) entitled *Quality Assurance Requirements for Nuclear Facility Applications.* Project- and activity-specific information concerning ORNL's application of the standard's requirements is provided in Document #QAP-ORNL-NR&D-01, *Quality Assurance Plan for Nuclear Research and Development Conducted at the Oak Ridge National Laboratory.*

6. ACKNOWLEDGMENTS

This work was supported by the US Department of Energy, Office of Nuclear Energy, Advanced Research and Technology—Materials Program. The author wishes to acknowledge the technical assistance of Ashli Clark and Rick R. Lowden.

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[2] T. D. Burchell, R. D. Lowden, and K. W. Monaghan, *Grade 2114: Preliminary Report of Selected Elastic and Mechanical Properties*, ORNL/TM-2018/1038, October 2018.

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[4] H. Li. A.S.L. Fok, and B. J. Marsden, "Evaluation of the Brazilian Disc Test in Fracture Strength Measurement of Nuclear Graphite," in *Management of Ageing in Graphite Reactor Cores*, G. B. Neighbour, ed., pp.75–82 (2007).

[5] ASTM D7779, *Standard Test Method for Determination of Fracture Toughness of Graphite at Ambient Temperature*, Annual Book of ASTM Standards, ASTM International, West Conshohocken, PA 19426-2959, USA

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[7] "QuickCalcs," GraphPad, https://www.graphpad.com/quickcalcs/ttest1/?Format=SD. Accessed January 2020.

APPENDIX A. EXPERIMENTAL DATA

Table A-1. Tensile strength data from D8280 Brazilian disc testing.

SPEC Number	orientation of C749 spec	C 749 Tensile Strength, MPa	dia 1 -0 (mm)	dia 2 - 90 (mm)	Mean dia (mm)	t1	t2 (center) (mm)	t3	mean t (mm)	Mass (mg)	Vol (mm^3)	Density (g/cm^3)	Crack Location and direction	failure Load (N)	D8289 Tensile Strength MPa
5A1P1P2T	AG	24.87	6.042	6.043	6.043	3.019	3.019	3.012	3.017	156.42	86.507	1.808	Center to off center left	869.80	28.28
5A1P1P4T	AG	30.42	6.050	6.055	6.053	2.982	2.989	2.989	2.987	154.75	85.930	1.801	Two vertical cracks	897.00	29.41
5A1P1P6T	AG	26.83	6.044	6.042	6.043	2.999	3.002	3.001	3.001	155.56	86.062	1.808	Vertical Center	875.50	28.62
5A1P4P2T	AG	21.54	6.055	6.061	6.058	2.975	2.978	2.980	2.978	155.22	85.827	1.809	Vertical Center	835.60	27.46
5A1P4P4T	AG	26.80	6.031	6.032	6.032	3.004	3.006	3.002	3.004	155.32	85.830	1.810	Vertical Center	908.90	29.73
5A1P4P6T	AG	30.79	6.045	6.044	6.045	3.004	3.002	3.001	3.002	155.89	86.153	1.809	Vertical top to split lower	897.90	29.33
5AIT2L1T	WG	23.01	6.042	6.050	6.046	2.965	2.971	2.972	2.969	154.41	85.248	1.811	Vertical Center	859.30	28.37
5AIT2L3T	WG	28.10	6.033	6.035	6.034	3.012	3.014	3.015	3.014	155.94	86.178	1.810	Off center left-Diagonal to off center right	881.70	28.74
5AIT2L5T	WG	31.57	6.038	6.043	6.041	2.999	3.003	2.998	3.000	155.47	85.972	1.808	Off center left-Diagonal to off center right	913.70	29.88
5AIT3L1T	WG	31.87	6.042	6.035	6.039	3.038	3.037	3.035	3.037	157.24	86.965	1.808	Vertical Center	882.70	28.53
5AIT3L3T	WG	28.10	6.047	6.043	6.045	2.999	3.003	3.002	3.001	155.84	86.138	1.809	Vertical Center	912.30	29.80
5AIT3L5T	WG	31.36	6.033	6.032	6.033	2.997	2.998	2.994	2.996	154.73	85.640	1.807	Vertical Center	834.40	27.36
5B3P1P2T	AG	31.67	6.037	6.042	6.040	2.996	3.001	2.996	2.998	156.47	85.877	1.822	Vertical Center	885.10	28.98
5B3P1P4T	AG	26.05	6.043	6.045	6.044	3.026	3.029	3.029	3.028	157.70	86.875	1.815	Forked upper to center lower	906.60	29.36
5B3P1P6T	AG	32.06	6.052	6.052	6.052	2.960	2.959	2.958	2.959	153.61	85.120	1.805	Vertical Center	849.70	28.12
5B3P4P2T	AG	30.89	6.025	6.025	6.025	3.050	3.050	3.048	3.049	158.09	86.938	1.818	Vertical Center	908.50	29.31
5B3P4P4T	AG	23.22	6.031	6.039	6.035	2.991	2.997	2.995	2.994	156.08	85.653	1.822	Vertical Center	903.20	29.62
5B3P4P6T	AG	29.50	6.014	6.014	6.014	3.058	3.059	3.050	3.056	157.09	86.801	1.810	Vertical upper to right off center	930.90	30.02
5B3T2L1T	WG	29.69	6.041	6.043	6.042	3.002	3.008	3.007	3.006	156.72	86.177	1.819	Vertical Center	908.50	29.65
5B3T2L3T	WG	30.37	6.048	6.044	6.046	2.972	2.975	2.976	2.974	154.73	85.392	1.812	Forked uper to center lower	905.10	29.83
5B3T2L5T	WG	29.58	6.053	6.059	6.056	2.969	2.974	2.974	2.972	155.58	85.617	1.817	Vertical Center	923.30	30.40
5B3T3L1T	WG	25.11	6.038	6.042	6.040	3.031	3.034	3.027	3.031	156.99	86.836	1.808	upper left to lower right	907.10	29.37
5B3T3L3T	WG	31.90	6.050	6.044	6.047	2.995	2.997	2.987	2.993	156.21	85.956	1.817	Forked upper to center lower	912.32	29.88
5B3T3L5T	WG	31.90	6.038	6.044	6.041	3.005	3.014	3.015	3.011	157.12	86.311	1.820	Vertical Center	919.90	29.97