High temperature biaxial compressive strength of a commercial isotropic ultrafine grain graphite

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Abstract. Graphite is widely used in industrial applications, thanks to its excellent high temperature resistance. Therefore, its mechanical strength under combined stress at high temperature is an important issue for structural design. This work reports biaxial compressive data of an isotropic ultrafine grain graphite, the POCO EDM-3. Uniaxial tests have been first performed at room temperature with plane specimens, in order to obtain the stress strain curve for this material. Compressive tests have been then carried out with different notched geometries, both at room temperature and at 1000°C, in order to evaluate the biaxial compressive strength. Since plastic deformation was observed in uniaxial compressive tests with plane samples, elastic-plastic simulations have been performed with the FE code Ansys®, in order to assess the stress state at failure. The applicability of the Von Mises yield criterion has been evaluated, by comparing the equivalent Von Mises stresses at failure in different notched geometries. The tests demonstrated that, at room temperature, this type of graphite plastically deform in compression and that the Von Mises criterion can be used in order to evaluate the stress state. Moreover, the comparison between the failure loads at room temperature and at 1000°C showed that the resistance increases as the temperature increases.

1. Introduction

Thanks to high resistance at high temperature, low thermal expansion and high thermal conductivity, graphite is often used to design components working at high temperature. For its excellent thermal shock resistance, it is therefore used in a variety of industrial and research environments, such as metallurgical or the renewable energy industries. It is also chosen for the design of some devices in physical accelerators research facilities, such as the SPES project, currently in the installation phase at the Legnaro Laboratories of INFN, Italy ([1][2][3]). The project is aimed at the construction of a facility for the production of radioactive ion beams, for medical and physical research.

Numerous investigations on high temperature mechanical and thermal properties have been conducted on different types of graphite. Greenstreet [4], Price [5] and Green [6] presented reviews about the published mechanical properties data; Malmstrom [7] and Wagner [8] analysed some mechanical properties at high temperature; Smith [9], Martens[10], Green [11] and Gillin [12] performed uniaxial tensile or compressive tests over 2000°C. Biaxial mechanical properties at room
temperature have been presented by Broutman [13], Ely [14], Weng [15], Ho [16] and Eto [17], while Babcock [18] and Jortner [19] performed also high temperature biaxial tests, over 2000°C.

In these studies, different types of graphite have been analysed, as reported in table 1. However, the following conclusions are supported by all results: resistance increases with temperature up to 2500°C and, at higher temperature, significantly decreases; resistance increases with density; elastic modulus increases with temperature.

Nevertheless, some aspects are not completely clarified: graphite is generally considered as a brittle material ([20], [21], [22], [23]) and therefore an elastic behaviour can be expected. Moreover, in biaxial strength studies, failure criteria suitable for brittle materials have been often chosen, such as the Coulomb-Mohr theory, as the best interpolation with the experimental data ([16], [24], [25]). However, some authors highlighted a plastic deformation during compressive tests, also at room temperature ([12], [18], [19]). Moreover, Christensen [26] states that, relative to failure, all materials can behave either in a ductile or a brittle manner, depending upon the state of stress and other environmental influences.

In addition, graphite physical properties and characteristics may vary depending on the raw materials, the process, the microstructure and the particle size ([20],[21]).For these reasons, it is necessary to perform a dedicated characterization of a given graphite. In particular, in the present work, the POCO EDM-3 isotropic ultrafine grain graphite is studied.

Uniaxial compressive tests have been performed, in order to obtain the stress-strain curve at room temperature. The results showed a plastic deformation in compression for this type of graphite. In order to evaluate the biaxial resistance, hourglass-shaped specimens, characterized by different hourglass radius, have been tested in compression both at room temperature and at 1000°C. The validity of Von Mises criterion at room temperature has been evaluated by means of elastic-plastic simulations, imposing the failure loads registered during the tests.

Table 1. Designation and characteristics of graphite analysed in previous studies.

| Author          | Material Designation | Manufacturer                      | Description                                                                 |
|-----------------|----------------------|-----------------------------------|----------------------------------------------------------------------------|
| Malmstrom et al. [7] | AGX                  | National Carbon Company           | Extruded and graphitized at 2600°C; anisotropic; ρ=1.58 g/cm³                |
|                 | C-18                 | National Carbon Company           | Molded and graphitized at 2600°C; anisotropic; ρ=1.60 g/cm³                 |
|                 | SA-25                | National Carbon Company           | Molded and graphitized at 3000°C; isotropic with small crystallites; ρ=1.55 g/cm³ |
|                 | AUF                  | National Carbon Company           | Extruded and graphitized at 3000°C; anisotropic; ρ=1.67 g/cm³               |
|                 | AWG                  | National Carbon Company           | Molded and graphitized at 3000°C; anisotropic; ρ=1.76 g/cm³                 |
| Wagner et al. [8] | H4LM                 | Great Lake Carbon Company         | Extruded; maximum particle size 838.2 µm; ρ=1.72 g/cm³                      |
|                 | CK                   | Los Alamos Scientific Laboratories | Molded; ρ=1.71 g/cm³                                                        |
|                 | LDH                  | Los Alamos Scientific Laboratories | Molded; Uranium content 125 mg/ cm³ carbon; ρ=1.73 g/cm³                    |
|                 | LDC                  | Los Alamos Scientific Laboratories | Molded; Uranium content 250 mg/ cm³ carbon; ρ=1.66 g/cm³                    |
| Smith [9]       | H4LM                 | Great Lake Carbon Company         | Extruded; maximum particle size 838.2 µm; ρ=1.72 g/cm³                      |
2. Materials and methods

2.1. Testing Equipment

Two different testing equipment have been used in this study. The uniaxial compressive tests have been performed using the MTS 858 Mini Bionix II servohydraulic testing system. For the biaxial strength tests, a previously designed equipment, housed inside an aluminum chamber, has been modified, and it is shown in figure 1. This apparatus has been designed to operate in vacuum chamber and permits high temperature tests. The specimen lies on the fixed punch and it is compressed by the movable punch. Tantalum end blocks, foreseen for each punch, allow to withstand the high temperature reached during the tests. Heating is obtained by Joule effect, through tantalum foils attached to copper clamps. The temperature on the specimen cross section surface is measured by means of an optical pyrometer and a disappearing filament pyrometer, aligned with the observation windows. A bellows maintains the vacuum, of the order of 10⁻⁵ mbar inside the chamber to avoid materials oxidation. An HBM-U9C 10 kN tension-compression load cell, installed thanks to two threaded connections, measures the compressive force. The load is applied manually: a joint allows the rotation of a screw, which is brazed to a steel component that loads two axial bearings. Four bushes are fixed on a plate and allow to convert the screw rotary motion to a linear motion. The axial load is then transferred to the force sensor and to the specimen, by means of a cylindrical nut.
2.2. Tested material
The POCO EDM-3 graphite offers a uniform ultrafine microstructure, less than 5 µm, and pores typically less than 1 µm. This graphite is isotropic therefore it has no preferred grain orientation. The declared apparent density can vary between 1.67 g/cm$^3$ and 1.76 g/cm$^3$. Density influences the strength of graphite: a higher density leads in fact to an higher strength [27]. However, also in the same graphite billet, a density variation can be found. For this reason, each samples have been carefully measured by means of a Sartorius hydrostatic balance. Even though the graphite is porous, the intrusion of water into the porosity is slow and the accuracy with this method is acceptable if the submerged weight is taken quickly. A density of 1.73 ± 0.01 g/cm$^3$ have been considered acceptable for this study.

2.3. Specimen Design
Several studies have been made in order to clarify graphite behaviour under multiaxial stress but few definitive conclusions have been reached. Usually, tubular specimens were tested to failure under a combination of internal pressure and axial tension or compression. The failure points were then compared with the predictions of various theories for failure under multiaxial stress (table 2). Most studies were conducted at room temperature, while only Babcock [18] and Jortner [19] performed also high temperature biaxial tests.
Table 2. Comparison between different criteria analysed in previous studies.

| Maximum test temperature | Biaxial stress state | Suitable criteria |
|---------------------------|----------------------|-------------------|
| Broutman [13] RT          | Tension-tension      | Modified maximum strain energy theory |
|                           | Tension-compression  | Coulomb-Mohr theory |
| Ely [14] RT               | Tension-tension      | Modified maximum strain energy theory |
|                           | Tension-compression  | Modified maximum strain energy theory |
| Weng [15] RT              | Tension-compression  | Modified maximum strain energy theory |
|                           | Compression-compression | Buckling problems |
|                           | Compression-tension  | Modified maximum strain energy theory |
| Ho [16] RT                | Tension-tension      | Modified Coulomb-Mohr theory |
|                           | Tension-compression  | Modified Coulomb-Mohr theory |
|                           | Compression-compression | Modified Coulomb-Mohr theory |
| Eto [17] RT               | Tension-tension      | Modified maximum strain energy theory |
| Babcock [18] 2760°C       | Tension-compression  | Coulomb-Mohr theory |
| Jortner [19] 2200°C       | Tension-tension      | Weibull’s criterion |
|                           | Tension-compression  | Maximum shear stress theory |

In most cases, the tension-tension and the tension-compression quadrants in the biaxial stress plane were analysed. In fact, in the compression-compression quadrant, buckling problems often arose during tests, preventing to obtain valid data ([15], [18]). It seems that the biaxial compressive stress state cannot be measured accurately with thin-walled cylindrical specimens that is the method adopted in the aforementioned studies.

For these reason, in this study, a different approach has been adopted. The idea was to obtain a biaxial compressive state, by axially compressing hourglass specimens. Due to the hourglass radius, also a second principal stress (the hoop stress) originated in the sample. By varying the geometrical parameters of the specimen, it was then possible to obtain different biaxial ratio (axial stress over hoop stress), translating in different point in the compression-compression quadrant of the biaxial stress plane. This approach was also suitable for high temperature tests.

Several notched geometries have been considered, characterized by different parameters such as the net section diameter and the hourglass radius. At the end, four geometries have been selected for the study (figure 2 and table 3).
Thermo-electrical simulations have been then performed in order to assess the temperature distribution along the specimens. For the analysis, the FE code ANSYS® has been used. Axisymmetric constraints were defined and the sample, the tantalum end block and the steel punch have been modelled. An input fixed current was imposed and, by taking advantage of the symmetry of the sample, a voltage constraint of 0V was imposed at the centre. Radiation heat transfer has been applied on specimen lateral surface to the surrounding space, at a fixed temperature of 25°C.

2.4. Testing procedures

The uniaxial tests have been performed with the MTS servohydraulic testing system. The strain was measured by means of a MTS axial contact extensometer, with a gage length of 5mm. During the test a strain rate of 0.02 min⁻¹ was imposed.

In order to evaluate the biaxial strength, the experimental apparatus described in par. 2.1 was used. The specimen was placed inside the aluminum chamber, on the tantalum end block of the fix punch. For the high temperature tests, tungsten or graphite discs have been interposed between the specimen and the tantalum end blocks, in order to avoid material deterioration. No influence of the contact material was expected for hourglass specimens [27]. When a vacuum of 10⁻⁵ mbar was reached, a pressure was applied to the sample, in order to decrease the electrical resistivity of the surfaces in contact and facilitate the current flow. The applied compressive load was approximately of 20 MPa on the reduced section. The desired heat power was then gradually reached, while the temperature was checked by means of

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**Figure 2. Plane and notched specimens.**

**Table 3. Geometrical parameters of uniaxial and biaxial specimens.**

| Geometry   | D [mm] | d [mm] | L_tot [mm] | l [mm] | R [mm] |
|------------|--------|--------|------------|--------|--------|
| Uniaxial   | 6      | 5      | 20         | 10     | 2.5    |
| Hourglass I | 6      | 4      | 16         | 8      | 8.5    |
| Hourglass III | 6    | 4     | 10         | 4      | 2.5    |
| Hourglass IV | 6     | 4     | 10         | 2      | 1      |
| Hourglass VI | 6     | 3     | 10         | 1      | 0.5    |
two pyrometers. It was then possible to start the compressive test, by manually applying the load up to failure.

3. Results

3.1. Uniaxial tests at room temperature
Analysing the available data in literature, it was not clear if a linear elastic model could be adopted for this type of graphite in compression states. For this reason, uniaxial compression tests have been performed in order to shed light on the mechanical behaviour of this material. Unnotched samples, presented in table 3 and figure 2 and named “uniaxial”, have been therefore tested in compression, while strain was measured by means of an extensometer. The resulting stress strain curves are reported in figure 3.

![Figure 3](image)

**Figure 3.** Experimental stress-strain curves obtained at room temperature for EDM-3 graphite.

This graphite can be seen to strain significantly before the fracture point, with a value of strain approximately 4.5% and 140 MPa stress.

3.2. Biaxial strength at room temperature
Hourglass specimens have been tested at room temperature. In the light of uniaxial test results, elastic-plastic simulations were performed in order to evaluate the stress field at failure. The experimental stress-strain curve has been implemented in structural simulations and the suitability of the Von Mises yield criterion has been evaluated: the registered loads at failure were imposed in structural simulations and the equivalent Von Mises stresses have been compared. In figure 4, an example of Von Mises stress distribution for each geometry is reported, while figure 5 presents the corresponding failure points on the stress-strain curve.
**Figure 4.** Example of equivalent Von Mises stress distribution at failure load for each hourglass geometry.

**Figure 5.** Failure points on the stress-strain curve. The experimental curve (solid line) has been extended following the same slope (dashed line).

The experimental curve has been extended in Ansys®, following the same slope, in order to allow any extrapolations. The biaxial fracture stresses corresponding to failure points are reported in figure 6.
3.3. Biaxial strength at high temperature

Hourglass specimens have been tested also at 1000°C. Graphite increases its elastic modulus and its resistance with temperature. Therefore, the stress-strain curve obtained at room temperature was no longer valid: it was not possible to determine the equivalent stress at failure by means of elastic-plastic simulation. However, it has been possible to compare the load at failure, in order to highlight an increase of strength with temperature. Figure 7 presents the average trend of the compressive load with temperature. The data are presented as nominal pressure on net section in order to decrease variability due to samples different dimensions. No data at high temperature have been obtained for Hourglass VI, due to problems in determining the failure load without a clear view of the sample.

Figure 6. Biaxial fracture stresses at room temperature, POCO EDM-3 graphite.
4. Discussion

4.1. Plastic deformation at room temperature
The POCO EDM-3 graphite shows a significant plastic deformation at room temperature. For a lot with density equal to $1.73 \pm 0.01 \, \text{g/cm}^3$, the average stress at failure is approximately 140 MPa with approximately 4.5% of strain. A yield strength around 100 MPa can be defined using the 0.2% offset method. This results are in line with other data presented by Gillin [12], Babock [18] and Jortner [19].

4.2. Elastic-plastic simulations
The elastic-plastic simulations were performed assuming a Von Mises yield criterion. The experimental failure loads have been then applied and the equivalent Von Mises stresses at failure have been compared. As reported in figure 5, all geometries show a Von Mises stress approximately 140 MPa, in accordance with the uniaxial tests. This seems to validate the choice of this yield criterion. Moreover, the Von Mises total mechanical strain is not too different if compared with 4.5% uniaxial strain. Therefore, no significant extrapolations of the stress-strain curve have been performed in the simulations.

Assessed the suitability of the Von Mises yield criterion, it has been possible to analyse the biaxial stress state at failure for the different geometries. As shown in figure 6, it seems that resistance increases as the stress state approach the equibiaxial stress state, where the ratio between the axial and the hoop stresses approaches the unit value.

4.3. Biaxial strength increase with temperature
The tests demonstrated that, as the temperature increases, the compressive failure load increases: at 1000°C, Hourglass I shows an average increase of 16%, Hourglass III of 18% while Hourglass IV of 8.5% with respect to room temperature values. This behaviour is consistent with POCO indications ([20]) and it is in agreement with data in literature ([11],[12],[27]). However, in order to properly define the stress state at failure by dedicated elastic-plastic simulations as for room temperature, it would be necessary to obtain the stress-strain curve at 1000°C.
5. Conclusions
In this study both uniaxial and biaxial compression strength of an isotropic ultrafine grain graphite, the POCO EDM-3, have been presented. Hourglass-shaped specimens have been axially compressed both at room temperature and at 1000°C in order to evaluate the biaxial compressive strength, while plane samples have been used in room temperature uniaxial tests. The uniaxial results show a plastic deformation, with approximately 140 MPa stress and 4.5% strain at failure. The obtained stress-strain curve was implemented in the FE code Ansys®, and elastic-plastic simulations have been performed by imposing the failure loads registered during tests with notched samples. The applicability of the Von Mises yield criterion has been verified at room temperature: for all different hourglass geometries, the equivalent Von Mises stress at failure is approximately 140 MPa. At 1000°C, an increase in strength has been registered for notched specimens of approximately 16% in Hourglass I, 18% in Hourglass III and of 8.5% in Hourglass IV with respect to room temperature values. However, it is necessary to obtain a high temperature stress-strain curve in order to evaluate the applicability of the Von Mises yield criterion also at 1000°C.

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