J Mater Sci (2010) 45:4401–4405
DOI 10.1007/s10853-010-4417-4

EUROMAT 2009

Comparison of shear strength tests on AV119 epoxy-joined ceramics

Andrea Ventrella · Milena Salvo · Massimiliano Avalle · Monica Ferraris

Received: 15 October 2009 / Accepted: 13 March 2010 / Published online: 30 March 2010

Abstract The results of an experimental investigation on epoxy-joined CVD SiC and alumina tested in shear and apparent shear mode by four different configurations are presented. Ceramics have been joined by an epoxy adhesive (AV119), which is not to be considered as the final joining material for high temperature applications, but just as a model brittle joining material chosen to obtain several joined samples in a reasonable time. Advantages and disadvantages of each configuration are discussed and compared to results obtained with the same epoxy-joined carbon/carbon composites, tested by the same shear tests.

Introduction

Advanced ceramics present brittle nature and consequent poor workability, which limit their extensive use. In order to avoid this limitation, joining of small parts is employed to obtain the final components: for this reason, joining of ceramic components is a true key enabling process. The joining of ceramics may be of either a ceramic/ceramic or a ceramic/metal type and joining materials can be brazing alloys, glasses or composites [1–4].

Adhesive joining materials (e.g. epoxy resins) are used to join ceramics only for low temperature applications (e.g. lower than 150 °C): however, due to the advantages of adhesive bonding over traditional joining techniques (e.g. brazing), adhesively bonded joints are widely used in automotive, aerospace, electronic and packaging industries [5].

Very different is the purpose of joining SiC and Al_2O_3 with an epoxy resin (Araldite AV119) in this work: as discussed for AV119-joined C/C composites in [6] the epoxy adhesive has been used here as a 'model' brittle [7] joining material to obtain many joined samples in a reasonable time. Several joined samples are needed to compare in a statistically relevant way four different shear and apparent shear tests and to discuss the results obtained here with those presented in ref. [6].

Various experimental test set-up can be found in the literature to estimate the shear strength of a joint. Even though asymmetrical four point bending test is the only one recommended by the ASTM for obtaining a pure shear strength of joined advanced ceramics [8], single-lap or offset single-lap tests in compression [9] are widely used methods to obtain an apparent shear strength. A completely different testing method is torsion [10, 11]: with torsion tests, a pure shear strength can be measured without the limitations in the joining material strength, as imposed by the asymmetrical four point bending test [8]. However, torsion tests on joined ceramics is not an ASTM standard.

The presence of many different shear strength tests in the literature makes a comparison quite impossible. Furthermore, since a homogeneous shear stress state is never obtained within a joint, evaluating the shear strength of the same joining material with different test methods could lead to different results [12]. Even larger differences in shear strength can be obtained when the same joining material is used to join different substrates: both topics will be discussed in this article, by comparing results obtained by testing epoxy-joined (AV119) SiC and Al_2O_3 by four

A. Ventrella · M. Salvo · M. Ferraris (✉)
Politecnico di Torino, DISMIC, corso Duca degli Abruzzi 24, 10129 Turin, Italy
e-mail: monica.ferraris@polito.it
URL: http://www.composites.polito.it

M. Avalle
Politecnico di Torino, DIMEC, corso Duca degli Abruzzi 24, 10129 Turin, Italy

different shear strength tests and comparing these results with those obtained on a similar testing campaign on epoxy-joined (AV119) C/C [6].

More than 100 joined samples have been prepared and tested at room temperature by single-lap compressive shear test (SL), offset single-lap compressive shear test (SLO), offset double-lap compressive shear test (DLO), torsion test (respectively, TS (square section samples) and TC (cylindrical section samples)).

Experimental

The ceramics are alumina (Al_2O_3, non-filled dots in Figs. 1, 2, 3, 4) (Bettini Textile, Italy) and chemical vapour deposition silicon carbide (CVD SiC, filled dots in Figs. 1, 2, 3) (Rohm and Haas, USA). Samples have been cut to obtain specimens as in Figs. 1, 2, 3, 4. Araldite AV119 (Huntsman, USA) [7] has been used as joining material on acetone and ultrasonic cleaned ceramic surfaces with a curing time of 1 h at 130 °C.

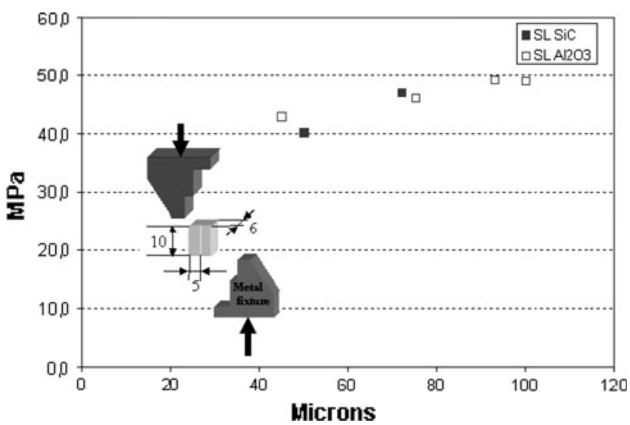

Fig. 1 Apparent shear strength evaluated with the compressive single-lap test (SL) on joined ceramics versus joint thickness

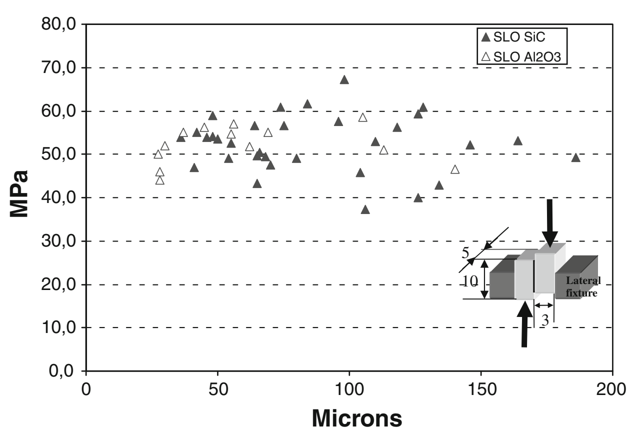

Fig. 2 Apparent shear strength evaluated with the compressive single-lap offset test (SLO) on joined ceramics versus joint thickness

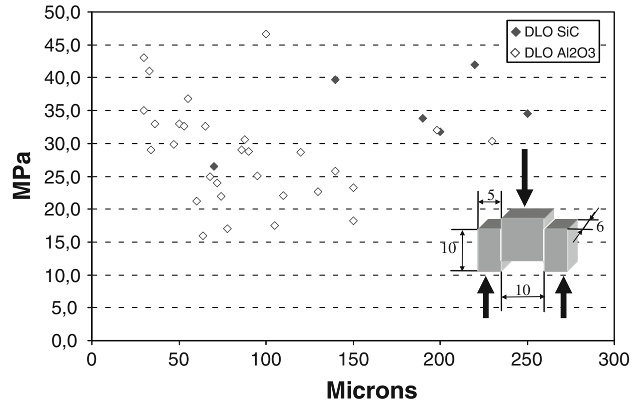

Fig. 3 Apparent shear strength evaluated with double-lap offset test (DLO) on joined ceramics versus joint thickness

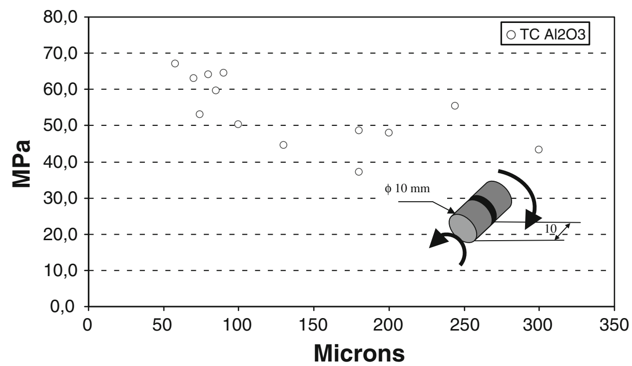

Fig. 4 Apparent shear strength evaluated with the torsion test (TC) on joined Al_2O_3 rods versus joint thickness

The ceramics surface roughness was measured by a surface profiler (KLA-Tencor P-11) and found to be, respectively, $R_a = 0.5631$ μm for Al_2O_3 and $R_a = 0.1056$ μm for SiC.

The joint thickness of each samples has been measured by optical or electron microscopy in order to investigate the relationship between shear strength and thickness.

A mechanical testing machine (SINTEC D/10) was used to perform the different shear tests in displacement control with a cross head speed of 0.5 mm/min. All the tests were carried out up to failure of the specimen. The average strength has been obtained by simple calculation of the ratio between the maximum load and the joint area. The results are displayed in Figs. 1, 2, 3, 4 showing shear strength versus joint thickness. Sample size has been reported in Figs. 1, 2, 3, 4 and are the same as in [6, 13].

Stereo microscopy and SEM have been used on each sample after shear tests to check the fracture surface: only the samples where the adhesive was observed on both the fractured surfaces were considered without taking into account tests showing fracture starting or propagating in the ceramic substrate.

Results and discussion

Single-lap compressive shear test (SL)

This test is a modification of the ASTM D1002-05 standard [14], which is recommended to test the apparent shear strength between two thin sheets of metal in tensile mode, according to the ASTM standard, in compression mode in this article.

The parallelepiped specimen obtained by joining two small parts of ceramics (Fig. 1, Al_2O_3 non-filled dots, CVD SiC, filled dots) has been glued to metal fixtures. These fixtures were placed between the loading plates of the testing machine and the specimen loaded in compression by a displacement applied on the top section of the apparatus. Due to the use of another adhesive also to hold the specimen within the fixtures, this test is only feasible at room temperature. Another issue is the alignment of fixtures to the joint: if not perfectly aligned, results are meaningless because of the presence of relevant opening normal stresses [6, 13].

Figure 1 shows the obtained results in the range of 45.8 ± 3.5 MPa with a remarkably poor test efficiency (number of samples broken inside the joint/total number of prepared samples, %) of 25% (Table 1), regardless to the joined ceramic (alumina or SiC).

One of the major drawbacks with the SL method is its limited measurement range: although samples are easy to obtain and to be broken in the joint for relatively weak joining materials, many difficulties have been encountered when measuring moderately strong joints. In this case, the failure always occurs between the fixtures and the specimen and never inside the joined area, independently from the kind of joined ceramic (alumina or SiC).

When the apparent shear strength of the joined samples approaches 50 MPa [15], this test cannot be used. Here, we are near this value and the tests efficiency is extremely low, particularly if compared to a test efficiency of 55% (Table 1) obtained for C/C joined with the same AV119 and tested with the same SL method and sample size [6].

Table 1 Mechanical test results and test efficiency (number of samples broken inside the joint/total number of prepared samples, %) for AV119-joined C/C samples [5] and AV119-joined ceramics

	C/C (MPa)	Test efficiency (%)	Ceramics (MPa)	Test efficiency (%)
SL	18.3 ± 1.9	55	45.8 ± 3.5	25
SLO	20.4 ± 2.0	80	52.3 ± 6.0	75
DLO	12.2 ± 0.9	85	29.4 ± 7.6	70
TC	19.7 ± 5.4	70	53.7 ± 9.4	80

A multiplicative factor of 2.5 has been found between SL tests on AV119-joined C/C and AV119-joined ceramics of the same size: this is due to the chemical and morphological difference of the two faying surfaces, porous for C/C and much smoother for alumina and silicon carbide.

Single-lap offset compressive shear test (SLO)

This single-lap test is obtained by joining the two parallelepipeds with a longitudinal offset (1.5 mm): the loading in apparent shear is obtained by loading in compression the offset surfaces (as sketched in Fig. 2 and in refs [6, 13, 15]). It is a modification of an ASTM standard used to test adhesives for wood with the help of two lateral stainless steel fixtures for a loose hold of the joined sample [16]. With this method, the joint-fixture alignment is not an issue as it was for SL [6], and SLO can be used also at high temperature.

Figure 2 (Al_2O_3 non-filled dots, CVD SiC, filled dots) shows the obtained results in the range of 52.3 ± 6.0 MPa with a test efficiency of 75% (Table 1). These results confirm what was discussed above: when approaching 50 MPa of apparent shear strength the SL is not suitable to obtain broken samples in the joined area, and SLO offers a valid and straightforward alternative. Again, as discussed above, multiplicative factor of 2.6 has been found between SLO tests on AV119-joined C/C and AV119-joined ceramics of the same size. Two statistical populations were found for SLO tests on AV119-joined C/C in ref. [6] depending on when the two lateral fixtures contributed to the mechanical strength of the joint.

The same drawback has not been found for AV119-joined ceramics, where the specimen-fixture friction has been carefully avoided by the use of a graphite foil. The high test efficiency (75%) makes this test extremely attractive in measuring apparent shear strength of joined ceramics. If the joining material strength is similar to that of the materials to be joined [15], also SLO can be unsuitable, but it is not the case with AV119-joined ceramics.

Double-lap offset compressive shear test (DLO)

With this test geometry (Fig. 3 and ref. [6]), the sample is obtained by joining three parallelepipeds with a central longitudinal offset. Loading in apparent shear is obtained by applying a displacement on the offset surface. The apparent shear strength is calculated by dividing the critical applied force to the sum of the two joined areas. Figure 3 (Al_2O_3 non-filled dots, CVD SiC, filled dots) shows the obtained results in the range of 29.4 ± 7.6 MPa with a test efficiency of 70% (Table 1). These results confirm what was discussed in ref. [6] where FE results indicated that the stress distribution within the middle of the joint in DLO was non symmetrical and can give results significatively lower than the values obtained with other tests. Also in this case, 29.4 ± 7.6 MPa is consistently lower that what was measured for SL and SLO. A multiplicative factor of 2.4 (discussed above) is still observable between AV119-joined C/C and AV119-joined ceramics.

Another drawback for this test has been found in its geometry dependence: if the DLO is done on samples obtained by joining three parallelepipeds of the same size ($10 \times 3 \times 5$ mm^3) (not reported in Fig. 3), the measured apparent shear is close to the one obtained by SL (52 MPa).

Torsion tests (TS, TC)

The torsion test method is proposed in some ASTM standards but none of them is directly applicable to joined ceramics [10, 11]. These two standards have been adapted to joined SiC [15, 17], first by preparing square section (TS) butt-joined bars or cylinders or pipes (TC), then by mechanically shaping the joined samples in an hour—glass shape with or without a reduced joined area. A dedicated equipment was designed and built at Politecnico di Torino (Italy) for torsion tests. In this work, TC test have been used on AV119-joined alumina rods 10 mm diameter and 20 mm height (Fig. 4); square section samples (TS) gave to the fracture of the ceramic instead of the joined area. A pure shear stress distribution is obtained with this tests: the stress distribution is non-uniform, since the shear stress acts in the tangential-axial plane and it is proportional to the distance from the cylinder axis. Experimental values (Fig. 4) gave 53.7 ± 9.4 MPa for the maximum shear stress at the outer radius of the specimen, with a test efficiency of 80% (Table 1), comparable to that observed for AV119-joined C/C. The multiplicative factor of about 2.7 is still observable between AV119-joined C/C and AV119-joined alumina for reasons discussed above.

As in ref. [6], a large scatter in the results is probably due to the fact that with torsion the maximum stress occurs on the external surface of the sample and surface defects are more likely to promote the propagation of fracture thus causing sudden failure of the sample.

Since torsion test is the only one able to give a pure shear value, more joined samples are currently under testing in order to find a shear versus thickness trend. Moreover, in order to validate the shear strength measured by torsion, asymmetrical four point bending tests (ASTM C1469-00) with V- or U-shaped notches are ongoing on AV119-joined ceramics.

Weibull analysis

Weibull analysis for SLO, DLO and TC on joined ceramics is shown in Fig. 5, where τ_{max} is the apparent shear strength and F_j is the cumulative distribution function [18]; the Weibull analysis is a powerful method to visualize both strength and data dispersion for a given test: the higher is the slope of the data linear fitting, the lower is their dispersion.

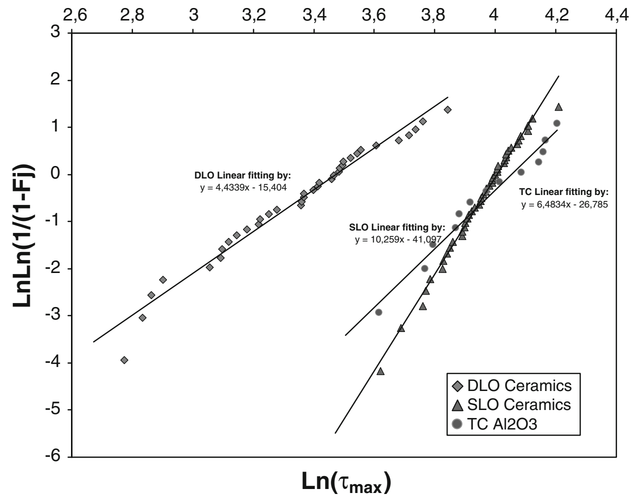

Fig. 5 Weibull analysis of the experimental results obtained by the three different shear test configurations (SLO, DLO, TC)

A relatively high dispersion of data is evident for the three tests, in particular for DLO and TC, while SLO shows a lower data dispersion. DLO (slope = 4.4339) has the largest scatter of data, probably due to the non-symmetrical stress distribution within the middle of the joint (as discussed in "Double-lap offset compressive shear test" section and in ref. [6]).

As discussed above ("Torsion tests (TS, TC)" section), the reason why TC has large scatter of data (slope = 6.483) is probably due to the maximum stress, which occurs on the external joined surface, where the defective rich surface can induce sample failure.

SLO (slope = 10.259) has a relatively lower scatter of data, provided that specimen-fixture friction is carefully avoided.

Finally, there is no meaningful statistical difference between the strength of the SLO and TC tests (as also shown in Table 1), while as expected and discussed above ("Double-lap offset compressive shear test" section), the DLO results pertain to a different statistical population with lower apparent strength and more dispersed data, due to the presence of two joined surfaces instead of one, which can both induce the failure.

Conclusions

Single-lap test (SL) demonstrated an already observed threshold of 50 MPa, above which it is unsuitable to test apparent shear strength [15] by this test. The single-lap off-set (SLO) gave reliable results with AV119-joined ceramics, with an encouraging test efficiency, thus proving that it can be a suitable way of testing and measuring apparent shear strength. However, more tests have to be done to measure its suitability on stronger joints. The double-lap off-set test (DLO) gave the lowest apparent shear strength values, thus confirming its unsuitability, as found also for AV119-joined C/C [6]. A sample geometry dependence of the measured apparent shear strength has also been verified.

If a pure shear strength is necessary, torsion tests on cylinders (TC) seems to be the most promising way of testing: possible evolutions of the simple torsion on butt-joined cylinders may be necessary in case of joining materials stronger than AV119.

Results obtained on joined alumina or SiC did not show any remarkable difference; conversely, a factor of about 2.5 has been found by comparing these results with those obtained on AV119-joined C/C tested by the same tests, thus confirming the role of interface between AV119 and different substrates (C/C or ceramics) on shear strength.

It must be underlined that none of the tests showed a reliable trend of shear strength versus thickness: apparent shear measured by SL seems to increase with thickness from 40 to 100 μm, SLO and DLO do not show any correlation and TC seems to decrease by increasing thickness from 50 to 300 μm.

In conclusion, it has been shown that apparent shear strength is strongly dependent on the kind of test used to measure it and on the substrate to be joined: it is worth noting that the same sequence of apparent (or pure) shear strength has been found for the four tests used on AV119-joined C/C and ceramics: DLO $\ll$ SL $<$ SLO, TC, thus demonstrating that, when keeping all parameters constant (joined sample size, joining material), the only variations are given by the materials to be joined and by the test used to measure their shear strength. This should be taken into account when proposing or comparing new joining materials and techniques.

Acknowledgements This work comes from the final projects of several students in Mechanical Engineering at Politecnico di Torino, II Facoltà, Vercelli, Italy: the authors would like to thank them all for their help in the preparation of the samples. The authors gratefully acknowledge Dr. Y. Katoh (Oak Ridge National Laboratory, ORNL, USA) and Dr. T. Hinoki (Kyoto University, Japan) for their helpful discussions. This work was supported in part by the EU Network of Excellence project Knowledge-based Multicomponent Materials for Durable and Safe Performance (KMM-NoE) under the contract no. NMP3-CT-2004-502243.

References

1. Cockeram BV (2005) J Am Ceram Soc 88(7):1892
2. Loehman RE, Tomsia AP (1988) Am Ceram Soc Bull 67(2):375
3. Nicholas MG, Mortimer DA (1985) Sci Technol 1(9):657
4. Malzbender J, Moench J, Steinbrech RW, Koppitz T, Gross SM, Remmel J (2007) J Mater Sci 42:6297. doi:10.1007/s10853-006-1178-1
5. Lo′pez-Puente J, Arias A, Zaera R, Navarro C (2005) Int J Impact Eng 32:321
6. Ferraris M, Ventrella A, Salvo M, Avalle M, Pavia F, Martin E (2009) Composites B 41(2010):182
7. Araldite AV119 epoxy adhesive paste, Product data sheet, Huntsman, 2004
8. ASTM C1469-00 (2005) Standard test method for shear strength of joints of advanced ceramics at ambient temperature
9. Amara D, Levallois F, Baziard Y, Petit JA (1996) J Adhes Sci Technol 10:1153
10. ASTM F734-95 (2006) Standard test method for shear strength of fusion bonded polycarbonate aerospace glazing material
11. F1362-97 (2003) Standard test method for shear strength and shear modulus of aerospace glazing interlayer materials
12. Serizawa FD, Lewinsohn CA, Singh M, Murakawa H (2007) J Nucl Mater 367–370:1223
13. Casalegno V, Chen Q, Chen Q, Salvo M, Smeacetto F, Ventrella A, Ferraris M (2010) Key Eng Mater (accepted)
14. ASTM D1002-05. Apparent shear strength of single-lap-joint adhesively bonded metal specimens by tension loading (metal-to-metal)
15. Ferraris M (2009) J Nucl Mater (submitted)
16. ASTM D905-03. Standard test method for strength properties of adhesive bonds in shear by compression loading
17. Ferraris M, Salvo M, Casalegno V, Rizzo S, Ventrella A (2010) Ceram Trans (accepted)
18. Weibull W (1939) Proc R Swedish Inst Eng Res 151:1