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ON THE AREOSPACE-GRADE ADHESIVES SHEAR STRENGTH TESTING WITH ASTM D5656 TEST AS AN EXAMPLE

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Abstract

This article presents adhesive shearing test methods, focusing especially on the ASTM D5656 method. These methods will be briefly characterized and compared. The most important concerns about the D5656 method are described. With the use of ASTM D1002 and D5656 methods, the influence of adherend surface preparation on shearing properties of the bond is evaluated. Compared to sandblasting only, sandblasting followed by the FPL process (sulfochromate etching of aluminum) increased shear strength of joints by 35 % for ASTM D1002 tests and by 48% for D5656 tests. Comparing these two methods, shear strength obtained in D5656 tests is about two times higher than in D1002 tests. The cause for this phenomena is much larger adherend thickness in the D5656 method, which provides the coupons with increased stiffness. Shear modulus, calculated with 3 different calculation methods, showed differences in obtained results, which points to necessary actualization of D5656 standard.

Keywords: adhesives, shear modulus, ASTM D5656, shear strength

1. INTRODUCTION

The basis and goal for modern aircrafts is the combination of low weight of the structure with high strength and efficiency. These features are obtained by using advanced materials (i.e. high strength aluminum alloys, carbon fiber reinforced composites) and developing special construction solutions. One of these solutions is increased use of adhesively bonded joints in place of mechanical joints (fasteners, rivets etc.). The main advantage of bonded joints is their low weight when compared to mechanical joints [1, 10]. Nonetheless, using adhesively bonded joints imposes new difficulties, both in production and testing or modelling of aircraft structures. Accurate testing of mechanical properties of adhesives, especially shear modulus, is critical for developing an effective design for aircraft structures.

Problems arising during mechanical testing of adhesives properties can be divided into two categories: macro- and microscopic. The most prominent macroscopic problem is very large influence of surface preparation of bonded material (grinding, chemical etching, etc.) on bonded joint properties, as well as on technological aspects of production (process time, special equipment required, etc.). Out of microscopic aspects of adhesives testing, especially in shear modulus tests, most important seem to be the uniformity of stress distribution in loaded joint and adhesion at adhesive/adherend interface. In the past, it was common to assume that stress in loaded joint is distributed uniformly. Nowadays, thanks to finite element analyses, such assumptions are deemed not credible. [2, 11, 12]. One of the effects of this new approach, in the case of ASTM D5656 tests [3], is the introduction of correction factors to shear modulus calculations, taking into account non-uniformities in stress distribution [4÷6]. The main problem with using these corrections is the fact that they still haven't been officially approved by standard committees so their usage can be questioned.

Aside from the mentioned D5656 test (thick adherend shear test, TAST), shear strength of an adhesive can be measured using a variety of other methods, both *in-situ* (testing samples of cast adhesive), as well as bonded joints tests. In-situ tests, for example *bulk torsion test,* aren't widely used, mostly due to difficulties with sample manufacturing (especially with film adhesives) and the requirement of torque-applying test machine for torsion tests. Therefore, bonded joint tests are usually used, for instance *butt torsion test* (Fig. 1a), *napkin ring test* (Fig. 1b) or single shear joint tests, ½" *lap shear test* (ASTM D1002 - Fig. 1c) with its improved version *thick adherend shear test* (Fig. 1d). Because both *Butt torsion* and *napkin ring test* have the same limitations, *bulk torsion test* and single lap shear tests are default tests for adhesive shear strength and modulus evaluation. The main advantage of ½" lap shear *test* - its simplicity - is also its main disadvantage: coupons are easy to manufacture and test, but their very low stiffness, leading to nonuniform stress distribution in the joint, limit this test to development and comparison tests. The only test which allows to (relatively) easily determine adhesive shear strength and modulus is ASTM D5656 method and its modified version, ISO 11003 test [6].

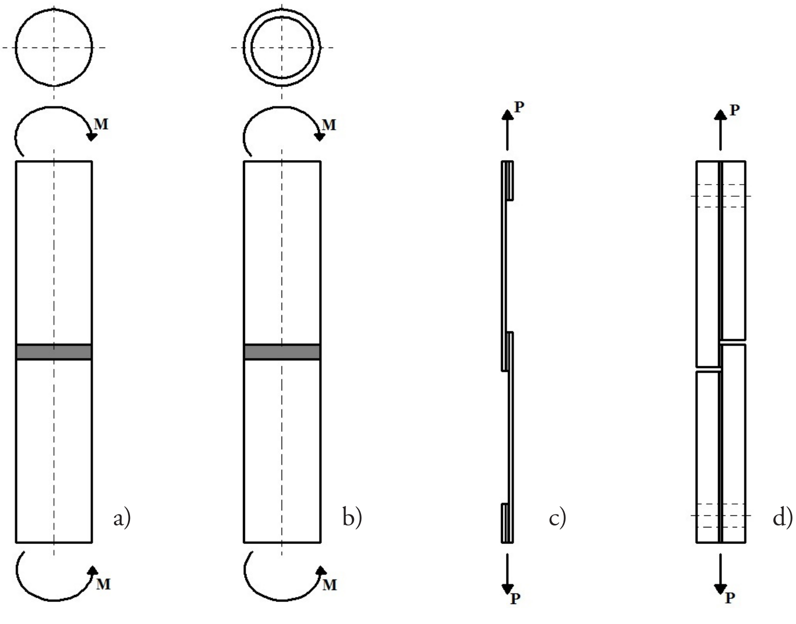

Figure 1. Schematics of different adhesive shear test methods: a – *butt torsion*, b – *napkin ring*, c – ½" lap shear, d – *thick adherend shear test,* author's work

The D5656 test method is relatively complicated and can be influenced by many factors. Specimen preparation process requires high precision cutting and drilling – misplacement of mounting holes will cause uneven loading of the specimen, leading to nonuniform stress distribution in the joint. The most challenging problem, in D5656 test as well as in adhesives' shear testing in general, is the accurate measurement of shearing strain in the joint. Original version of D5656 method introduced KGR-1 type extensometers, designed for joint shear strain measurement (Fig. 2) [7]. Here, extensometers measure displacement of bonded adherends relative to each other. This method of measurement has 2 main problems: slipping of extensometer pins during the late stage of the test, and the fact that extensometers measure not the strain of the adhesive itself, but the strain of the adhesive and layer of the adherend between the adhesive and extensometer contact point. Therefore, strain correction is needed. It is done by measuring the strain of a pure aluminum specimen with simulated bondline thickness (assuming uniform stress distribution in material) at a given load. Obtained metal strain is then subtracted from strain measured during the test.

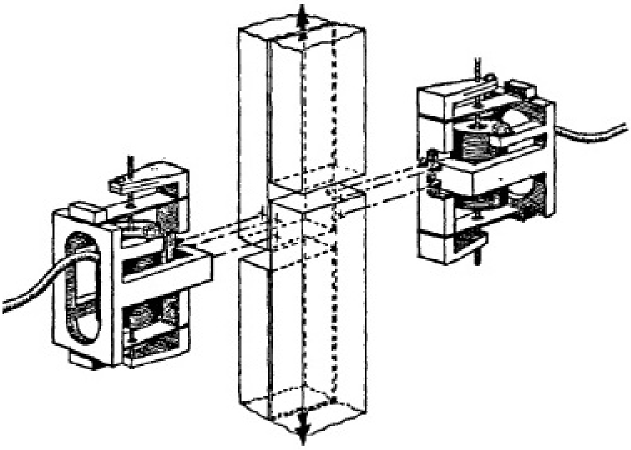

Figure 2. KGR-1 type extensometers and their placement on the specimen [3]

KGR-1 extensometers are expensive, therefore some researchers try to use other ways by converting standard linear extensometers into joint strain measuring devices [4]. Nonetheless, this measurements still need metal displacement corrections, as well as calculation corrections that account for nonuninform stress distribution inside the joint. The digital image correlation (DIC) methods are promising alternatives to standard strain measurements but they still have not been standardized which limits their usage to strictly scientific purposes [8].

2. METHODOLOGY

The influence of the surface preparation of 2017A aluminum alloy on shear strength and modulus of 3M AF163-2K film adhesive (293 g/m^2 aerial weight and 0.24 mm thickness) was tested. Surface preparation methods were 1) a 60 µm grain diameter alumina sandblasting alone and 2) a 60 µm grain diameter alumina sandblasting joined with chromic etching, known as the FPL etching. FPL process was done according to adhesive manufacturer guidelines, with etching solution composition given in Table 1.

Table 1. The FPL solution composition (per 1 litre)

Ingredient	Amount
Distilled water	700 ml + supplement to 1 l
Sodium dichromate	47 g
Sulfuric acid 96%	300 g
Aluminum chips	1,5 g

For process control, 5 samples of each preparation process were tested according to D1002 test standard (½" lap shear test) [9]. Thick adherend shear specimens were tested in the same manner, 4 specimens per series, using a modified D5656 method. For shear strain measurements, instead of KGR-1, standard linear extensometers modified according to [4], presented on Figure 3, were used. These modifications include a 4 contact pin configuration (instead of the standard 3 pins) and adding mounting holes drilled into the sides of the specimen, which significantly decreases extensometer slippage during the test. Measured aluminum displacement (for shear strain correction) was 0.00365 mm for 4464 N load.

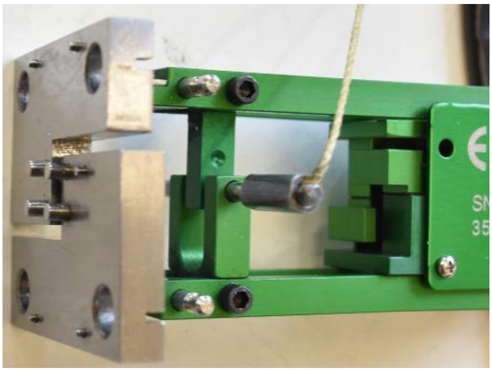

Figure. 3. Extensometer modifications for shear strain measurement, author's work

Metal displacement during the test (d_m) was calculated using equation 1:

$$d_m = \frac{p-t}{t} \times M \times \frac{L}{4464} \tag{1}$$

where: p – distance between extensometer contact points on both sides of the adhesive, mm; t – adhesive thickness, mm; M – metal displacement at 4464 N load, mm; L – given load , N. Shear strain (γ_i) was calculated using equation 2:

$$\gamma_i = \frac{d_a - d_m}{t} \tag{2}$$

where: d_a – joint displacement; mm, d_m – metal displacement, mm; t – adhesive thickness, mm.

Shear stress δ_i was calculated according to:

$$\delta_i = \frac{F_i}{l \times w} \tag{3}$$

where: F_i – load, N; l – joint length, mm; w – joint width, mm.

Shear modulus G_c was calculated according to equation 4:

$$G_c = \frac{\delta_1 - \delta_2}{\gamma_1 - \gamma_2} \tag{4}$$

where: $\delta_{1,}$ δ_2 – stress at points 1 and 2, in linear portion of stress-strain curve; $\varepsilon_{1,}$ ε_2 – adhesive strain at these points.

To test accuracy, two different calculation corrections were applied to shear moduli calculated with equation 4. The first, based on article [4], is applied to metal displacement during the test to correct non-uniformities in joint strain:

$$F_c = -C_f \times t_{sim} + 1{,}065 \tag{5}$$

where: F_c – correction factor; C_f – constant, equal to 1,73 for adhesive thickness given in inches, or 0.0683 for adhesive thickness given in mm; t_{sim} – simulated adhesive thickness in pristine aluminum coupon, mm. A correction is made by multiplying aluminum displacement from eq. 1 by coefficient F_c.

The second correction is based on [5] and is aimed at correcting non-uniformities in adhesive strain. This correction is based on finite element analysis of joint shearing. It is composed of two coefficients:

$$C_1 = 1{,}03t^{0{,}068} \tag{6}$$

$$C_2 = -0{,}000483 + 0{,}073e^{-3{,}29t} \tag{7}$$

Correction is applied to shear modulus according to:

$$G_{rec} = C_1 \times G_{ASTM} + C_2 \tag{8}$$

where: G_{ASTM} – shear modulus calculated with D5656 standard.

3. TEST RESULTS

3.1. ½” *lap shear test results*

Single lap shear strength was tested according D1002 test standard. Results in Table 2 show strong influence of surface preparation method on joint strength.

Table 2. ½” *lap shear* test results

	SANDBLASTING		FPL PROCESS	
Coupon label	**Failure load,** N	**Shear strength,** MPa	**Failure load,** N	**Shear strength,** MPa
Coupon 1	4910	14.7	7032	20.4
Coupon 2	5379	16.2	6994	20.3
Coupon 3	4502	13.5	6593	19.1
Coupon 4	4470	13.6	6511	18.7
Coupon 5	4823	14.7	6768	19.6
Average	**4817**	**14.5**	**6780**	**19.6**
Std dev.	**369**	**1.1**	**233**	**0.7**
Coeff. of variation	**7.66%**	**7.51%**	**3.43%**	**3.77%**

The average shear strength of joints with aluminum being sandblasted only was 14.5 MPa, while for joints with aluminum after sandblasting followed by the FPL process was 19.6 MPa. This means 35% joint strength increase after using the FPL process. Coefficients of variation decreased for coupons after FPL process, compared to coupons after sandblasting only. Therefore, joint quality is also more uniform and repeatable after using the FPL process.

3.2. Thick *adherend shear* test results

Tests were primarily carried out using the thick adherend shear tests according to ASTM D5656 standard. Shearing of the joint has two stages, 1) linear, where adhesive strain is elastic and stress-strain relation is linear (shear modulus) and 2) plastic stage, where shear strain is non-elastic and stress increases much more slowly than in the elastic stage. Figure 4 presents a typical stress-strain curve for a thick adherend shear test with three characteristic points.

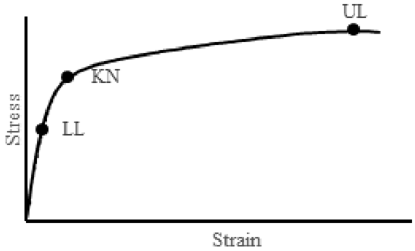

Figure 4. Typical shear stress-strain curve, author’s work

LL point in Figure 4 is the linear limit (the end of the elastic stage), KN point is the knee point on stress-strain curve and the UL point is the ultimate stress point. Results of thick adherend shear tests are presented in Tables 3 and 4.

Table 3. ASTM D5656 shear test results for coupons after sandblasting only.

Coupon label	**LL point strain**	**LL point stress,** MPa	**KN point strain**	**KN point stress,** MPa	**UL point strain**	**UL point stress,** MPa	**Shear modulus** MPa
PIA 1	0.052	18.76	0.067	22.53	0.133	25.63	420.0
PIA 2	0.055	14.56	0.114	20.77	0.799	27.03	254.4
PIA 3	0.051	17.24	0.078	25.22	0.281	29.08	539.9
PIA 4	0.022	14.15	0.097	22.15	0.932	28.91	521.6
Average	**0.045**	**16.18**	**0.089**	**22.68**	**0.536**	**27.66**	**434.0**
Std dev.	**0.015**	**2.200**	**0.021**	**1.862**	**0.389**	**1.643**	**130.8**
Coeff. of variation	**34.34%**	**13.60%**	**23.34%**	**8.21%**	**72.50%**	**5.94%**	**30.14%**

Table 4. ASTM D5656 shear test results for coupons after FPL process.

Coupon label	**LL point strain**	**LL point stress,** MPa	**KN point strain**	**KN point stress,** MPa	**UL point strain**	**UL point stress,** MPa	**Shear modulus,** MPa
FPL 1	0.030	19.74	0.091	31.08	1.050	42.12	611.8
FPL 2	0.052	24.80	0.115	30.64	1.000	40.66	375.6
FPL 3	0.043	23.97	0.115	31.63	0.970	41.11	544.4
FPL 4	0.045	22.16	0.094	30.88	0.674	40.05	662.8
Average	**0.043**	**22.68**	**0.104**	**31.06**	**0.924**	**40.99**	**548.7**
Std dev.	**0.009**	**2.24**	**0.013**	**0.42**	**0.170**	**0.87**	**125.1**
Coeff. of variation	**21.61%**	**9.89%**	**12.58%**	**1.36%**	**18.36%**	**2.13%**	**22.81%**

Comparing these two series, there is clear influence of the FPL process on shear strength and modulus of the adhesive. After using the FPL process, the average LL-point stress had increased by 40 % (from 16.18 MPa to 22.68 MPa) and KN-point stress had increased by 37 % (from 22.68 MPa to 31.06 MPa). Ultimate shear strength also increased by 48%. It shows that using sandblasting as the only surface preparation process before bonding is insufficient for achieving high strength joints. Similarly to D1002 tests, coefficients of variation were lower for all measured properties in cases where the coupons underwent FPL process surface preparation. Figure 5 shows a comparison of stress-strain curves for both test series. Measured average shear modulus was 26% higher after using the FPL process, but both of the test series came with large coefficients of variation, which shows a significant range of measured values. For coupons that underwent sandblasting only, obtained values for shear modulus were between 254 and 540 MPa; for FPL process coupons were in the range of 376 to 663 MPa. Combined with the large scatter of measured LL-point strain values, it shows possible influence of extensometer placement and clearances occurring at contact points on obtained modulus values.

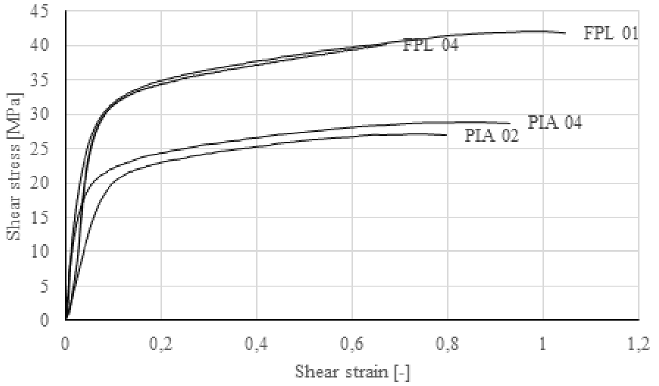

Figure 5. Comparison of stress-strain curves for both types of coupons, author's work

The summary of conducted tests is presented in Figure 6. The figure shows increased shear strength in both of the tests after using the FPL surface treatment. For D5656-tested coupons, there was a 48% increase in strength, compared to 35% increase in the case of the D1002 test. The other phenomena observed here is greater measured shear strength using D5656 method, independent of aluminum surface preparation process. For sandblasted-only coupons, shear strength obtained via the D5656 test was 91% higher, than values obtained with D1002 method. For coupons that underwent the FPL treatment, this difference increased to 109%. It can be related to much higher thickness of coupons used in D5656 test. Increased thickness provides higher stiffness of the coupon, which correlates to reduced bending and twisting of the joint during the test, which promotes more uniform shear state in the bondline.

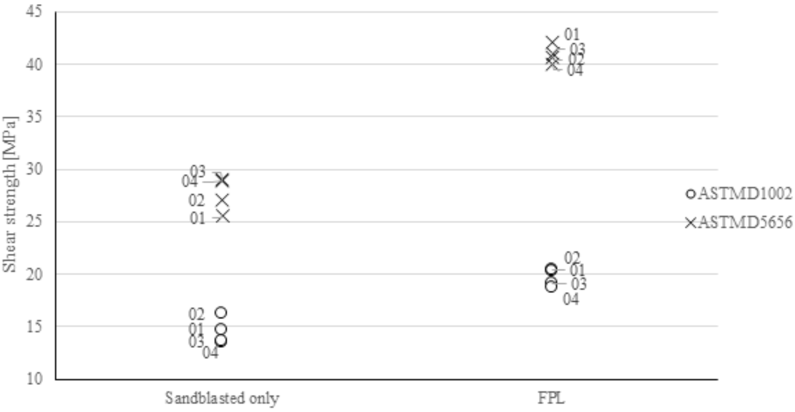

Figure 6. Comparison of shear strength of joints prepared with two different aluminum Surface treatment processes, tested with D5656 and D1002 test methods

Here, different shear modulus calculation correction factors have to be taken into account. Table 5 presents results of corrections made according to equation 5 (correction factor 1) and equations 6,7 and 8 (correction factor 2).

Table 5. Comparison of shear moduli for both of tested series, calculated with the use of aforementioned correction factors.

	Sandblasting only			**FPL treatment**		
Coupon label	**G acc. to D5656,** MPa	**G with c. f. 1,** MPa	**G with c. f. 2,** MPa	**G acc. to D5656,** MPa	**G with c. f. 1,** MPa	**G with c. f. 2,** MPa
Coupon 1	420.0	427	393.1	611.8	629	566.9
Coupon 2	254.4	256.9	240.4	375.6	382	347.8
Coupon 3	539.9	550	509.3	544.4	556	505.2
Coupon 4	521.6	532	487.4	662.8	682	615.7
Average	**434.0**	**441.5**	**407.6**	**548.7**	**562.3**	**508.9**
Std dev.	**130.9**	**134.5**	**122.3**	**125.1**	**130.8**	**116.5**
Coeff. of variation	**30.14%**	**30.46%**	**30.01%**	**22.81%**	**23.26%**	**22.90%**

Values of moduli calculated with correction factor 1 are similar to those calculated by the D5656 standard only. The differences were 1.7% increase for average modulus of coupons after sandblasting only, and 2.5% increase after the FPL process treatment. On the other hand, correction factor 2 leads to a decrease in obtained shear moduli values by 6.1% for coupons after sandblasting and 7.3% for coupons after the FPL treatment. Applying correction factors alters obtained values, which may lead to discrepancies between experimental and applied properties of the adhesive. Use of correction factors is still not recognized by the D5656 standard, therefore only values calculated according to this standard are universally accepted. However, introducing correction factors by some researchers points to ongoing development in the area of adhesive shear modulus measurements.

4. CONCLUSIONS

Results from the adhesive shear tests performed validate the following conclusions:

1. The combination of sandblasting with the FPL process as an aluminum surface preparation prior to bonding positively affects the shear strength of the joint. Coupons prepared with the use of the FPL process showed 35% and 48% increase in shear strength measured via D1002 and D5656 test methods respectively, compared to coupons after sandblasting only.
2. The FPL treatment has a positive influence on joint uniformity. For both tests, the series of coupons that were treated using the FPL process showed lower coefficients of variations for shear strength – in the D5656 test it was 2.13% compared to 5.94% for coupons after sandblasting only.
3. When comparing results for D1002 with D5656 test methods, values obtained in the latter test were higher and closer to the theoretical shear strength of the adhesive. The difference between these two test methods was as high as 91 % for coupons after sandblasting only and 109 % for coupons after the FPL treatment. Increased aluminum thickness is the main cause for greater stiffness in D5656 test coupons, leading to more uniformity and less bending in the joint as compared to D1002 coupons.
4. Differences in values of shear moduli obtained via different calculation methods and correction factors, point out that D5656 test method needs a new revision, accounting test results and FEM simulations results.

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PROBLEMATYKA BADANIA WYTRZYMAŁOŚCI KLEJÓW LOTNICZYCH NA ŚCINANIE NA PRZYKŁADZIE METODY ASTM D5656

Streszczenie

W niniejszym artykule opisano metody badań wytrzymałości na ścinanie klejów, ze szczególnym uwzględnieniem metody ASTM D5656. Dokonano porównania tych metod i krótkiej ich charakterystyki. Opisano najważniejsze problemy związane ze stosowaniem metody ASTM D5656. Zbadano również wpływ przygotowania powierzchni aluminium na otrzymywane wartości wytrzymałości spoiny na ścinanie, mierzone metodami ASTM D1002 i ASTM D5656. Porównując z piaskowaniem, połączenie piaskowania aluminium z procesem FPL (chromianowanie aluminium) zwiększyło wytrzymałość spoin na ścinanie o 35 % dla prób według normy ASTM D1002 i o 48 % dla metody D5656. Spośród tych dwóch metod, wytrzymałość na ścinanie uzyskiwana w badaniach wg normy D5656 jest średnio dwukrotnie wyższa, niż ta uzyskiwane w badaniach wg normy D1002. Przyczyną jest użycie znacznie grubszych substratów i większa sztywność spoiny w normie D5656. Przeprowadzenie obliczeń modułu ścinania kleju z uwzględnieniem 3 różnych metod wykazało różnice w otrzymywanych wynikach, co wskazuje na konieczność aktualizacji normy D5656.

Słowa kluczowe: kleje, moduł ścinania, ASTM D5656, wytrzymałość na ścinanie.