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Analysis of manufacturing parameters on the shear strength of aluminium/GFRP co-cured and adhesively bonded single-lap joints

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Appendices

Appendix A – Literature Review

Many early researchers (Volkersen, 1938; Goland & Reissner, 1944; Hart-Smith, 1973; Adams & Peppiatt, 1974; Bigwood & Crocombe, 1990) have investigated the shear and out-of-plane tensile stress distributions within the adhesive layer for single-lap joints. From these studies, it is well know that due to loading eccentricity and differential straining of the substrates, the shear stress distribution in single-lap joints are typically non-uniform.

Since these studies, other researchers (Cheng *et al.* 1991; Da Silva *et al.* 2006; Kwang-Soo Kima, 2006; de Morais *et al.* 2007; Kahraman *et al.* 2008; Lee *et al.* 2009; Pereira *et al.* 2010; Reis *et al.* 2011; Asgari Mehrabadi & Ganguli, 2012; Pinto *et al.* 2014; Reis *et al.* 2011) have studied the influence of various manufacturing parameters on the shear behaviour in single-lap joints. Even though the majority of these studies examine the process of adhesive bonding, the configuration of the single-lap joint selected for evaluation of co-curing is similar to those used in adhesive bonding; hence, the information available on these joints is still applicable.

In recent years, the effect of the co-curing manufacturing process has been studied. In the few related studies on co-cured single-lap joints (Shin *et al.* 2003; Kwang-Soo Kima, 2006; Matsuzaki *et al.* 2008a; Matsuzaki *et al.* 2008b; Tzetzis, 2012), much of the work is focused around surface pre-treatment and surface roughness. These studies report that surface pre-treatment is one of the most important parameters influencing joint strength. Pereira *et al.* (2010) found that joint strength increases with a decrease in surface roughness.

Shin *et al.* (2003) studied co-cured single-lap joints using steel and carbon fibreepoxy composite adherends. Initial failure mechanism of the co-cured single-lap joints was analysed using stress distributions obtained from finite element analysis (FEA). It was found that out-of-plane tensile and shear stresses play an important role in the failure of the co-cured joints (Shin *et al.* 2003).

Kwang-Soo Kima (2006) reports that the structural performance and reliability of the co-curing method is better than that of secondary bonding. However, in his study the joint strength of the co-cured single-lap joints was found to be lower than the secondary bonded ones, due to premature delamination failure. The same results were found in the study conducted by Seong *et al.* (2008) who studied composite-to-composite single-lap joints. It was found that joint strength increases with adherend thickness (Seong *et al.* 2008). In the study conducted by Kwang-Soo Kima (2006), progressive failure of the adhesive layer and early crack growth delays delamination failure in the secondary bonded specimens. It was also found that as surface roughness and bondline thickness decreases, joint strength increases (Kwang-Soo Kima, 2006). Other studies (Bigwood & Crocombe, 1990; Kwang-Soo Kima, 2006; Kahraman *et al.* 2008) report similar results.

To simplify the manufacturing process and improve joint strength, Matsuzaki *et al.* (2008a), proposed a bolted/co-cured hybrid joining method for joining metal-tocomposite joints. The method combines co-curing and bolted joints without damaging reinforcing fibres in the composite adherend. It was found that the hybrid joints initially experience adhesive failure and then the hybrid joint behaves as a bolted joint until joint failure (Matsuzaki *et al.* 2008a). The hybrid joints were found to improve joint strength in comparison to the co-cured joints. However, the method uses several bolts to enhance fracture toughness, and this is problematic in terms of weight saving design. Matsuzaki *et al.* (2008b) proposed a novel method for reinforcing metal-to-composite co-cured joints using inter-adherend (IA) fibres. It was shown that the IA fibre performs as a bridge and suppresses crack propagation, and as a consequence, the displacement to failure and static strength are significantly increased (Matsuzaki *et al.* 2008b).

In a recent study on co-cured single-lap joints, Tzetzis (2012) investigated the mechanisms that govern adhesion using surface profilometry, contact angle and surface energy measurements, X-ray photoelectron spectroscopy (XPS) and scanning electron microscopy. It was found that removal of internal contamination of the bonded surfaces, increased bonding ability. It was also shown that joint strength is not proportional to the adhesion strength of the bulk adhesive, but failure dictated by the interlaminar shear strength of the composite part, which coincides with the literature reported by Kwang-Soo Kima (2006) and Seong *et al.* (2008).

Appendix B – Material Properties

Material Property	Value	Units
Structural Epoxy Adhesive		
Elastic Modulus	2415	MPa
Poisson's Ratio	0.35	N/A
Mass Density	1100	kg/m^3
Tensile Strength	28	MPa
Compressive Strength	104	MPa
Thermal Conductivity	0.188	W/(m⋅K)
Epoxy/E-Glass Fiber, 0/90 Woven Fabric Lamina		
Elastic Modulus in X	37722.18	MPa
Elastic Modulus in Y	37722.18	MPa
Elastic Modulus in Z	9132.38	MPa
Poisson's Ratio in XY	0.2663	N/A
Poisson's Ratio in YZ	0.2663	N/A
Poisson's Ratio in XZ	0.4273	N/A
Shear Modulus in XY	3357.48	MPa
Shear Modulus in YZ	3199.55	MPa
Shear Modulus in XZ	3357.75	MPa
Mass Density	1937.6	kg/m^3
Tensile Strength in X	1075	MPa
Tensile Strength in Y	1075	MPa
Compressive Strength in X	725	MPa
Compressive Strength in Y	725	MPa
Shear Strength in XY	88.47	MPa
Yield Strength	1075	MPa
1050 H19 Aluminium Alloy		
Elastic Modulus	69000	MPa
Poisson's Ratio	0.325	N/A
Shear Modulus	25000	MPa
Mass Density	2680	kg/m^3
Tensile Strength	166	MPa
Compressive Strength	157	MPa
Yield Strength	157	MPa

Appendix C – Surface Roughness Methodology and Experimental Results



Specimen	Sample	SRp (µm)	SRv (µm)	SRz (µm)	SRa (µm)	SRq (µm)
	Average	28.36	31.67	60.03	3.95	5.91
	Max	31.60	35.19	66.04	4.45	6.57
Mechanically Blasted	Min	24.87	28.59	54.04	3.48	5.23
	Range	6.73	6.60	12.00	0.97	1.34
	S.d	2.87	2.65	4.91	0.41	0.55
Mechanically Abraded	Average	13.58	14.86	28.45	2.30	3.34
	Max	16.34	16.01	32.19	2.58	3.78
	Min	11.40	13.59	25.12	1.97	2.89
	Range	4.94	2.41	7.07	0.61	0.89
	S.d	2.13	0.96	2.96	0.25	0.35

Appendix D – Typical Load/Extension Curve



Appendix E - Statistical Methodology

Winsorizing of raw data

Prior to mean testing, sample outliers were investigated by identifying sample maximum and minimum values within each dataset. For each sample, a Lilliefors normal distribution test was conducted at the 1% significance level, in order to determine the normality of the data. Two-sample F-tests were then conducted to clarify if the variances of the original and winsorized data are equal.

After clarifying the normality and population variance of each sample, two-sample ttests were further conducted in order to compare the means between the original and winsorized data. Acceptance of the null hypothesis, indicates that the data in the two samples comes from independent random samples and normal distributions, with equal means and equal but unknown variances. The alternative hypothesis is that the data between the samples comes from populations with unequal means. A result of h=1 rejects the null hypothesis at the 1% significance level, and 0 otherwise.

Finally a box-and-whisker plot for all samples were generated and mean comparisons expressed as a percentage were calculated. From these results and the statistical analysis any noticeable outliers were identified and eliminated from the forthcoming analysis.

Two-sample testing

Statistical analysis 1 to 6 shown in Table 3.1 were analysed using two-sample t-tests in MATLAB R2015b. Statistical analysis 1 to 4 investigates the influence of the bonding process (i.e. co-curing vs. adhesive bonding) on the tensile lap-shear strength for a composite adherend thickness of 20, 15 and 10 plies respectively. Within analysis 1 to 3, all samples were prepared by mechanical abrasion. In statistical analysis 4, the influence of the bonding process is compared when specimens are prepared by mechanical blasting. The influence of surface preparation is later studied in analysis 5 and 6 for both bonding processes.

For two-sample statistical hypothesis testing, two-sample F-tests were firstly conducted to determine if the variances of the two samples are equal at the 1% significance level. For the tests involving populations with equal variance, two-sample t-test were then conducted. This method of statistical testing clarifies whether the two independent samples come from populations with equal or unequal means. A large p-value indicates that the difference between sample means is insignificant, hence the tensile lap-shear strengths are similar and the null hypothesis is accepted, h=0. A small p-value indicates that difference between sample means is significant, hence the tensile lap-shear strengths are not the same and this suggests rejection of the null hypothesis, h=1. For additional confirmation, the degree of overlap in a boxplot comparison was used to confirm these results.

For the tests involving populations with unequal variance, unequal two-sample t-test were conducted at the 1% significance level. For further validation, log transformation testing was conducted. Taking the log transformation improves the stability and linearity of the populations by reducing skewness, this additionally eliminates any bias from the analysis. Using the log transformation data, once again two-sample F-tests for equal variance and two-sample t-tests were conducted to validate results. Finally, for populations with unequal variance, non-parametric Kruskal-Wallis tests are performed to assess one-way analysis of variance (ANOVA).

Three-sample testing

Three-sample tests (Statistical Analysis 7 to 11) shown in Table 3.1 were conducted by means of one-way ANOVA statistical hypothesis testing. But firstly, for each analysis, Bartlett's test for homogeneity of variances (homoscedastic) was conducted to estimate whether more than two groups are homoscedastic. A large p-value indicates acceptance of the null hypothesis that the samples come from normal distributions with the same variance. The alternative hypothesis is that at least two of the data samples do not have equal variances.

One-way ANOVA and multiple comparison mean testing was finally conducted in order to determine which pairs of group means are significantly different. Small pvalues indicate that the differences between sample means are significant, and this suggests rejection of the null hypothesis. Large p-values favour the null hypothesis and suggest that the difference between sample means are insignificant.

Further comparisons using multiple comparison of means allows for clarification as to which sample means are different, as performing multiple two-sample t-tests to determine which pairs of means are significantly different would be highly inefficient. From this test, combinations involving confidence intervals that do not include zero and small corresponding p-values, indicate that the differences in means are significant and the null hypothesis is rejected. Conversely, for combinations involving confidence intervals that include zero, and large corresponding p-values, the null hypothesis cannot be rejected. Confidence interval graphical plots were finally used to validate these results.

Appendix F - Statistical Results - Winsorizing of Raw Data

Sample Number	Lilliefors Normal Distribution Test				
	kstat	critval	р	h	
Α	0.1409	0.3326	0.5000	0	
В	0.1709	0.3326	0.5000	0	
С	0.1461	0.3326	0.5000	0	
D1	0.0000	0.3171	0.3074	0	
D2	0.0000	0.3034	0.3872	0	
D3	0.0000	0.3326	0.0211	0	
E1	0.1191	0.3034	0.5000	0	
E2	0.1072	0.3034	0.5000	0	
E3	0.0000	0.3034	0.1562	0	
F1	0.1321	0.3034	0.5000	0	
F2	0.1545	0.3034	0.5000	0	
F3	0.1592	0.3034	0.5000	0	
G1	0.3046	0.3171	0.0165	0	
G2	0.1653	0.3326	0.5000	0	

Lilliefors Normal Distribution Test

Two-sample F-test for Equal Variance

Sample Number	Two-sample F-test for Equal Variance				
	р	h			
А	0.6070	0			
В	0.4446	0			
С	0.1781	0			
D1	0.6980	0			
D2	0.1556	0			
D3	0.0064	1			
E1	0.6990	0			
E2	0.4457	0			
E3	0.8288	0			
F1	0.5666	0			
F2	0.7222	0			
F3	0.4167	0			
G1	0.5441	0			
G2	0.6034	0			

Two-sample t-test

Sample Number	Two-sample t-test				
	ci1	ci2	р	h	
А	-0.9691	0.9381	0.9613	0	
В	-0.5831	0.6547	0.8628	0	
С	-0.2591	0.2409	0.913	0	
D1	-2.5062	2.5158	0.9955	0	
D2	-1.3478	1.4891	0.8862	0	
D3 (unequal)	-1.5352	2.3504	0.5064	0	
E1	-1.6964	1.7342	0.9747	0	
E2	-1.484	1.3931	0.9276	0	
E3	-1.6947	1.7329	0.9744	0	
F1	-2.0441	2.0783	0.981	0	
F2	-1.76	1.7568	0.9979	0	
F3	-1.9446	1.823	0.926	0	
G1	-0.522	0.4486	0.825	0	
G2	-1.0448	1.1333	0.9033	0	

Mean Comparisons (% Difference)

Sample Number	Mean Test 1 (MPa)	Mean Test 2 (MPa)	Percentage Difference (%)	Outlires
А	5.1574	5.1729	0.3000	
В	2.8348	2.7990	-1.2620	
С	2.4101	2.4192	0.3788	
D1	5.7278	5.7229	-0.0845	
D2	5.9332	5.8626	-1.1905	
D3	3.0927	2.6852	-13.1783	D3.9
E1	5.5238	5.5049	-0.3420	
E2	4.3708	4.4162	1.0397	
E3	4.2748	4.2558	-0.4466	
F1	4.0564	4.0393	-0.4213	
F2	3.0857	3.0873	0.0513	
F3	4.5829	4.6437	1.3273	
G1	11.1599	11.1967	0.3292	
G2	7.9725	7.9283	-0.5548	

Appendix G - Statistical Results - Two-sample Testing

Analysis Test Number	Sample Combination		Number Sample Combination Two-sample F-test for E		for Equal Variance
	Sample 1	Sample 2	р	h	
1	А	D2	0.0970	0	
2	В	E2	0.0146	0	
3	С	F2	2.2910E-05	1	
4	G1	G2	0.0652	0	
5	G1	F2	0.0010	1	
6	G2	С	0.0017	1	

Two-sample F-test for Equal Variance

Two-sample F-test for Equal Variance (Log Transformation Data)

Analysis Test Number	Sample Combination		Two-sample F-test for Equal Var	iance (Log Transformation Data)
	Sample 1	Sample 2	р	h
3	С	F2	0.0001	1
5	G1	F2	2.1146E-08	1
6	G2	С	0.6847	0

Two-sample t-test

Analysis Test Number	Sample Combination			Two-sam	ple t-test	
	Sample 1	Sample 2	ci1	ci2	р	h
1	Α	D2	-2.1679	0.6164	0.1231	0
2	В	E2	-2.8002	-0.2718	0.0027	1
3 (unequal)	С	F2	-2.0577	0.7064	0.1491	0
4	G1	G2	2.3894	3.9855	5.6322E-09	1
5 (unequal)	G1	F2	6.6869	9.4615	3.0820E-09	1
6 (unequal)	G2	С	4.6756	6.4492	3.1916E-08	1

Two-sample t-test (Log Transformation Data)

Analysis Test Number	Sample Combination		Two-sam	ple t-test (Lo	g Transforma	tion Data)
	Sample 1	Sample 2	ci1	ci2	р	h
3 (unequal)	С	F2	-0.6549	0.3556	0.3661	0
5 (unequal)	G1	F2	0.8802	1.8900	8.8327E-06	1
6 (unequal)	G2	С	1.0708	1.3199	1.3344E-13	1

Kruskal-Wallis Test

Analysis Test Number	Sample Co	mbination	Kruskal-Wallis Test
	Sample 1	Sample 2	р
3 (unequal)	С	F2	0.4239
5 (unequal)	G1	F2	0.0002
6 (unequal)	G2	С	0.0008

Two-sample Mean Comparisons

Analysis Test Number	Sample Combination		Mean Comparison (MPa)		Percentage Increase (%)
	Sample 1	Sample 2	Sample 1	Sample 2	Sample 1 vs. 2
1	А	D2	5.1574	5.9332	15.0418
2	В	E2	2.8348	4.3708	54.1856
3	С	F2	2.4101	3.0857	28.0336
4	G1	G2	11.1599	7.9725	39.9802
5	G1	F2	11.1599	3.0857	261.6617
6	G2	С	7.9725	2.4101	230.7958

Appendix H - Statistical Results – Three-sample Testing

Bartlett's Test for Homogeneity of Variance

Analysis Test Number	Sam	ple Combina	tion	Bartlett's Test									
	Sample 1	Sample 2	Sample 3	Bartlett's Statistic	р	h							
7	А	В	С	8.4272	0.0148	1							
8	D2	E2	F2	0.2000	0.9048	0							
9	D1	D2	D3	5.6238	0.0601	1							
10	E1	E2	E3	0.1560	0.9249	0							
11	F1	F2	F3	0.3062	0.8580	0							

One-way ANOVA

Analysis Test Number	Sam	ple Combina	tion	One-way ANOVA										
	Sample 1	Sample 2	Sample 3	F-statistic	р	h								
7	A	В	С	85.9376	7.7250E-11	1								
8	D2	E2	F2	13.1669	0.0001	1								
9	D1	D2	D3	15.5932	3.1500E-05	1								
10	E1	E2	E3	3.0664	0.0631	0								
11	F1	F2	F3	2.5587	0.0960	0								

Multiple Comparison Mean Tests

		Statis	tical Analysis 7											
Sample Comparisons Multiple Comparison Mean Test														
Sample 1	Sample 2	ci1	Diff in Means	ci2	р									
С	В	-0.9933	-0.4247	0.1440	0.1685									
С	А	-3.3160	-2.7473	-2.1787	1.1496E-09									
В	А	-2.8913	-2.3227	-1.7540	4.3976E-09									

		Statis	tical Analysis 8		•								
Sample Co	omparisons	Multiple Comparison Mean Test											
Sample 1	Sample 2	ci1	Diff in Means	parison Mean Test ci2 p 0.0929 0.0711 -1.4695 6.3253E-05 -0.1844 0.0239	р								
F2	E2	-2.6630	-1.2851	0.0929	0.0711								
F2	D2	-4.2254	-2.8475	-1.4695	6.3253E-05								
E2	D2	-2.9404	-1.5624	-0.1844	0.0239								

Statistical Analysis 9														
Sample Co	omparisons	Multiple Comparison Mean Test												
Sample 1	Sample 2	ci1	Diff in Means	ci2	р									
D1	D2	-2.7324	-0.7782	1.1759	0.5909									
D1	D3	1.4084	3.3625	5.3167	6.1965E-04									
D2	D3	2.1866	4.1408	6.0949	4.4682E-05									

		Statist	ical Analysis 10		
Sample Co	mparisons		Multiple Compa	rison Mean Test	
Sample 1	Sample 2	ci1	Diff in Means	ci2	р
D1	D2	-0.2387	1.1529	2.5446	0.1187
D1	D3	-0.1428	1.2489	2.6406	0.0849
D2	D3	-1.2957	0.0960	1.4877	0.9840

	•	Analysis	Test Number 11												
Sample Co	omparisons		Multiple Comparison Mean Test												
Sample 1	Sample 2	ci1	Diff in Means	ci2	р										
F1	F2	-0.6942	0.9706	2.6355	0.3325										
F1	F3	-2.1913	-0.5265	1.1383	0.7158										
F2	F3	-3.1620	-1.4971	0.1677	0.0841										

Three-sample Mean Comparisons

Analysis Test Number	S	ample Combinatio	ึ่งท
	Sample 1	Sample 2	Sample 3
7	A	В	C
8	D2	E2	F2
9	D1	D2	D3
10	E1	E2	E3
11	F1	F2	F3
Analysis Test Number	Me	an Comparison (N	1Pa)
	Sample 1	Sample 2	Sample 3
7	5.1574	2.8348	2.4101
8	5.9332	4.3708	3.0857
9	5.7278	5.9332	2.5606
10	5.5238	4.3708	4.2748
11	4.0564	3.0857	4.5829
Analysis Test Number	Pe	rcentage Increase	(%)
	Sample 1 vs. 2	Sample 2 vs. 3	Sample 3 vs. 1
7	81.9352	17.6205	113.9930
8	35.7465	41.6454	92.2787
9	3.5871	131.7102	123.6864
10	26.3785	2.2446	29.2152
11	31.4557	48.5180	12.9795

Appendix I - Manufacturing Standard Combination Table

Adhesive Bonding Process

		AREA:	SINGLE	OPER	ATO	R		S	ТА	ND	AR	DIS	ED	iss	ue D	ate							0	RIGH	WATC	8	C	HEC	KED.		штн	ORISI
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JOB CC	NTENT	TO						1		T/	ABL	.Е		TA	ктт	ME																
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	-			MANUAL	AUTO	WAT	MALK	L.,	5	10	1	5 2	0 2	5 3	30	35	40) 4	5	50	55	6	0	1							í.	
1	Mo	uld To	ol Preparation	0.5												111				111	1				Ш							
2	Con	sumab	les Preparation	0.5																												
3	Lay-	up & V	acuum Bagging	1												Ш					Ш											
4	Lam	ninate	under Vacuum		4																				Ш							
5		Wai	ting for 4			4															Ш				Ш							
6	Mix Inf	usion Re	esin/Infuse Laminate	0.5					П	TT						m	TT	III	111	III	TT	m	TT	TI	ΠŤ	III	ttt	Ħ	Ш	III	TT	m
7	Lar	ninate	Curing Cycle		24											III	III			III	TT	Ш			Ħ					III		III
8		Wait	ing for 7			23.5			Í							m	III	Ш		Ť	Π		TT	TT	III	Ш	III	Ħ	III	ŤΠ	T	IIII
9	Part Re	moval 8	Sizing of Adherends	4					İ											111	III				III							IIIT
10	Surfac	e Prep	(Both Adherends)	1																	Ш				Ш							III
11	Setup/	Calibrate	Adhesive Bonding #G	3												1	П				Ш				Ш							
12	Cu	ring of	Bonded Joint		11		\vdash	m		Ш			IIII		TII	III					Ш	Ш	TIT		ΠT	III	m		Ш	III		m
13		Waiti	ng for 12			11	\vdash						TTT			П					Ш				Π		Π			Ш		Ш
14	Part R	eroval a	nd Trim Excess Resin	1																												
			U																													
								KEY	C.					1	~	~				E		+	T	ota	1 8	ivai	lab	le	tin	ne	(se	ec)
	1	TOTAL	S	11.5	39	38.5		-	100	-			Water	0						1	AK	1	1	C	Cus	ton	her	[Den	ian	d	

Co-curing Process

		AREA:	SINGLE	OPER	ATO	R		ST	TAT	ND	AR	DIS	SEC		ssue	Date				-				DH	KIN	NTOR		CHE	CKED	5,9	NUTH	DRISS
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	-	-	ill andresses	MANUAL	AUTO	WAT	WALK		5	10	1	5 2	20	25	30	35	4	0 4	45	50	5	5	60							1	tre	
1	Mo	ould To	ol Preparation	0.5																				Ш	Ш							
z	Con	sumab	les Preparation	0.5	-		\vdash						111																			
3	Surfac	e Prep	(Metal Adherends)	1			\vdash																Ш		Ш							
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Appendix J – Mean (x̄) Statistical

Process Control (SPC) Charts





Appendix K - Test Statistics

The Lilliefors test statistic (D^*) is:

$$D^* = max \left| \hat{F}(x) - G(x) \right|$$

where $\hat{F}(x)$ is the empirical cumulative distribution function (CDF) of the sample data and G(x) is the CDF of the hypothesised distribution with estimated parameters equal to the sample parameters.

For the two-sample F-tests, the test statistic is:

$$F = \frac{{s_1}^2}{{s_2}^2}$$

where s_1 and s_2 are the sample standard deviations. The test statistic (F) is a ratio of the two sample variances.

The two-sample t-test, test statistic (t) is:

$$t = \frac{\bar{x} - \bar{y}}{\sqrt{\frac{{S_1}^2}{n} + \frac{{S_2}^2}{m}}}$$

where \bar{x} and \bar{y} are the sample means, s_1 and s_2 are the sample standard deviations, and n and m are the sample sizes.

Within Bartlett's test for homogeneity of variance, the test statistic (T) is:

$$T = \frac{(N-k)\ln(s_p^2) - \sum_{i=1}^k (N_i - 1)\ln(s_i^2)}{1 + \left(\frac{1}{3(k-1)}\right)\left(\left(\sum_{i=1}^k \frac{1}{N_i - 1}\right) - \frac{1}{(n-k)}\right)}$$

where s_i is the variance of the ith group, N is the total sample size, N_i is the sample size of the ith group, k is the number of groups, and s_p^2 is the pooled variance. The pooled variance is defined as:

$$s_p^2 = \sum_{i=1}^{k} (N_i - 1) s_i^2 / (N - k)$$

The test statistic has a chi-square distribution with k - 1 degrees of freedom under the null hypothesis.

Test statistics obtained from 'The MathWorks Inc, (2016)'.