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# FRACTURE BEHAVIOR OF TYPICAL STRUCTURAL ADHESIVE JOINTS UNDER QUASI-STATIC AND CYCLIC LOADINGS

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## ABSTRACT

Structural adhesive joints are expected to retain integrity in their entire service-life that normally involves cyclic loading concurrent with environmental exposure. Under such a severe working condition, effective determination of fatigue life at different temperatures is crucial for reliable joint design. The main goal of this work was thus defined as evaluation of fatigue performance of adhesive joints at their extreme working temperatures in order to be compared with their fracture properties under static loading. A series of standard double-cantilever-beam (DCB) specimens have been bonded by three structural 3M epoxy adhesives selected from different applications. The specimens were tested under monotonic and cyclic opening loads (mode-I) in order to evaluate the quasi-static and fatigue performances of selected adhesives at room temperature, 80°C and -40°C. The test results revealed that the fatigue damage occurred at relatively low load levels when compared to quasi-static fracture forces. At room temperature, the maximum cyclic fatigue forces varied between 25% and 40% of corresponding quasi-static fracture loads of selected adhesives. More significant reductions in adhesive mechanical performances were observed at 80°C. At cryogenic temperature, the adhesives had their own characterizations; mainly increasing the fatigue resistance but very sensitive to testing parameters such as loading rate or crosshead speed. In conclusion, the experimental observations showed a significant influence of fatigue loading on adhesive joints mechanical performances that should be considered in joint design, particularly at non-ambient temperatures.

## INTRODUCTION

Structural adhesives are now progressively used in different applications where the high strength bonds are required. Aircraft fuselages, ground transportation frames, renewable-energy like wind generators and adhesively bonded panels in construction applications are some good examples for structural adhesive joint applications. In many cases, it is often required to transfer cyclic loads concurrent with environmental exposure. Although this type of service condition is possibly the most destructive form of mechanical loading, yet the majority of durability tests and consequently the adhesive joint designs are based on the quasi-static test results.

At such destructive loading condition, the knowledge of fatigue behavior becomes strategic for designers to rely on adhesive joining for structural applications particularly in automotive industries. Design of reliable structural adhesive joints requires the effective determination of fatigue life at corresponding environmental conditions. Therefore, this research work was planned to aim the evaluation of fatigue performances of typical adhesive joints in a comparison with their static fracture properties. The specific goal of this work was then a comparison between fatigue behaviors of three selected 3M adhesives and their static fracture properties at their extreme working temperatures.

## SPECIMENS AND EXPERIMENTS

A fracture-based approach is used in this work to evaluate the temperature-dependency in monotonic and fatigue behaviors of three structural adhesives of 3M selected from different applications. Adhesive-A was a one-part epoxy adhesive with a lower elastic modulus that was developed to efficiently work at a wide range of working temperatures. Adhesive-B was a general-purpose non-sag two-part epoxy adhesive and the third adhesive was a film-based one part epoxy adhesive (Adhesive-C). The elastic properties of selected 3M adhesives are tabulated in Fig. 1. Three double-cantilever-beam specimens (DCB) were fabricated by 3M for each combination of selected adhesive and working temperature totally 27 adhesive specimens. The specimens were cut from the bonded aluminum plates (2024-T351, 1/2"x12"x12") that were treated as an FPL etch and phosphoric acid anodization process prior to bonding. A thin-layer primer was also applied and fully cured before applying the adhesive. In order to control the adhesive bondline thickness, the 150  $\mu\text{m}$  brass shims (635  $\mu\text{m}$  for Adhesive-C) were placed along three sides of aluminum plates and on the fourth side a Teflon tape was applied as a precrack. The DCB specimens were cut from bonded aluminum plates in accordance to ASTM standard D3433-05 with a final geometry schematically shown in Fig.1.

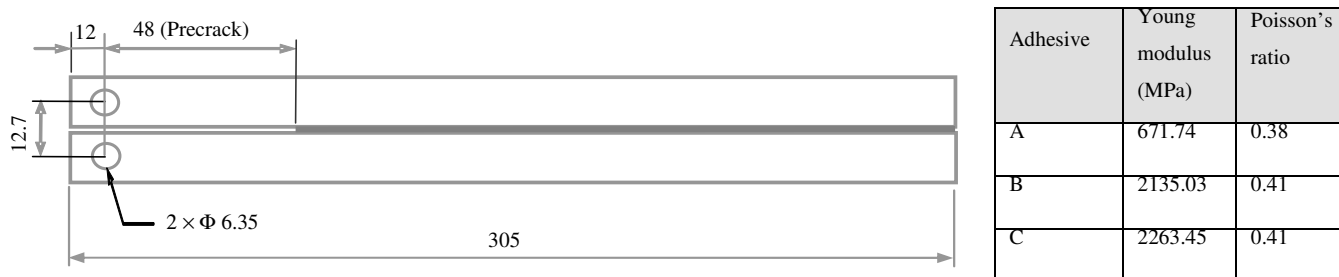


Fig. 1: Geometry of DCB specimens and adhesives elastic properties (All dimensions are in millimeter).

The specimens were tested under opening monotonic and cyclic loads (mode-I) by employing a novel fracture test technique in which some crack detection sensors were installed on one face of DCB specimen for controlling the testing machines by their signals. The machines alternatively changed the loading case from monotonic forces to variable-amplitude cyclic loads as crack broke a sensor. Two closed-loop servo hydraulic systems (810 MTS) together with the optical crack length measuring systems were used in this work. A dual-actuator (2 x 15kN) hydraulic machine equipped with two unidirectional hydraulic actuators was used for the tests at room temperature and 80°C as shown in Fig. 2a. This machine was especially designed for adhesive joint strength evaluation under complex loading modes which was equipped with a custom-made temperature chamber for the tests at higher temperatures. For the tests at -40°C, a standard 50kN hydraulic system equipped with liquid nitrogen (LN2) injected chamber was employed as shown in Fig. 2b. Due to limited space in this chamber, a loading fixture was fabricated to convert vertical actuator load into horizontal loads and allow

longer DCB specimens to be mounted vertically in the chamber. Fig. 3 shows both testing setups with the typical adhesive joints installed. It should be mentioned that the conformity of the test results obtained from different loading setups was verified through a series of validation tests with 50 kN load frame at room temperature and 80°C.

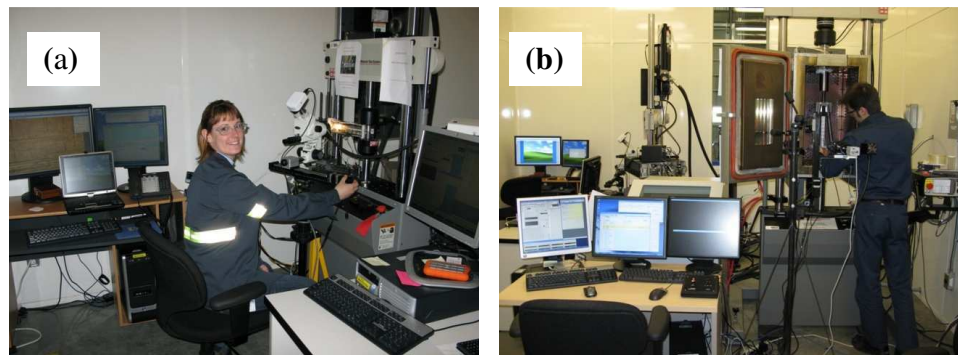


Fig. 2: MTS servo-hydraulic test units, (a) dual-actuator 2x15kN, (b) 50kN system.

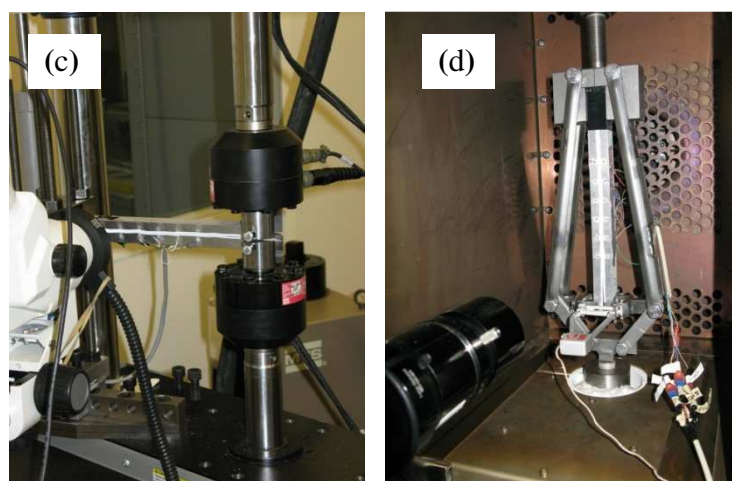


Fig. 3: Testing setup on (c) dual-actuator unit, (d) 50kN unit that was only used in cryogenic tests.

Each fracture test was started by a series of monotonic loading stages at room temperature in order to determine the quasi-static resistance of tested specimen. Five or six variable-amplitude cyclic tests were then performed under force control at a load ratio of 0.1 and a frequency of 8Hz. Prior to each cyclic loading, at least five more monotonic loadings were again applied to sharpen the crack tip and also to determine the local quasi-static resistance at testing temperature. Fig. 4 schematically shows the loading stages during a typical fracture test of this study. In quasi-static test stages (Quasi 1, Quasi2, ...), the testing machine was programmed for automatic fracture detection via the variation of joint compliance. After reaching the fracture point, the opening displacement was kept constant in 30 seconds for a finite crack propagation that was normally around 2 to 3 mm. The actuators were then returned back to 100 N load level before starting the next quasi-static stage. The maximum cycle force in each cyclic test (Cyclic1, Cyclic2, ...) was a percentage of local fracture force varying between 80% and 35% for the first and the last cyclic tests, respectively.

The advantage of this testing method is determining both the adhesive quasi-static and cyclic performances by using a single specimen. This ensures that the mechanical performances are determined under the same conditions of pretreatment, curing and fabrication. It also eliminates the operator interference and also the need of optical crack length measurements or other sophisticated experimental method for crack length determination.

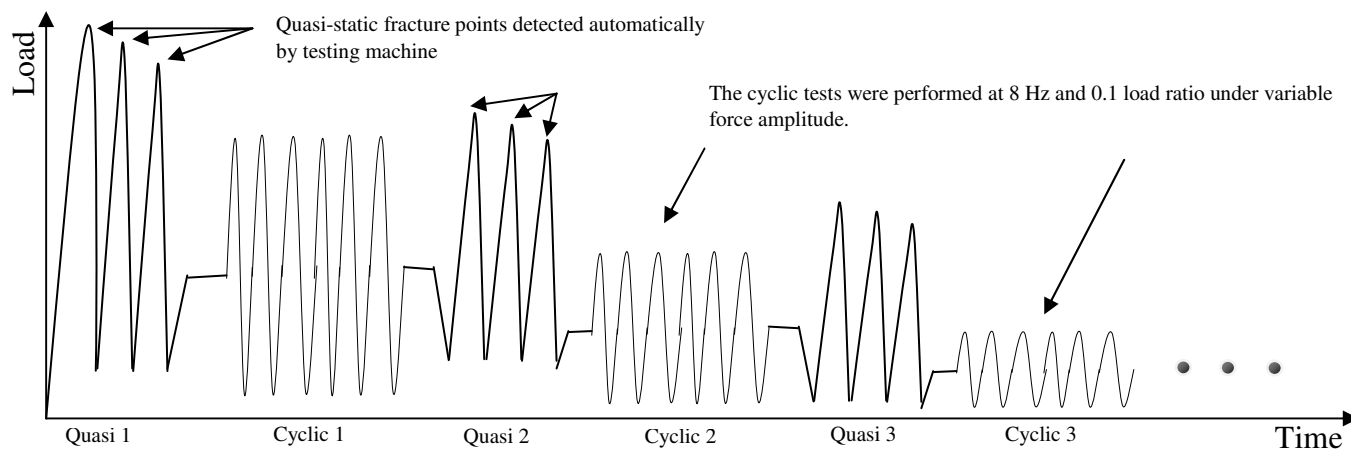


Fig. 4: Quasi-static and fatigue test sequences triggered by crack detection sensors.

The fracture surfaces were carefully examined after each test for any sign of interfacial or semi-interfacial fracture as shown typically in Fig. 5. This image clearly demonstrates a test sequence as well as quasi-static and cyclic zones. The cohesive failures represent the quasi-static zones while the crack propagation in the cyclic areas was dominantly interfacial or semi-interfacial for the specimen shown.

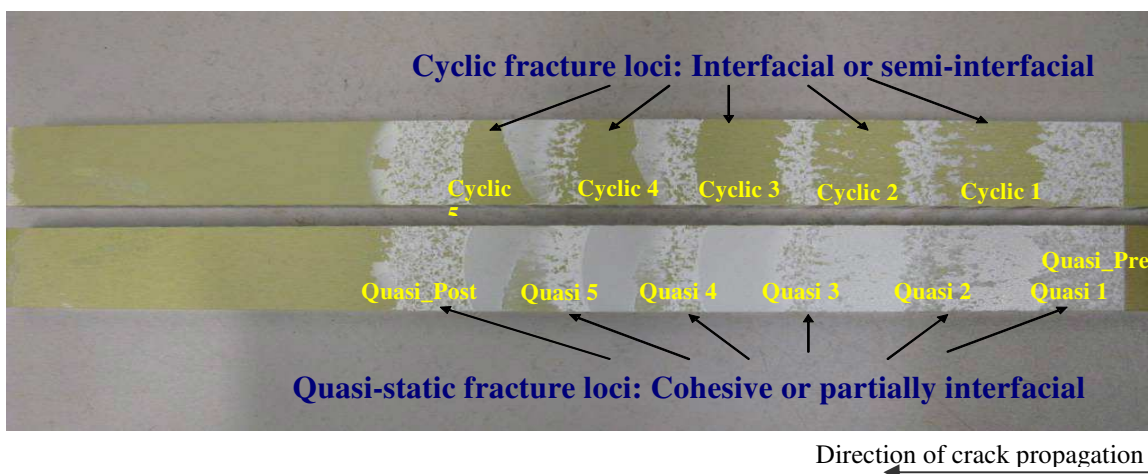
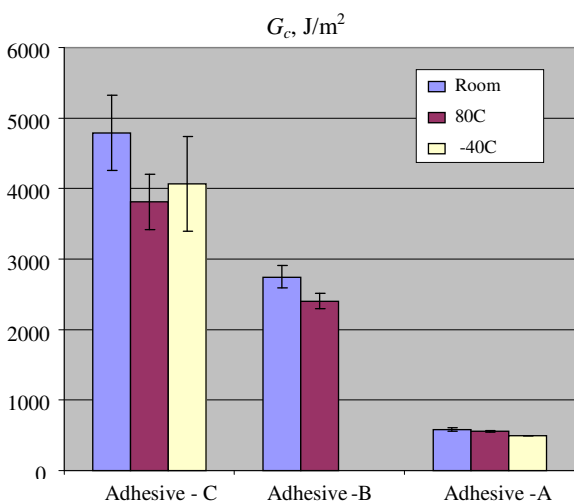


Fig. 5: Typical fracture surface for a specimen bonded by Adhesive-B.

## RESULTS AND DISCUSSION

The critical strain energy release rate  $G_c$  was calculated from fracture force and joint geometry at every monotonic loading stage and is used here as an index for quasi-static performance of tested specimen. The variation of  $G_c$  as a function of crack length, i.e.  $R$ -curve, was plotted for each fracture test. The average of  $G_c$  values over all the quasi-static stages at any temperature was calculated and tabulated in Table 1. A comparison is also made for the adhesive  $G_c$  values at different temperatures in Fig. 6. The highest average  $G_c$  value at any testing temperature corresponds to Adhesive-C that had the highest elastic modulus as well. Adhesive-A demonstrated a more stable behavior to temperature changes. It also had the lowest elastic modulus and the minimum deviation in the data as well. Moderate fracture toughness was obtained for Adhesive-B at room temperature and at 80°C but interfacial fracture to the primer layer was observed for this adhesive when tested at -40°C. Hence, its cryogenic performance was not fully determined in this study. The maximum fracture force of 4600N was measured for Adhesive-C at room temperature that was almost 3 and 1.5 times of corresponding fracture forces for Adhesives-A and Adhesive-B, respectively. Regarding the temperature dependency, we may say that Adhesive-A was almost insensitive to temperature changes, but a reduction of 12% and 20% in  $G_c$  values were observed at 80°C for Adhesive-B and Adhesive-C, respectively. At cryogenic temperature, no data was collected for Adhesive-B, while a 15% reduction in  $G_c$  value was determined at -40°C for Adhesive-C. In general, the fracture behavior of adhesives at cryogenic temperature was very sensitive to testing parameters such as loading rate or crosshead speed.



**Table 1:** The average  $G_c$  plateau values in J/m<sup>2</sup> at different temperatures.

Temperature	Adhesive A	Adhesive B	Adhesive C
Room	585	2749	4788
80°C	555	2404	3812
-40°C	494	N/A	4069

**Fig. 6:** The average  $G_c$  values for the tested adhesives at different temperatures.

For the cyclic tests, on the other hand, a power law curve was fitted to each fatigue curve that represents the variation of  $G_{max}$ , the maximum energy release rate in each loading cycle, as a function of  $da/dN$  or crack speed. The coefficient of power law is directly related to adhesive quasi-static  $G_c$  value, while the power factor could be assumed as an index for fatigue performance. In general, the fatigue performance of an adhesive can only be determined when its threshold strain energy release rate  $G_{th}$  is determined. But, one may say that the lower value of power-law factor represents smoother fatigue curve and then a better normalized fatigue resistance relative to its quasi-static  $G_c$  value. The power-law factors for the tested adhesives are tabulated in Table 2,

while Fig. 7 shows a typical power law curves fitted to fatigue data points obtained for Adhesive-C at three different testing temperatures. Evidently, there is a temperature dependency in power-law coefficients obtained for each adhesive that means the fatigue performances are affected by temperature variation. The maximum cyclic force  $F_{max}$ , at which a crack propagation of less than millimeter was observed after  $10^6$  cycles, was also determined and normalized to corresponding fracture force. Table 2 gives a summary of power-law factors (first row for each temperature) and normalized  $F_{max}$  for different adhesives at various temperatures. The most elevated  $F_{max}$  was 45% of corresponding fracture force that belongs to Adhesive-C at  $-40^\circ\text{C}$ . It occurred at 20% of its quasi-static  $G_c$  value ( $G_{max} = 815 \text{ J/m}^2$  that should be compared to  $G_c = 4069 \text{ J/m}^2$ ). Fig. 8 shows a comparison between fatigue curves obtained for the three adhesives at room temperature that clearly shows the best fatigue performance of this study corresponds to Adhesive-C.

**Table 2:** Power-law factor (1<sup>st</sup> row) and normalized  $F_{max}$  (2<sup>nd</sup> row) at  $10^6$  cycles for the adhesives tested at different temperatures.

Temp.	Adhesive A	Adhesive B	Adhesive C
Room	0.1782 39%	0.2032 26%	0.1311 39%
80°C	0.1377 41%	0.2663 17%	0.1928 22%
-40°C	0.1377 45%	---	0.0794 45%

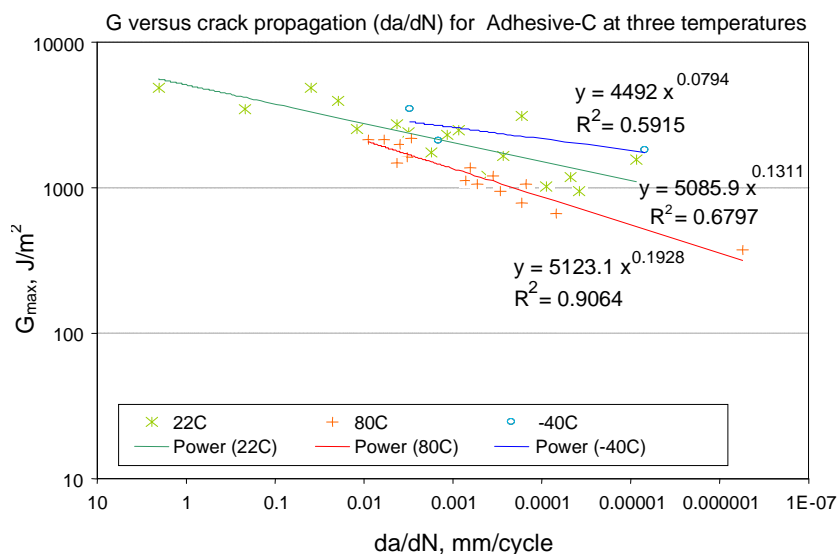


Fig. 7: Temperature dependency in fatigue performance of Adhesive-C.

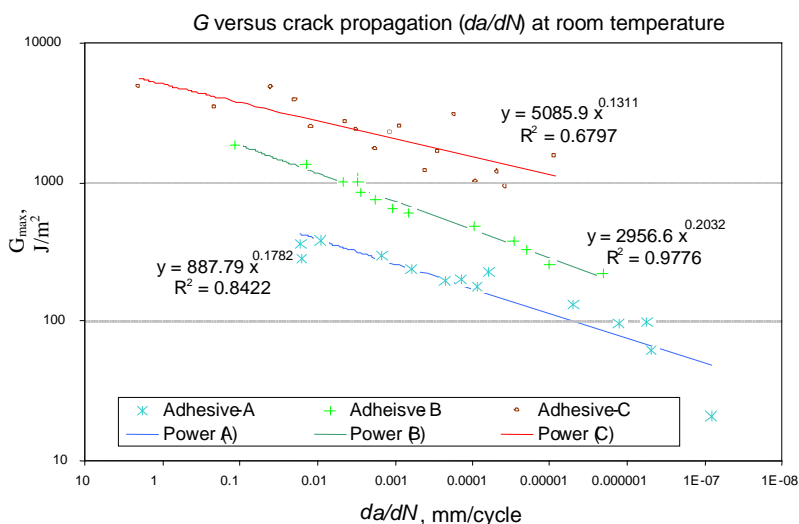


Fig. 8: Fatigue performances of selected adhesives at room temperature.

## CONCLUSIONS

A series of quasi-static and fatigue tests have been conducted in this work on double-cantilever-beam specimens (DCB) in order to evaluate the mechanical performance of three 3M adhesives under monotonic and cyclic loading cases at room temperature, 80°C, and -40°C. The test results of this study revealed that the best fatigue performance corresponded to Adhesive-C at different temperatures. Both quasi-static and fatigue performances of adhesives were affected by temperature changes. Indeed, the reductions of 5%, 13% and 20% were determined from quasi-static tests on Adhesives-A, B and C at 80°C, respectively. While the reductions of 16% and 15% were determined from quasi-static tests at -40°C for Adhesives-A and C. The fatigue performances of adhesives, on the other hand, were mostly decreased at 80°C, but an increase was observed at cryogenic temperature for the tested adhesives. The maximum fatigue forces were varying between 26% and 45% of the quasi-static fracture force at different temperatures. Therefore, we can conclude that there is a significant influence of fatigue loading on mechanical performance of adhesive joints that should be considered in adhesive joint design particularly at higher temperatures.

## REFERENCES

1. Kinloch and Osiyemi, Predicting the fatigue life of adhesively-bonded joints, *The J. of Adhesion*, 1993, 43, pp. 79-90
2. Ashcroft et al., Mode I fracture of epoxy bonded composite joints: 2. Fatigue loading, *Int. J. Adhesion and Adhesives*, 2002, 22, pp. 151-167.
3. Erpolat et al., A study of adhesively bonded joints subjected to constant and variable amplitude fatigue, *Int. J. Fatigue*, 2004, 26, pp. 1189-1196.

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