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MICROWAVE DIELECTRIC ANALYSIS ON ADHESIVE DISBOND IN ACRYLIC GLASS (POLY (METHYL METHACRYLATE)) AT KU-BAND

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ABSTRACT

A microwave dielectric spectroscopy for detecting adhesive disbonds between acrylic glass (aka Poly (methyl methacrylate)) was discussed. The adhesive bond was developed using epoxy resin and acrylate. The level of joint disbond can be quantified using Young Modulus. In this work, the strength of bond is affected by radius of air void within adhesive bond. A high-frequency electromagnetic wave propagated through two joint acrylic glass with acrylate and epoxy adhesive using waveguide adaptor WR90 in conjunction with professional network analyser. This electromagnetic wave is reflected and transmitted at the bond interface due to mismatch impedance at adhesive bond. The output is a dielectric properties that characterizes the bond interface. The increment of Young Modulus leads to increment of dielectric constant and loss factor for epoxy resin and acrylates, respectively.

Keywords: adhesive disbond, dielectric constant, loss factor, young modulus.

INTRODUCTION

Body of aircraft is generally made of composite for long time and its usage is growing drastically. For the sake of reduction of fuel consumption, aviation industry is investing massive resources to reduce weight of construction material for aircraft body. One of the major issue in making aircraft body is to determine adhesiveness of joint between two composite panels. Reliable adhesiveness of joint at aircraft is crucial to pertain their service life. Disbond of joint in aircraft lap joint is usual but annoying in aviation industry. This flaw can cause massive maintenance fee. If it was overlooked, it might lead to severe tragedies, e.g. air crash or aviation accident. Mechanical adhesive bond inspection methods used in the aviation industry are not suitable because most of these methods are destructive. As a result, it is in urge to assess quality of adhesiveness during maintenance using an efficient detection method.

Acrylics (Polymethyl-Methacrylate or PMMA) is an amorphous thermoplastic which is transparent thermoplastic which used often as a replacement of glass in many application due to its optical properties and safety [1]. It is unaffected by moisture and displays a high strength-to-weight ratio. Chemically, it is the synthetic polymer of methyl methacrylate and its chemical formula is $(C_5O_2H_8)_n$. PMMA is always preference in many applications due to its moderate properties, versatile and cheap in cost. PMMA can be joined using epoxy and acrylate adhesive for much kind of purposes. Exposure of electromagnetic radiation would not cause any variation on mechanical properties of PMMA [2]. Characteristic of transparency will cause only absorption of light and ultraviolet radiation which is insufficient to cause molecular disbond in PMMA. As a result, it provides high weather resistance, especially long term exposure to extreme weather condition, e.g. sunlight and heat. This material used to be helicopter windows, motorcycle wind-deflectors, caravan glazing and automotive rear lights.

Currently, ultrasonics, thermography, X-radiography, shearography and etc were the research focus among researchers worldwide in detecting adhesiveness or adhesive failure at joint. The ultrasonic methods were widely implemented used during the manufacturing process [3]-[4]. However, a skillful operator is needed to ensure the optimum operation. Hence, the training is necessary to nurture a certified operator. Large grain size that found in bond of certain materials might results in energy lost. It subsequently shields the presence of defects at bond. It could lead to spurious indications and the misreading of signals.

Thermography [5]-[6] is difficult to generate accurate data from models that have no significant thermophysical and radiometric properties. Cameras used in thermography cannot be operated optimally at temperature below -50°C. The pre-measurement is time-consuming as it must ensure the heat distribution must be in equilibrium. In addition, the inhomogeneity on surface of material can cause inconsistent measurement.

Shearography [7]-[8] and x-ray radiography [9]-[10] have been implemented regularly in disbond detection. Meanwhile, background lightning and thermal noise could lead to the inaccurate measurement in shearography. X-ray radiography exhibit less sensitivity in detecting defect on flat surface.

The adhesive properties of epoxy resins are described by electric forces between the epoxy resin and the surface of the substrate. This electric force is due to polar component that presented in resin [11-12] and surface of the substrate. It leads to bonds between reactive sites in the resin and reactive or polar sites on the surface of the substrate. Component of hydroxyl groups that can be found in chain of epoxy resins bond strongly with oxide or hydroxyl surfaces through polar attractions. Surfaces of most of the inorganic material have high energy due to polarity.

Acrylates is a type of vinyl polymer. Acrylate monomers contain vinyl groups. This group is directly attached to the carbonyl carbon of the ester group. Some polymers (especially copolymers) contain the free acid, acrylic acid. The alpha and beta carbons are presented in vinyl group. High polar moment of carbonyl attract electron density from vinyl group. Hence, the alpha carbon has lower electron density than the beta carbon [13]. It has a huge effect on the polar moment of the monomer. In other words, electric polarization becomes possible for acrylates (and methacrylates as well).

Recently, the non-destructive technique. The typical frequency range for spectrum microwave is from 3 kHz to 300 advancement of microwave technologies is advocating the implementation of microwave techniques in solving engineering problem, especially development of GHz. The instrumentation becomes more complicated, when the frequency is higher [14]. There are many advantages by implementing microwave inspection, e.g. safe due to usage of non-ionizing radiation, simple operation procedure, low cost and etc. Many studied have been conducted in mechanical application [15]-[17]. The major concern of these studies is to develop a non-destructive, scientific, efficient, low cost, reliable, and portable detection system for adhesiveness failure.

METHODOLOGY

Sample Preparation

Block PMMA were prepared based on the dimension of waveguide WR62 (Ku Band) as specimen in this work. The specimen for Ku band was prepared in dimension as described in Figure-1(a). A jig was used to facilitate specimen preparation. The specimen can be fabricated through the adhesion on single surface of two PMMA. This jig was equipped with micrometer screw gauge which help to ensure thickness of adhesiveness between PMMA in 1 mm. Three specimens were prepared in small, moderate and large defect. It is determined according to radius of air bubble as listed in Table-1. The radius of air bubble is examined and measured through optical microscope.

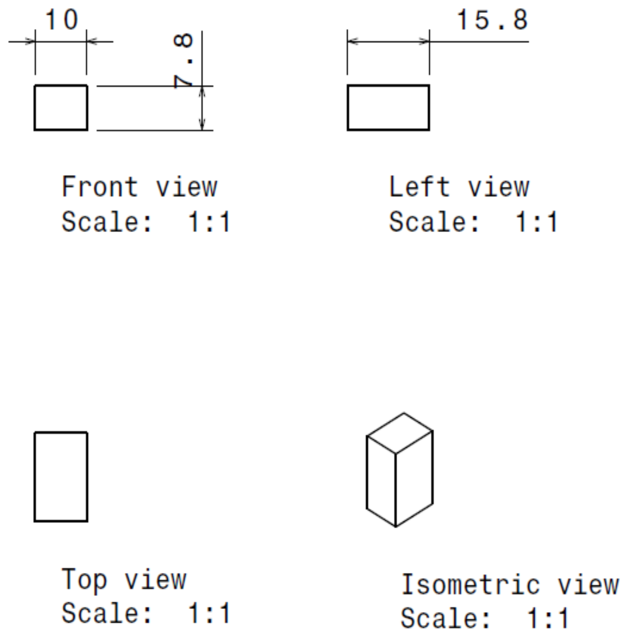

Figure-1. Technical drawing of specimen for specimen with dimension 15.8 × 7.8 mm.

Table-1. Average radius of air void in adhesive bond for specimen.

Dimension	Defect level	Radius of air void, mm	
		Epoxy	Acrylate
15.8 × 7.8 mm	Small defect	0.03	0.032
	Moderate defect	0.06	0.058
	Large defect	0.069	0.07

Waveguide

The technical drawing for this waveguide WR62 is as shown in Figure-2. The operating frequency for waveguide WR62 is within 12.4 GHz to 18 GHz (Ku-Band). In addition, the dimension for cross sectional area of sample plate holder must be in accordance with waveguide.

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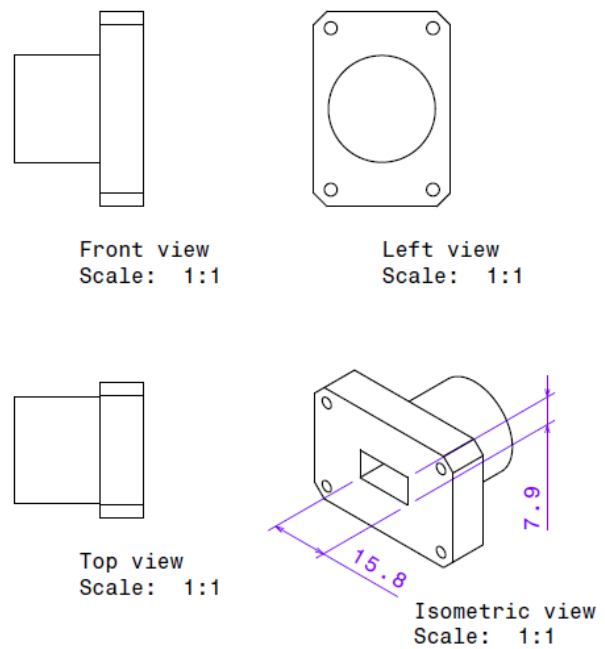

Figure-2. Technical Drawing of WR62 waveguide.

Experimental Setup

The dielectric and transmission-reflection measurement system are developed using X-band and Ku-band waveguide in conjunction with Agilent E8362B P-series network analyzer. The configuration of measurement setup is as shown in Figure-3. The specimens which prepared in required dimensions as shown in Figure-1 and Table-1 is placed in a sample holder. The sample in holder is connected with waveguide adaptors. The waveguide adaptors are connected to port 1 and port 2 of network analyzer, respectively prior to measurement.

In addition, Universal Testing Machine (UTM) was used to gauge the strength of bonding between PMMA due to adhesiveness. The gauge length for all specimens is 15mm to facilitate measurement of tensile strength. In this test, failure stress, σ_f (MPa) was measured for every specimen which prepared with different grade of defect. These defects can be determined through radius of bubble presented in bonding joint. Failure stress is a maximum stress for UTM to break the adhesion of specimen.

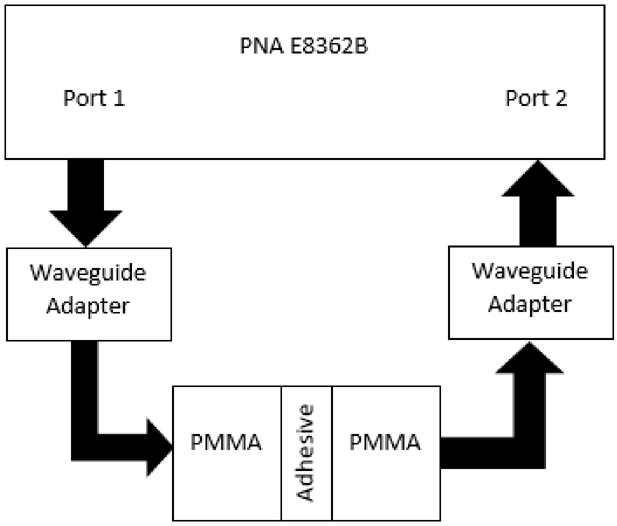

Figure-3. Working Flow of PNA.

RESULTS AND DISCUSSIONS

Tensile Test

Three sample with different degree of defects were prepared for tensile test. They have been quantified by using radius of air void. It can be noticed that larger defect exhibit larger radius of air void as listed in Table-1. The presence of air void cause defect to the joint bond (epoxy and acrylate) between PMMA. It weakens the structure of joint bond where interconnection between PMMA is inadequate. Subsequently, the large defect exhibit lower stress at failure if compare with small and moderate defect. It can be seen at Table-1 that large defect indicates the lowest Young's modulus. It implies that large defect cause low stiffness of joint bond. The joint bond between PMMA due to epoxy and acrylate has high tendency of deformation with considerably low of Young modulus. It can be justified by lowest Young modulus among the category of defect. In contrary, The Young's modulus which exhibited by small defect is the highest. In other words, high stiffness that attributed to high Young's modulus lead to highest stress at failure. The deformation is quantified by strain at failure. In this work, the strain at failure is similar for these three levels of defect. However, the applied stress (force per unit area) is the highest for small defect. Suffice to say, the joint bond for small defect is the strongest and deformation is hardly to be detected with considerably low applied force.

Table-2. The Young's Modulus, strain and stress at failure for specimens with epoxy adhesive.

Category of defect	Strain at failure (mm/mm)	Stress at failure (MPa)	Young's Modulus (MPa)
Small	0.050	11.73	380.65
Moderate	0.047	9.27	368.05
Large	0.027	8.50	306.43

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Table-3. The Young's Modulus, strain and stress at failure for specimens with acrylate adhesive.

Category of defect	Strain at failure (mm/mm)	Stress at failure (MPa)	Young Modulus (MPa)
Small	0.049	14.271	444.391
Moderate	0.046	11.012	397.922
Large	0.024	8.552	359.684

DIELECTRIC MEASUREMENT

Epoxy Adhesive

In Figure-4, the level of defect has been quantified by stress at failure as listed in Table-1. ε' for these three samples increases with frequency. When the applied field is switched during oscillation, the component of hydroxyl groups that can be found in chain of epoxy resins (polar component) takes characteristic time for polarization to adjust. This period of time is known as relaxation time, τ. When the applied field oscillate at direction with frequency lower than relaxation frequency (number of complete oscillation made in 1 s, 1/τ) of hydroxyl group, the dipole orientation can keep up with oscillated field. As frequency increases, the dipole orientation keep up with the oscillated field progressively. The synchronization between applied field and dipole orientation take place. This polarization mechanism contribute to increase ε'.

On the other hand, it can be noticed that sample with Young modulus of 380.65 MPa and 368.05 MPa exhibit the highest ε' over frequency. It is due to smaller air void which is represented by shorter radius of air void in joint bond (epoxy resin). These samples are indistinguishable as shown in Figure-1(a). The ε' of sample with Young modulus of 306.43 MPa is the lowest among other samples with joint bond using epoxy resin. It can be referred to Table-1 and Table-2 for further detail. The presence of air void causes the ε' decline. When the air void is significant in size, air void might dominate the sample. Hence, the larger air void (lower Young modulus) result in lower ε'. In addition, ε' is similar for Young modulus at 368.05 MPa and 380.65 MPa. It might due to like dispersion of air void and structure within joint bond.

Unlike ε', ε" exhibit two peaks at 14 GHz and 17 GHz as shown in Figure-4(b). The presence of these two peaks reflect the switch of polarization mechanism. The involved polarization mechanism might be interfacial polarization and dipole polarization. Interfacial polarization occur might be due to presence of air void in joint bond. Loss factor is high around the relaxation frequencies of the polarisation mechanisms. The inconsistencies between relaxation frequency and operating frequency of applied field, causing friction among the different molecules that present in sample. Dispersion of relaxation frequency due to these different molecule in inhomogeneous sample results in heating.

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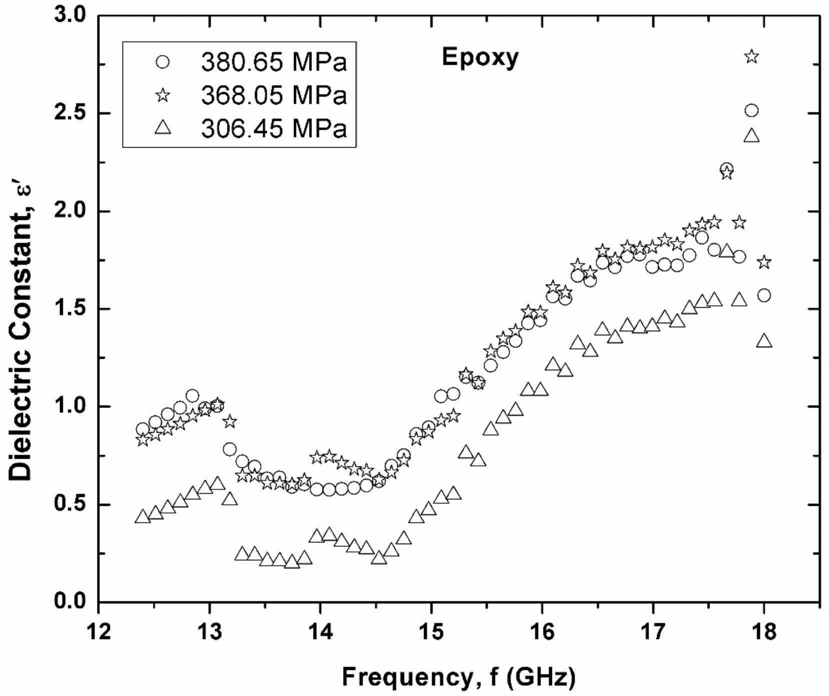
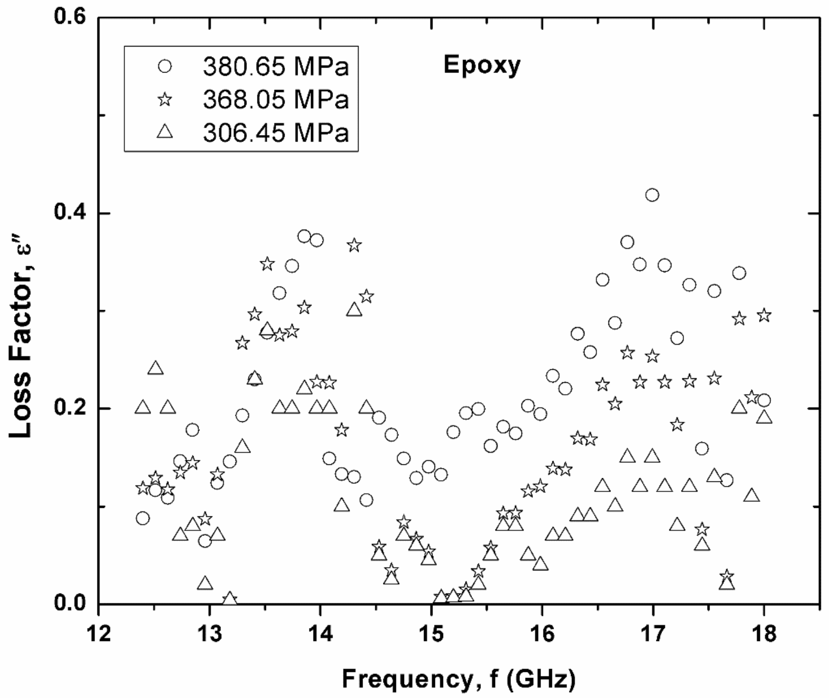


(a)



(b)

Figure-4. (a) Dielectric constant ε' and (b) loss factor, ε'' for small, moderate and large defect of epoxy adhesive in Ku-Band.

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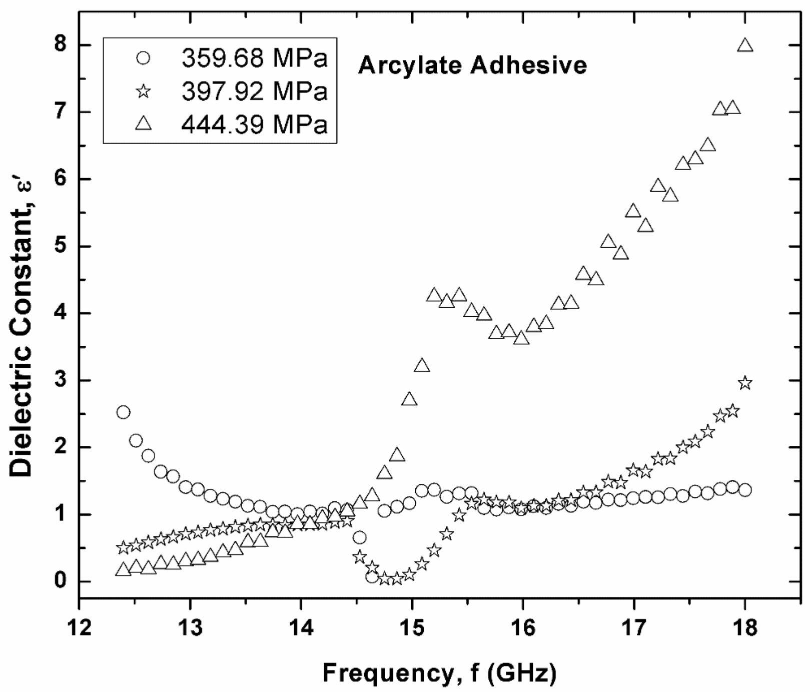
Acrylate Adhesive

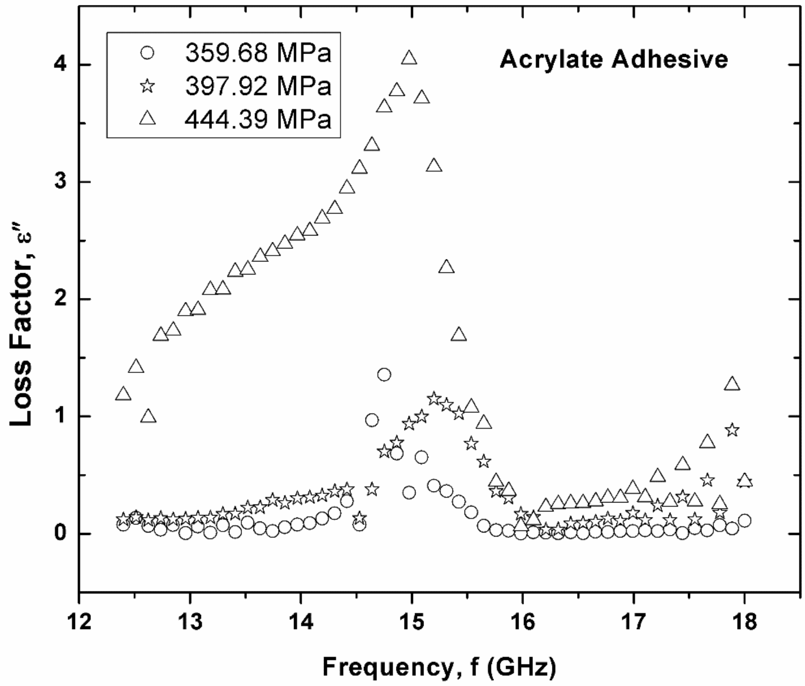
The small, moderate defect in joint bond made of acrylate adhesive is quantified by Young modulus. Similarly, larger air void (large defect) indicates higher Young modulus as tabulated in Table-3. In Figure-5(a), it can be observed that ε' behave like in Figure-4(a) where ε' increases with frequency. ε' increases when Young modulus decreases. As a result, specimen with Young modulus of 444.39 MPa (the highest Young modulus) exhibit highest ε' among the specimens and vice versa. More significant air void at joint bond lead to smaller Young modulus, as the air void dominate the joint bond. Carbonyl group with high polar moment in acrylate adhesive experience dipole rotation (orientation polarization). On the other hand, the presence of air void implies that interfacial polarization take place. However, it is less significant than orientation polarization that attributed to interaction between oscillating field and carbonyl group with high polar moment. It contributes to the highest ε' over frequency.

In the meantime, ε" exhibit peak at 15 GHz for these three specimens. Theoretically, ε" is higher in materials with higher ε'. It can be justified by specimen with Young modulus of 444.39 MPa where this specimen exhibit the highest ε' and ε". On the other hand, ε" increase over frequency from 12.5 GHz to 15 GHz. The polarization occur and keep up gradually with frequency of applied field within this frequency range. ε" is downside when frequency > 15 GHz. When frequency > 15 GHz, ε' still increases when frequency increases. Nevertheless, ε" decline over frequency. When relaxation frequency exceed operating frequency, the oscillating field lag behind the polarization. As a result, the direction switch has been delayed. The delay in time causes the friction ceases. Subsequently, ε" decline when frequency > 15 GHz.

Overall, ε' and ε" of acrylate as adhesive are higher than epoxy resin. Carbonyl group in acrylate has higher average dipole moment than hydroxyl group [18]. Higher dipole moment of functional group indicate higher polarization.



(a)



(b)

Figure-5. (a)Dielectric constant ε' and (b)loss factor, ε" for small, moderate and large defect (Young modulus) of acrylate adhesive in Ku-Band.

CONCLUSIONS

A transmission-reflection microwave measurement system which consists of two WR62 waveguide adaptors in conjunction with PNA is developed for the sake of joint disbond measurement. The measured results are presented in ε' and ε". It can be observed that ε' increase when frequency increases for epoxy resin and acrylate. Meanwhile, ε" exhibit a peak in the mid of frequency range (12 GHz to 18 GHz) for epoxy resin. ε" exhibit two peaks over frequency for acrylate. Smaller Young Modulus is attributed to larger size of air void in adhesive bond. Suffice to say, the measured dielectric properties present considerably good of performance in assessing the level of joint disbond. Larger size of air void which can be quantified by larger Young Modulus implies increment and decrement ε' and ε", respectively due to significant effect of air void for both types of adhesive and vice versa.

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