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A Preliminary Study on the Effects of Photoinitiator and UV Curing Exposure Time to the Mechanical and Physical Properties of the Epoxy and Vinyl Ester Fiber Glass Laminated Composites

J. Ramli[1], A.S. Hadi[1], A.R. Jeefferie[2] & M.M Mahat[3]
[1]Faculty of Mechanical Engineering, Universiti Malaysia Pahang, Lebuhraya Tun Razak, 26300, Kuantan, Pahang Darul Makmur, Malaysia.
[2]Engineering Materials Department, Faculty of Manufacturing Engineering, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100, Durian Tunggal, Melaka, Malaysia.
[3]Faculty of Applied Sciences, Universiti Teknologi MARA, 40450, Shah Alam, Selangor Darul Ehsan, Malaysia.
Corresponding Author E-mail: ramli@ump.edu.my

***Abstract*—Ultra-violet (UV) curing process is introduced in the curing of polymer matrix composites (PMC) for the application in producing bullet proof vest. Two types of adhesives or matrix materials were used: epoxy and vinyl ester. Each of them were mixed together with different types of photoinitiator; Bisacyl Phosphine Oxide (BaPO) and Alpha Hydroxyl Ketone Peroxide (AHK) at 1.0 and 10.0 per hundred of resin (phr) from the total proportion of the mixture. Sandwich construction of the PMC was done by hand lay-up process where the mixture was wiped on the fiber layers. Samples were then cured under various duration times which are 3, 6, 9 and 12 minutes to compare the quality of composites under the exposure of the UV light. The laminate was then tested to determine its characteristics of physical properties and its behavior to applied loads. Morphological observation through Scanning Electron Microscope (SEM) was performed in order to evaluate the quality of adhesion between each fiber layer and matrix wetting behavior. Vinyl ester is not recommended to be used as the matrix since two days is required to make it fully cured unlike epoxy which was cured rapidly once exposed under UV light. The physical testing shows that optimum density for the effect of photoinitiator obtained at 1.0phr for epoxy and 10.0phr for vinyl ester. This could give benefit to the manufacturer since it gives lower weight compared to virgin matrix materials. On the other hands, the effect of curing time shows the optimum result for density was obtained at 12 minutes of curing time. The effect of photoinitiator to the tensile strength shows optimum results at 1.0 phr for both epoxy and vinyl ester while curing time between 6 to 9 minutes is proposed to be optimum for the exposure of composites under UV light. Effect of photoinitiator to the hardness of the PMC is found optimum at the present of 1.0 phr for both matrix materials and optimum at 3 minutes to be exposed under UV light for the effect of curing time.**

***Index Terms*—UV Curing, Photoinitiator, Curing Time Effects, Mechanical, Physical, Morphological**

I. INTRODUCTION

Ultraviolet (UV) curing is known to be a hardening process of liquid material when it is exposed to UV radiation. The curing process of polymeric material by using UV light has played an important role in the processing of polymer material processing since the process is more advantageous than heat cure. Although curing by this technique is relatively slow compared to heat curing, the result is high strength and high impact properties of Polymer Matrix Composites (PMC) due to crystallinity enhancement of the polymers through cross-linking mechanism.

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J. Ramli and A.S. Hadi are the academic members of Faculty of Mechanical Engineering, Universiti Malaysia Pahang (UMP), Lebuhraya Tun Razak, 26300, Pekan, Pahang, Malaysia. The main author is now working at UMP. The corresponding author can be contacted through email at ramli@ump.edu.my while A.S. Hadi can be contacted at hadisalaam@ump.edu.my.

A.R. Jeefferie is an academic member of Faculty of Manufacturing Engineering, Universiti Teknikal Malaysia Melaka (UTeM), Hang Tuah Jaya, 76100, Durian Tunggal, Melaka, Malaysia. He can be contacted at jeefferie@utem.edu.my.

M.M Mahat is with Faculty of Applied Sciences, Universiti Teknologi MARA (UiTM), 40450, Shah Alam, Selangor Darul Ehsan, Malaysia. He can be contacted through email at mmuzamir@salam.uitm.edu.my.

One of the applications of the curing process of polymeric material by ultraviolet light can be seen in producing personal protective equipment (PPE) for instance bullet proof vest. This research is conducted as the preliminary stage in producing the bullet proof vest where the PMC will be used to wrap an alumina plate in order to act as holder when high ballistic impact hit the bullet proof vest. This will avoid alumina plate break into pieces once bullet is triggered to hit them.

The objective of this research is to develop an outside part of bullet proof vest and to assess the ability of the UV light in converting the thermosetting material to become harden or cure under UV exposure with the assistance of photoinitiator. In addition, it is also to study the parameter involve in the UV curing process of polymeric materials and their effects to the properties of the resulted products. This study has its own importance and benefits, which are to provide the fundamental understanding on the effects of UV curing to the physical and mechanical properties for the application of bullet proof vest manufacturing technique. The output of the cured PMC will then be studied and analyzed.

Although a particular substance to be processed may vary widely depending upon its application and the final use, they are basically composed of polymer. UV curing, a conversion process of polymeric materials from a liquid to solid by UV light is a popular alternative instead of conventional drying. The number and variety of applications for UV curable inks, coatings, and adhesives, continue to expand at a rapid pace and pose new design challenges to increase cure efficiency, speed, and the physical properties of the cured polymer film.

UV curing is highly adaptable to painting and coating, decorating, and assembling of a great variety of products owing to some of its key attributes. It is a low temperature process where heat is not required and a high speed process cure is nearly instantaneous. In addition, it is energy efficient processes whereby energy is invested only in the curing reaction, not in heating [1].

UV curing adhesive also have two components. One part is the adhesive resin itself and the second part already mixed in is called a photoinitiator. The secret of the photoinitiator is that, it will not react with the resin by itself. The photoinitiator must absorb UV light before anything can happen. When the UV light is delivered, the photoinitiator will undergo a chemical reaction and produce products that cause the adhesive to harden better. One type of photoinitiator used in this study is BAPO with Irgacure 819 as its trade name.

BAPO is a versatile photoinitiator for radical photo polymerization of unsaturated resins upon exposure to UV light. It has demonstrated useful application in white pigmented formulations, the curing of glass fiber reinforced polyester/styrene systems and for clearcoats for outdoor use with light stabilizers. The outstanding absorption properties of BAPO also allow curing of thick sections [2]. On the other hands, AHK is the second photoinitiator that used in this research with trade name Irgacure 184. This is highly efficient non yellowing photoinitiator, which is used to initiate the photopolymerisation of chemically unsaturated prepolymers in combination with mono or multifunctional vinyl monomers [2].

UV light has two important characteristics which are wavelength and intensity. For the photoinitiator to react correctly, it must be exposed to light of the correct wavelength and of sufficient intensity. Otherwise, the chemical reaction may not happen completely. The result will be poor or inconsistent in the adhesive performance [3].UV curing is dependent on photon molecule collision. The successful implementation of any UV curing process is dependent on the ease or difficulty of projecting photons into a curable material to activate photoinitiator molecules [1]. The optical properties of the curable material (polymeric resin) and the optical characteristics of the lamp must be matched to produce an effective UV curing system. In addition, there are several optical and thermodynamic characteristics of the material to be cured that interact with the radiant energy from the UV lamp and have significant impact on the process [1].

Typically, the radiant energy produced by the UV lamp is focused by a reflector onto the coating or ink. The UV energy striking the surface causes the photoinitiator to trigger the polymerization reaction. The material is usually solidified or dried when it exits the UV cure zone. The time, and consequently the space, required for cure is significantly less than thermal drying methods. Because the process relies on UV light to initiate the crosslinking of molecules, it does not evaporate any solvents nor significantly heat the substrate [4].

The application, particularly the end product it produces, will determine the requirements of the physical properties of the cured photochemistry. Target properties, such as opacity or hiding, film thickness, hardness or flexibility, resistance to abrasion or scratching, and adhesion, are only a few that are determined by the end product and the coating, decorating, or bonding process used. Identification of the substrate is not only important to properties, such as adhesion and flexibility, but will have a significant effect on the response to infrared radiation [2]. UV processing offers several advantages over other finishing methods.

Typical product lines involve coatings (on wood, metal, paper and plastic), inks (for letterpress, lithographic, gravure and screen printing) and adhesives (for film, foil or paper substrates). The industries using these technologies are diverse and varied, including automotive components, medical products, electronics, CDs and DVDs, two piece and three piece can print, pipe and tube coating, furniture, fiber optics, flooring, packaging and containers [4].

II. METHODOLOGY

A. *Raw Material and Samples Preparation*

Polymeric resins are the main raw material that was mixed together with photoinitiator. Both epoxy and vinyl ester act as binder in the mixture. It has been bought from Wee Tee Tong

Chemicals Pte. Ltd, Singapore. Meanwhile, photoinitiator material has been sponsored by Ciba Specialty Chemicals, Singapore. The photoinitiator was added to the matrix materials in the amount of part per hundred of resin (phr), with ratio of 1:3.

There are four types of samples that involved in this research. Sample A is a reference sample produced without photoinitiator while sample B, C and D is the samples added with photoinitiator material in certain ratio. Details on sample formulation are described in the following Table 1. Each sample A, B, C and D was mixed together and stirred to obtain homogenous solution. The total weight of resin is 120 gram. The composition ratio of BAPO to AHK is 1:3. At the first stage of sweeping process, a layer of woven roving was placed on a flat surface. Once mixture of resin and photoinitiator is homogenized, it was swept over the top of the glass using brush. After that, another layer was put on the first glass laminate and the same procedure was repeated until a laminate of l0 layers are produced. This sandwich construction was done repeatedly for another sample. After the 10 layers of woven roving stick together, the laminate was then undergoing vacuum bagging process where it was put in a vacuum chamber.

TABLE I
DETAILS ON SAMPLE FORMULATION AND CURING STRATEGY

Sample	Details for each sample
A	*1. Woven roving + Vinyl ester* *2. Woven roving + Epoxy*
B	*Woven roving + Vinyl ester + BAPO + AHK ;* *(BAPO + AHK = 1.0 phr)* *1. 3 minutes* *2. 6 minutes* *3. 9 minutes* *4. 12 minutes*
C	*Woven roving + Epoxy + BAPO + AHK ;* *(BAPO + AHK = 1.0 phr)* *1. 3 minutes* *2. 6 minutes* *3. 9 minutes* *4. 12 minutes*
D	*1. Woven roving + Vinyl ester + BAPO + AHK ;* *(BAPO + AHK = 10.0 phr)* *2. Woven roving + Epoxy + BAPO + AHK ;* *(BAPO + AHK = 10.0 phr)*

By performing this step, the adhesion between the layers was improved and the entrapped air was expelled. After the vacuum bagging stage, the glass laminate was exposed to UV light. Once the process completed, sample was taken out and ready for testing and analysis procedure. For sample A and D the exposure time under UV light is 6 minutes while for sample B and C, the exposure time for each resin vinyl ester and epoxy was varied to 3, 6, 9 and 12 minutes.

B. Sample Testing

Density Test

Density test was carried out in order to study the physical properties of UV cured laminate composites produced with parameter that has been varied for instance type of adhesive used to develop sandwich construction between each layer of glass, the time of curing and the amount of photoinitiator added in the adhesive resin. This test was conducted by applying the Archimedes principle.

Tensile Test

Tensile test was carried out in accordance to ASTM D 3039 standards. The test was carried out at a crosshead speed of 2 mm per minute. Specimens for the tensile test were produced by cutting out laminate strips measuring 200 mm x 25 mm.

The specimens were cut 3 mm oversize and final dimensions obtained by grinding and using sand paper to produce smoother end surface. Aluminum end tabs of 3.2 mm thick and measuring 50 mm x 25 mm was locally bonded onto the both ends of each laminate.

A total of 12 specimen strips were cut from each of the composite laminates produced. For this testing, three series of sample were produced. Series 1 is sample A produced without photoinitiator material added in the adhesive resin with 6 minutes of curing time. Series 2 are sample B and C where the curing time was varied to four different exposure time under the UV light and photoinitiator material at 1.0 phr was added to the resin.

Third series is sample D where the adhesive resin of this sample was added with 10.0 phr photoinitiator and the curing time is 6 minutes. The purpose of this testing is to study the effects between the absence and presence of photoinitiator in the resin and to study the effects in curing by increasing the exposure time under UV light exposure. Figure 1 shows samples for tensile testing.

Fig. 1. Specimen for tensile test ASTM D3039

Hardness Test

Hardness test was performed to the samples in order to know the resistance of surface penetration to the glass laminates that has been cured under UV light. The hardness test was conducted in the Brinell hardness test mode with diameter of indenter is 10 mm and 100 N of loads.

C. *Morphological Observation by Scanning Electron Microscope (SEM)*

The purpose of this observation is to determine if proper adhesion and fiber wet out occurred in each of the laminates. SEM used for this observation is the variable pressure scanning electron microscope.

III. RESULT AND DISCUSSION

Physical Testing of Density Behavior

The purpose of the density test is to observe the effect on the physical properties of UV cured laminate composites produced by different type of resin, to study the variation of curing time under the UV light and the presence of photoinitiator amount in the polymeric resin. Generally, the density of the vinyl ester used is 1.35 g/cm^3 while for the epoxy; the density is 1.50 g/cm^3. Figure 2 shows the plotted data of density versus sample at different amount of photoinitiator addition.

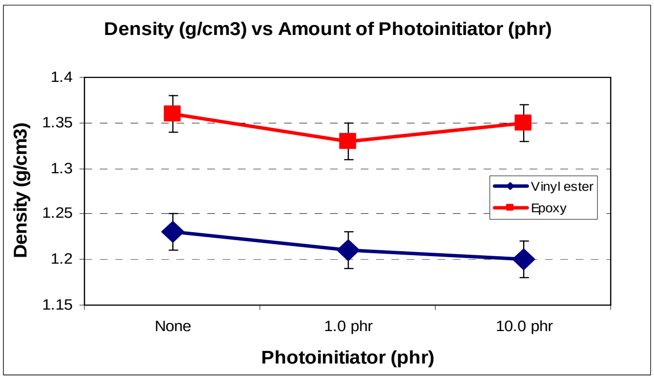


Fig. 2. Density at different amount of photoinitiator addition

Samples using epoxy as an adhesive have greater density as compared to samples using vinyl ester. This result is in accordance with the density data of both virgin resins where epoxy's density has greater value than vynil ester. The plot also indicates that the resulted density of the laminate composite is not affected by the presence of photoinitiator. It might be due to the little amount of photoinitiator weight used.

In this case, photoinitiator exists in the resin as an additive material. It acts as the catalyst to initiate the process of curing, thus, resulting in a monotonic change of density for each sample and not far varied compared to the sample produced without photoinitiator. It tells that, the presence of photoinitiator materials in the mixture is not affecting the value of density of the produced sample.

Figure 3 shows the effects of curing time under the UV light exposure to produce composites of samples B and C. Based to the plot, the density value for sample B seems to increase constantly with the increasing of curing duration.

For sample C, the plotted data shows a bit increment as a time of curing is increased. This indicates that the exposure time under the UV light affects the density value of UV cured laminates. It may caused by the atom of resin that might have enough energy to move in order to make better arrangement between them while curing is in progress. In addition, this will allow the fibers to make better linkage due to curing with polymer chain as time of exposure under UV light increases. Chain in the resin will have more time to arrange the fiber closer to each other. Thus, the probability for the void to exist was minimized by longer exposure time of UV light. The void occurred preferentially at the ply interfaces and grew quite large [5].

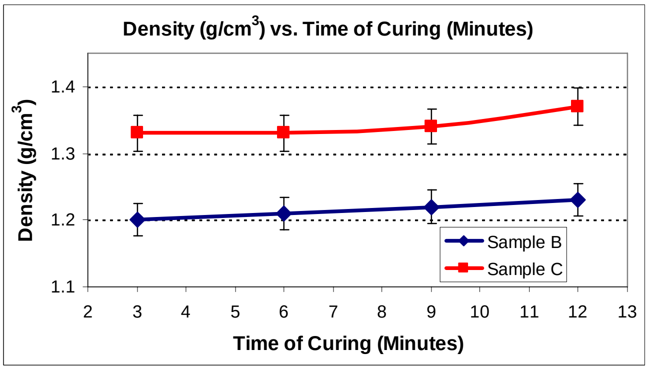


Fig. 3. Density of samples at various curing time exposure

Mechanical Testing of Tensile Strength

Figure 4 shows the plotted data for tensile strength versus samples at different amount of photoinitiator. From the graph, the tensile strength for the sample wiped with epoxy have greater values compared to sample that used vinyl ester as their adhesive material.

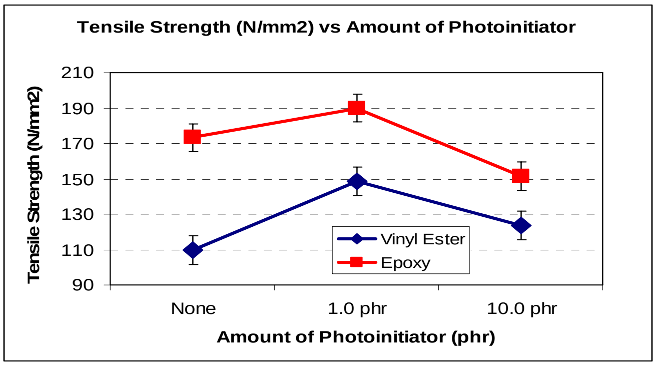


Fig. 4. Tensile strength of samples at different amount of photoinitiator

The graph indicates that, for the series using vinyl ester resin and epoxy as adhesive, the tensile strength was optimum at 1.0 phr of photoinitiator addition. Proper adhesions between the glass fiber woven roving and the polymeric resin are achieved at this point and lead to higher value of tensile strength. Good adhesion between both phases indicates lower voids presence in the fabricated composites. However, with the presence of 10.0 phr of photoinitiator in the resin, the tensile strength was decreased significantly for both sample produced by epoxy or vinyl-ester. This indicates that, the presence of photoinitiator in higher amount will not contribute to the increase in tensile strength behavior of the fabricated composites.

The function of photoinitiator in the resin is to act as energy absorber to initiate the process of curing. This implies that, the presence of photoinitiator in the mixture cannot be in a large amount since it is just an accelerator to initiate the curing process. This is because chain of resin that bonds the fibers together becomes weaker as the amount of photoinitiator increases to a huge amount. This is the reason why photoinitiator is added as an additive material in the mixture at a little amount. The reduction of mechanical properties might due to the part warpage caused by over-curing introduced by larger amount of photoinitiator addition [5].

Figure 5 shows the plotted data for tensile strength versus time of curing for both sample in group B and C. 1.0 phr photoinitiator material was added into the adhesive resin for sample B and C. From the graph, both of the tensile strength curves showed the strength peaks at certain value of exposure time. Therefore, in overall, distribution data shows that, series of sample C have greater tensile strength compared to the series of sample B.

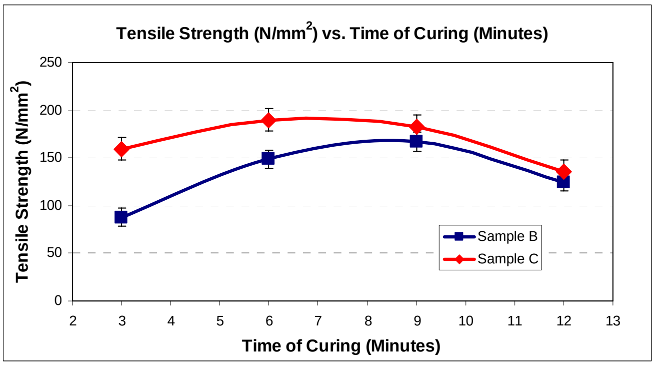


Fig. 5. Tensile strength of samples at different curing time exposure

From the plotted graph, the value of tensile strength for 3 minutes of curing exposure is the lowest compared to all data obtained in the testing result for the series of sample B. This might due to not enough curing time of 3 minutes for the adhesive to complete the transformation phase from liquid to solid under the UV light exposure. Thus, it produces weak bonding between each fiber between the polymer matrixes. The glass fiber woven roving inside the laminate is still wet, thus not strongly bonded between each other.

For 6 minutes of exposure time, the mechanical tensile strength of composites for both polymer matrixes were getting stronger compared to 3 minutes of curing exposure. The wet fibers become completely dried, making the adhesion bonded strongly.

For further 3 minutes of curing exposure, for the sample B3, the tensile strength was found to increase further, however for curing time up to 12 minutes, the tensile strength decreased. At 12 minutes of curing time the fibers become charred where it will not act as reinforcement and the ability to bond between each particle become limited.

For sample C, 3 minutes of curing under the UV light exposure shows greater tensile strength. This is due to the adhesive itself which is epoxy that has better properties compared to vinyl ester. The adhesive between the fibers is strongly bonded for epoxy resin. However, the limitation of the epoxy is that, it will not cure as rapid as vinyl ester. After UV curing step, it needs another 2 days of drying period at room temperature to be fully cured and hardened.

After another 3 minutes of curing exposure, the tensile strength was increased further. This means that, higher force is needed to break the sample because better properties were achieved for sample C.

As the exposure time increased, the result showed a constant value of tensile strength but there was a bit reduction occurred at 9 minutes of exposure time. This means that for epoxy resin with 1.0 phr of photoinitiator addition, the sample achieves peak value between 6 to 9 minutes of exposure time. As the exposure time increased until 12 minutes, the value of tensile strength was decreased.

Hardness Test Evaluation

Figure 6 shows the plotted data of hardness versus samples that were produced in different amount of photoinitiator addition. The graph consists of two series of samples. Series 1 is the sample that uses vinyl ester as adhesive while series 2 is the sample that uses epoxy as adhesive material and all were cured at 6 minutes under the UV light exposure.

Based on the graph, for vinyl ester sample A1 which produced without photoinitiator, the hardness is lower compared to the sample that was added with photoinitiator material, which is sample D1.

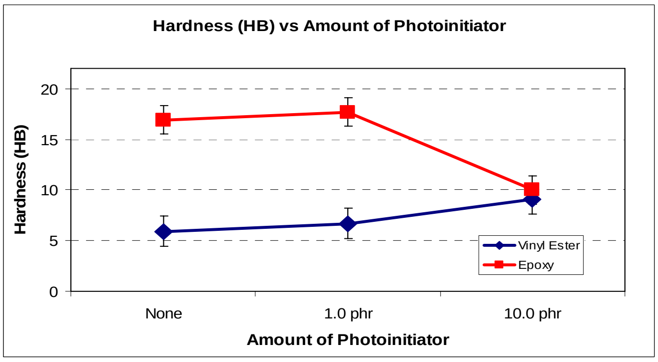


Fig. 6. Hardness of Samples at Different Amount of Photoinitiator.

For series that used epoxy as adhesive resin, the hardness between sample with and without photoinitiator shows a decrement in its value. The hardness for sample A2 is much higher compared to the sample D2. This is because; the distribution of adhesive resin during the wiping process was not even from one part to another. This again contributes to the variation of hardness value for the two samples with and without photoinitiator materials.

Thus, from the result, it can be concluded that the value of hardness is not affected by the presence of photoinitiator material.

Based on Figure 6, the plotted graph indicates that vinyl ester have lower value in term of hardness as compared to laminate composite that used epoxy as adhesive. For sample A1, the hardness data obtained is the lowest where no photoinitiator was added to the resin.

For sample B2 where 1.0 phr photoinitiator was added to the resin, the graph shows an increment. The value elevated from 5.9HB to 6.7HB. This shows that hardness of material increases with the presence of photoinitiator. Perhaps, the fibers inside the laminate were bonded tightly with the presence of photoinitiator. For sample D2, where photoinitiator was added with 10.0 phr, the hardness was increased too. This might be due to the effectiveness of photoinitiator to act as energy absorber to initiate the process of curing and produces harder laminate.

For series that using epoxy as adhesive resin, the hardness of laminate is greater as compared to laminate that used vinyl ester as adhesive. For 6 minutes of curing without photoinitiator addition, the data obtained is 16.9HB. For sample C2, with 6 minutes of curing time and the adhesive added with 1.0 phr photoinitiator, the hardness of laminate composite was increased to 17.7HB. This shows that, the presence of photoinitiator helps the composite laminate to become harder, hence, to have better properties. After 10.0 phr photoinitiator added to the resin, sample D2 shows a drastic decrement in its value. Addition of 10.0 phr of photoinitiator in the epoxy is large enough to act as accelerator where at one point it reaches yield point and out of it real function as photoinitiator.

Figure 7 shows the plotted data for hardness of material that was cured under the UV light with different exposure time. Series of sample B represent samples that were wiped with vinyl ester as adhesive resin and series of sample C represent samples that were wiped by epoxy as adhesive material.

Based on Figure 7, the plotted graph shows that epoxy which is represented as series of sample B have greater hardness value. For series of vinyl ester sample, sample B1 shows that, with 3 minutes of exposure time, the hardness is better compared to sample B2 even B2 was exposed under the UV light for longer period which is 6 minutes. The comparison can be clarified by the idea of uneven adhesive distribution on glass laminate during the wiping process. The resin of adhesive was probably wiped on the glass layers in an uneven condition where the resin distribution may slightly varied at different places on the same plate.

In addition, void may exist in the fiber layers that resulted in lower hardness value. Thus, during hardness test, the result obtained shows that the variation in hardness is depends on the resin distribution on glass laminate. Thicker adhesive wiped on glass laminate may have greater hardness value. For sample B3, which is 9 minutes exposure time under UV light, there is a bit increment in hardness values as compared to the 6 minutes of curing time.

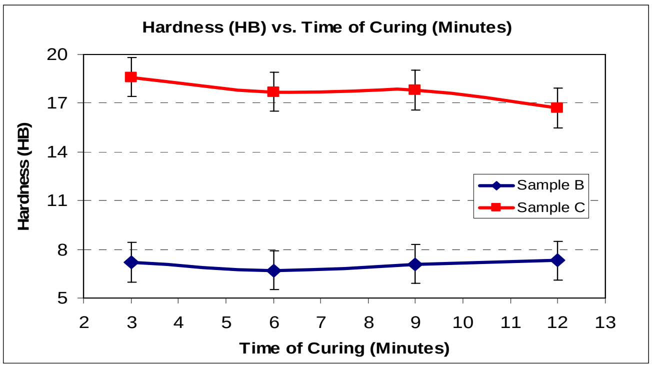


Fig. 7. Hardness of Samples at Different Curing Time

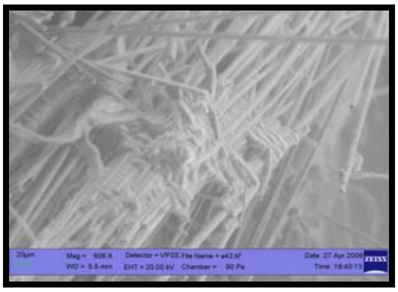
However, for 6 minutes of curing time, the value of hardness for sample C2 shows a bit decrement. The value increases slightly at 9 minutes of curing time for sample C3 but decreases again for sample C4 down to 16.7 HB. Thus, from the data obtained, it is indicated that, with the increasing of curing time, only series of sample that used vinyl ester as adhesive shows an increment in term of hardness. In contrast, the value of hardness for epoxy is decreased. Thus, in overall, it can be clarified that the variation of curing time gives no effect to the hardness of UV cured laminate.

Morphology Analysis

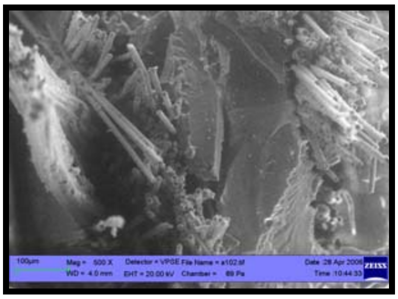
In addition to the physical and mechanical properties, morphological observation through the Scanning Electron Microscope (SEM) was also carried out on both of UV cured laminates that use vinyl ester and epoxy as adhesive material.

The micrographs of the selected UV cured laminate are shown in Figure 8 and it was taken at the point of tensile fracture. It clearly shows that the fiber strands have very little resin impregnated along their length. This could probably be indicative of improper adhesion between the fiber and the matrix during the curing time. The surface of fiber is covered by the matrix in an uneven condition.

In addition, the existing of void can also be seen. The presence of voids affects the result especially to the mechanical properties of the UV cured laminate. However, at the point of fracture the fiber strands appear to pull out cleanly with little evidence of resin impregnated along their length. However, no fiber wet out found in the fracture surface of the sample when observation was conducted. All samples have good well dried adhesives indicate that the matrix solution was cured enough before testing is conducted.



Sample B2



Sample C4

Fig. 8. Micrograph of selected sample of UV cured laminated composites

IV. CONCLUSION

In this research, several observations like mechanical and physical properties have been done to the UV cured laminated composites with respect to the processing parameter of UV curing exposure time and the effect of photoinitiator addition to matrix materials. Two types of adhesive materials used indicate that the vinyl ester was not suitable to be used as adhesive since it needs 2 days to be cured after being exposed under the UV light exposure.

On the other hands, epoxy can cure rapidly after 3 minutes of exposure time under the UV light. Thus, the use of epoxy in the application of UV cured laminate composite is a right option if compared to vinyl ester. Moreover, it was clearly showed that the density of epoxy was higher compared to vinyl ester. Presence of photoinitiator in the matrix materials was found not really affecting the density of the produced UV cured laminated composite.

However, as the exposure time under the UV light was increased, the density of the PMC was increased as well. This tells that, voids within the substrate were reduced or eliminated due to curing mechanism. For tensile strength, the value was higher for epoxy in comparison to vinyl ester and the value was not affected by the presence of photoinitiator.

In addition, the tensile strength was reached it peaks value between 6 to 12 minutes of curing time. An additional of 1.0phr photoinitiator shows the increment in tensile strength but caused sudden decrement in its value with 10.0phr of photoinitiator was added. Hardness of UV cured laminate was found not affected by the presence of photoinitiator. The curing time to the substrate also not affects the value of hardness. Furthermore, through the morphological observation by SEM, it was found that proper adhesion is not achieved between the layers of fiber and the adhesive matrix. Overall, further study need to be carried out in order to fully discover the potential of proposed materials and curing technique as well as the role of photoinitiator especially for bullet proof vest application. Results and properties obtained from this preliminary investigation is still need to be expanded by some other study to achieve the optimum information with regards to the contribution of UV light in converting the matrix materials from liquid to solid.

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