

Fiber Reinforced Polyester Resins Polymerized by Microwave Source

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Polyester resin based composite materials are widely used in the manufacture of fiberglass boats. Production time of fiberglass laminate components could be strongly reduced by using an intense energy source as well as microwaves. In this work a polyester resin was used with 2% by weight of catalyst and reinforced with chopped or woven glass fabric. Pure resin and composite samples were cured by microwaves exposition for different radiation times. A three point bending test was performed on all the cured samples by using an universal testing machine and the resulting fracture surfaces were observed by means of scanning electron microscopy (SEM). The results of mechanical and microscopy analyses evidenced that microwave activation lowers curing time of the composite while good mechanical properties were retained. Microwaves exposition time is crucial for mechanical performance of the composite. It was evidenced that short exposition times suffice for resin activation while long exposure times cause fast cross linking and premature matrix fracture. Furthermore high-radiation times induce bubbles growth or defects nucleation within the sample, decreasing composite performance. On the basis of such results microwave curing activation of polyester resin based composites could be proposed as a valid alternative method for faster processing of laminated materials employed for large-scale applications.

Keywords composite materials, flexural tests, microwaves curing, polyester resin

1. Introduction

In the last years, structural design has been gradually orienting towards structures and manufacturing methods that allow realization of more rigid and light structures by using high performance materials. With this aim, polymeric composites have begun to substitute traditional materials.

A fundamental limit of polymeric composite materials is represented by the curing process necessary to obtain the thermosetting matrix solidification. The right choice of the curing system is extremely important in optimizing performance-manufacturing cost relationship. Traditionally composite materials curing is obtained by means of a thermal treatment based on energy transfer by conduction, convection or radiation (Ref 1). Resin and fibers, having very low-thermal conductivity, require long thermal process durations. Exothermic cross-linking reactions generate an additional internal heat that is slowly dissipated through conduction (Ref 2). The result of all these factors is a nonhomogeneous temperature distribution in the material.

Microwaves have been investigated as an efficient alternative energy source for polymers and composites processing, due to their relevant advantages over conventional curing process

(Ref 3). The microwave energy is transferred directly to the materials, through a dielectric interaction with the electromagnetic field. Since microwave radiation easily penetrates polymer materials, heat can be generated throughout the entire material volume. Consequently it is possible to achieve rapid and uniform heating of thick solids (Ref 2). The most common microwave effect consists in the cure kinetics acceleration of thermosetting resins (Ref 4). The “microwave effect” in thermosetting polymers strictly depends on the resin type and on the alignment induced by the electric field on dipolar groups. As the material undergoes cross linking, the dielectric properties change as a consequence of changes in polymer network structure. The initial liquid resin couples well with microwaves; as cross-linking proceeds and viscosity increases, dielectric loss decreases since the ability of dipoles to orient in the electric field is reduced. These changes in dielectric properties require an optimization of the cure cycle in microwave processing (Ref 2).

Several Authors have already studied microwave curing of thermoset resins, especially epoxy resin, and their composites in a variety of applications. About resin characterization, Prasad Yarlagadda and Hsu (Ref 3) studied and compared mechanical properties of both microwave cured and conventionally cured epoxy resins. The study evidenced that the epoxy resins, microwave cured, resulted in higher glass transition temperature than conventional heating. Similarly Tanrattanakul and Tiaw (Ref 4) compared the mechanical properties of epoxy resins cured by thermal heating and microwave heating evidencing equivalent or better mechanical properties for samples cured by microwaves.

Bogdal and Gorczyk (Ref 5) studied a new microwave-assisted method of synthesis of solid epoxy resins (SER) with desired epoxy-groups content, leading to a reaction time reduction in comparison to the conventional heating.

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Table 1 Fabrics and resin codes and composition

Material type	Commercial code	Sample code	Areal density, g/m ²	No. of layers	Resin content, g	Composite thickness, mm
Fabric	MAT 450	MAT-3	450	3	33.5	3.2
	MAT 450	MAT-5	450	5	49.0	4.8
	MAT 450	MAT-7	450	7	66.2	6.3
	COMBI 500/300	COMBI-1	500/300	1	31.4	2.3
	COMBI 500/300	COMBI-2	500/300	2	69.6	4.4
	COMBI 500/300	COMBI-3	500/300	3	99.4	7.0
Resin	Polyester	RESIN	10.0	...

About the composite applications, Shulls et al. (Ref 6) described the microwave processing technique as being able to ensure uniformity of polymer composite material properties. Further development in the polymer composite technology coupled with microwave curing for large-scale applications is reported by Johnson et al. (Ref 7). They developed, with good results, a specialized in-line microwave resin preheating system prior to injection to decrease thermal quench and to reduce the cycle time in the resin transfer moulding. Other Researchers (Ref 8) focused their attention on a novel industrial microwave system that integrates advantageously the basic processing steps of the composite lay-up with the microwave curing process of the composite structure.

Despite the success or the good experimental results obtained by these combined technology, additional work is necessary to increase the knowledge of the relationship between structure-property-process parameters for composite microwave curing. The risks of microwave use in the industrial process for thermoset resin curing must be also evaluated (Ref 9). However not many examples on curing process in large-scale applications, like in the marine field, are available. Polyester resin composites reinforced with fiberglass are commonly used in yachts building with manual room-temperature manufacturing processes, involving high-curing times. In order to increase the production efficiency (with a consequent business growth) manufacturers could be interested in new technologies able to reduce manufacturing times or to increase performances and quality of the final product (Ref 10). From this point of view, the fast curing time induced by microwaves is highly attractive.

In this work, the microwave activated cure reaction of a polyester “marine” resin, pure and mixed with fiberglass, was investigated. Mechanical static three-point flexural tests were performed on pure resin and on laminates as well as on room temperature self-cured samples. Scanning electron microscopy was used to correlate the mechanical performance to the materials morphology induced by the microwave treatment.

2. Experimental

2.1 Sample Preparation

A polyester resin, (Crystic 474PA, Scott Bader), currently employed in marine applications was used; it was added with a 2% catalyst (Butanox 50) under continuously stirring in order to obtain a good dispersion.

Two types of reinforcement were used:

1. the “MAT 450” (a chopped glass fiber fabric with a surface areal density of 450 g/m²);

2. the “COMBI 500/300” (a glass fiber fabric, characterized one side by a 500 g/m² balanced warp/weft continuous woven fibers and the other side with a mat 300).

The composite laminates (10×10 cm²) made by MAT 450 or COMBI 500/300 fabrics and 65 wt.% of polyester resin, have been prepared by manual lay-up.

Different thickness of the composite laminates were obtained by increasing the number of layers; more specifically 3, 5, and 7 layers of MAT 450 and of 1, 2, and 3 layers of COMBI 500/300 were employed. Fabric and resin details are resumed in Table 1. Samples self-cured at room temperature for different times of both the pure resin as the composite laminates were also made. From now, the self-cured samples will be named with the code “SC” followed by the curing days at room temperature.

2.2 Microwave Curing

Microwave curing of pure resin and composite samples was conducted in a microwave oven (Samsung CM 1829) with working frequency of 2.45 GHz and a power output of 1800 W.

Ten grams of the polyester resin samples were cured by microwaves for different times from 2 to 20 s. In order to have comparable results, microwave curing times used for composite samples were calculated to keep the same “exposition time/grams of resin” used for polyester resin alone. Therefore, microwave exposition ranged from 0 to 2.0 s/g for all the composite laminates tested, whatever the layer number and the reinforcement type was.

The microwave cured samples were named with the code “MW” followed by a number indicating the s/g ratio used in the treatment.

2.3 Mechanical Characterization

Mechanical flexural tests were performed both on pure resin and on composites according to ASTM D790, by using a Lloyd Universal Testing Machine model LR10. The test measurements were carried out at room temperature, in three-point bending mode. The quoted results were the average of five test results. Test parameters, relative to the studied samples, are detailed in Table 2. Microwave cured samples were mechanically tested after 24 h from microwave irradiation.

2.4 Scanning Electron Microscope (SEM) Analysis

A JEOL mod. JSM-5600 LV SEM with an acceleration voltage of 15 kV was used to examine the fracture surfaces of the pure polyester resin samples. A thin section of the fracture surface, obtained by three point flexural test, was cut and

mounted on an aluminum stub with a conductive adhesive film. Each sample was sputter coated with gold before the morphological analysis.

3. Results and Discussion

Mechanical characterization of SC and MW cured samples was used to evaluate the reticulated structure of the pure resin and the composite materials formed during the quick cross-linking induced by microwaves. SEM analysis of the fractured surfaces of the self-cured resin samples helped to a better understanding of material features after microwave curing.

For the sake of clarity, the resin and composite characterization results will be separately discussed in the following.

3.1 Resin Characterization

In Fig. 1, flexural modulus (E_f), strength (σ_{\max}) and failure strain (ϵ_f) values of the room temperature self-cured polyester resin vs. curing time (in the range of 0.5-20 days) are plotted. The plots show as the resin stiffness and strength progressively increase with increasing curing time while failure strain

Table 2 Three point flexural test setup

Sample code	Dimension, mm ³	Span, mm	Crosshead, mm/min
RESIN	6×4×50	23.6	1
MAT-3	25×3.2×100	80	4
MAT-5	10×4.8×100	80	2.1
MAT-7	10×6.3×100	80	2.1
COMBI-1	25×2.3×100	80	4
COMBI-2	10×4.4×100	80	2.1
COMBI-3	10×7.0×100	80	2.1

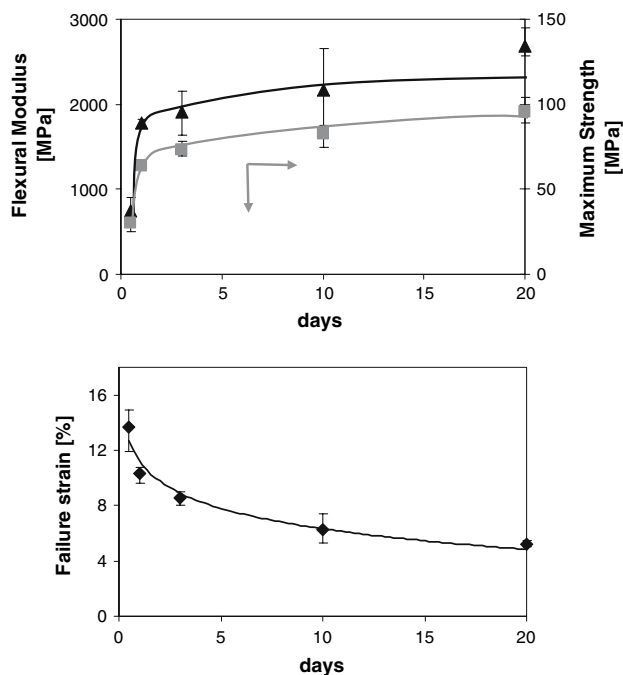


Fig. 1 Flexural modulus, strength, and failure strain vs. polyester resin self-curing time

decreases. After 10 days curing, the E_f , σ_{\max} , and ϵ_f reached almost constant values (Table 3). This time was assumed as the referring curing time for the pure polyester resin samples, indicated in the following as “RESIN-SC10”, and the related mechanical parameters (E_f , σ_{\max} , and ϵ_f) used for comparison with microwave cured samples performances.

Mechanical performances of the microwave cured resin are presented in Fig. 2 in terms of maximum strength and flexural modulus vs. the microwave exposition (s/g).

In both of the graphs three regions can be identified:

- Zone I. Up to a value of 1.0 s/g, the strength and the modulus show low and quite constant values: i.e., the mechanical properties of the resin seem not to be influenced by the microwave treatment.
- Zone II. Between 1.0 and 1.5 s/g, the resin quickly solidifies and the mechanical properties reach the highest values, even if slightly lower than the values obtained for the RESIN-SC 10 (horizontal dotted line in the graphs).

Table 3 Three point flexural test modulus, maximum strength, and failure strain of SC resin and composites

Sample code	Flexural modulus, MPa	Maximum strength, MPa	Failure strain, %
RESIN-SC10	2167±505.5	83±7.3	6.2±1.1
MAT-3-SC10	6959±397.1	187±14.3	4.2±0.4
MAT-5-SC10	5844±359.1	181±12.6	4.2±0.2
MAT-7-SC10	7363±307.5	215±6.3	4.1±0.0
COMBI-1-SC10	3920±61.2	102±4.5	6.7±0.3
COMBI-2-SC10	8060±584.4	236±5.8	4.3±0.6
COMBI-3-SC10	11,130±441	315±17	4.5±0.8

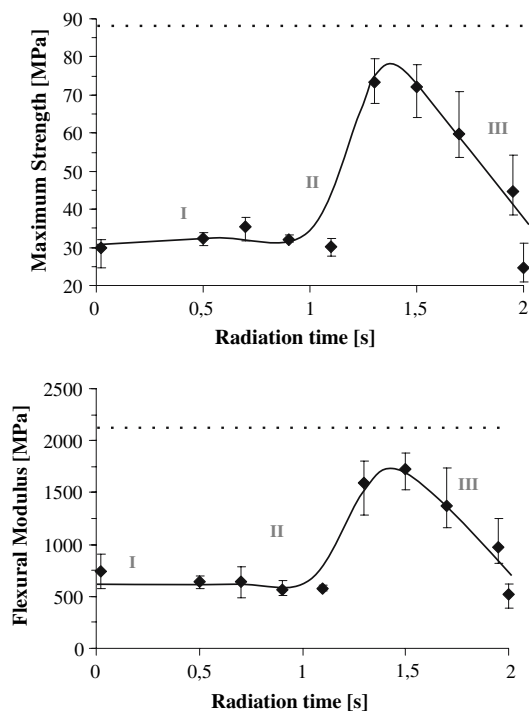


Fig. 2 Strength and flexural modulus vs. polyester resin MW exposure time

- Zone III. After 1.5 s/g, the mechanical properties steeply decrease. Ripples and bubbles appeared on the samples surface.

Fully reticulated thermosetting polymers do not interact with microwaves or they do only weakly, so they are considered “transparent” to microwaves while before curing the monomeric precursor readily absorbs microwave energy and converts it into heat. MW interaction is mainly due to the presence of styrene in the resin formulation that acts as solvent and as reagent in the cross-linking reaction. As the cure reaction proceeds, microwaves interaction with the polymer decreases due to the formation of the polymer network that blocks styrene molecules in the structure and leads to matrix solidification (Ref 9).

Microwaves can drastically reduce curing time due to a more efficient and volumetric polymer heating, but if the radiated energy is too low (Zone I) thermal curing not will be fully activated and the solid resin will not fully cross-link. In the opposite case, as a consequence of an excessive microwave exposition (Zone III), styrene heating will be so rapid that it can start to bulky evaporate causing bubbles formation inside the matrix. The premature styrene evaporation is, again, a cause of poor cross linking between the polymeric chains while internal bubbling generates cracks and voids in the cured polymer.

Microwave exposition at 1.5 s/g has given the better compromise between mechanical properties of the resin and curing time (Zone II) at this value it can be considered that the quantity of thermal energy given was able to accelerate the cure process avoiding an excessive styrene evaporation. The results in terms of flexural modulus, maximum strength and failure strain for the sample RESIN-MW1.5 are reported in Table 4.

SEM images of fracture surfaces of the RESIN-SC10 and the RESIN-MW1.5 are shown in Fig. 3(a) and (b). Fracture morphologies are similar: a mixed brittle-ductile behavior can be observed.

Some details of the microphotographs of Fig. 3 are compared in the Fig. 4(a)-(c) (relative to the RESIN SC10 sample) and in the Fig. 4(d)-(f) (relative to the RESIN MW1.5 sample). In the ductile areas, the surfaces appear rough with a series of “hackle markings” and “scallops” (Fig. 4a, d). The scallops are concave resin areas. The hackle markings are irregular lines that take place in thermosetting resin when shear stresses dominate the fracture process. They are attributed to coalescence of many tension-induced microcracks inclined at an angle to the overall fracture plane (Ref 11). These lines extended approximately along the crack-propagation direction and are related with the arrest point of the crack front (Ref 12, 13). In the brittle areas the morphology changes, the hackle

Table 4 Three point flexural test modulus, maximum strength, and failure strain of resin and composites microwave cured at the optimal exposition time

Sample code	Flexural modulus, MPa	Maximum strength, MPa	Failure strain, %
RESIN MW1.5	1727 ± 183.3	72 ± 7.2	6.8 ± 0.2
MAT-3 MW0.8	6069 ± 449.3	169 ± 12.5	3.9 ± 0.2
MAT-5 MW 0.8	5357 ± 230.7	164 ± 11.1	4.3 ± 0.3
MAT-7MW 0.5	6134 ± 453.2	180 ± 12.6	4.4 ± 0.1
COMBI-1 MW1.0	3261 ± 370.2	78 ± 10.4	4.7 ± 0.3
COMBI-2 MW0.8	6374 ± 670.5	212 ± 9.0	5.3 ± 0.9
COMBI-3 MW0.2	8872 ± 729.9	277 ± 23.4	5.1 ± 0.2

markings and scallops disappears. Near the crack, striations are formed (Fig. 4b, e). In other areas, a progressive transition to a smooth fracture surface occurs, as shown in Fig. 4(c) and (f), typical of a cleavage-like brittle fracture (Ref 14).

An example of fracture surface of an microwave over-exposed sample RESIN-MW2.0 (from Zone III of Fig. 2) is presented in Fig. 5. A smooth fracture surface and a big hole suggests a full brittle behavior of the material which contains defects due to styrene bubbling into the cured matrix. The correlation between the low-mechanical performances and the fracture mechanics is evident in this case.

Even at the “ideal” microwave exposition resin mechanical performance remains nevertheless lower than that of the self-cured sample (RESIN SC10). The fractured surfaces of samples RESIN SC10 and RESIN MW1.5 showed however similar morphology and no further porosity caused by fast styrene evaporation was evident on the MW sample surface (Fig. 3). Moreover, the mechanical performance of RESIN-MW1.5 samples tested after 10 days did not reach the values obtained after ten days of self-curing. One reason of the lower performances of the MW cured samples could be due to the styrene homopolymerization during the microwave curing reaction that could causes insaturations in the polyester resin (Ref 15). Further investigation is needed to evaluate if microwave activation can promote styrene homopolymerization inside the polyester matrix.

3.2 Composite Characterization

Composite laminates prepared with different layers of COMBI and MAT fabrics have been microwave cured for

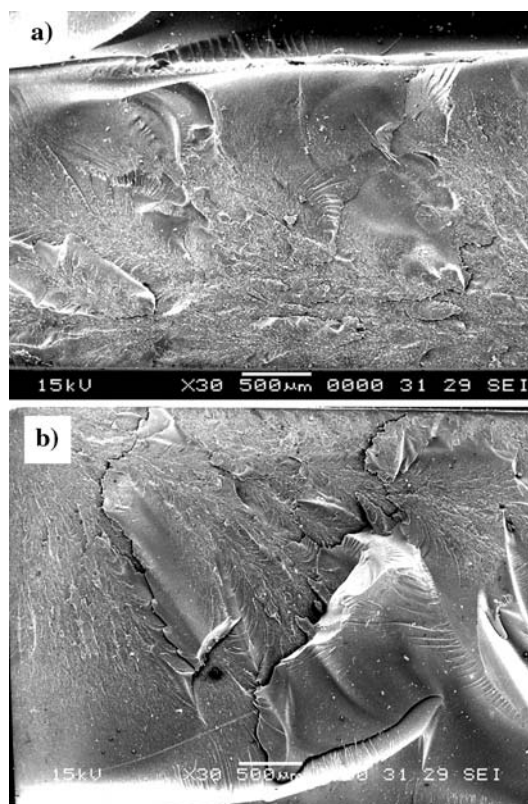


Fig. 3 SEM micrographs of RESIN-SC10 (a) and RESIN-MW1.5 (b)

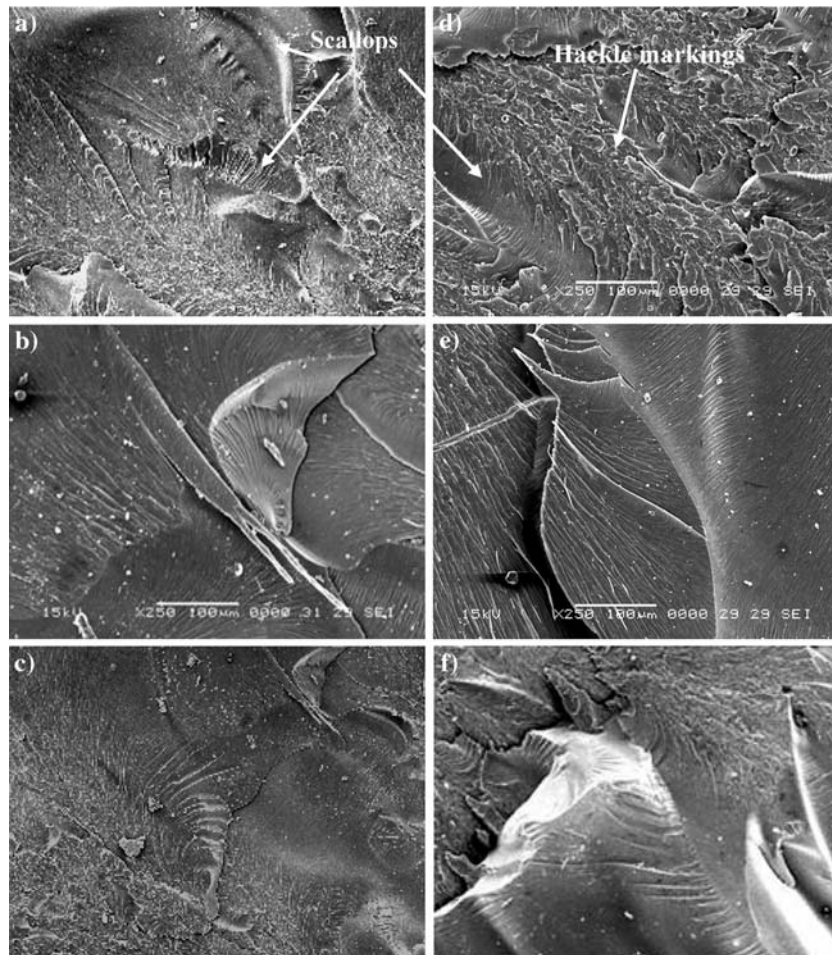


Fig. 4 SEM micrographs of fracture surface after a three point flexural test of RESIN-SC10 (a, b, c) and RESIN-MW1.5 (d, e, f)

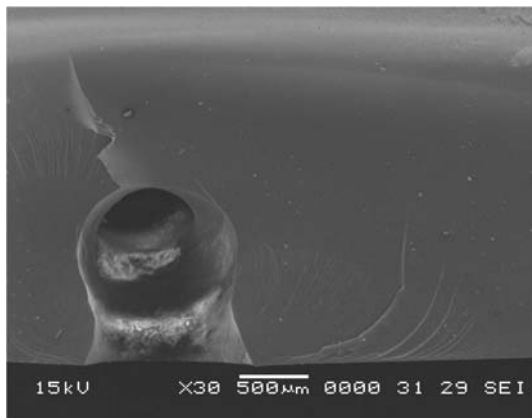


Fig. 5 SEM micrographs of RESIN-MW2.0

different times calculated to keep the same range of 0-2.0 s/g-of-resin (s/g) used for the polymeric matrix treatment. This was done in order to have comparable results because, as shown in Table 1, COMBI and MAT composites required different quantity of resin according to the fiber reinforcement used.

In Fig. 6, the maximum strength and the flexural modulus vs. the s/g ratio for the MAT (Fig. 6a) and the COMBI (Fig. 6b) laminates are depicted. The first point of each curve represents the average value for the sample not exposed to the microwaves

and self-cured for 24 h. From these plots the following points can be highlighted.

- Both the flexural modulus and the maximum strength plots show, independently from the number and typology of layers, a maximum indicating an ideal time (for gram) of microwave exposition.
- In Fig. 6(a), MAT-7 reached the maximum mechanical performances at 0.5 s/g, while MAT-5 and MAT-3 at 0.8 s/g. Similarly in Fig. 6(b), COMBI-3 showed the higher values at 0.2 s/g, COMBI-2 at 0.8 s/g and COMBI-1 at 1.0 s/g. In each laminate typology, MAT and COMBI, the higher the number of layers, the lower the ideal s/g parameter resulted.
- It is worthwhile to notice that in all the six tested laminate typologies, the maximum in the flexural modulus and in the maximum strength were reached for values of s/g lower than the ideal values (1.5 s/g) found for the pure resin.
- Finally, all MAT and COMBI laminates cured by microwaves evidenced a small degradation of the mechanical performances if compared with the self-cured analogues (Table 3), similarly to what observed in Fig. 2 for the pure resin.

The main difference between the pure resin and the composites is the presence of the fiberglass reinforcement immersed in the polymeric matrix. From a microwave point of view, fiberglass

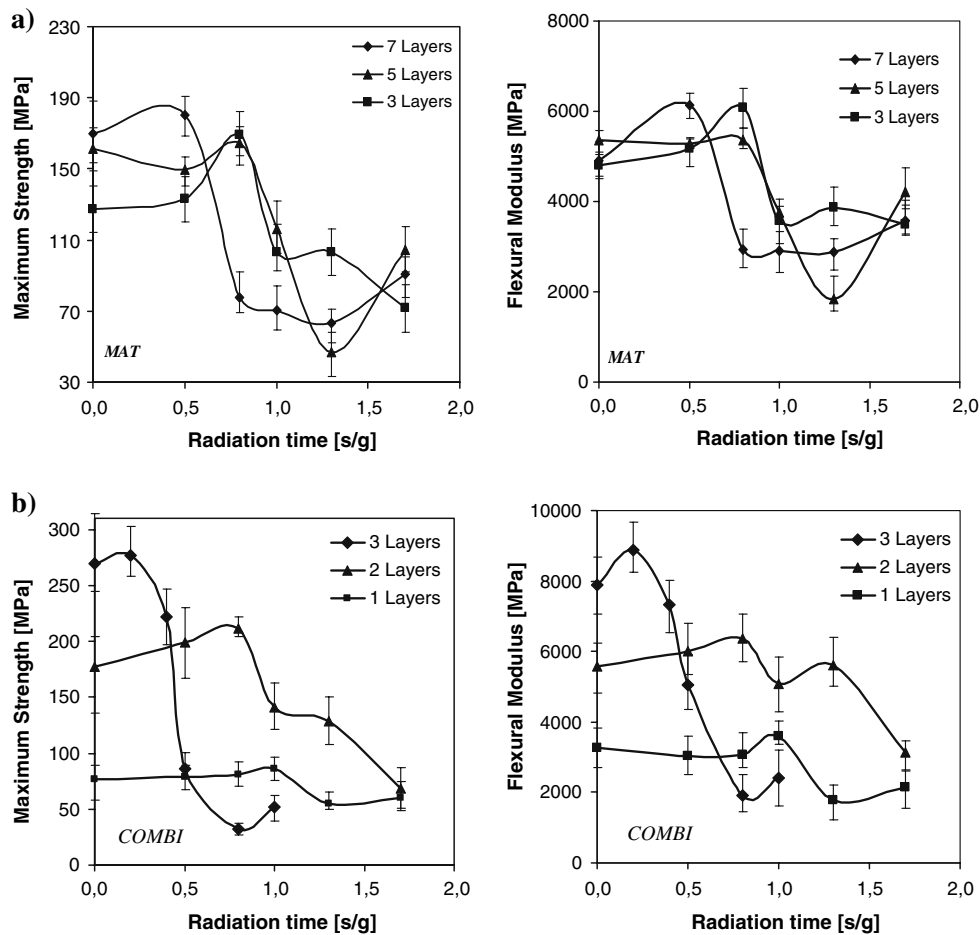


Fig. 6 Flexural modulus and strength of MAT (a) and COMBI (b) laminates

is almost transparent to the oscillating electromagnetic field at 2.45 GHz so his contribute to the whole composite heating is minimum. The composites reticulation and solidification too is mainly determined by the styrene heating and evaporation under microwave irradiation.

In order to better understand about the microwave activation effect on the composite material, an analysis of the fracture mechanism was performed on MAT and COMBI samples after the three point bending tests.

In Fig. 7(a)-(c), pictures of the MAT7 samples (microwave irradiated at the time of 0.0, 0.5, and 0.8 s/g, respectively) are showed. Each picture shows two different views of the same sample, lateral and top. In top view picture it is possible to identify a central zone where the sample/pusher contact occurred. The MAT7 sample without any microwave curing treatment (MW 0.0 s/g) and that one cured at low-microwave exposition times (MW 0.5 s/g) broke with a cohesive mechanism. The fibers did not detach from the matrix suggesting a good adhesion between the layers. The sample cured at high-microwave exposition times (MW 0.8 s/g), showed evident delamination phenomena, indicative of a poor fibers/matrix interaction that brought to a premature laminate mechanical failure at low stresses (around 70 MPa). As it was observed for the pure resin, an evident degradation of mechanical performances of the laminate is correlated to an over-exposition to microwaves. Voids or bubbles formation inside the matrix due to the styrene overheating could be the main cause of the observed delamination.

MAT3 e MAT5 samples cured at their optimal microwave exposition time (0.8 s/g) are shown in Fig. 8. The failure mode of the MAT3 MW0.8 and of the MAT5 MW0.8 samples was similar to that of the MAT7 MW0.5 sample. Indeed, the failure was cohesive and no evidence of delamination phenomena was found in the two samples, confirming the good fiber/matrix interaction reached.

In Fig. 9(a)-(c), pictures of COMBI laminates cured at their optimal microwave exposition time (COMBI1 MW1.0, COMBI2 MW0.8, COMBI3 MW0.2) are shown. As in the previous case, the highest mechanical performances of COMBI samples obtained at the ideal microwave curing times corresponded to a cohesive failure mode without evidence of delamination between layers. Even more, at different microwave exposition times the laminates containing the COMBI reinforcement showed the same failure mode than the MAT samples. Delamination phenomena were always observed at high-exposition times.

Plotting the results in terms of MW exposition time (s) vs. grams-of-resin for MAT and COMBI samples, having the highest mechanical performances, some new features can be highlighted. In Fig. 10 the two curves obtained reprocessing the data are shown: the first value (15 s-10 g) represents the pure resin result, to be considered as a reference point for both of the composites, while in each point of the plots the relative laminate layers are indicated.

The trend of the two plots in Fig. 10 is similar: microwave curing time increases with resin weight and laminate layers almost linearly up to a maximum. Beyond these limit values the

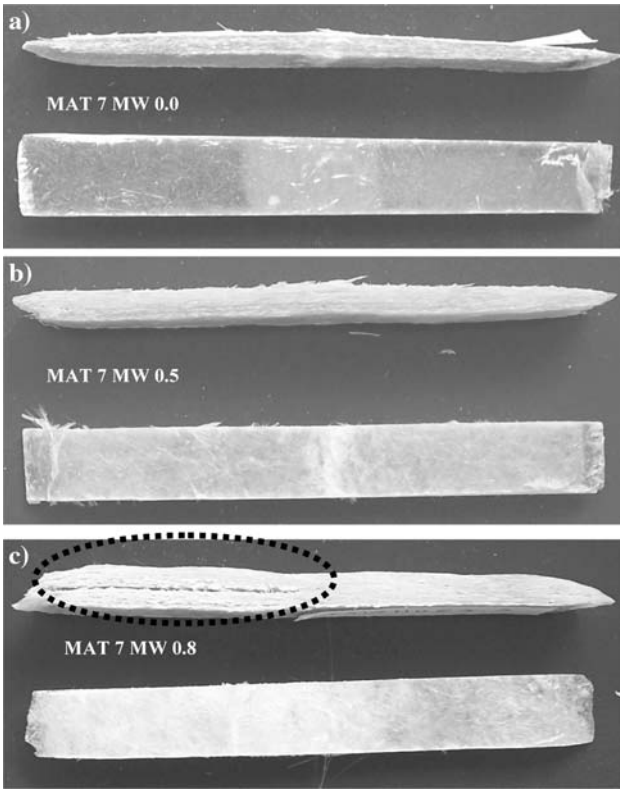


Fig. 7 Lateral and frontal views of MAT7 samples, microwave irradiated at 0.0 s/g (a), at 0.5 s/g (b), at 0.8 s/g (c), after the three point flexural test

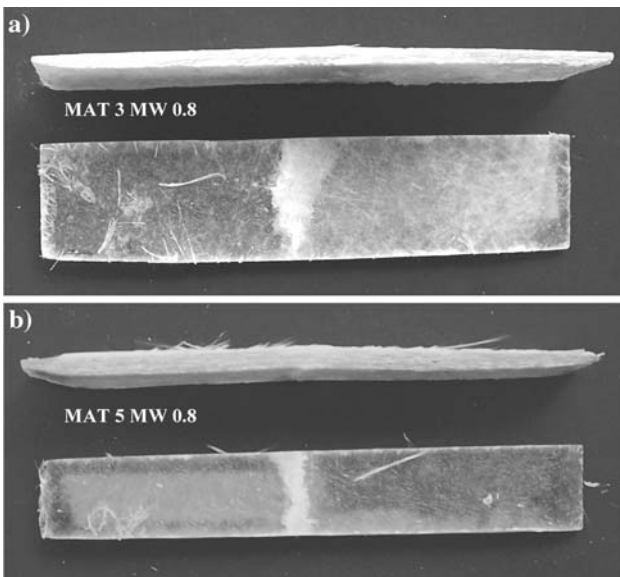


Fig. 8 Lateral and frontal views of MAT3 (a) and MAT5 (b) samples microwave irradiated at 0.8 s/g, after the three point flexural test

curing times steeply decrease. This effect is more evident for COMBI than for MAT.

According to what is observed above, the correlation between the increasing numbers of layers and the earlier obtainment of maximum performances can be explained with

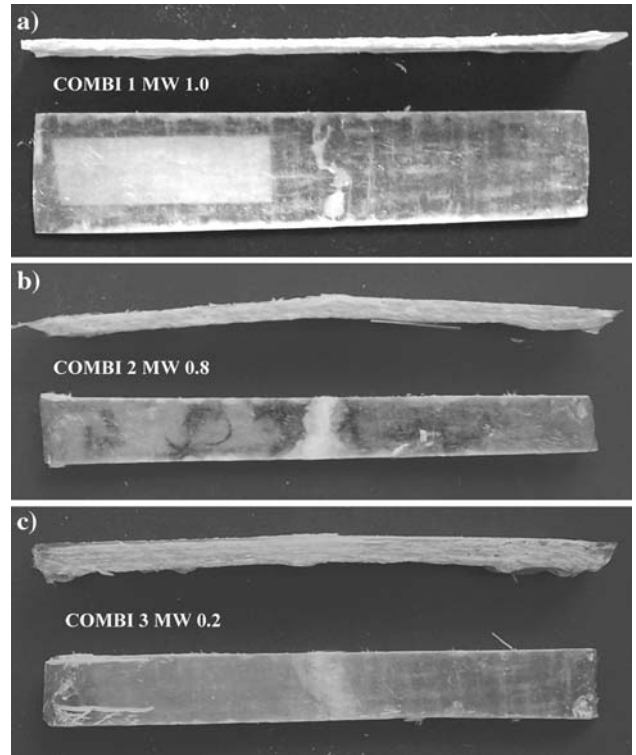


Fig. 9 Lateral and frontal views of COMBI1 (a), COMBI2 (b), and COMBI3 (c) laminate microwave irradiated at optimal exposition time, after three point flexural test

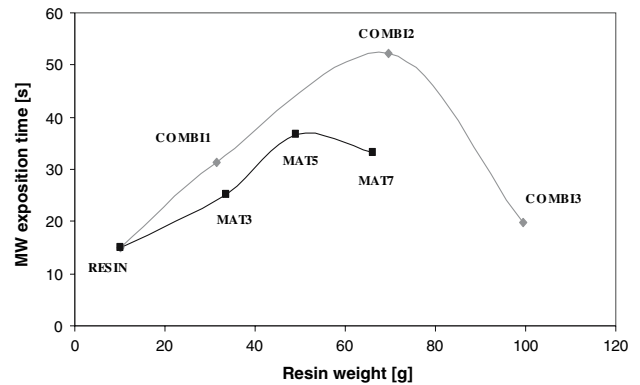


Fig. 10 Resin weight vs. the optimal microwave exposition time for MAT and COMBI laminates

the reaction heat developed during the curing. Curing process is exothermic and, once started, the generated heat accelerates the reaction itself in an autocatalytic progression. This heating “build-up” become more effective by increasing composite thickness, independently from the type of heating source. The microwave irradiation, thanks to the high penetration in the polymeric bulk, induces a fast and massive styrene activation in the matrix, accelerating all the heating mechanisms. This effect of “thermal runaway” makes the microwave treatment more efficient than other heating methods. Increasing the composite thickness, the incident radiation effectively kept by the material increases contributing to a better energy usage. The thickness influence is evident in Fig. 10, where the MAT exposition

values are lower than for the COMBI, with the MAT composites made of more layers than the COMBI: more thickness means more radiation trapped and more thermal effect.

4. Conclusions

The effect of the microwave source on the curing process of a commercial polyester resin used in nautical manufacturing process was investigated. Polyester resin and glass fiber/polyester resin composite were microwave cured and cured without any energetic input (self-cured).

The experimental results suggested that microwave energy strongly decreases the time necessary to obtain a fully cured resin, with respect to a self-cured resin. In fact, an irradiation time of about 1.5 s/g was sufficient, following a stabilization period of 24 h, to obtain a cured resin with mechanical properties comparable with that obtained after 10 days of self-curing. Furthermore, microwave energy strongly decreases the time needed to obtain a cured composite laminate reinforced with MAT and COMBI fabric. In this case the irradiation times were in the range 0.2 s/g-0.8 s/g followed by a period of 24 h for completing laminates hardening. A short curing time has many economical advantages, related to lower production times, as an increase in the productivity and as a reduction in the manufacturing costs.

This study evidenced also the importance in determining an optimal MW exposure time: a short time could be enough to complete resin cross linking but a fast energy input could lead to a remarkable degradation of the laminate. In this last case, delamination at the layers interface and the premature mechanical failure could occur.

Reinforcements with high fiber areal density (COMBI) stiffens and strengths the composite laminate better than a low-fiber areal density (MAT). The COMBI fabric needs an optimal reinforcement layer number, and consequently a high-resin content, in order to give the best reinforcement action. On the contrary, the MAT fabric gives all its best reinforcement action even at low layers number and, consequently, even at low-resin content.

High-laminates thickness, and so high layers numbers and high resin content, are preferable since a phenomenon of “thermal runaway” adds to the activation microwave effect.

Finally, it was observed that the mechanical properties of the materials (resin and laminates) cured with their optimal MW exposure time never reach that of self-cured ones. The styrene

homopolymerization mechanism could be the reason of this discrepancy.

Mechanical performances of microwave-reticulated materials are well acceptable for their use in the marine industry and the lower performances can be compensated by reducing the whole process time.

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