VACCUUM INFUSION PROCESSING OF COMPOSITES WITH INTEGRATED DAMPING ELEMENTS

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ABSTRACT: NiTi alloy wires were embedded during the infusion processing of woven carbon fibre reinforced plastic (CFRP) composite plates with the purpose to passively increase their damping. Two types of NiTi wires, having the same diameter of 203 μm, were considered, one superelastic at room temperature, the other one martensitic. For the first wire type, a martensitic transformation was induced by applying a pre-strain of 2.5% before embedding the wires. A metallic mould with a frame was designed for shape memory alloys (SMA) wire placement and pre-straining during infusion process. The internal stress build-up during cure and post-cure of composite and the impact on the cure progress of the presence of SMA wires was evaluated using optical fibre Bragg gratings (FBG) sensors.

KEYWORDS: Infusion, damping, shape memory alloy, optical fibre sensor.

INTRODUCTION

Adaptive composite with embedded small diameter wires into composite materials are reaching potential applications as actuator for shape control and vibration damping in a variety of industries, including aerospace, civil and automobile [1]. SMAs show a strong damping behaviour in the martensitic phase and a large reversible and hysteretic deformation potential in the austenitic phase (superelastic behaviour). The origin of this high damping capacity is generally linked to the presence and the mobility of twins between different variants of martensite or of austenite-martensite boundaries [2], respectively. Reversible phase transformation can be induced either by changing temperature or stress. The integration of SMA wires should guarantee the most efficient damping effect but must also be compatible with the composite processing route. The choice of processing must, in turn, also facilitate the SMA wires integration. Autoclave process is generally used in high performance applications, such as aerospace [3] Resin infusion based processes such as resin transfer moulding (RTM) or vacuum infusion are however gaining market share compared to autoclave process thanks to their higher versatility permitting the manufacturing of complex and high quality composite parts in fairly short cycle times. This article presents the development of a vacuum infusion process for structural smart composites with embedded SMA wires.

MATERIALS AND EXPERIMENTS

Materials

The matrix was a standard epoxy resin system based on the Dow® D.E.R.332 resin mixed with a suitable hardener. The weight fraction of hardener in the system was fixed at 18 %. Recommended cure schedule was 70°C for two hours, and post-cure at 140°C

for two hours. IM7 carbon fibre 5 HS woven fabrics from Hexcel® Corporation were selected. They permit to obtain quasi-isotropic mechanical properties and to reach high in-plane mechanical properties, when compared to other woven fabrics [4].
Two SMA wire types with different compositions (Table 1) were used. They were chosen for their properties at room temperature, i.e. superelastic and martensitic, respectively. Martensitic wires were provided by SAES Smart Material Getters Group and superelastic ones by Memry Corporation GmbH. Their surface was chosen oxidized for a better adhesion, and the SMA/host interface quality was verified through pull-out tests in a previous study [5]. In the same study, differential scanning calorimetry (DSC) analyses were performed to evaluate the wire transition temperatures [5]. Martensitic transformation temperatures are summarized in Table 1 for both wire types. For martensitic wires, austenite will form in the material at the curing and post-curing temperatures. As austenite lattice parameters are smaller than those of martensite, the material will tend to shrink as soon as the A_s temperature is exceeded. Consequently, both martensitic and austenitic wires should be maintained during all curing and post-curing, to prevent them from shrinking before complete reticulation of the epoxy matrix.

Table 1 SMA austenite-martensitic transformation temperatures

Type of wire (composition)	M_s (°C)	M_f (°C)	A_s (°C)	A_f (°C)
Austenite (Ni: 50.8%; Ti: 49.2 %)	8.5	-19.6	-8.2	17.4
Martensite (Ni: 50.4%; Ti: 49.6 %)	29.3	16	21.5	34.3

Experimental Procedure

Resin Chemorheology

The resin curing kinetics were measured using an ARES dynamic torsional rheometer. Elastic shear modulus G', viscous shear modulus G" and complex shear viscosity η* were recorded during the testing of the originally uncured epoxy system. Tests were performed using 25 mm diameter plates, at 6.28 rad/s, with adjustable strain amplitude. The study was performed for two different temperatures: 45°C and 70°C, corresponding to the mixing/injection and cure temperature, respectively. The heating rate was set to 20°C/min.

Compaction Test

The maximum achievable fibre volume fraction V_f was measured through the compression of dry fabrics. Tests were carried out on a universal UTS testing machine at constant compression rate of 0.1 mm/min up to a load of 12 kN. Two different lays-up (5 and 7 plies) of squared 5 HS fabrics were tested. To respect the final composite lay-up a layer was oriented at ± 45°. V_f was calculated from the relation $V_f = \rho_a n / \rho_f H$ where m_{of} is the fabric areal weight, n the number of plies, ρ_f the density of the fibres and H is the corresponding thickness of the compressed assembly.

Composite processing

A metallic frame allowing control of wire position was designed (Fig. 1), so that two layers of aligned wires could be placed on each side of a stack of carbon fabric. Additional layers of carbon (at least one) are then added on each external side, to reach a fully symmetric assembly and protect the SMA wires. Impregnation was performed at 45°C while curing was at 70°C for 2 hours before a post-cure of 2 hours at 140°C. The

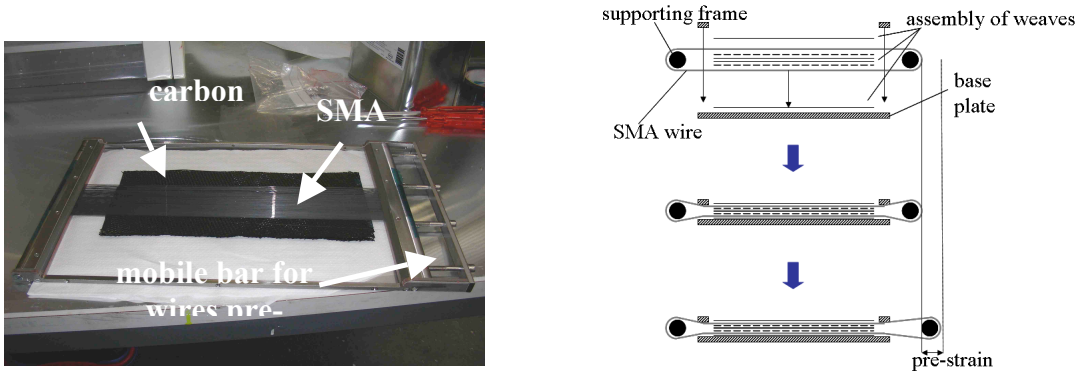


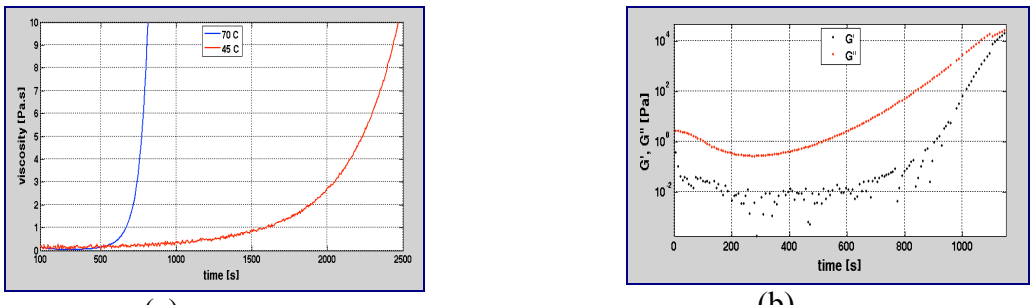
Fig. 1 Metallic frame for SMA wires positioning and procedure for pre-strain

symmetrical composite lay-up was: $[(0/90)/\{SMA\}/(+45/-45)/(0/90)]_s$ [1]. Prior to injection, resin was degassed, at 45ºC, before and after mixing with hardener. A resin flow mesh was applied at the surface of the peel-ply over the carbon fabrics lay-up. Freekote® 770 demoulding agent was used on the mould surface. FBGs with a gauge length of 4 mm written in silica fibres SMF-28e® from Corning were used to follow the build-up of internal stresses from strain measurements along the SMA wires direction. Before embedment, FBG sensors were annealed at 160 °C during 24 hours [6]. FBG were positioned in-between SMA wires. A sm125-500 FBG interrogator from Micron Optics provided the FBG response at 1 Hz. Three types of CFRP plates were made. One without any wire and the others with SMA wires in martensite (SMA-M) and austenite (SMA-S) state, at room temperature, respectively. The FBG peak shift was converted into strain, subtracting the effect of temperature as indicated in ref. [6].

EXPERIMENTAL RESULTS

Resin Chemorheology

Fig.2 (a) shows the evolution of η∗ with time at 45 and 70°C. Viscosity should be typically below 1.0 Pa.s for manufacturing. The available time is thus about 10 minutes at 70°C, and 27 minutes at 45°C. Gel time (t_{gel}) at 70°C was determined from G' and G'' intersection (Fig. 2 (b)) to be about 20 minutes. The pressure differential that is applied for compaction of the resin-fibre system should be kept until vitrification (~3*t_{gel}), when the resin effectively solidifies and transforms into a glassy material. For precaution vacuum was applied along the entire process.



(a) (b)

Fig. 2 (a) Effect of temperature on the viscosity evolution of the resin, (b) Evolution of elastic and viscous shear moduli of the resin at 70°C.

[1] As the HS fabric is non-symmetric (acting almost as two half plies stacked together with stitches, one with 80% fibres at 0° and 20% fibres at 90°, and another one with 20% fibres at 0° and 80% fibres), the orientation of the plies are given with the orientation of the half plies. So, the "(x/y)" and "/" give the half ply and the ply separation respectively.

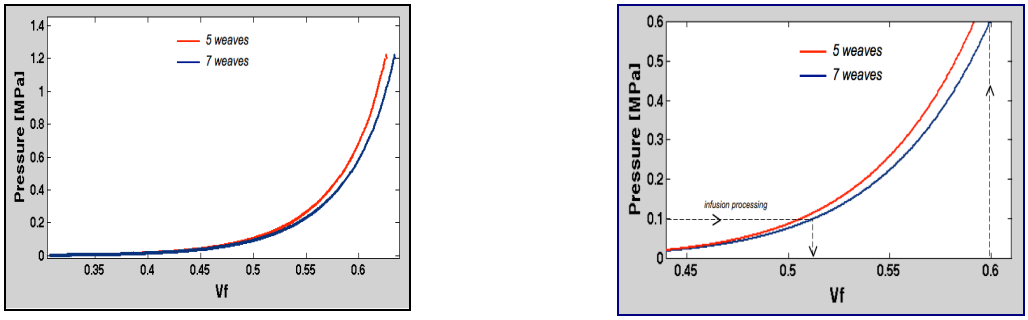


Fig.3 Results of dry compression on 5 HS weaves, with a stack of 5 or 7 weaves (0/90 and +45/-45 plies alternated)

Compaction Test

The influence of the number of weaves is observed from Fig. 3; increased fabric nesting is found with 7 weaves. With vacuum processing, the maximum achievable V_f is ~ 51%, as the maximum applied pressure with a vacuum bag is 0.1MPa. For high performance industries such as aerospace this value is too low. For such application RTM in a closed mould should be considered.

Composite manufacturing

Fig. 4 (a) and Fig. 4 (b) show the evolution of the total strain, $\Delta\varepsilon_{tot}$, for the three plates during cure and post-cure, respectively. The temperature evolution during the manufacture of the plate without SMA is given for indication. The strains were set zero at the beginning of the injection. Some strain was noticed after the application of vacuum that could be related to the preform compaction. It is also worthy to underline that the viscous nature of the resin in the initial phase of the curing process may inhibit complete strain transfer to the FBG sensor. Therefore, the information provided by the sensor before gel point must be interpreted with caution. FBG strain responses after gel time follow similar trends for the three plates during cure. The strain measurement also captured the separation of the laminates from the mould during the cool down at around 2.5 hours, due to the difference in coefficients of thermal expansion between the composite and the metallic tool [7].

The FBG interrogation was unfortunately interrupted too soon during the cool-down phase of the SMA-M and SMA-S plates post-cure, at 40 ºC (> M_s) and 100 ºC, respectively. FBG responses during post-cure of CFRP and SMA-M plates permit however to underline the mould-composite interaction that has significant impact on the internal stresses build-up during the cool-down [6,7]. The strain measurement during post-cure reveals a mix between the constraining of the CFRP, the constraining of the SMA wire and the expansion due to the frame dilatation.

Void content was measured from micrographic observations of the cross section of the plates. Mean fibre and SMA-wire volume fraction were measured using mass and thickness of the final plate, knowing the initial mass and density of fibres, wires and resin. They are reported in Table 2.

Table 2 Fibre and wire volume fraction, void content

Sample type	Vf	Vw	Void content
No-SMA	53.3	0	7.1±1.9
SMA-S	42.9	5	6.2±0.3
SMA-M	44.9	5.2	6.5±0.3

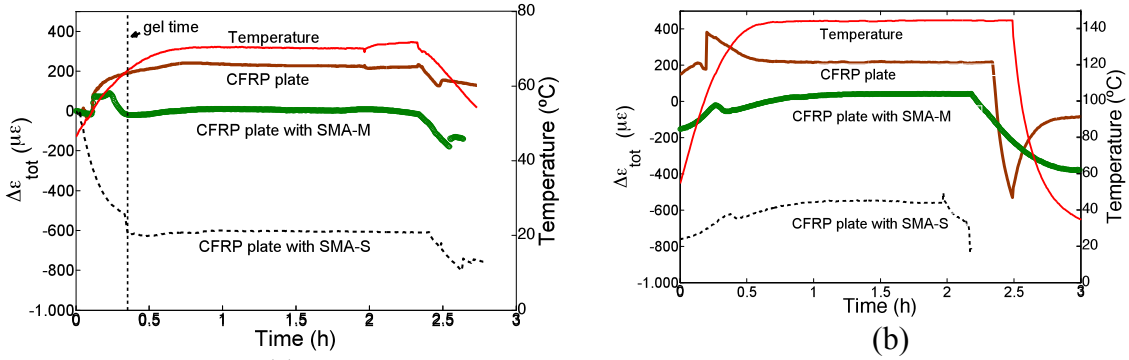


Fig. 4 Strain evolution along (a) cure and (b) post-cure

CONCLUSIONS

A procedure was developed for efficient SMA wire embedment in CFRP woven plate manufactured by infusion. A metallic frame was designed that guaranteed the wire positioning and superelastic wires pre-straining. FBG sensor measurement showed that the strain in the composite during cure is not much affected by the presence of the wires. The effectiveness of the embedment of the SMA wires by vacuum infusion process was verified from the experimental evaluation of passive damping enhancement from free vibration tests [5].

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REFERENCES

1. V. Michaud, “Can Shape Memory Alloys and their Composites be smart?”, *Scripta Materialia*, Vol. 50, pp. 249-253 (2004).
2. J.V. Van Humbeeck and S. Kustov, “Active and passive damping of noise and vibrations through shape memory alloys: applications and mechanisms”, *Smart Materials and Structures*, Vol. 14, pp. S171-S185 (2005).
3. J.A. Balta, et al., “Smart composites with embedded SMA actuators and FBG sensors: activation and control”, *Smart Mat. and Struct.*, Vol. 14, pp. 457-465 (2005).
4. R.A. Saunders, C. Lekakou and M.G. Bader, “Compression in the processing of polymer composites 1. A mechanical and microstructural study for different glass fabrics and resins”, *Composites Science and Technology*, Vol. 59, pp. 983-993 (1999)
5. R. de Oliveira, et al., “Passive damping of composites with embedded shape memory alloy wires”, *Proc. of the Smart Structures and NDE,* San Diego, USA, (2010).
6. R. de Oliveira, et al. “Experimental investigation of the effect of the mould thermal expansion on the development of internal stress during composite processing”, *Composite Part A*, Vol. 39, pp. 1083–1090 (2008).
7. L. Khoun, R. de Oliveira, V. Michaud and P. Hubert, “Measurement of process-induced strains by fibre Bragg grating optical sensor in resin transfer moulding”, *Proc. of the 17th International Conference on Composite Materials (ICCM17)*, (2009).