

## Progress on Processing and Design of Composites with Embedded Shape Memory Alloy Wires.

V. Michaud and J.-A. E. Månson

Laboratoire de Technologie des Composites et Polymères (LTC), Ecole Polytechnique Fédérale de Lausanne (EPFL),  
CH-1015, Lausanne, Switzerland,

Fax: 00 41 21 693 58 80, e-mail: veronique.michaud@epfl.ch

Shape memory alloy wires are nowadays available in small diameters, allowing their direct integration into polymer composite materials without altering the integrity of the structure. These hybrid materials offer the potential to adapt their shape, thermal or vibration response, if one can master both their design and processing to reliably produce the desired level of activation. Recent progress obtained in the framework of a concerted European effort towards a fundamental understanding of the manufacturing and design of SMA composites is hereby presented. The model material is a Kevlar-epoxy host containing 150 $\mu$ m diameter pre-strained NiTiCu wires. Additionally, embedded Fiber Bragg Grating optical fibers are used for in-situ strain measurement. Particular emphasis is placed on the processing issues including the choice of materials and of a manufacturing route for the embedment of pre-strained wires, the development of internal stresses in the host material during post-cure and activation, and the evaluation of the interfacial strength required to sustain the activation induced stresses. Evolution of the composite strain, recovery stresses, and resonance vibration shift as a function of temperature and wire volume fraction is compared with the results of design tools developed during the project, demonstrating the potential and limitations of the chosen approach.

Key words: Shape Memory Alloy, adaptive composites, Fiber Bragg gratings, composite processing

### 1. INTRODUCTION

The development of composite materials has evolved in the past 40 years from the initial search for very high specific properties alone, to the need to maintain high properties while reducing manufacturing time and production costs, to recently include the need to integrate added functionality in the composite part. Since conventional structural composite materials cannot fulfill this last requirement alone, adaptive or smart composite materials, which integrate actuators and sensors, may well represent a next step in composite materials development. A potential class of such materials is fiber reinforced polymer composites containing thin Shape Memory Alloy (SMA) wires as actuating elements [1-3]. Shape Memory Alloys have been available for 40 years and have found applications as actuators [4,5], but have been only recently manufactured as high quality wires with diameters below 0.2 mm. Thin SMA wires may thus be integrated into the host composite material, without altering its structural integrity. In comparison to other actuating technologies, SMAs provide the following advantages: high reversible strains (up to 6%), high damping capacity, large reversible change of mechanical and physical characteristics, and most importantly, the ability to generate high recovery stresses [6]. Their main drawback is that their transformation is induced by

temperature or stress, greatly limiting their response time, controlled by heat transfer kinetics.

As a result, composite materials with SMA wires demonstrate added functional effects such as a shape change, a controlled overall thermal expansion or a shift in the natural vibration frequency upon activation. The working principle is that pre-strained martensitic shape memory alloys tend to recover their initial shape when heated above their transformation temperature. If the wires are clamped externally or they are embedded into a host material, the wire strain recovery is limited, and large recovery stresses are instead generated, which may lead to the effects cited above.

Pioneering work in this area was performed in the 90's [5,7] but these materials have still found very little industrial applications. One reason is of course the fact that the price of the SMA wires was and still is rather high (about 0.3 €/m for a thin wire). Another cause is that practical tools for the material design and reliable processing of these materials are not yet available [8]. Research is thus still needed and ongoing in the fields of SMA wire optimization, material design tools and processing science. In particular, a concerted European effort was launched several years ago towards a fundamental understanding for manufacturing and design of SMA composites, entitled "Adaptive composites with embedded shape memory alloy wires (ADAPT)" [9-11]. This article illustrates some of the critical issues on the

manufacturing and design of composite containing SMA wires, mostly based on recent results obtained in the above-mentioned project. The case study chosen here is to design a material, which adapts its resonance vibration frequency in clamped configuration as needed. The wires are hence always positioned in the neutral axis of the beams to prevent bending. The model material is a unidirectional composite host containing 150 $\mu\text{m}$  diameter pre-strained SMA wires. Additionally, embedded Fiber Bragg Grating optical fibers are used for in-situ strain measurement. Particular emphasis is placed on the processing issues including the choice of materials and of a manufacturing route for the embedment of pre-strained wires, and on the design issues, involving the development of material models. Evolution of the composite strain, recovery stresses, and resonance vibration shift as a function of temperature and wire volume fraction is finally compared with the results of design tools developed during the project, demonstrating the potential and limitations of the chosen approach.

## 2. MANUFACTURING

### 2.1 Host material requirements

The host material should be a structural composite material, which remains elastic until well above the activation temperature, hence with a high  $T_g$  if a thermoset matrix is chosen. The processing temperature, however, should remain below the activation temperature of the wire, if possible, to prevent any wire contraction, or the need to clamp the wires, during cure. The material should be insulating to prevent short circuits during Joule activation of the wires. Since a vibration shift effect is sought, it is preferred to use a composite material with a negative CTE, to make sure that the contracting effect of the wires will not be offset, at the beginning at least, by a thermal dilatation of the host. Finally, the host composite modulus is chosen rather low in this case study, to magnify the effects of the wire activation. Based on these requirements, the chosen host material is a Kevlar29-epoxy LTM217 unidirectional composite (ACG, UK). The material is cured at 70 °C for 12 hours, and post-cured at 140°C for 4 hours, to yield a final  $T_g$  of about 180°C as measured by Dynamic Mechanical Analysis. The modulus of the final composite is about 40GPa, and the CTE = $-3 \cdot 10^{-6} / ^\circ\text{K}$ . It was unfortunately not possible at that time to find a commercial epoxy system, which could cure at a lower temperature for a reasonable amount of time, and yield a very high final  $T_g$ .

### 2.2 Wire requirements

The requirements placed on the SMA wire are that they would be martensitic at room temperature, to allow a shape memory effect upon heating, with a relatively low transformation temperature (between 30 and 50 °C). The activation efficiency should be high ( $d\sigma/dT$  should be large), the hysteresis low, for better control in the heating and cooling mode and the wires should exhibit a stable behavior. Finally, the wires should be thin enough to be

embedded into a composite material, and provide a surface that is suitable for adhesion with the matrix.

Based on these requirements, a large number of SMA wires with a diameter of 150 $\mu\text{m}$  were investigated [11] in terms of thermo-mechanical behaviour, transformation range, and adhesion characteristics with the epoxy material. The best materials with low hysteresis for this application are R-phase NiTi wires (ths: Furukawa) if one seeks a high activation efficiency over a narrow temperature range and NiTiCu wires with about 12 wt%Cu (ths: Furukawa and mes: Memry) if one seeks a more progressive effect together with a very good stability [11,12]. An example of the recovery stress-temperature behavior of the ths wire is given in Figure 1. Note that for almost all wires, an initial pre-strain of about 2% was already present from the cold drawing process, so the wires exhibited recovery stresses, even at no additional pre-strain. Also, an optimal value of about 3 to 5 % was found for the NiTiCu wires, above which plastic deformation occurs. Regarding the wire-matrix adhesion, it is found that the oxide layer present on the surface provides a very good adhesion, leading even to a cohesive failure at the resin-wire interface for the ths wire [13].

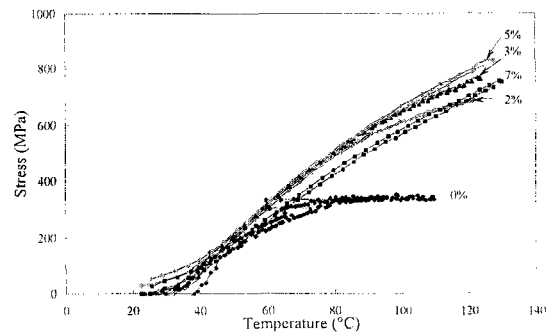


Figure 1: Recovery stress versus temperature for the single wire ths, for various levels of pre-strain.

### 2.3 Process requirements

Since the cure temperature of the epoxy resin is above the transformation temperature of the wires, it is necessary to maintain the wires in place during cure. This is performed with help of a specially designed frame, in which the wires are aligned, pre-strained and maintained [10,13]. Note that some recent developments by Otsuka et al. proposing to use heavily cold-drawn wires to shift their transformation temperature during processing, and shift it back with flash heating may provide an elegant technique to alleviate the need for a maintaining frame [14]. The maximum number of wires possible is 1 wire every 500 $\mu\text{m}$ . In a typical experiment, the wires are placed in one or two frames, depending on the number of wire layers desired, cleaned and heated to 100°C to release potential deformation of the wire during handling, and pre-strained by the desired amount. Plies of prepregs are placed on each side of the wires. The Kevlar fiber orientation is always kept the same as the SMA wire orientation, at least in the layers directly

next to the wires.

The final assembly is placed in a vacuum bag, and cured in an autoclave under vacuum. The resin is cured for 12 hours at 70°C with a heat up rate of 4°C/min. The post-curing is 1 or 4 hours at 140°C with a heating ramp of 20°C/hour. The resulting material is generally well consolidated. In some additional experiments, a fiber optic with a Bragg grating is also introduced in place of one of the wires, as the fiber optic also has a diameter of 150 μm. This allows an in-situ measurement of the stress evolution during post-cure of the material in the frame [15]. It is found that a large compressive strain of about -40MPa developed in the host composite during the heating ramp up to 140°C, probably due to the its negative CTE. After the end of the dwell at 140°C, during cool-down, some of these stresses relax, and the final stress state is about -30 MPa. Similar results were found by partners of the project using Raman Spectroscopy techniques [16,17]. As a result, it is important to notice that the usual modeling assumption that the stress build-up occurs only during cool-down of the composite is wrong, as a significant level of stress is already present after the cure, and the heat-up ramp of the post-cure.

### 3. MATERIAL DESIGN

The next requirement to produce an adaptive composite with the desired properties is to establish the tools, which enable the material design. In our case, with the choice of host material and wire, the issue is to choose the best material combination in terms of wire volume fraction, wire pre-strain, wire arrangement, etc.. to provide the desired activation level in terms of resonance frequency vibration shift. This, in turn, requires the use of adequate models.

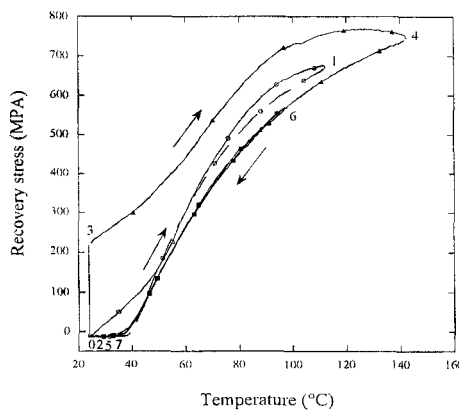


Figure 2: Recovery stress of a single mes wire as a function of temperature for a simulated processing cycle: 0-1-2 preheat the wire in the frame prior to embedding, 3: pre-strain 3 %, 4-5: cure/post-cure up to 140°C, 6-7: activation up to 100°C.

#### 3.1 SMA wire modeling

The most critical issue is of course to find a suitable model for the wire thermomechanical behavior. A number of models are available in the literature, which are reviewed in [6,18]. In the present work, the model RC-Loop developed by Sittner [19,20] is used as a starting point. It is a phenomenological model, easy to implement, which requires a set of preliminary tests on the single wire to fit its behavior in an appropriate manner. Its advantage for the use with composites resides in the ability to model hysteresis loops between two temperatures. Examples of the model are given in refs [20,21]. In addition, wire modeling was done for some experiments by directly fitting the behavior of the single wire during a simulated processing cycle, as shown in Figure 2 for the mes wire.

#### 3.2 Matrix modeling

Under the assumption that the host material remains elastic, and that its properties do not change in the activation temperature range, the material data needed for composite behavior modeling are the matrix elastic properties ( $E$  modulus if the problem is in pure tension), and the thermal properties, CTE.

#### 3.3 Composite modeling

The composite strain is obtained by a simple force balance, such that

$$\epsilon_{comp}(\sigma, T) = \frac{\sigma_m}{E^c} - \alpha^c (T - T_{rs}) \quad (1)$$

where  $T_{rs}$  is the temperature at which the constrained thermal load starts,  $\alpha^c$  the CTE of the host composite,  $E^c$  the Young's modulus of the host composite, and  $\sigma_m$  the stress in the host composite. The latter is related to the stress exerted by a wire  $\sigma_w$ , which is calculated at each step with RC Loop or using a direct fit to Fig.2, as follows:

$$\sigma^m = \frac{V_f}{1 - V_f} \sigma_w \quad (2)$$

where  $V_f$  is the wire volume fraction. Finally, the modulus of the entire composite is written with a rule of mixtures from the moduli of the host composite and wire:

$$E^{comp} = V_f E^w + (1 - V_f) E^c \quad (3)$$

The matrix modulus,  $E^c$ , is considered to be temperature independent in a first approximation, while the Young's modulus of the wire,  $E^w$ , is temperature dependent and hysteretic. The thermal dependence of the SMA composite beam Young's modulus  $E^{comp} = E^{comp}(T)$  hence inherits the non-linearity and hysteresis from the Martensitic Transformation of the embedded SMA wire.

3.3 Vibration model

The resonance frequency of a clamped beam is known to depend on the axial force in the beam. The modeling of the adaptive composite is thus carried out in a first approach by neglecting any additional damping effect which may be due to the temperature change and the interface friction. Neglecting the rotational inertia and the effect of shear deformation of the beam, and assuming that the beam is elastic, with constant properties in the temperature range, and that the interface is perfect, the vibration modes of a clamped composite beam verify the following equation [13,22]:

$$-2\sqrt{\rho A \lambda} \frac{F_{tot}}{\sqrt{EI}} \sin(\alpha l) \sinh(\gamma l) + 2\sqrt{\rho A \lambda} \cos(\alpha l) \cosh(\gamma l) = 0 \quad (4)$$

$$\alpha = \sqrt{\frac{(-F_{tot} + \sqrt{F_{tot}^2 + 4EI\rho A \lambda})}{2EI}}$$

$$\gamma = \sqrt{\frac{(F_{tot} + \sqrt{F_{tot}^2 + 4EI\rho A \lambda})}{2EI}}$$

where  $F_{tot}$  is the axial force in the beam,  $\rho A$  is the mass per unit length,  $l$  the distance between the clamps,  $\lambda = (2\pi f)^2$ ,  $f$  is the vibration frequency,  $E$  is the composite modulus, and  $I$  is the moment of inertia of the composite beam. The relationship between the vibration frequency  $f$  and the total axial force is obtained by the solution of this implicit equation, using Mathematica™.

4. RESULTS

4.1 Activation Strain

Many results were obtained for the activation strain of the adaptive composite, as a function of wire volume fraction, pre-strain and type [11]. An example is given in Figure 3, for a sample with 5.5% of this wire pre-strained 3%, where the strain is measured with an optical fiber. The experimental strain is compared with the theoretical strain calculated from Eq. (1-3), and with the contribution of the thermal expansion only. It is shown that with 5.5% wires, an already significant deviation from the thermal contribution is observed, and the strain is as expected continuously decreasing almost linearly. Agreement between the calculated strain and the experimental value is very good, confirming that the wire behavior fully dictates the composite behavior, and that simple models apply in this case. Comparisons with RC-loop model have also been performed, to quantify the influence of the wire volume fraction [15,20]. The agreement is not so good in terms of absolute values, as it is often difficult to fully capture the wire behavior with RC-Loop, however, the opening of the hysteresis loop is clearly demonstrated when using the RC-Loop simulation.

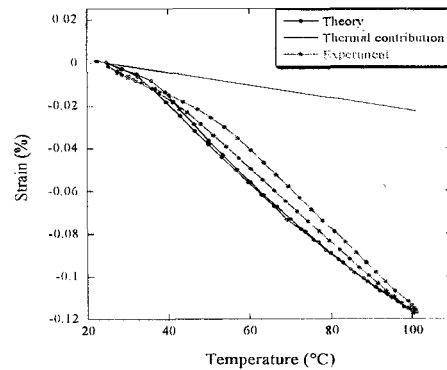


Figure 3: Strain versus temperature for a composite containing 5.5% of this wire, pre-strained 3%.

4.2 Recovery forces

The response of the composite samples activated by electrical current heating was measured using a set-up previously developed by Bidaux et al. [23]. A composite sample, 15 cm long and 1 cm wide is typically mounted on a U-shaped sample holder, equipped with strain gauges to measure the deflection of the holder, hence the force exerted by the composite on the grips. Direct electrical current passes through the wires connected in series, and the temperature is recorded with a thermocouple pasted onto the sample surface. The U-shape holder used to measure the recovery force is not infinitely rigid, as it is thinner on the arm which is equipped with the strain gauges, in order to gain enough precision on the measured deflection. The compliance of the set-up thus needs to be taken into account when comparing the results.

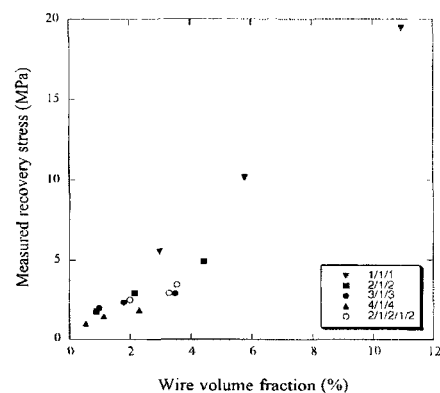


Figure 4 : Measured recovery stress at 90°C as a function of wire volume fraction for composites with mes wire, prestrained 3%, and various lay-up configurations : 1, 2, 3, or 4 layers of pre-preg on each side of the wire layers, or with 2 layers of wires (2/1/2/1/2)

Experimental measurements, coupled with finite element calculations of the holder have shown that the compliance of the holder is  $k=540$  N/mm. Taking this into account, the stress of the beam in a fully clamped configuration with no strain allowed, is related to the stress measured in the U-shape holder by a simple force balance:

$$\sigma_{total} = \sigma_{meas} \left( 1 + \frac{E_r S_c}{kl} \right) \quad (5)$$

where  $\sigma_{meas}$  is the stress measured in the experimental set-up,  $S_c$  the area of the beam cross-section,  $k$  the compliance of the set-up and  $l$  the clamped length of the beam. The recovery force of various composites was measured as a function of temperature, in clamped-clamped configuration. Figure 4 shows an example of raw results of the measured recovery stress at 90°C as a function of the wire volume fraction, for various samples with the mes wire, pre-strained 3%. The arrangement of the wires does not influence the magnitude of the activation, and the volume fraction certainly is the main parameter, as expected from the force balance equations. A linear relationship is found when taking into account the compliance of the frame [11,13]. As a result, it is possible to model the composite behavior, for this simple configuration by using Eq.(4) together with Hook's law.

4.3 Resonance vibration frequency shift

To measure the resonance vibration of the composite samples, the U-shape sample holder is fixed on a shaker, which produces vibrations perpendicular to the plane of the composite with a variable frequency. The vibration amplitude of the composite is recorded with a laser using the Doppler effect, and the resonance vibration frequency is recorded for a given temperature. Figure 5 presents an example of frequency response as a function of

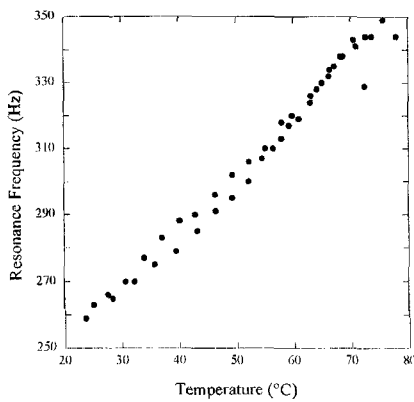


Figure 5: Resonance frequency versus temperature for a composite with 3% wires, pre-strained 3%.

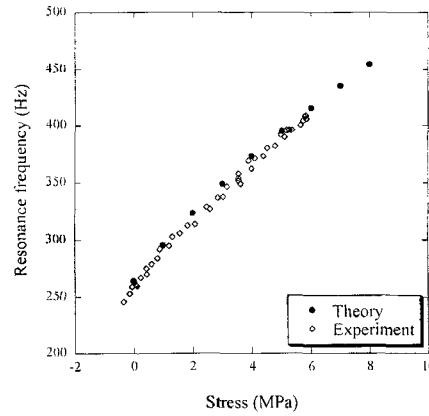


Figure 6: Resonance frequency as a function of measured activation stress for a composite with 5.5% this wire, prestrained 3%.

temperature, for a sample with 3% of this wire, pre-strained 3%. A behavior similar to that of the recovery stress response is found. This is confirmed by another example given in Figure 6, for a sample with 5.5% of this wires pre-strained 3%, presenting the resonance vibration frequency as a function of measured recovery stress. The theoretical curve presented alongside is obtained from Eq. (4) and shows very good agreement. We noticed however that the theoretical curve is always slightly above the experimental curve, in particular for the high temperature range hence high stress range. This indicates that there is a slight effect of modulus variation or damping induced by the temperature [10].

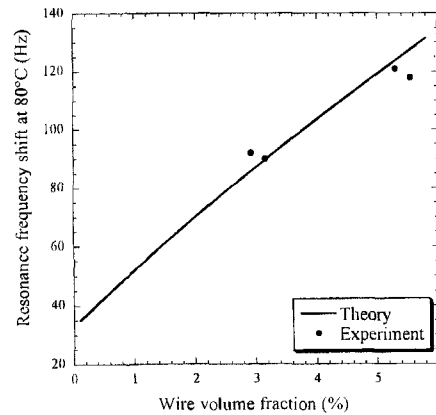


Figure 7: Resonance frequency shift at 80°C as a function of wire volume fraction, for a composite containing 3% wires, pre-strained 3%.

Finally, all tools are ready to predict the behavior of an adaptive composite in terms of resonance frequency shift at a given temperature, as a function of wire volume fraction. Such a simulation is presented in Figure 7, for activation at 80°C, assuming that the section of the sample and its modulus do not depend on the volume fraction wires. A reasonable agreement is obtained with the experimental data points presented alongside, indicating that the simple modeling approach is satisfactory in a first attempt to design with adaptive composites. This analysis was applied to the design of a winglet as a demonstrator in collaboration with the ADAPT partners [24].

## 5. CONCLUSION

Based on the above presented results, and on the fast growing body of literature on the topic of adaptive composite, it is clear that we have progressed towards a better control of adaptive composite manufacturing, and a set of design tools which are now proven in simple configurations. A number of challenges have been uncovered at various levels: (i) manufacturing techniques are still very hand-labor consuming and thus far from mass-production, (ii) precise modeling of the wire behavior is still in progress, (iii) design of complex shapes with SMA actuators is still not yet at a practical engineering level, (iv) more work is needed to take into account second order effects, such as the matrix viscoelastic properties, friction and damping effects, heat gradients effects. None of these seem however impossible to overcome if an industrial application is at hand. Finally, these materials still suffer from thermal management effects, which limit their response time and fields of use. Passive applications, or the use of other types of Shape Memory Alloys may then be a more viable solution if temperature variations are not allowed [25].

## ACKNOWLEDGEMENTS

This work was carried out in the frame of the ADAPT Brite/EuRam Project, supported the European Commission. The Swiss partners of the project were funded by the Swiss Federal Education and Science Office. The partners of the ADAPT project (EADS, Daimler-Chrysler, British Aerospace, KULeuven, ICE-HT in Patras) are gratefully acknowledged for their collaboration. Dr. R. Gotthardt and Dr. M. Parlinska of IGA-EPFL are specially acknowledged for collaboration within the ADAPT project and the set-up of the vibration measurements. Additional collaboration with the LMAF at EPFL is also acknowledged. Finally, Dr P. Sittner from the Academy of Science of the Czech Republic in Prague is sincerely thanked for the development of RCLoop and fruitful collaboration.

## REFERENCES

- [1] A.L. Roytburd, J. Slutsker and M. Wuttig, Chapter 5.23 of *Comprehensive Composite Materials*, volume 5, Elsevier, pp.507-522, 2000.
- [2] M.V. Gandhi and B.S.Thomson, "Smart materials and structures", Chapman and Hall, 1992.
- [3] C. Boller, *Encyclopedia of materials, Science and technology*, Elsevier Science Ltd, 2001, pp.1376-1382.
- [5] Z. G. Wei, R. Sandstrom and S.Miyazaki: *J. Mat. Sci.* **33** (1983) 3763-3783.
- [4] Z.G.Wei, R. Sandström, S. Miyazaki, *J. Mat. Sci.*, **33**, pp.3743-3762, 1998.
- [6] K.Otsuka, C. M. Wayman, "Shape memory materials", Cambridge University Press, Cambridge U.K., 1998
- [7] J.-E. Bidaux, J.-A.E. Manson, R. Gotthardt, *Mat.Res.Soc.Symp. Vol.459*, pp. 107-117, (1997).
- [8] C. Boller, *Mat.Res.Soc.Symp.Proc.Vol.604, Materials for Smart Systems III*, 2000. Materials Research Society, p.87-98.
- [9] J. Schrooten, V. Michaud, Y. Zheng, J.-A. Balta and J.A. Manson, *Proceedings of SPIE Smart Structures and Materials*, San Diego US, paper 4698-59,2002.
- [10] V. Michaud, J. Schrooten, M. Parlinska, R. Gotthardt and J.E. Bidaux, *SPIE Smart Structures and Materials*, 17-21 March 2002, San Diego US, paper 4698-60, 2002.
- [11] J. Schrooten, V. Michaud, J. Parthenios, G. Psarras, C. Galiotis, R. Gotthardt, J.A. E. Manson, J. Van Humbeeck, *Materials Transactions JIM*, Vol.43 No.5 (2002) pp.961-973.
- [12] K. Tsoi, R. Stalmans and J. Schrooten, *Acta Materialia*, **50**, 14, p.3535-3544, Sept 2002..
- [13] J.A. Balta, V. J. Michaud, J-A. E. Manson, M. Parlinska, and R. Gotthardt, *proceedings of ECCM 10*, Brugge, June 2002, CD version.
- [14] Y.Xu, K.Otsuka, H.Yoshida, H.Nagai, R. Oishi, H.Horikawa and T. Kishi, *Advanced Engineering Materials*, vol4, N9, 2002, p.683-686.
- [15] V. Michaud, J.A. Balta, F. Bosia, J.Botsis and J.A-E. Manson, *proceedings of ICCM 14*, San Diego, paper # 1621, July 2003.
- [16] J. Parthenios, G.C. Psarras and C. Galiotis,, *Composites Part A*, **32**, pp.1735-1747, 2001.
- [17] G. C. Psarras, J. Parthenios and C. Galiotis: *J. Mater. Sci.*, **36**, 535-546, 2001.
- [18] A.J.Zak, M.P. Cartmell, W.M. Ostachowicz and M. Wiercigroch, *Smart Materials and Structures*, **12** (2003), 338-346.
- [19] P. Sittner, R.Stalmans and M.Tokuda: *Smart Materials and Structures* **9** (2000) 452-469.
- [20] P.Sittner, V. Michaud and J. Schrooten, *Materials Transactions JIM*, Vol.43 No.5 (2002) pp.984-993.
- [21] P. Sittner and R. Stalmans: *JOM* **52** (2000) *Special Issue-Millennial Materials Technologies*, pp.15-20.
- [22] M. Parlinska, J.A. Balta, V. Michaud, J.E.Bidaux, J.A.-E. Manson and R.Gotthardt, *Journal de Physique IV*, vol 11 (PR8), pp.129-134, nov. 2001.
- [23] J.-E. Bidaux, J.-A.E. Manson, R. Gotthardt, *Mat.Res.Soc.Symp. Vol.459*, pp. 107-117, (1997).
- [24] J. Simpson and C. Boller, , *SPIE Smart Structures and Materials*, March 2002, San Diego US, paper 4698-45.
- [25] V. Michaud, *Scripta Materialia*, in press, 2004.