DEVELOPMENT AND CHARACTERISATION OF HIGH DAMPING MAGNESIUM BASED COMPOSITES

Christine Mayencourt and Robert Schaller

Institut de Génie Atomique, Ecole Polytechnique Fédérale de Lausanne,
1015 Lausanne, Switzerland

SUMMARY: C / Mg and SiC / Mg have been produced by gas pressure infiltration of fibre preforms by a Mg-Si alloy. The Young's modulus of these composites corresponds to the mixture law. The damping capacity of the magnesium matrix is preserved and is 10 to 100 times higher than in an industrial magnesium alloy (AZ63). Moreover, the mechanical loss spectra obtained by mechanical spectroscopy are modified by transitory phenomena which appear during thermal cycling. The transient response informs us about the thermal stress relaxation mechanism occurring at ceramic-metal interfaces and thereby about the interface quality and the fatigue behaviour. The comparison between the experimental results and a model of dislocation motion allows one to interpret the energy dissipation as due to reversible dislocation motion, which is controlled by a solid friction mechanism. This interpretation is confirmed by transmission electron microscopy observations during "in situ" thermal cycling. Furthermore, the composites do not exhibit thermal fatigue over more than 50 thermal cycles (100K-400K).

KEYWORDS: Damping capacity, Young’s modulus, fatigue, magnesium, unidirectional fibres, dislocations, microstructure.

INTRODUCTION

Numerous problems in engineering are caused by mechanical vibrations. They affect the good running of electronic devices, they limit the precision of machine-tools, they generate fatigue of materials or simply induce noise pollution. Especially in transport systems, there is a need of lightweight materials which exhibit simultaneously a high-damping capacity and good mechanical properties such as a high modulus, a high mechanical strength, good creep resistance or good fatigue resistance [1]. These properties are often incompatible [2], but composites can join them if each phase plays a specific role [3]. Thus, metal-matrix composites (MMCs) play an increasing role as construction materials because of their high specific properties, in particular for aerospace applications.

However, the introduction of ceramic fibres or particles in a metal matrix often leads to a decrease in fatigue resistance or toughness. The effect of stresses on mechanical behaviour of MMCs can be considerable. Damage accumulation can occur by cracking of reinforcement [4], by debonding along the interfaces [5] or by crack propagation in the matrix [6]. Mechanical

stress is not the only factor which induces fatigue. Thermal stresses, build up at interfaces by thermal expansion coefficient mismatch, can be very important [7]. As the mismatch in thermal expansion coefficient can be large, thermal stresses can exceed the yield stress during temperature changes and may induce significant degradation of mechanical properties. Hence, the effect of thermal variations are very similar to non-stationary stresses which induce mechanical fatigue. Such phenomena can degrade the mechanical properties during thermal treatments or simply during cooling from the processing temperature [8]. However, the mechanism of thermal stress relaxation can also be creation and motion of dislocations [9, 10, 11], preserving the mechanical properties. Thus, it is important to know which of the microscopic relaxation mechanisms is active, in order to predict a possible degradation of the composite properties.

Pure magnesium is known to exhibit a very high damping capacity [12, 13, 14], but its mechanical strength is low. This metal can be hardened by introducing second phase precipitates which are able to pin dislocations, in order to avoid plastic deformation and thereby to increase the tensile strength. However, if the mechanical properties are better in magnesium alloys than in pure magnesium, the damping capacity is lost in most of them. The magnesium matrix has to be relatively pure to exhibit a high-damping capacity. Therefore, a pure magnesium matrix reinforced with high-strength fibres is a good candidate for a high-damping composite: magnesium matrix being responsible for the high damping and fibres being responsible for the high mechanical strength. Moreover, the high damping capacity is expected to increase the fatigue resistance and the toughness by dissipating energy through dislocation vibration instead of crack initiation and propagation [15, 16].

In this context, two phase composites, which consist in a Mg-Si alloy reinforced with C or SiC aligned fibres, have been processed. The composites have been mainly studied by mechanical spectroscopy and transmission electron microscopy. Mechanical spectroscopy permits one to quantify the damping capacity of the composites. Furthermore, this technique has been proved to be an efficient non-destructive method to investigate interfaces [6]. Actually, interface thermal stresses arising during heating or cooling induce an additional response in mechanical spectroscopy measurements [9, 17], superimposed to the isothermal equilibrium damping. This transient additional response should reveal the active mechanism for thermal stress relaxation. This paper aims to compare mechanical spectroscopy measurements and transmission electron microscopy results with a theoretical model [11], which relates the thermal stress relaxation with the motion of dislocations.

EXPERIMENTAL PROCEDURES

Before infiltration, the fibres were treated to remove the sizing agent (30 min. at 730°C). The surface of fibres was observed by atomic force microscopy (AFM), in order to insure their complete desizing. Single fibre was extracted from a yarn and glued on a glass slide. The observations were performed on a M5 (Park Scientific Instrument) with a maximum scan range of 100 µm.

Mg-based composites were processed by gas pressure infiltration [18] of two different preforms by a magnesium alloy (Mg-Si 2 wt%). Preforms are composed by long unidirectional C or SiC fibres. Both ceramic preform and magnesium alloy were preheated at 720°C. A pressure of 2 MPa was applied during infiltration. Then the composite is rapidly cooled down by air circulation.

After infiltration, plate-shaped specimens were cut from the solidified rods by spark machining, in such a way that the sample length is parallel to the fibres. The Young's modulus was deduced from the flexure vibration resonance frequency of a sample. The optical pictures of composites together with the Young's modulus values allows on to evaluate the quality of infiltration process.

Mechanical loss spectra are obtained from measurements in a forced, inverted torsion pendulum [19]. The energy dissipation is given by the mechanical loss angle tan(ϕ), where ϕ is the phase lag between the applied stress and the strain. In other words, the mechanical loss angle tan(ϕ) is a measure of the damping capacity of materials. Further, spectra obtained during thermal cycling at different frequencies or different heating rates are compared with a theoretical model [11] in order to identify the thermal stress mechanism. After being fixed in the measurement setup, the samples are first cooled down to 100 K and then annealed "in situ" for 4 hours at 600 K, in order to get a stable behaviour. When not indicated, the measurement parameters were ω=0.5 Hz, ε=2 x 10^{-5} and $\dot{T}$ =2 K/min.

Besides, some transmission electron microscopy (TEM) experiments have been done during "in situ" thermal cycling. Samples were cut by spark machining at a thickness of 200 µm, then mechanically polished to approximately 60 µm and finally ion beam thinned at 5 kV, 0.5 mA. The observations were carried out in a CM20 (Philips) at 200 keV. Movies, which displayed dislocation network in the magnesium matrix and dislocation motion, have been recorded during heating and cooling.

Finally, a first thermal fatigue test has been done on the C/Mg composite. During mechanical spectroscopy measurement, the sample has been submitted to more than 50 thermal cycles from 100K to 400K.

RESULTS

Composite processing

During the preform preparation, the fibres were treated to remove the sizing agent (30 min. at 730°C). The surface of fibres was observed by atomic force microscopy (AFM) in order to verify the desizing (Fig. 1). One can observe that the sizing agent has been almost completely removed by the treatment, the arrows point the sizing residues. As the fibres are heated again before the infiltration, one can consider that the sizing agent is perfectly removed for the infiltration. The surface of the C fibre is grooved and the surface of the SiC fibre is smooth.

C / Mg and SiC / Mg were obtained by gas pressure infiltration. The infiltration has been fully achieved in both cases. The C and the SiC fibres are completely embedded in the matrix and the fibre surface is not damaged (Fig. 2). One can also see some short and fine Mg_2Si second phase precipitates in the matrix, visible especially on the C / Mg picture but present in both composites. A volumetric fibre concentration of about 30% C and 40% SiC was calculated by image analysis. From the volumetric fibre concentration and the Young's modulus of the matrix (45 GPa) and the fibres (640 GPa for C, 200 GPa for SiC), one can evaluate the composite Young's modulus. The mixture law gives a Young's modulus of 224.7 GPa for C/Mg and 107 GPa for SiC/Mg. Experimentally, a Young's modulus of 239 ± 40 GPa for C/Mg and 99 ± 20 GPa for SiC/Mg were obtained by a resonance method.

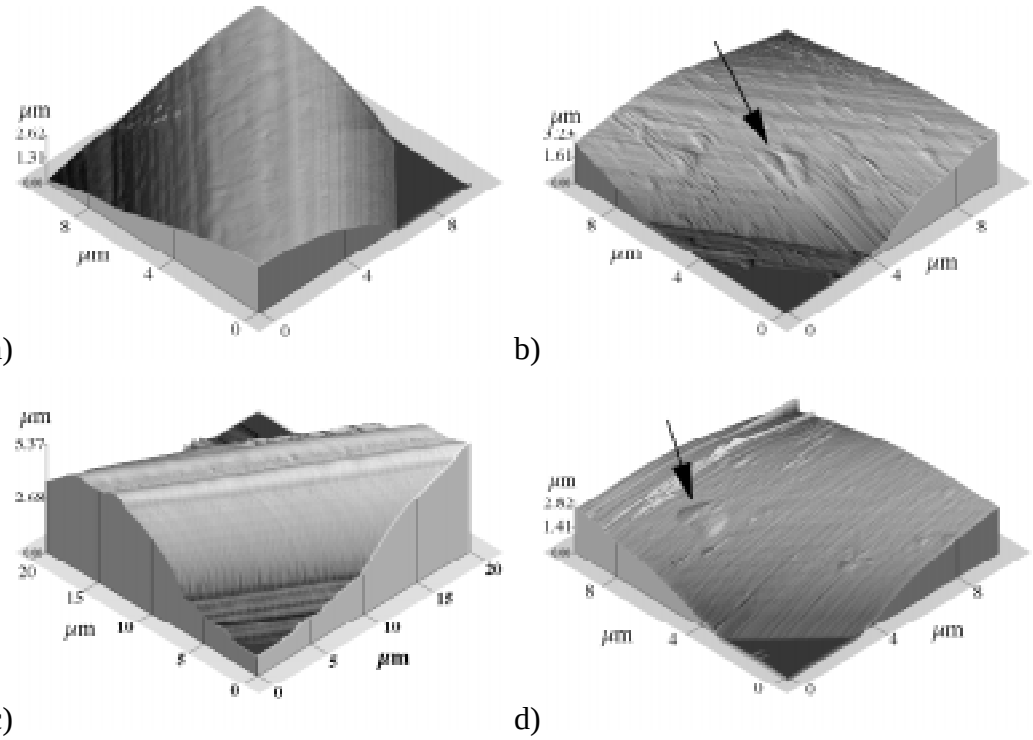

Fig. 1: AFM images of: a) C fibre as received, b) C fibre after desizing, c) SiC fibre as received, d) SiC fibre after desizing. Arrows indicate the sizing residues.

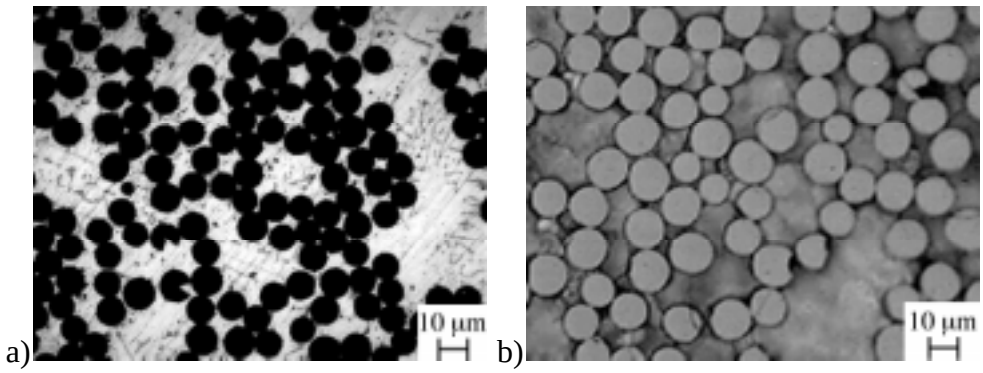

Fig. 2: Transverse optical image of a) a C/Mg sample, b) a SiC/Mg sample.

Damping properties

The composites are characterised by a high damping capacity. Fig. 3 shows the mechanical loss angle as a function of the temperature in both composites, the spectrum for an industrial alloy (AZ63) is displayed as a comparison. The damping capacity level is lower in C / Mg than in SiC / Mg, but largely higher than in the industrial alloy. The mechanical loss spectra have a similar shape in both composites. The mechanical loss is rather constant over the whole temperature range and presents a large hysteresis during thermal cycling: for temperature higher than 350 K damping is higher during heating than cooling, an inversion of this behaviour occurs at lower temperature. A maximum in SiC/Mg appears between 100 and 300 K, and a small second one appears at 380 K. The maximum in C/Mg is broader and covers over the whole temperature range between 100 and 500 K.

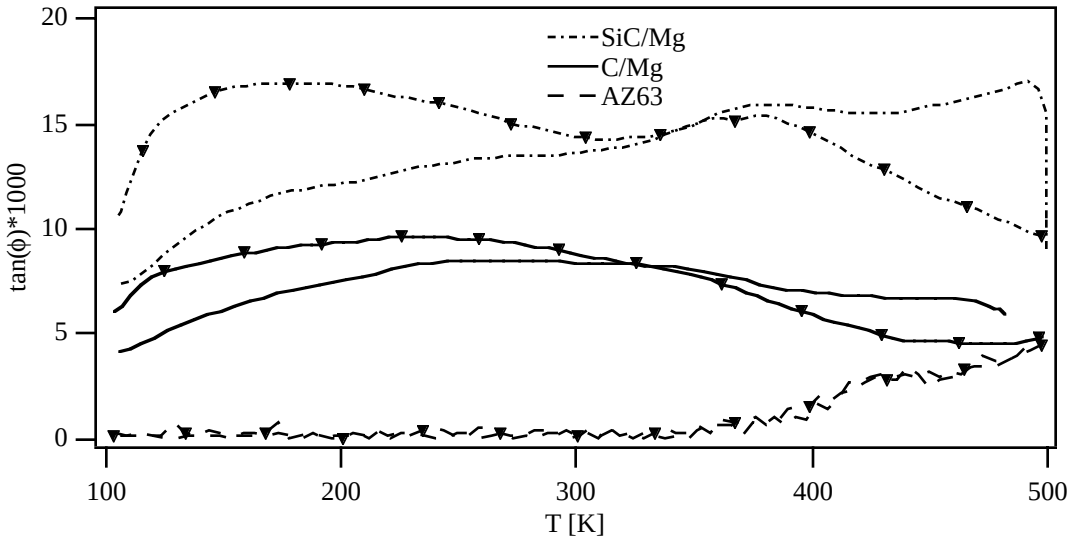

Fig. 3: Mechanical loss angle versus temperature in a SiC/Mg and C/Mg. Markers indicates cooling runs.

Mechanical loss spectra have a strong dependence on the temperature variation rate ($\dot{T}$) and on the frequency (ω). Fig. 4 displays spectra measured in a C / Mg sample during cooling at (a) different cooling rates and (b) different frequencies. One can clearly see that the mechanical loss increases with $\dot{T}$ and decreases with ω. Spectra of SiC / Mg samples present the same behaviour.

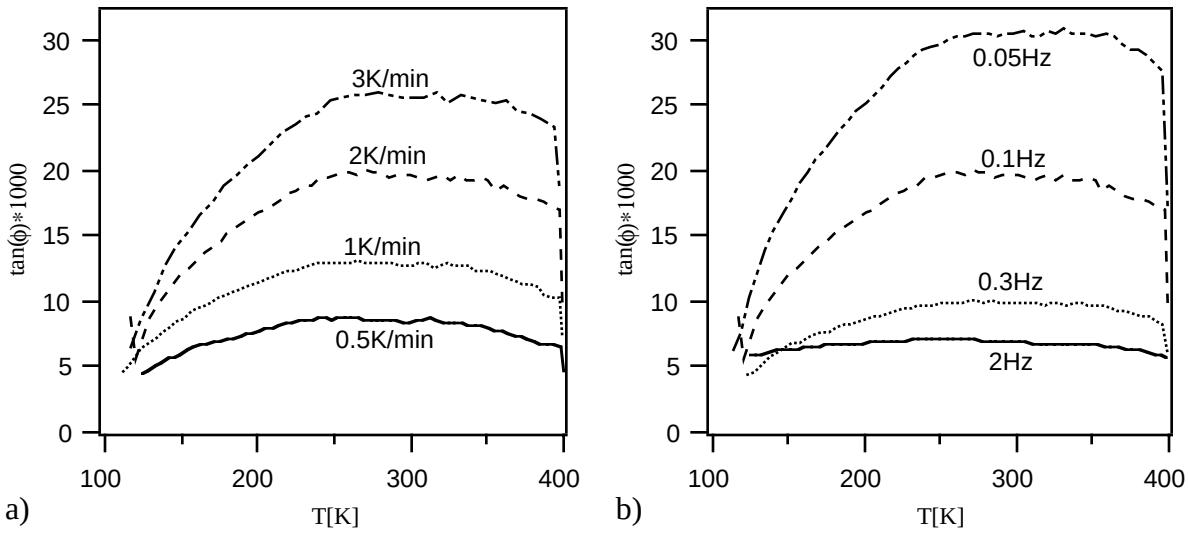

Fig. 4: Mechanical loss angle spectra measured in the C / Mg sample. a) At different heating rate with a frequency of 0.01 Hz. b) At different frequency during heating at 2 K/min. Tan(ϕ) increases with $\dot{T}$ and decreases when ω increases.

Furthermore, the relation between the mechanical loss and $\dot{T}$ or ω is non linear. Fig. 7 displays points measured at 350 K on spectra taken at different heating rate or frequency for both C / Mg and SiC / Mg. Tan(ϕ) depends not simply on $\dot{T}$ or ω, but depends on the ratio $\dot{T}/\omega$, and this dependency is non linear. The dependency law is the same for both samples, but the intensity of the phenomenon is more important in the SiC / Mg sample than in the C / Mg sample.

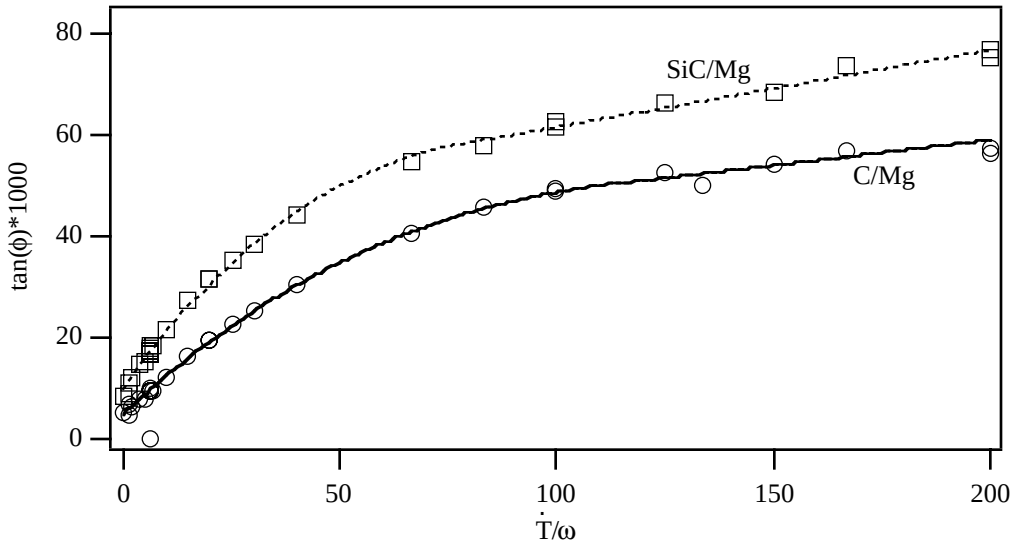

Fig. 5: Mechanical loss angle as a function of $\dot{T}/\omega$ in a C / Mg sample and a SiC / Mg sample. The curves are calculated from the theoretical model of dislocations which dissipate energy by friction mechanism. Tan(ϕ) increases with $\dot{T}/\omega$. The curves fit well the experimental points.

A model has been recently developped in order to interpret the non linear dependence of the mechanical loss on $\dot{T}/\omega$ [11]. The model is based on the hypothesis that the thermal stress relaxation is due to dislocation motion, which is controlled by a solid friction mechanism. The theoretical curves calculated from the model are also displayed on Fig. 5. The curves fit perfectly the experimental points in both composites.

Dislocation network

The interpretation of a solid friction mechanism is corroborated by a transmission electron microscopy experiment (TEM). A thinned SiC / Mg sample has been filmed by TEM during "in situ" thermal cycling between room temperature and 250°C. The dislocation motion has been observed. The motion is not a regular gliding but clearly a "step by step" motion. Fig. 6 displays pictures taken from the film during heating. An arrow repairs a dislocation. One can observed that the dislocation does not move between 57°C and 61°C, and has suddenly moved between 61°C and 62°C. The movie can be watched on the web site http://igahpse.epfl.ch/mmc/dislocation_e.html. No dislocation creation has been noticed.

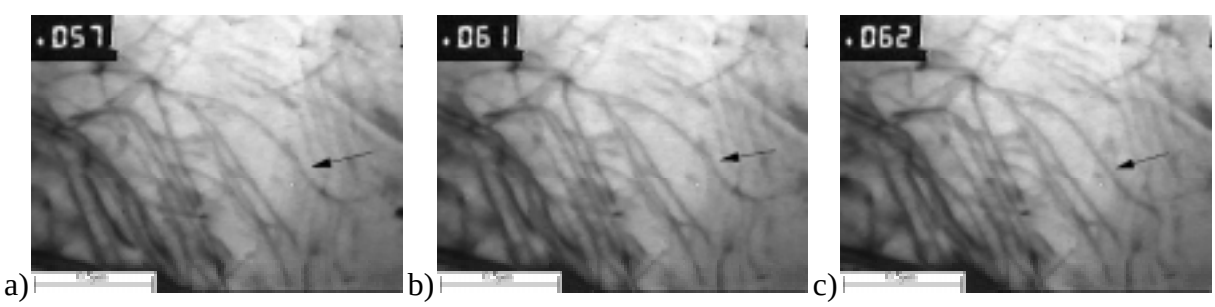

Fig. 6: Images obtained by TEM during "in situ" heating. Temperature is indicated in Celsius degrees on each image: a) 57°C, b) 61°C, c) 62°C. Arrow repairs a dislocation, which does not move between 57°C and 61°C, and has suddenly moved at 62°C.

The dislocation motion is a reversible phenomenon. Fig. 7 displays pictures taken during cooling. The same dislocation as during heating is repaired by an arrow. One observes a similar

motion in the opposite direction. The dislocation is at a fix position between 46°C and 43°C and suddenly is at another position at 42°C. Due to the reversibility of dislocation motion, the same dislocations are present during heating and cooling. The dislocations get globally the same positions, but at a lower temperature during cooling than during heating, due to the hysteresis of the phenomenon.

Fig. 7: Images obtained by TEM during "in situ" cooling. Temperature is indicated in Celsius degrees on each image: a) 46°C, b) 43°C, c) 42°C. Arrow repairs a dislocation, which did not move between 46°C and 43°C, and had suddenly move at 42°C.

Thermal cycling

Such a reversible phenomenon should increases the thermal fatigue resistance of the composites. Although no real thermal fatigue tests were done, some results suggest that the thermal fatigue of the magnesium-based composites is limited. Fig. 8 displays the mechanical loss measured in a C / Mg sample after fifty thermal cycles between 100 and 400K. It does not exhibit any change from the first cycle.

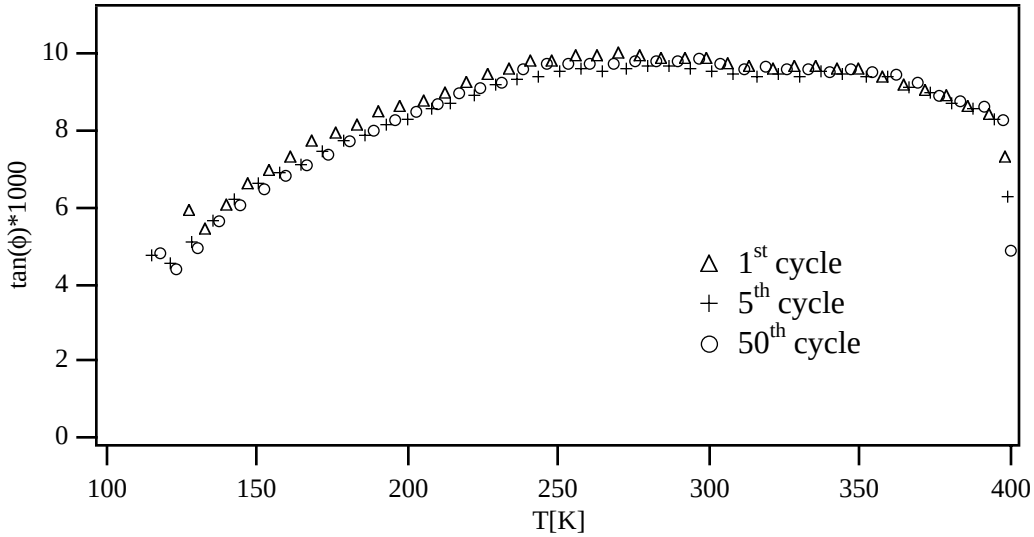

Fig. 8: Mechanical loss vs temperature in a C/Mg sample, after 1, 5 and 50 thermal cycles. The spectra are not modified.

DISCUSSION

Both composites present a similar structure, with aligned long fibres. In both cases the processing is satisfying. The micrographs of all the samples (Fig. 2) show a clean interface between the matrix and the reinforcement. The fibres are well bonded, no porosity is visible at

the optical scale, the fibre surface seems to be not damaged. This indicates that the infiltration process is achieved. In the C/Mg composite the Young's modulus is five times higher and in the SiC/Mg two times higher than the one of pure magnesium, with a negligible increase in weight. In both cases, this corresponds to the mixture law. Because of the structure of long and aligned fibres, the composite can be considered as a system of springs in parallel. The Young's modulus is not greatly influenced by the interface properties [20]. Thus, the mixture law is a good first approximation of the composite properties. Even if the good agreement between the measurement and the calculation does not give information concerning the interface quality, this allows one to conclude that the fibre properties are not degraded during the processing.

The samples exhibit a high damping, as was desired. Moreover, the damping does not depend on the frequency [21], and depends only weakly on the temperature (Fig. 3). This behaviour is coherent with a damping, which originates in the magnesium matrix and which is due to the dislocation vibrations controlled by pinning-depinning from point defects. This has already been observed in magnesium and some magnesium alloys [13, 14] and magnesium-based composites [21, 22, 23]. Such a high damping, independent on the frequency and temperature is an advantage for applications where the utilisation conditions can vary. The damping level also indicates the matrix purity [13]: if the matrix contain too much point defects (solute atoms), they firmly pinned the dislocations and the breakaway is not possible. The damping capacity of SiC/Mg and C/Mg is slightly lower than the damping capacity of pure magnesium [12]. However, the decrease of damping can be explained by the high volumetric fibre content. This means that the composites can combine the properties of damping and strengthening because each phase plays a specific role. The fibres are responsible for the strengthening and the magnesium matrix is responsible for the damping; the mechanisms are independent [3]. Thus, damping is due to dislocation vibration in the matrix and plastic deformation is avoided by the fibres limiting the long distance movement of dislocations. Besides, a correlation between the damping level and the toughness has recently been proposed [16]. The toughness is related to the energy dissipation at the crack tip. Thus, the good ability to dissipate energy, here through dislocation motion in the matrix, is beneficial to improve the fracture resistance by dissipating energy at the crack tip.

Mechanical spectroscopy measurements performed during thermal cycling in magnesium-based composites show a non linear dependence of the mechanical loss angle on $\dot{T}/\omega$. Tan(Φ) can be decomposed in two parts: an intrinsic damping of the material at thermal equilibrium and an additional transient mechanical loss which depends on $\dot{T}/\omega$. Such a decomposition of tan (Φ) has already been used in order to analyse spectra obtained in other materials [9]. The transient mechanical loss is commonly interpreted as due to the thermal stress relaxation, generated by the thermal expansion coefficient mismatch between the different phases. Such a mismatch can generate thermal fatigue, as explained in the introduction. A model based on dislocation motion [11] has been used to identify the relaxation mechanism. This model assumes that the stress relaxation is due to dislocations, which are pushed away from the fibre-matrix interface by thermal stresses. The dislocation motion is controlled by a hysteretic solid friction mechanism in the matrix. Fig. 5 indicates that this model is appropriate in the case of magnesium-based composites reinforced by C fibres or SiC fibres. Actually, the theoretical curves calculated with the help of the model fit perfectly the experimental points. Moreover, this interpretation of the thermal stress relaxation is confirmed by the transmission electron microscopy images (Fig. 6 and 7). Further development of the model should determine more precisely the meaning of the curve parameters (slope and bowing), and to relate them to composite parameters as the fibre content or the dislocation density, with the help of TEM results. The good agreement between the model and the experimental results allows one to conclude that the thermal stress relaxation in magnesium-based composites is due to dislocation gliding in the matrix. The dislocation

motion is controlled by a solid friction mechanism. This mechanisms is reversible and hysteretic (Fig. 6 and 7). That allows one to explain the large hysteresis observed between cooling and heating (Fig. 3) as due to the inversion of the thermal stresses when $\dot{T}$ sign changes.
Because of the reversibility of the dislocation motion, such a mechanism should preserve the interface bonding and limit thermal fatigue.

Actually, real fatigue tests were not performed in the specimens, but thermal cycling over 50 runs did not affect the mechanical loss spectra. Such an absence of thermal fatigue has also been observed in other Mg-based composites [15]. This indicates that the thermal stress relaxation by dislocation motion is a property of magnesium. This make high-damping magnesium matrix very promising for fatigue reduction.

CONCLUSION

Magnesium-based composite materials have been processed and characterised. They exhibits enhanced mechanical properties and preserved damping properties as compared with pure magnesium. The high-damping capacity is interpreted as due to dislocation dissipative mechanisms in the Mg matrix and the good mechanical properties are related to the presence of high modulus fibres. A theoretical model has been successfully applied to experimental results of thermal behaviour. The comparison between the model and the experimental results allows one to conclude that the thermal stress relaxation in magnesium-based composites is due to dislocation gliding in the matrix controlled by a solid friction mechanism. This interpretation is confirmed by in situ TEM studies. Such a mechanism should preserve the interface bonding and limit thermal fatigue. Actually, no thermal fatigue is observed after 50 thermal cycles.

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