Energy Damping in Magnesium Alloy Composites Reinforced with TiC or Ti₂AlC

Particles

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Abstract

In part I of this study, we reported on the synthesis and mechanical properties of Mg alloy composites reinforced with TiC or Ti₂AlC particles. In this, part II, we explore their damping as a function of the nature and volume fraction of the reinforcing particles and the matrix alloy composition. Using Mg alloys instead of pure Mg and reducing the reinforcement particle size reduced the damping capabilities of these composites. In all cases, a tradeoff was found between a composite's stiffness and its damping capability - as measured by the area enclosed in its fully and spontaneously reversible cyclic compressive stress-strain curves. Low stiffness materials, such as pure Mg and its alloys, dissipated significant amounts of energy, but only at lower stresses. The TiC reinforced composites dissipated the least energy. However, by combining two solids, Mg or its alloys with Ti₂AlC, that are both inherently damping, it is possible to dissipate roughly 25% of total applied mechanical energy, while simultaneously maintaining ultimate compressive stresses and stiffness values that were almost as good as those of the TiC reinforced composites. However, since below 200 MPa, Ti₂AlC does not dissipate much energy, the difference between the two was only manifested at stresses > 200 MPa. When the interfaces were strong, cyclic hardening and no fatigue were observed; when the interfaces were weak, cyclic softening and fatigue were recorded. Most importantly, we show that the energy needed to extend or bow out the dislocations – that are presumably strongly pinned and postulated to be the mechanism for energy dissipation – is a linear function of the applied stresses for the end members – Mg and Ti₂AlC – and is even more pronounced for the composites.

Keywords: Composites; Energy damping; Magnesium alloys; Carbide reinforcements; MAX phases

1. Introduction

High damping materials are important in structural applications in which reducing mechanical vibrations and noise are needed. Moreover, in different applications, such as automotive and aerospace, lightweight materials with high damping capability and high mechanical strength and modulus are needed. However, higher damping materials usually have lower strengths and/or moduli. Metal matrix composites, MMC, have been used in order to improve the damping properties of metals and at the same time enhance the mechanical properties such as stiffness and strengths.

It is possible to tailor the damping properties of MMCs firstly by applying reinforcements with high damping capacities and/or controlling their volume fractions and geometries, and secondly by modifying the matrix microstructure to create sources for energy dissipation, such as defects and interfaces. The damping capacities of MMCs were mostly ascribed to the presence of large numbers of defects [1, 2]. In general, the presence of a second phase can create two types of defects: large interfacial areas between the composite components [3, 4] and high dislocation densities in the regions adjacent to the interfaces, that can form during processing or deformation [2]. Interfacial damping results from frictional energy loss caused by sliding at the reinforcement/matrix interface. In general, weak interfaces can lead to more energy dissipation [4, 5]. Among different shapes of reinforcement used in MMCs, one of the highest damping improvement was reported when particulate reinforcements were used in MMCs [4].

Residual thermal strain, as a result of the large coefficient of thermal expansion, CTE, mismatch between matrix and reinforcement, can also result in high densities of dislocations in the former [1]. In the case of MMCs reinforced with ceramics particulates,

the CTE values of the metal matrix are typically in the range of $25 \times 10^{-6} \text{ K}^{-1}$ while those for the ceramic reinforcement are closer to 3 to $10 \times 10^{-6} \text{ K}^{-1}$ [2, 6]. This large CTE difference can result in a high density of dislocations induced during cooling from processing temperatures.

By applying reinforcements with high damping capabilities, it is also possible to further enhance the MMCs damping capacities, which in turn can be controlled by varying the reinforcement volume fraction, size and shape. Zhang *et al.* reported that 1 vol.% SiC-Al alloy composite, fabricated via spray deposition, had the same damping properties as monolithic spray deposited Al alloy. However, when 1 vol.% of graphite particulates were used, instead of SiC, the damping capacity of the composite was doubled, which was attributed to the inherent damping of graphite [4].

In contrast to Al, the hexagonal close-packed metals, Ti, Mg, Co, Zr and Zn, inherently have good damping capabilities. Zhou *et al.* showed that when polycrystalline Co [7], Mg [8] or Ti [9], samples are cyclically loaded in simple compression, fully reversible loops formed above a threshold stress, σ_t . The size and shape of these loops was found to be a strong function of grain size; large grained samples had significantly larger damping capacities. The size of the loops was also a function of pre-plastic deformation that was attributed to the fact that plastic deformation reduced the grain size, which in turn increased σ_t and ultimately reduced damping. In these studies, the damping behavior of these hexagonal metals was explained by a dislocation-based model and they were all classified as kinking non-linear elastic, KNE, solids [7-9]. The KNE model, which describes the damping mechanism in these solids, is discussed in reference [10].

Among all the structural metallic materials, pure cast Mg has the highest damping

capability [11]. That was explained by the large average grain size of as-cast Mg and also by the low concentration of alloying elements, both of which lead to longer and easier dislocation movement on basal planes and easier twin formation [8, 12].

In general, the elastic and mechanical properties of pure Mg and Mg-alloys are relatively low for structural materials. One approach to enhancing these properties is to fabricate Mg matrix composite. However, since Mg damping is strongly grain size dependent [8, 10], and fabricating Mg-matrix composites reduces its grain size, in almost all reports on the latter, typically improvements in mechanical properties come at the expense of lowering the damping capabilities [13, 14].

As noted above, one approach to enhance the damping properties in MMCs is to use reinforcements that exhibit high damping capabilities. In previous work, Amini et al., showed that in pure Mg matrix composites, reinforced with 50 vol.% ceramic particulates, changing the reinforcement type from SiC to Ti_2AlC or Ti_3SiC_2 can double the damping properties of these composites. The ternary carbides, Ti_2AlC and Ti_3SiC_2 , belong to MAX phases (see below) with inherent damping capacities [15, 16]. At 700 ± 10 MPa the ultimate compressive strength, UCS, of 50 vol.% Ti_2AlC -Mg was reported to be significantly higher than those of the 50 vol.% Ti_3SiC_2 -Mg and 50 vol.% SiC-Mg composites, which was attributed to the presence of nano-Mg grains in the Ti_2AlC reinforced composite. As explained in part I of this study [17], further work has shown that not all the Mg grains are at the nano scale. Instead, the presence of nano Mg grains, observed in previous work, was most probably due to Mg infiltration of pre-existing nano sized delaminations within the Ti_2AlC particles [17, 18]. In part I of this study it was also concluded that the high strengths obtained were due to strong matrix/reinforcement

interfaces as a result of the presence of Al in Ti_2AlC and also finer reinforcement particle sizes [17].

The ternary carbide, Ti_2AlC , belongs to a large 70+ family of ternary carbides – with a general formula $\text{M}_{n+1}\text{AX}_n$, (MAX) where $n = 1, 2$ or 3 ; M is an early transition metal, A is an A-group element (mostly groups 13 and 14) and X is either carbon and/or nitrogen [19-21]. The M_{n+1}X_n layers are interleaved with layers of pure A-group elements. The MAX phases combine some of the best attributes of metals and ceramics. Like metals, they are electrically and thermally conductive, most readily machinable [22, 23], not susceptible to thermal shock, plastic at high temperatures, and exceptionally damage tolerant [24]. Like ceramics, some of them are elastically rigid (Young's moduli > 300 GPa), lightweight (≈ 4 gm/cc) and maintain their strengths to high temperatures. Ti_2AlC is also creep, fatigue and oxidation resistant [25-27]. In addition, MAX phases have energy damping capacities. In certain cases, up to 25 % of the mechanical energy can be dissipated during cyclic loading even at stresses of the order of 1 GPa [15]. The reasons Ti_2AlC was chosen for this work are its relatively high elastic moduli, low density, machinability and high damping capabilities [10, 19].

In part I of this study, the fabrication and the mechanical properties of pressureless melt infiltrated Mg alloy composites, reinforced with TiC and Ti_2AlC particles – with two particle sizes – were reported [17]. The volume fractions of the reinforcement phases were ≈ 50 vol.% for all the composites. In addition to pure Mg, three Al-containing Mg alloys, AZ31, AZ61 and AZ91, were used as matrices. At 1028 ± 5 MPa, the UCSs of a fine-grained, FG, TiC-AZ61 is one of the highest UCSs ever reported for a TiC-Mg composite. The elastic modulus, at a stress of 100 MPa, E_{100} , and

the Vickers hardness, V_H , of this composite were measured to be 174 ± 5 GPa and 3.4 ± 0.3 GPa, respectively.

In the Ti_2AlC reinforced composites, the best mechanical properties were achieved when AZ61 was used as the matrix in FG- Ti_2AlC composites. In this case E_{100} , UCS and V_H were reported 136 ± 6 GPa, 760 ± 9 MPa and 2.1 ± 1 GPa, respectively. It was also concluded that the optimum Al content is ≥ 6 wt.%. Also, reducing the reinforcement particle size in both the Ti_2AlC - and TiC -composites, significantly enhanced E_{100} , V_H , yield strength, YS and UCS values, which was attributed to the refinement of the matrix grains and to the presence of finer particles in the matrix. The high elastic and mechanical properties were thus ascribed to the presence of Al, which resulted in a strong matrix/reinforcement interface and grain refinement of the Mg matrices due the presence of fine reinforcement particles.

The purpose of this work is to report on the damping properties of the aforementioned composites. The effects of matrix and reinforcement compositions and particle size on the damping properties of these composites are explored. To investigate the effect of reinforcement volume fraction, pure Mg matrix reinforced with 5 and 20 vol.% reinforcement - Ti_2AlC and TiC – composites were also fabricated and their damping properties measured and compared to their 50 vol.% counterparts.

3. Experimental Details

3.1. Materials

Pure Mg (99.8% pure) in powder (- 325 mesh) and as rods, AZ31B (3 wt.% Al, 1 wt.% Zn) both purchased from Alfa Aesar (Ward Hill, MA) and AZ61L (6 wt.% Al, 1

wt.% Zn, low Mn) and AZ91D (9 wt.% Al, 1 wt.% Zn) both supplied by Thixomat (Livonia, MI) were used as the matrix materials. TiC powders (Alfa Aesar, Ward Hill, MA) and Ti₂AlC powders (Kanthal, Sweden) were used as reinforcements. Details about the reinforcement particle sizes can be found in part I of this study [17].

3.2. Processing

Composites with three different volume fractions of the reinforcement at 52±2 vol.%, 20±2 vol.% and 5±1 vol.% were fabricated. The 52±2 vol.% reinforcement composites (henceforth referred to as 50 vol.%) were made via pressureless melt infiltration, MI. Two different particle sizes of TiC and Ti₂AlC powders were used. The composites were made using TiC powder with d_{av} = 4.7 µm and 0.4 µm, henceforth will be referred to as coarse grain 50 vol.%, CG50- and fine grain 50 vol.%, FG50-, TiC composite, respectively. The composites with 50 vol.% of Ti₂AlC with d_{av} = 1.5 µm and 0.5 µm henceforth will be referred to as CG50- and FG50-Ti₂AlC composites, respectively. More details of the processing of these composites can be found in the part I [17].

The 20±2 and 5±1 vol.% reinforcement composites were made via powder metallurgy and only pure Mg powders were used. These composites henceforth will be referred to as 20- and 5- Ti₂AlC-Mg or TiC-Mg composites. A proper mixture of reinforcement particles, Mg powder and zirconia milling balls were sealed in a plastic jar and the latter was sealed in a steel can inside a glove box filled with argon, Ar, gas. The mixtures were then removed from the glove box and ball-milled for 12 h and cold pressed with a load corresponding to a stress of 50 MPa into rectangular bars (1.3 x 1.3 x 70

mm³). The porous bars were then placed in alumina crucibles (AdValue Technology, Tucson, AZ), the insides of which were lined with graphite foil to avoid reaction between Mg and alumina. The crucibles were in turn covered with alumina lids and placed in a graphite-heated vacuum-atmosphere furnace, (Series 3600, Centorr Vacuum Industries, Somerville, MA) heated at 10°C/min to 750°C, held at that temperature for 1 h, after which the furnace was turned off and the samples were furnace cooled. For comparison purposes, chunks of monolithic AZ61L were melted, following the same steps outlined above.

3.3. Microstructural characterization

The bulk composite samples were cross-sectioned, mounted and polished down to 1 µm with diamond slurries. The polished microstructures were imaged using a scanning electron microscope (SEM) (Zeiss Supra 50VP, Germany) equipped with an energy-dispersive spectroscope (EDS) (Oxford Inca X-Sight, Oxfordshire, UK).

3.4. Compression tests

All specimens were electro-discharge machined, EDMed. The room temperature UCSs were measured using an electromechanical testing machine (Instron 5600, Norwood, MA) on small (4x4x4 mm³) cubes that were EDMed. Six cubes of each microstructure were tested. Cylinders, 9.7 mm in diameter and 31 mm high, were EDMed and used to carry out cyclic uniaxial compression tests. In all cases, the samples' macroscopic strains were measured using an extensometer (2620-603 Instron, 10 mm gauge length with a 10 % full range) directly attached to the sample.

The following protocol was used for the cyclic compression experiments: Each cylindrical sample was loaded to a stress corresponding to $\sim 75\%$ of its UCS for two consecutive cycles before cyclically loading it by 50 MPa decrements from the maximum load during each cycle for the composites and by 20 MPa decrements for the pure AZ61 alloy. Immediately following the decremental cyclic compression tests, and to further investigate the low cycle fatigue properties of these composites each cylinder was cyclically loaded 100 times, with a strain rate of 0.01 s^{-1} , to a stress corresponding to $\leq 70\%$ of their UCSs.

4. Results

4.1. Microstructural observation

The microstructures of all specimens revealed a relatively homogenous distribution of the reinforcement in the composites. The 50 vol.% reinforced composite's microstructures were shown in part I of this study [17]. Figures 1a and b, respectively, show the microstructures of the polished surfaces of 20- and 5-Ti₂AlC-AZ61 composites, respectively. Figures 1c and d, respectively, show the microstructure of the 20- and 5-TiC-Mg composite tested herein. In all cases, the darker regions correspond to the Mg matrix and the brighter regions correspond to the carbide reinforcements.

4.2. Elastic moduli

Figure 2a, shows typical complete cyclic compression tests - two cycles to the maximum stress followed by decremental loading - for the FG50-Ti₂AlC-AZ61, CG50-Ti₂AlC-AZ31, 5-TiC-Mg and as-cast AZ61. All first cycle stress-strain curves for the other 50 vol.% composites discussed herein were shown in part I of this study [17]. In all

cases the first cycle is open, registering plastic strains of the order of 0.5 to 3% (Fig. 2a), after which, all subsequent cycles at stresses lower than the maximum stress are closed. The first loading cycles - without the hysteresis loops - of the 5-Ti₂AlC-Mg and 20-TiC-Mg composites are also plotted in Fig. 2a.

In the case of 50 vol.% reinforcement composites, hysteresis loops were only observed at stresses higher than 100 MPa. Below 100 MPa, the relationship between stress and strain appears to be linear (see Fig. 2b). The elastic modulus values reported herein, and henceforth referred to as E_{100} - are the slopes of the stress-strain curves at a stress of 100 MPa (see below). In case of the 20 and 5 vol.% reinforcement composites and pure AZ61, the elastic moduli were measured below 50 MPa, since at 100 MPa, a hysteresis loop was observed. The E_{100} values are listed in Table 1. According to the rule of mixture, using the same reinforcement type, and vol.% in a given matrix should result in elastic moduli that are almost identical. In this work, it follows that all composites with a given reinforcement type should all have the same elastic moduli – assuming the alloys have the same moduli as pure Mg, which is not a bad assumption. Experimentally, however, the slopes of the initial linear segments of the stress-strain curves for the different Mg matrices are different. Said otherwise they cannot represent the true Young modulus values. To avoid misinterpretation of our results, we refer to slope of the linear part of the stress-strain curves - up to 100 MPa - as E_{100} . Why E_{100} varies as much as it does is unclear at this time. Possible reasons include plastic deformation of the soft Mg matrices at stresses lower than 100 MPa, that do not lead to stress-strain loops and/or the loops are so small as to be below the detectability limit of our extensometer. Other possibilities include misalignment of our machine or other unknown experimental

problems.

Figure 2b plots the stress-strain cycles, at various stresses, for a CG50-Ti₂AlC-AZ31 composite sample. The loops are shifted horizontally for clarity. Defining an elastic modulus for loops such as those shown in Fig. 3b is somewhat problematic. However, one can define an effective elastic modulus, E_f , obtained by least squares fits of the entire data set. This procedure results in diagonal dashed lines bisecting the loops (Figs. 2b and 3). A schematic of how E_{100} and E_f were defined is shown in Fig. 3. Figure 2b clearly shows that E_f depends on the maximum applied stress for each loop and decreases with increasing stress. The average of E_f , $E_{f(av)}$, values are calculated by averaging all the E_f values over the entire applied stress range and shown in Table 1. From the design point of view, E_f is quite important since it is the effective modulus that would dictate, for example, how much a part would deflect at a given stress.

4.3. Damping

In all the composites fabricated in this study, above a threshold stress, σ_t , energy dissipation was observed during cyclic loading. Before proceeding much further, it is instructive to explain how the energy dissipation, and other related parameters, are measured. Referring to Fig. 3, the following procedure was followed. As a first step, the area of each stress-strain loop was measured. This area, W_d , is the energy dissipated per unit volume in each cycle. As explained earlier, E_{100} was defined as the slope of the initial linear part. The stress at which the loading curve deviated from linear is labeled the threshold stress, σ_t . Like all other such stress-strain loops [10], at the very beginning of unloading from the maximum load in each cycle, the slope of the stress-strain curve is

again equal to E_{100} . The strain between the two parallel lines with slopes E_{100} is the nonlinear strain, ϵ_{NL} (Fig. 3). When the latter is subtracted from the total strain, ϵ_{tot} , the linear elastic strain, ϵ_{LE} , can be calculated. As noted above, the least squares fits of the entire data set – shown as the diagonal dashed lines bisecting the loops (Fig. 3) – yields the effective modulus, E_f .

In order to find the best correlation between W_d and the maximum applied stress for each cycle W_d was plotted versus σ and power law fits assuming

$$W_d \propto \sigma^n$$

were used. The resulting fits, and their correlation coefficients, are shown in Figs. S1a and S1c and Fig. S2 in Supplementary Material. Based on these results, it is clear that for the most part n hovers around 3. To find the correlation between ϵ_{NL} and σ they were plotted and again a power law fit assuming

$$\epsilon_{NL} \propto \sigma^m$$

was used. The results are shown in Figs. S1b and d. In this case m hovers around 2.

The generally validity of these fits is best seen in Figs. 4a and b where W_d versus σ^3 and ϵ_{NL} versus σ^2 for all 50 vol.% Ti_2AlC composites, respectively, are plotted. In all cases the correlation coefficient of the least squares fits, R^2 , were > 0.98 . The solid lines correspond to the CG50- Ti_2AlC composite results; the dotted lines represent those of FG50- Ti_2AlC composites. For comparison's sake, the W_d and ϵ_{NL} values of monolithic AZ61 tested herein and two monolithic Ti_2AlC samples with fine, and coarse, grains reported earlier [16, 28] are also plotted in Figs. 4a and b.

Figures 4c and d plot the W_d vs. σ^3 and ϵ_{NL} vs. σ^2 results for both CG50-TiC and

FG50-TiC composites, shown with solid lines and dotted lines, respectively.

Figure 5 plots W_d versus σ^3 for the 5 and 20 vol.% reinforcement composites where the matrices are pure Mg. For the sake of comparison, the results of monolithic AZ61 and also pure Mg matrices reinforced with FG50-TiC or FG50-Ti₂AlC are shown as well.

At any given stress, the W_d values in the CG50-Ti₂AlC composites decrease as the matrix changes from pure Mg to AZ61 (red solid lines in Fig. 4a). The W_d values of AZ31 and AZ91 composites are almost identical and fall in between those of the Mg and AZ61 matrix composites (Fig. 4a). The same is true for the FG50-Ti₂AlC composites (Fig. 4a); the AZ61 matrix composites dissipate the least energy. In this series, however, the AZ91 matrix dissipates less energy than the AZ31 matrix, but is still higher than the AZ61 matrix composite. The same trend is observed for CG50-TiC, in which increasing the Al content of the matrix reduces W_d . However, in the case of FG50-TiC composites, the W_d values of all three Mg alloy matrices are almost at the same level and about half of those of the FG50-TiC samples in which the matrix is pure Mg (dotted lines in Fig. 4c).

4.4. 100 cycles of loading to a high stress

Figure 6a shows 100 cycles of compression loading to 500 MPa for CG50-Ti₂AlC-AZ61 and CG50-TiC-AZ61 and to 350 MPa for CG50-Ti₂AlC-Mg and CG50-TiC-Mg. Note that the stress-strain loops for the latter are shifted horizontally for clarity. The 2nd and the 100th cycles of each sample are superimposed over each other and compared in Figs. 6b and c.

5. Discussion

The most important achievement of this study is the fabrication of high strength Ti_2AlC -Mg alloy composites that can dissipate up to 25% of the applied mechanical energy at stresses of the order of ≥ 400 MPa (Fig. 4). As discussed below, this result is due to the capability of Ti_2AlC to dissipate energy at high stresses. In the TiC composites, despite their high strengths, damping was reduced compared to pure Mg and Ti_2AlC -Mg composites. It is important to note that the 50-Mg- Ti_2AlC composites reported herein dissipate almost twice as much energy as Mg-SiC [28] and Mg-TiC (this work) composites, with comparable mechanical and elastic properties.

Given the many variables explored herein, the effects of matrix composition, reinforcement type, reinforcement size, and, reinforcement volume fraction, on properties, in general, and damping, in particular, are discussed separately below.

5.1. *Effect of matrix composition on damping*

For all four composite series, changing the matrices from pure Mg to the Mg alloys, significantly reduced W_d . This is best seen by comparing the results - with the same color - in Figs. 4 (a) and (c). As discussed in detail in part I of this study, it was concluded that the addition of Al to the Mg matrices improved the matrix/reinforcement interfacial strengths. The results shown in Figs. 4a and c, confirm this conclusion since stronger interfaces should yield less damping, which in turn reduces W_d . The weak interface led to the highest damping for the pure Mg matrix composites. Furthermore, the

fact that changing the matrix from either AZ31 or AZ91 to AZ61 reduces W_d , suggests that the latter form the strongest interfaces.

5.2. Effect of reinforcement type on damping

Given that the FG50-Ti₂AlC and FG50-TiC composites have almost the same reinforcement particle sizes, the differences between their W_d values (compare dotted lines in Figs. 4a to those in Fig. 4c) can be ascribed to the fact that Ti₂AlC itself is a high damping solid [10, 16], while TiC is not. Before making that argument, however, the following other ideas for the differences have to be considered:

i) Differences in dislocation densities. In theory one possible way to increase the damping capacity of a material is to increase its dislocation density [1]. However, since the CTEs of Ti₂AlC and TiC are comparable it is difficult to argue that one reinforcement can result in significantly more dislocations than the other.

ii) Differences in matrix/reinforcement interfaces. In our previous work we argued that, the Mg/Ti₂AlC interfaces were stronger than their Mg/TiC counterparts. If that were the case, then the former composites would have been expected to dissipate less energy than the latter, which is not the case when the W_d values in Fig. 4a are compared with those in Fig. 4b. It follows that, here again, interface sliding cannot be responsible for the differences.

The most likely explanation for the damping differences has thus to be the inherent damping capacity of Ti₂AlC. As discussed previously, Ti₂AlC dissipates energy during cyclic loading [29, 30]. However, for Ti₂AlC to dissipate energy a threshold stress, σ_t , that depends on grain size, among other factors, has to be exceeded. For example, Zhou et al. reported σ_t for bulk Ti₂AlC, with average grain size thickness, $t_{(av)}$,

of $14 \pm 7 \text{ } \mu\text{m}$, to be 180 MPa [16]. Amini et al. reported a σ_t of 226 MPa for fine-grained ($t_{(av)} = 5 \pm 3 \text{ } \mu\text{m}$) polycrystalline samples [28].

Based on these considerations, it follows that below σ_t of Ti_2AlC , only the Mg-matrices should be contributing to W_d . Said otherwise, the differences in W_d values between the two families of composites should be more evident at higher applied stresses, as observed (Fig. 4). The W_d values of the Ti_2AlC and TiC composites are significantly different only at stresses $> 200 \text{ MPa}$. For example, at 550 MPa, W_d of the FG50- Ti_2AlC -AZ61 composite (0.27 MJ/m^3), is about 55% higher than that of its FG50-TiC counterpart. At stresses below 200 MPa, the W_d values in the both families of composites are comparable (Fig. 5). As discussed below, this effect is better observed when the reinforcement volume fraction, v_r , is low.

Another possible reason is that, at 440 GPa, the Young's modulus of TiC is $\approx 37 \%$ higher than that of Ti_2AlC . When the interfaces are strong, stiffer reinforcement, bear more of the stress, which reduces the stresses on the Mg matrices, that in turn, can reduce W_d .

5.3. Effect of reinforcement particle size on damping

In general, the presence of finer reinforcing particles can affect W_d in three major ways.

i) Effect of Ti_2AlC particle size: The W_d values for both FG50- Ti_2AlC and FG50-TiC composites (dotted lines in Figs. 4a and c) are lower than those of their corresponding coarse grained (CG50) counterparts (compare solid to dotted lines in Figs. 4a and c).

Like Mg, damping in Ti_2AlC is strongly grain size dependent. For example, Zhou

et al. reported W_d values for monolithic Ti_2AlC with $t_{(av)}$ (average grain thickness) of $14\pm 7\mu m$ at 336 MPa to be $0.085 MJ/m^3$ [16, 29]. However, at $0.017 MJ/m^3$, the W_d of a fine-grained Ti_2AlC ($t_{(av)} = 5\pm 3\mu m$), at the same stress level, was reported to be 80 % lower [28]. This grain size dependence can in turn explain the smaller W_d values for the FG50- Ti_2AlC composites compared to their CG50- Ti_2AlC counterparts.

ii) Effect of matrix/reinforcement interfacial area. If interfacial slip were a significant contributor to damping, then using finer reinforcing particles should result in an *increase* in W_d . The fact that the opposite is true is thus quite significant because it reveals that the damping sources are more within the grains than at their interfaces or grain boundaries. Said otherwise the contribution of interfacial friction to W_d , must be smaller than the friction due to dislocation motion within the grains.

iii) Matrix grain size: Like the Ti_2AlC reinforcing particles, reducing the Mg grain size, should reduce matrix damping. Based on this statement, one would expect the highest damping to occur in the CG50-TiC composites, in which the Mg grain size, reported in part I of this study, was the largest possible among all the 50 vol.% composites [17]. However, the W_d values of the latter are comparable to the FG- Ti_2AlC composites, wherein, at $0.5 \mu m$, the average Mg grain size is significantly finer (compare results for CG50-TiC in Fig. 4c to FG- Ti_2AlC composites in Fig. 4a). To understand why the W_d values for the FG- Ti_2AlC composites are higher than the CG50-TiC composites, one has to again invoke the idea that the Ti_2AlC particles themselves are contributing to the damping.

5.4. Effect of reinforcement volume fraction

Figure 5 plots the W_d values for the TiC and Ti_2AlC composites as a function of v_r and matrix compositions. From these results, it is clear that: i) the W_d values for $v_r = 5$ % are higher than those for $v_r = 20$ % and significantly higher than those with $v_r = 50$ %; ii) Up to ≈ 200 MPa, and for all samples, the type of reinforcement has little effect on W_d (Fig. 5). From these observations it is reasonable to conclude that at stresses of 200 MPa or lower, only the matrices are contributing to W_d . Given that the σ_t values for polycrystalline Ti_2AlC samples - whose grains were larger than the reinforcement particles used herein - were > 200 MPa, this conclusion is not too surprising [16, 28].

We note in passing, that had the un-reinforced AZ61 matrix not yielded at ≈ 220 MPa, it would have possessed the highest W_d values. Some may argue that one should simply use un-reinforced Mg for damping, since clearly the reinforcements reduce W_d . And while such an argument would be perfectly valid if a part were only subjected to low stresses, it does not hold at higher stresses.

It has been suggested that the rule of mixtures can be used to estimate the damping capacity of composites, from a knowledge of the damping capacities and respective volume fractions of their individual components [4]. Such an approach, however, cannot be used here because of the strong grain size dependency of W_d in Mg and the fact that we do not know the exact grain size of Mg in the matrices. The effect of grain size is best seen in Fig. 5, where it is obvious that W_d of the pure Mg composite, reinforced with 5 vol.% Ti_2AlC particles, is considerably lower than that of the un-reinforced AZ61. Following the rule of mixture, the addition of 5 vol.% reinforcement should not reduce W_d by 40% as compared to pure AZ61. This can be only explained by the presence of smaller Mg grains in the composite. Furthermore, in Fig. 4a, the W_d

values of AZ61 and Ti_2AlC compositions are shown, which if the rule of mixture were applied would clearly overestimate the W_d values for the Ti_2AlC -AZ61 composite.

Based on all these considerations one can conclude that at stresses > 200 MPa, at least some of the energy dissipated in those composites is due to energy dissipation in the Ti_2AlC reinforcing particles and that the Mg matrices grain sizes of the Mg matrix also have an important effect on W_d .

5.5. 100 cycles of loading to a high stress

Kontsos et al. reported on the fatigue behavior of Ti_2AlC -Mg composite in tension [26]. To further investigate the sources of energy dissipation and to test the interfacial strengths, select composite samples were cyclically loaded 100 times in compression. The results for CG50- Ti_2AlC -Mg and CG50- Ti_2AlC -AZ61 and CG50-TiC-Mg and CG50-TiC-AZ61 are compared in Fig. 6. With the notable exception of the CG50-Mg-TiC composite samples (both particle sizes) (Fig. 6a), for all other composites, the 100 stress-strain loops appeared to overlay each other with high fidelity (Fig. 6a). As shown in Figs. 6b and c, not only was there no degradation in properties, but *cyclic hardening* was observed in the following composites: CG50- Ti_2AlC -Mg, CG50- Ti_2AlC -AZ61 and CG50-TiC-AZ61. Such hardening categorically rules out microcracking as the main source of energy dissipation. These results provide further compelling evidence that the interfaces in these composites are relatively strong. It is worth noting in this context that the stresses to which most of these composites were loaded were of the order of ≈ 70 % of their UCS. In other words, the cycling stresses were relatively high.

In part I of this study [17], we argued that because – in the case of the CG50-Mg-TiC composites – neither matrix nor reinforcement contained Al, the interfaces were

relatively weak. This conclusion is indirectly confirmed by the response of this composite to cyclic loading. In contrast to all Al-containing compositions (Mg alloy matrices), upon cycling, a slow increase in strain with each cycle was observed (see loops labeled TiC-Mg in Fig. 6a). Similarly, the FG50-TiC-Mg composite, despite its higher strength and elastic modulus, also deformed continuously during the same 100 cycles (not shown). Together with the slow deformation, cyclic softening (Fig. 6b), was observed indicating that the interfaces in the Mg-TiC composite, in sharp contrast to all others, are susceptible to fatigue, presumably due the presence of weak interfaces.

Lastly in this section we note that bulk monolithic MAX phases do not show much cyclic work hardening, at least up to 100 cycles [15, 29]. However, cyclic hardening in Mg and its alloys is well known [31, 32]. As a result, it is reasonable to ascribe the cyclic hardening observed in the composites (Figs. 6b and c) to that of the Mg matrices.

Finally, it is important to discuss the results obtained here from a design aspect, which depends on the application and stress levels. If high damping capacity is desired, and the design stresses are low, then pure Mg matrices can be used. For high stresses, AZ61 matrices should be used. Applying Ti_2AlC as the reinforcement increase the damping capacity, regardless of the matrix.

5.6. Correlations between parameters

In order to understand the effect of dislocation damping on W_d and ϵ_{NL} , one needs to review basic dislocation theory. If one assumes, ϵ_{NL} in a polycrystalline material is due to reversible dislocation movement ($\bar{x}$) then [33]:

$$\varepsilon_{NL} = \frac{M}{2(1+\nu)} b \bar{x} \rho_m \quad (1)$$

where M , ν , b and ρ_m are the Taylor factor, Poisson's ratio, Burgers vector and dislocation density, respectively. $\bar{x}$ is the average dislocation movement in response to the applied stress. Equation 1 can also be written as:

$$\varepsilon_{NL} = \frac{M}{2(1+\nu)} b \bar{A} n \quad (2)$$

where n and $\bar{A}$ are the total number of dislocations per unit volume and the average area reversibly swept by a dislocation, respectively.

Similarly, if one assumes W_d to be due to dislocation motion then:

$$W_d = 2n\bar{A}\Omega \quad (3)$$

where Ω is the energy dissipated by a dislocation line sweeping a unit area. The factor of 2 accounts for the fact that energy is dissipated during both loading and unloading. Combining Eqs. 1, 2 and 3 leads to

$$W_d = \frac{2}{k} \frac{\Omega}{b} \varepsilon_{NL} \quad (4)$$

where $k = M/2(1+\nu)$. This relationship is quite powerful because it can be used to shed light on the energy needed to extend the dislocations responsible for ε_{NL} . Note that all information relating to the dislocations and their densities, etc., is embedded in ε_{NL} . Clearly, if Ω is a constant, then plots of W_d vs. ε_{NL} should yield straight lines, with a slope of $2\Omega/kb$, and by extension, $\frac{\partial W_d}{\partial \varepsilon_{NL}}$ would be a constant.

Based on Eq. 4, $\frac{\partial W_d}{\partial \varepsilon_{NL}}$ is proportional to Ω . To estimate, the former, W_d vs. ε_{NL} results were plotted (not shown) and fit by second order polynomials. The latter were

then differentiated and the results - viz. $\frac{\partial W_d}{\partial \varepsilon_{NL}}$ - were, in turn, plotted vs. σ . Figure 7a plots $\frac{\partial W_d}{\partial \varepsilon_{NL}}$ vs. σ , for monolithic Ti_2AlC , Ti_3AlC_2 and Mg (shown as inset) with different grain sizes reported in previous studies [8, 16, 28]. The same analysis was carried out on the 50 vol.% composites and pure AZ61 samples tested herein; the results are plotted in Fig. 7b. From these plots, it is clear that Ω is stress dependent. The results shown in Fig. 7a clearly show that for Ti_2AlC , Ti_3AlC_2 and pure Mg, Ω increases linearly with σ . In other words, it takes more energy to extend the dislocations segments presumed to result in the nonlinear response observed here with increasing σ . The same is true for monolithic AZ61 tested in this study – plotted in the bottom left corner in Fig. 7b.

In Fig. 7a, the rate of increase in Ω with σ was constant. When $\frac{\partial W_d}{\partial \varepsilon_{NL}}$ vs. σ for the composites tested herein are plotted (Fig. 7b), however, that is no longer the case; not only is Ω not constant, but its rate of increase, increases with increasing σ . This is an important result because it shows that, at a fundamental level, a synergetic effect for energy dissipation is only activated in the composites as compared to their monolithic counterparts. The exact reasons for this state of affairs, is unclear at this time. The activation of multiple damping sources is one possible explanation. Another is, as discussed below, it is most likely inherent to nature of the bowing out of dislocations, through obstacles or other dislocations. Said otherwise, dislocation interactions with other dislocations or defects. The interaction can pin the dislocation, and higher applied resolved shear stresses would then be needed to overcome the obstacle strengths. These comments notwithstanding, these important observations need to be studied in more detail to better understand them.

It is important to note that some of the observations made herein, are not entirely new. Roberts and Hartmann [34], loaded Mg single crystals with their basal planes near 45° to the direction of applied stress and observed – after a pre-strain of 0.66 – fully and spontaneously reversible stress-strain loops such as the ones shown here. In the same work, it was assumed, without much evidence, that the damping was associated with the bowing out of long lengths of dislocations strongly pinned at either dislocations nodes or regions of strong dislocation-dislocation interactions. We calculated their W_d values as a function of stress and strain and plotted the results in Fig. 8. From these results it is clear that:

- i) When the non-linear shear strain, γ_{NL} is plotted against the applied shear stress, τ , squared (Fig. 8a), a straight line passing through the origin – with a correlation coefficient > 0.998 – is obtained.
- ii) The relationship between W_d and γ_{NL} (Fig. 8b) is initially linear, but then becomes non-linear. These results – like ours – imply that the energy needed to extend, or bow, a dislocation is more than a linear function of stress.

It is important to point out that in this case the Mg was quite pure and the tests were carried out on single crystals in an orientation of easy slip and thus neither grain boundaries nor twins can be implicated. Based on these results it is reasonable to assume that the energy needed to bow out dislocations in the presence of other dislocations is indeed a function of applied stress, as concluded here. This is an important result that is maybe not well appreciated in the literature.

6. Summary and Conclusions

In general, adding reinforcement particles to Mg, results in composites with enhanced elastic and mechanical properties but, as shown here, at the expense of their damping characteristics. This was demonstrated herein by the addition of TiC particles to Mg. However, when Ti_2AlC , an inherently energy dissipative solid, is used as a reinforcement it is possible to improve both the mechanical properties and achieve high damping properties. Based on the results of this work it is reasonable to conclude that:

- a) The damping capacity of Mg matrix composites depends on matrix and reinforcement composition, size and volume fraction. In general, reducing either the matrix or reinforcement grain size reduces damping.
- b) When strong interfaces are formed, the damping is reduced. Weak interfaces, however, render the composites more prone to fatigue and lead to cyclic softening.
- c) The most likely mechanism for energy dissipation is the bowing out of dislocations that are strongly pinned. Inherent to this mechanism is the fact that as the applied stresses are increased, the energy dissipated also increases.
- d) Low stiffness solids, such as pure Mg and its alloys, dissipate significant amounts of energy, but only at lower stresses.
- e) Composites reinforced with Ti_2AlC are damping at higher stresses. However, since below 200 MPa, Ti_2AlC does not dissipate much energy, the difference in between it and the TiC-reinforced composites only manifest themselves at stresses > 200 MPa.

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Table 1. Elastic moduli measured from the stress-strain curves at a stress of 100 MPa (E_{100}), average effective elastic modulus ($E_{ff(av)}$), 0.2% yield strengths (YS), ultimate compression strengths (UCS) and Vickers hardness, V_H , values all the composites studied here. E_{100} is the slope of the stress-strain curves up to 100 MPa. Numbers in parentheses are the uncertainties on the last digit.

Material	E_{100} (GPa)	$E_{ff(av)}$ (GPa)	0.2 % YS (MPa)	UCS (MPa)	V_H (GPa)
AZ61‡	45(1)	37(7)	59(4)	220(20)	0.5(1)
CG50-Ti ₂ AlC-Mg	73(2)	67(4)	285(4)	600(10)	1.5(1)
CG50-Ti ₂ AlC-AZ31	106(4)	89(11)	285(4)	662(8)	1.7(1)
CG50-Ti ₂ AlC(R)-AZ61	112(3)	100(8)	331(6)	719(7)	2.0(1)
CG50-Ti ₂ AlC(N)-AZ61	120(3)	107(8)	416(4)	730(15)	2.1(1)
CG50-Ti ₂ AlC(P)-AZ61	123(2)	112(8)	545(3)	755(10)	2.1(1)
CG50-Ti ₃ AlC ₂ -Mg	71(2)	66(5)	254	606(14)	1.5(1)
FG50-Ti ₂ AlC-Mg	109(4)	95(3)	349(3)	688(18)	1.7(1)
FG50-Ti ₂ AlC-AZ31	124(4)	104(4)	346(4)	773(7)	1.8(1)
FG50-Ti ₂ AlC-AZ61	136(6)	114(4)	426(5)	760(9)	2.1(1)
FG50-Ti ₂ AlC-AZ91	123(5)	106(4)	408(4)	768(11)	2.2(1)
CG50-TiC-Mg	123(6)	108(10)	258(4)	500(10)	2.6(2)
CG50-TiC-AZ31	140(6)	122(9)	311(5)	560(14)	2.8(3)
CG50-TiC-AZ61	163(8)	134(13)	382(5)	740(6)	3.1(3)
FG50-TiC-Mg	143(5)	131(4)	421(10)	705(10)	2.9(3)
FG50-TiC-AZ31	169(7)	149(4)	479(14)	963(7)	3.1(3)
FG50-TiC-AZ61	174(5)	157(4)	523(8)	1028(5)	3.4(3)
FG50-TiC-AZ91	184(5)	162(5)	557(8)	1013(12)	3.4(2)
20-Ti ₂ AlC-Mg‡	55(3)	51(2)	122(1)	336(7)	0.9(1)
5-Ti ₂ AlC-Mg‡	54(2)	49(2)	128(2)	339(21)	0.9(1)
20-TiC-Mg‡	57(3)	52(2)	140(2)	285(5)	0.9(1)
5-TiC-Mg‡	42(2)	38(2)	123(1)	337(16)	0.88(2)

‡ Measured at 50 MPa.

Figure Captions

Fig. 1. Backscatter SEM micrographs of polished surfaces of: (a) 20-Ti₂AlC-Mg, (b) 5-Ti₂AlC-Mg, (c) 20-TiC-Mg, (d) 5-TiC-Mg composites.

Fig. 2. (a) Complete cyclic compressive stress–strain curve for FG50-Ti₂AlC-AZ61, CG50-Ti₂AlC-AZ31, 5-TiC-Mg and as-cast AZ61 samples. First cycle of compressive stress–strain curves for 5- and 20-Ti₂AlC-Mg and 20-TiC-Mg are also shown, (b) stress–strain cycles at various stresses for the CG50-Ti₂AlC-AZ31 composite shown in Fig. 2a. The curves are shifted horizontally for clarity. E_f (dashed lines inside the loops) are computed by least squares fits of each loop.

Fig. 3. Typical stress-strain curve for a KNE solid and definitions of non-linear strain, ϵ_{NL} , total strain, ϵ_{tot} and energy dissipated per cycle, W_d .

Fig. 4. (a) W_d vs. σ^3 and (b) ϵ_{NL} vs σ^2 , for all the CG50 and FG50-Ti₂AlC composites. (c) W_d vs. σ^3 and (d) ϵ_{NL} vs σ^2 , for all the CG50 and FG50-TiC composites.

Fig. 5. W_d vs. σ^3 for 20 and 5 vol.% composites and 50 vol.% composites with AZ61 matrices and monolithic AZ61.

Fig. 6. (a) Stress-strain curves for 100 cycles of compression, at a constant stress, on CG50-Ti₂AlC-Mg, CG50-Ti₂AlC-AZ61, CG50-TiC-Mg and CG50-TiC-AZ61. All the 100 cycles for each test are shown. CG50-TiC-Mg curves are shifted horizontally for clarity. The 2nd and 100th cycles of each test are shown for: (b) CG50-Ti₂AlC-Mg and CG50-TiC-Mg, (c) CG50-Ti₂AlC-AZ61 and CG50-TiC-AZ61 composites.

Fig. 7. $\frac{\partial W_d}{\partial \epsilon_{NL}}$ vs. σ plots for (a) Ti₃AlC₂, CG-Ti₂AlC [16] and FG-Ti₂AlC [28]. The inset shows pure Mg with different grain size [8]. The numbers in parantheses respresent Mg grain size; (b) for all the 50 vol.% reinforcement composites and monolithic AZ61.

Fig. 8. a) γ_{NL} vs. τ^2 , and, b) W_d vs. γ_{NL} for Mg single crystal. All results extracted from Fig. 3 in Ref. [34].

Fig. 1.

Fig. 2

Fig. 3

Fig. 4

Fig. 5

Fig. 6

Fig. 7

Fig. 8