Nilsén, Frans; Aaltio, Ilkka; Hannula, Simo Pekka

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Comparison of magnetic field controlled damping properties of single crystal Ni-Mn-Ga and Ni-Mn-Ga polymer hybrid composite structures

Frans Nilsén[1]*, Ilkka Aaltio[1,2], Simo-Pekka Hannula[1]

[1] Aalto University, Department of Chemistry and Materials Science, Kemistintie 1, 02150 Espoo
* Corresponding author E-mail address: frans.nilsen@aalto.fi
[2] Osaka University, Graduate School of Engineering, 2-1 Yamadaoka, Suita, Osaka 565-0871, Japan

Abstract

Magnetically controlled hybrid Ni-Mn-Ga composites are potential candidates for actuation and damping applications. The combination of ductile polymer and gas atomized large grained Ni-Mn-Ga powder has many advantages compared to bulk single crystals. These advantages include ease of manufacturing and freedom of shape, while still being magnetically controllable. In this report, Ni-Mn-Ga-epoxy hybrid composite structures are manufactured at three different filling ratios 25, 30 and 35 vol-% and damping properties of the composites are compared to those of 5M Ni-Mn-Ga single crystal. The damping properties are characterized using a laboratory made high-frequency dynamic mechanical testing instrument and a dynamic mechanic analyzer (DMA) in single cantilever mode. The mechanical cycling experiments revealed that the damping ability of the Ni-Mn-Ga composites depends on the filling ratio. The magnetic field induced stiffening observed in the mechanical cycling experiments of the single crystal sample at 100 Hz correlated roughly with that of the composite sample having filling ratio of 35 vol-%.

Keywords: A. Smart materials; A. Functional composites; B. Magnetic properties; E. Casting; Damping

1. Introduction

Ni-Mn-Ga Heusler single crystals displayed high and reversible magnetic-field-induced strains (MFIS) up to 6-11%, depending on the composition and martensitic structure of the alloy [1,2]. This makes the material suitable for magnetically controlled actuators, which can be used in damping applications [3]. So far, the costs and the difficulties related to manufacturing of Ni-Mn-Ga single crystals and the brittle nature of the material [4] have hindered its widespread use. As a viable alternative to bulk single crystals, magnetic shape memory (MSM)-hybrid composite structures have been proposed [5-16].

Previously, such Ni-Mn-Ga composite structures have been produced from particulates manufactured either by crushing [7-14] or cutting [15] of single crystals or from specialized processes such as spark erosion [9] or melt-spinning [16]. However, these do not solve the complexity related to single crystal manufacturing. Instead, large grained gas atomized Ni-Mn-Ga powder may be used as MSM-elements in the composite as suggested in the work utilizing the PECS processed porous MSM-hybrids [17]. It should be then possible to combine large grained gas atomized Ni-Mn-Ga powder with a ductile polymer for damping elements. Gas atomization is a high-volume manufacturing method for metallic powders, which produces round and smooth particles, with a narrow particle size distribution [18]. Previous research has shown that Ni-Mn-Ga composites containing round particles have higher damping capabilities, as the round particles do not have a shape effect in the magnetic anisotropy [9]. The properties of gas atomized Ni-Mn-Ga powders have been studied previously [19] and the powder has been used to manufacture hybrid Ni-Mn-Ga composites [20].

The main advantage of the hybrid composite structures is the easiness of manufacturing compared to the single crystals as the composites can be freely cast into different shapes and these shapes can then be joined together to create the needed shape for the end product. Such damping elements can be designed to match the needs of different applications. In this paper, the damping properties of 5M Ni-Mn-Ga single crystal are compared to the properties of Ni-Mn-Ga composites applying a laboratory built high-frequency dynamic mechanical analysis instrument (HF-DMA) [21] and a DMA in single cantilever mode [22].

2. Material and methods

The induction melted ingot having composition of $Ni_{49.3}Mn_{29.6}Ga_{21.1}$ was gas atomized at 1308°C using argon gas at a pressure of 50 bars. The powder was sieved to separate particles having diameter between 20 and 45 μm, with an average grain size of 28.2 μm. Because the fast cooling during the gas atomization process leads to chemically inhomogeneous fine grain structure, the powder needs to be annealed to induce grain growth and to homogenize the powder before composite manufacturing. The sieved powder was therefore heat-treated in alumina crucible by mixing it with NaCl (Ensure® from VWR) with the ratio of 10/90 vol-% respectively in argon atmosphere at 770°C for 28 hours. This salt was selected as it does not react with Ni-Mn-Ga, minimizes manganese loss during annealing and keeps the gas atomized powder from sintering during annealing.

After the heat-treatments, the salt was removed by water immersion and the powder surface was cleaned using 10 wt-% citric acid solution. The cleaned powder was magneto-mechanically trained by repeated compression of 10 MPa after orienting the particles perpendicularly to the compression direction with applied magnetic field of 1 T. The phase transformation and magnetic behavior was subsequently studied by using a laboratory built low-field *ac* magnetic susceptibility measuring system at heating and cooling speed of 5°C/min. The phase composition was studied with X-Ray Diffractometer (PANalytical X'Pert Pro XRD) using Cu-$K\alpha$ and the elemental composition with an energy dispersive analyzer (NSS EDX) attached to a scanning electron microscopy (Mira Tescan SEM 3 FEG-SEM).

The epoxy for the composites was chosen based on its mechanical properties, which need to match the stress to induce twin movement in Ni-Mn-Ga powder particles, as well as on its suitability for composite manufacturing. Polymers with yield strength between 1 and 10 MPa and Young's moduli of 1 to 1000 MPa were considered as potential matrix materials. The T_g of the epoxy was measured using TA Instruments Q2000 DSC in the temperature range of -80 to 180°C. The composites were manufactured by mixing 25, 30 and 35 vol-% of pretreated Ni-Mn-Ga powder with Loctite Hysol 9455. The mixture was then degassed using a vacuum chamber and cast into polyoxymethylene molds with dimensions of 5 x 6 x 25 mm. The powder particles were magnetically oriented to form chains along the long axis of the composite sample. The composite was then left to cure at room temperature for three days. Additionally, a reference hybrid composite was manufactured using gas atomized Ralloy® WR4 steel powder and the Hysol 9455 epoxy with a filling ration of 35 vol%, using the same composite manufacturing process and magnetic orientation. The single crystal stick used in the experiments was manufactured by Adaptamat Ltd. It was cut along the <100>$_p$ planes and its average chemical composition was $Ni_{50}Mn_{28.3}Ga_{21.7}$. The dimensions of the single crystal after compression along its longest axis in martensitic state were 1 x 2.5 x 20.1 mm.

The damping properties of the composites were studied using two different methods. First, the damping properties were studied with a TA Q800 DMA in the single cantilever mode at frequencies of 1, 5, 10, 50, 100 Hz at a peak-to-peak stress of 1 MPa with an average sample size of 3 mm x 5.3 mm x 25 mm. Reference measurements were done at 1 MPa with the steel hybrid composite and at 2 MPa using the Hysol 9455 sample without any added particles. To study the effect of the magnetic field in this DMA, a permanent magnetic circuit with a yoke made of $Fe_{54.1}Co_{45.9}$ alloy was constructed. The circuit produced an applied magnetic field of 0.3 T and could be attached around the sample so that the field was perpendicular to the direction of particle chains in the composite sample and the direction of the bending movement of the single cantilever beam.

Secondly, a laboratory built high frequency mechanical testing instrument (HF-DMA) was applied to measure the change in the composite strain with and without a magnetic field at frequencies of 21, 41, 61, 81 and 101 Hz with ±1 MPa in tension-compression mode. The original design of the laboratory built testing instrument is described in [21]. For the present experiments, it was redesigned such that a linear guide was added to the bottom grip to increase its range of movement to facilitate testing of larger samples. Additionally, the grips were re-aligned and a movable magnetic circuit using permanent magnets producing a 0.5 T perpendicular field to the sample was added to the frame. As previous energy harvesting experiments have showed [23], an applied field less than or equal to 0.6 T should be optimal for Ni-Mn-Ga twin movement. The size of the DMA sample holder and the air gap limited the design of the magnetic circuit, thus only 0.3 T applied magnetic field could be achieved even after adding an iron-cobalt alloy yoke. As there was more space for the magnets in the laboratory built HF-DMA a larger magnetic circuit was possible to place into it. The larger field can produce a more pronounced effect in the composites.

For the HF-DMA experiments, thin pieces with dimensions of 0.6 mm x 2.5 mm x 15 mm were cut from the composite samples using a slow speed diamond saw and a surgical knife. The sample was attached between the two grips in the HF-DMA testing instrument (Fig. 1). The lower grip was forced to vibrate up and down using a speaker element driven with a sinusoidal signal from a TT*i* TG550 function generator. The signal was amplified using a QSC RMX 2450 audio amplifier to reach the desired stress level. Testing was load controlled and the sample displacement was measured from the lower grip using laser displacement sensor, while the applied force was measured with a piezoelectric load cell at the upper grip. The 0.5 T applied field could be turned on and off during the cycling by moving the magnetic circuit.

The displacement, force and drive signals were recorded using Agilent technologies DSO-X 2024A oscilloscope. For the dynamic measurements carried out with DMA and HF-DMA at room temperature 20°C, the measurement time was kept short to minimize heating of the composite sample due to continuous oscillation in the magnetic field. This is important as the dynamic modulus and tan δ of the 5M Ni-Mn-Ga alloy is temperature dependent close to the martensite to austenite phase transformation temperature [22]. For the DMA measurements there were two samples with 20 vol%, one sample with 25 vol% and three samples of the 35 vol% composition. Finally, the damping properties of the single crystal was studied with the HF-DMA at frequencies of 21, 41, 61, 81, 102, 202 and 410 Hz and peak-to-peak stress of 2 MPa with and without the magnetic field. The single crystal sample was prepared by first measuring the orientation from the grown crystal using X'pert MRD and by then cutting the sample using EDM wire saw. Then the cut sample was wet ground using SiC-paper in the austenitic state using hot water and finally electropolished at -30°C in 10 wt.% nitric acid and methanol solution. The resulting sample had thus sharp edges and smooth surfaces.

Only one single crystal sample was measured with the HF-DMA, but the measurement was repeated multiple times

4. Results and Discussion

The X-ray measurements (Fig. 2a) showed that the structure of the powder at room temperature had changed from cubic (before annealing) to 5M martensite. As previously reported [17,19] the heat-treatment homogenized the powder and reduced stresses created during the gas atomization process. Additionally, the heat-treatment increased the average grain size in the powder from 1.7±0.2 μm to 17.9 ±8.5 μm. The EDX results, measured from cross-sections of powder particles, showed that during the annealing process the manganese content decreased only slightly from 29.6 to 29.3 at-%. Thus, the composition of the after annealed powder was $Ni_{49.3}Mn_{29.3}Ga_{21.4}$. The low field *ac* magnetic susceptibility measurement revealed a two-step reverse phase transformation that appeared during the heating of the powder (Fig. 2b). The phase transformation temperatures shown on the graph are the averages of start and finishing temperatures of the phase transformation.

The single cantilever experiments done on the commercial TA Q800 DMA showed that the composite filling ratio has a large effect on the damping properties and the magnetic field response (Fig. 3). A similar effect of filling ratio has been observed in previous Ni-Mn-Ga composite studies [9,13] and it is likely that a certain minimum filling ratio is needed in order the matrix to transmit the stress into the particles for the activation of the twin movement [9]. This has been confirmed in [10] where a composites with filling ratio between 30-40 vol.% displayed enhanced damping capability. However, at higher volume fractions the epoxy composites may become prone to cracking [11,12].

At the lowest filling ratio of 20 vol-% (Fig. 3a), the applied magnetic field had almost no effect on the tan δ of the composite and only minor difference could be seen at 100 Hz. However, when the filling ratio of the composite is increased (Figs. 3b and c) the magnetic field starts to have a larger effect. The highest filling ratio shows 108% higher tan δ with magnetic field on at 100 Hz compared to the value without applied magnetic field (Fig. 3f). The measured values of tan δ are comparable to those reported previously [8,4,11] for Ni-Mn-Ga -polyurethane and -epoxy composites, despite that the pure Hysol 9455 epoxy has a higher tan δ (Fig. 3d) than the Lord polyurethane [4] or the Bisphenol-a-epoxy [11]. When the current results are compared with melt-spun Ni-Mn-Ga composites [16], the measured tan δ is higher in the T_g to A_f (austenite finish) temperature range, however, at a higher temperature the tan δ of the melt spun composite is higher. This is expected as the melt spun ribbon has a bamboo-like grains structure and higher A_f temperature [16]. When the measured tan δ values are compared to the reference value of 0.10 for Ni-Mn-Ga 5M at 290 K [22] it can be seen that all the composites have higher tan δ, which is expected as the epoxy has higher tan δ than the Ni-Mn-Ga single crystal. However, when the measured tan δ values of the composites are compared to the reference sample Hysol 9455 (Fig. 3d), the influence the Ni-Mn-Ga particles in increasing the damping capability is clear. In the reference epoxy (Fig. 3d), the tan δ of the composite decreases rapidly with increasing frequency, while in the composite with the highest filling ratio (Fig. 3c), the tan δ increases with the magnetic field when the frequency is increased from 50 to 100 Hz. On the contrary, the steel hybrid reference (Fig. 3e) shows no noticeable effect of the applied field to the tan δ. The measured tan δ of the steel reference is also lower than that of Ni-Mn-G hybrid composite. Additionally, in the Ni-Mn-Ga hybrid composite the storage modulus E’ increases 1.3 times and the loss modulus 2.7 times by the applied magnetic field. This implies that the increased damping capability of the composite is likely due to the suppressed twin boundary movement in the filler particles, as the magnetic field tends to align the c-axis (here same as the magnetic easy axis) of the twins, thereby increasing the stiffness of the composite. It is also notable, that the results show that the Ni-Mn-Ga hybrid composites exhibit adequate damping in both axial and (transversal) bending loading conditions.

The dynamic measurements of the composite structures, were reproducible with the tan δ having average standard deviation of only 0.01 %. For the HF-DMA measurements there were two samples of each composition with the strain having average standard deviation of 0.1 %. Additionally, the results of the laboratory built HF-DMA experiments (Fig. 4) are consistent, even if not totally comparable, with the single cantilever DMA results as the stiffening effect increases consistently with the composite filling ratio (Fig. 4b-c). As in the single cantilever experiments, the largest change in strain appears at above 50 Hz and the highest magnetic field induced change is at f=100 Hz (Fig. 4c). In the two different dynamic measurement setups, the differences induced by the two magnetic circuits are likely not very large. As revealed by the magnetization measurements carried out with the heat-treated Ni-Mn-Ga powder, the magnetization of the heat-treated gas atomized powder reaches saturation magnetization approximately between 0.6 T and 0.8 T applied field [19]. The heat-treated Ni-Mn-Ga powder from the same batch was used in [19,20] and in the present Ni-Mn-Ga composites. The saturation magnetization of the gas atomized powder was 43 emu/g after heat-treatment, while at applied field of 0.3 T the magnetization was 32 emu/g. Thus using the permanent magnet with 0.3 T applied magnetic field, the achieved magnetostress is 25% smaller than the highest achievable magnetostress. As the magnetostress for twin reorientation is highest at the saturation magnetization [24].

The difference in the single cantilever and the laboratory built HF-DMA experiments, mainly with the 25 vol-% composite (Fig. 4a), can be attributed to three different factors. Firstly, the stress field inside the composite can be assumed to be more uniform in the uniaxial HF-DMA experiments compared to the single cantilever experiments. In the single cantilever experiments, the bending creates a gradually increasing a tensile stress towards the surface on one side, a zero-stress region at the neutral axis (center) and a gradually increasing compressive stress to the opposite side of the sample. While in the HF-DMA experiment, the macroscopic uniaxial stress is uniform - either tensile or compressive in the entire sample. In the experiments, the calculated peak stress at the surface of the single cantilever test for the MSM hybrid composites is on average 6.6 ±0.5 MPa while the peak uniaxial stress is 1 MPa. While for the reference steel hybrid composite, the peak stress at the surface during bending was only 2.5 ±0.02 MPa. As it is necessary to overcome the twinning stress to start the expected twin movement in large grains of the particles, the more pronounced stiffening effect of the field in the single cantilever experiments compared to the uniaxial test results can be due to the increased stress in bending. Secondly, as the Ni-Mn-Ga particle chains are aligned along the field lines throughout the relatively large composite samples, the inter-chain distance cannot be precisely controlled. Thus, the smaller sample size of the HF-DMA experiments leads to larger variation in the results. As such, it is possible that not all the particle chains in the smaller samples are working as intended, while in the larger single cantilever experiments a few non-optimal particle chains do not affect the results compared to the hundreds of chains in the larger samples, which were observed by microscopic examination. Additionally, the viscoelastic nature of the chosen epoxy affects the results. However, in spite of the influence of these differences, the effect of the composite filling ratio can be seen in both the single cantilever DMA and in the uniaxial HF-DMA results.

The T_g of the Hysol epoxy was 18.2°C. However as shown by Lahelin *et al.* [8], the T_g temperature can be increased in composites because Ni-Mn-Ga particles can affect crosslinking during the curing process. Similar effect has been seen in other Ni-Mn-Ga composite structures [9] with increasing particle fraction. As the vibration experiments, DMA and HF-DMA, were performed at room temperature (20˚C), the epoxy was either at the viscous state or right at the T_g temperature during testing. According to previous research of Ni-Mn-Ga composites [8-10,13,16] it can be synergistically beneficial if the T_g of the matrix and the martensite to austenite phase transformation temperature of Ni-Mn-Ga particles are close to each other and the DMA test temperature, as both materials have enhanced damping capability at those temperatures. Previously the heating up of Ni-Mn-Ga composites was studied and the observed heating was only 2 °C at 300 Hz long term cycling [21].

The potential overheating of the composite needs to be taken into account in the application design. However, when designing shock and vibration dampeners the working temperature needs to be taken into account usually as well, since increased temperatures can effect even fluid based dampeners [26]. A reasonable working temperature range for the composite to achieve the highest vibration damping potential is from the glass transition temperature of 18.2°C to the A_T temperature, which is the middle point for the austenite transformation, since the austenite transformation temperature is the highest temperature where the composites damping can be controlled with the magnetic field. A similar temperature range for the Ni-Mn-Ga composites could be seen in the work done by Lahelin *et al.* [8] where the polymer had increased damping capabilities between 20°C and the austenite transformation finishing temperature. Thus, the epoxy matrix defines the lowest working temperature while the Ni-Mn-Ga composition defines the highest working temperature. It is expected that the Ni-Mn-Ga particles could stay magnetically active as low as -108 °C [27] as that is the lowest working temperature reported for magnetic shape memory phenomenon for 10M structure.

The results of the dynamic mechanical testing of the single crystal Ni-Mn-Ga show that the cycling frequency has a profound effect on the strain during the load controlled mechanical cycling (Fig. 5). When the mechanical cycling frequency is increased, the strain of the single crystal without the magnetic field increases up to f=81 Hz, while the strain with the field on stays almost constant through the whole frequency range. When the cycling frequency is increased further, the strain without the magnetic field starts decreasing until it reaches the strain with the magnetic field at f=420 Hz. This means that at high mechanical cycling frequencies twin movement at moderate applied stress levels is not possible as the strain values with and without the field are the same. This frequency dependent change in the mechanical cycling properties of the material, as the dynamic mass increases at increasing cycling frequency, is quite significant. It is likely that as the cycling frequency is between 50 and 200 Hz the dynamic mass in the testing instrument is in the optimal range for twin boundary movement. When the frequency is increases beyond 200 Hz the dynamic mass in the testing instrument starts reducing the vibration energy as the vibration source in the laboratory built testing instrument is load controlled and thus is not able to increase with the dynamic mass. When the highest strain value without magnetic field at f=61 Hz is compared to the corresponding strain value at f=410 Hz the difference is over 62%. Additionally, this same dependency on the testing frequency can also be observed in the composite measurements (Figs. 3 and 4) as in all experiments on the composite the difference in strain increases with the frequency. Thus, there is an optimal mechanical cycling frequency range for the twin boundary movement in 5M Ni-Mn-Ga in the laboratory built HF-DMA instrument. If the change in strain due to magnetic field in the single crystal is compared to the change in strain in the 35 vol-% composite at 100 Hz, it can be observed that the change in strain is 3.7 times larger in the single crystal. This agrees roughly with the 35 vol-% filling ratio hybrid Ni-Mn-Ga composite, as approximately only 1/3 of the composite is Ni-Mn-Ga. The results for the single crystal measurements were reproducible with the strain, with magnetic field, having an average standard deviation of 0.1 %.

5. Conclusion

We have demonstrated that axially oriented epoxy composite structures made from gas atomized Ni-Mn-Ga powder and Hysol 9455 -epoxy have comparable stiffening effect in the externally applied magnetic field as single crystal Ni-Mn-Ga at forced cyclic tensile/compressive vibration. In the composites, the magnitude of the magnetic field induced stiffening is directly related to the filling ratio. The composites can be used in damping applications, as they are easy to manufacture and have adequate mechanical properties. The vibration damping in Ni-Mn-Ga composites can be controlled by applied magnetic field also during transverse loading, which opens new possibilities in damping application design as previously Ni-Mn-Ga actuators were mainly limited to axial loading. Additionally, the composites can be cast to different shapes, which make developing damping components easier. The mechanical cycling experiments of the single crystal Ni-Mn-Ga revealed that the magnetic field induced stiffening can be used to control vibration amplitude remarkably, in particular at the frequency range from 50 to 200 Hz.

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Figure captions

Figure 1. Laboratory built HF-DMA testing machine.

Figure 2. (a) X-ray spectra of the as atomized powder and annealed powder, (b) magnetic susceptibility measurement curve for treated powder showing phase transformation temperatures and two-step reverse transformation during the heating up cycle.

Figure 3. DMA measurement results (TA Q800). Storage modulus (E'), loss modulus (E"), strain (%) and tan δ with and without field of Ni-Mn-Ga hybrid composites: (a) filling ratio of 25 vol-%, (b) filling ratio of 30 vol-%, (c) filling ratio of 35 vol-%. (d) Reference epoxy sample, (e) steel powder filled reference hybrid composite and (f) magnetic field induced change (in percentage) in tan δ, and in storage and loss modulus of Ni-Mn-Ha hybrid composites at tested frequencies.

Figure 4. Results obtained by the laboratory built HF-DMA showing the magnetic field induced change in strain in the composite and the actual strain at ±1 MPa with trend lines, with and without the magnetic field for: a filling ratio of (a) 25 vol-% Ni-Mn-Ga, (b) 30 vol-% and (c) 35 vol-% Ni-Mn-Ga.

Figure 5. Magnetic field induced change in strain of the single crystal and strain with and without the applied magnetic field at peak-to-peak stress of 2 MPa with trend lines.