Ni-Ti SMA-Reinforced Al Composites

G.A. Porter, P.K. Liaw, T.N. Tiegs, and K.H. Wu

A shape-memory alloy, nickel-titanium, has been distributed throughout an aluminum matrix, using powder-metallurgy processing, in the hope of using the shape-memory effect to achieve strengthening and improve the fatigue resistance, as compared to the aluminum matrix. The shape-memory effect was activated by cold rolling the samples at –30°C. Upon reheating to the austenite phase, the Ni-Ti was expected to return to its original shape while embedded in the aluminum matrix. It is thought that this action created residual, internal stresses around each particle, which strengthened the material. The yield and ultimate strengths, and the fatigue lives of the Ni-Ti reinforced aluminum composites, have been improved considerably, as compared to the unreinforced material. The cross-sectional microstructures of the composites, as well as the modes of crack growth, have been examined with a scanning electron microscope (SEM) to identify fatigue and fracture mechanisms.

INTRODUCTION

A shape-memory alloy (SMA) has the unique property of being able to recover its original shape after deformation has occurred. Once regarded as a phenomenon, the unique shape-memory behavior is a proven result of a phase change in the material. This phase change from a softer martensite to a harder austenite is temperature-dependent, and its micromechanism has been theoretically explained in detail by authors such as Wayman[1] and Nishiyama.[2] For example, if a shape-memory alloy is cooled below a certain temperature labeled as the martensitic starting temperature (M_s), a phase change to martensite occurs. If there is deformation of the material while in the martensite phase, then the material will return to its original shape upon reheating to another specific temperature (usually higher than the M_s temperature) labeled as the austenitic starting temperature (A_s). Up to 6% to 10% deformation can be reversed through this process, thus providing enough shape recovery to allow many applications of the shape-memory properties. Table I gives a list of some of the more frequent uses of shape-memory alloys.

The purpose of this study was to create a nickel-titanium/aluminum metal-matrix-composite material that would have mechanical properties superior to those of the aluminum matrix. The goal was to fabricate this composite material using isostatic or hot-pressing, powder-metallurgy processing techniques. We desired to obtain a material with a satisfactory density (>97% of theoretical density), and, if possible, use the shape-memory effect of the Ni-Ti to introduce residual stresses into the matrix to improve the strength and fatigue resistance.

PARTICULATE STRENGTHENING MECHANISMS

This work represents the formulation of a new idea pertaining to particulate strengthening mechanisms. At the Oak Ridge National Laboratory, Tiegs[4] had the idea of dispersing the Ni-Ti shape-memory alloy, in the form of a powder, throughout an aluminum matrix in the hope of using the shape-memory effect to achieve strengthening in the aluminum matrix. Some research has been done in this field that has focused mainly on the use of SMA fibers.[5–7] Articles describing some previous work done in the manufacturing and testing of Al/Ni-Ti SMA metal-matrix composites have been reviewed, and some of the major findings have been summarized.[4-8] Only one reference[9] could be found on any work done using a SMA in the form of a powder in a matrix, and the research was limited to the basic manufacturing of a material with the shape-memory effect intact. Unfortunately, the authors did not conduct mechanical-property characterization tests beyond the observation of the dampening properties. Therefore, since the current work not only develops a functional SMA reinforced composite, but also examines the mechanical properties of such a composite, this article offers information previously unknown and unavailable.

The theoretical foundation for this strengthening mechanism is portrayed in Figure 1. The Ni-Ti shape-memory alloy, in the form of a powder, is dispersed throughout an aluminum matrix to create the composite material. The composite is chilled below the M_s temperature (around –20°C) to produce martensite in the Ni-Ti particles, and then cold rolled to produce a 10% deformation. Since the martensitic phase of the Ni-Ti has a lower yield strength and modulus than the aluminum matrix, these particles may also be deformed during cold rolling. Upon reheating above the A_s temperature, a phase transformation is generated and the shape-memory effect is exhibited by the Ni-Ti particles. The particles return to their original shapes (within 8% to 9% of deformation), while embedded within the aluminum matrix, which has a much lesser degree of strain. This action will create residual, internal stresses around each Ni-Ti particle; tensile stresses in the longitudinal and transverse directions; and compressive stresses in the through-thickness direction. This arrangement of stresses will increase the yield strength and strengthen the material in a similar fashion as thermal stresses strengthen a ceramic-particle reinforced metal-matrix composite upon cooling from the manufacturing temperature.

Besides the stresses produced from the Ni-Ti particles as a consequence of the shape-memory effect, there is an additional influence on these stress fields resulting from the differing thermal expansion coefficients of the composite material constituents. Since the coefficient for aluminum (23.6 μm/°C) is much greater than that of Ni-Ti (11.0 μm/°C), it can be expected that the Ni-Ti particles will undergo compression during the cooling of the composite from manufacture. This effect is enhanced when the M_s

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temperature is reached, as the martensitic phase of the Ni-Ti has a still lesser coefficient of thermal expansion (6.6 μm/°C) than the austenitic phase. Because of the presence of the compressive stress, it is expected that an additional amount of strengthening may be achieved from the combination of these properties.

Moreover, the shape change of the Ni-Ti particles resulting from the shape-memory effect and the residual stresses resulting from the differing thermal expansion coefficients both contribute to produce stress fields surrounding the particles. These stress fields are expected to disrupt the homogenous plastic flow of the material during the deformation of the composite and interfere with the propagation of a crack at its tip, causing deflection and branching. Thus, the yield and ultimate strengths, as well as the fatigue life of the composite, will be increased in comparison to the unreinforced matrix material.

The actual development of such a material has been lengthy and obstacle-ridden. The problem of the diffusion of aluminum into the SMA particle during hot pressing was extensive and most troublesome.[10] Much of the research involved the development of a diffusion-barrier between the Ni-Ti powders and aluminum matrix. This was finally accomplished by the enhancement of the interface with a thin oxidation layer. This procedure required special attention to prevent the oxygen from diffusing into the particles and upsetting the shape-memory effect. Some of the other major problems encountered in the production of the material included

- Poor Ni-Ti particle to aluminum-matrix bonds, which became sources of crack initiation.
- Insufficient product densities due to the inadequate compaction of powders in the die mold, which led to extensive fracturing during deformation.
- Diffusion, leading to reactions between the Ni-Ti and the aluminum matrix, resulting in the loss of the shape-memory effect and formation of brittle intermetallic compounds.

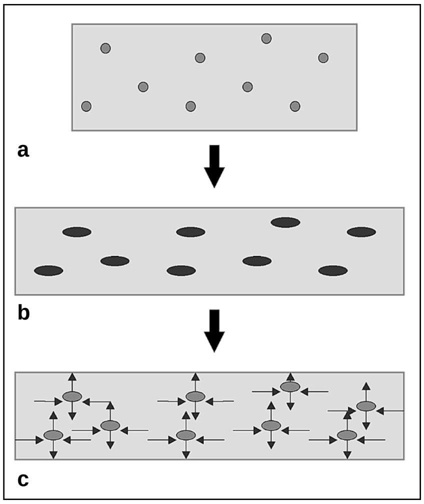

Figure 1. A schematic of the behavior of the shape-memory alloy particles and their possible effect on the matrix. (a) Dispersion of a shape-memory alloy in a metal matrix, (b) deformation below the transition temperature, and (c) raising the temperature, enacting the shape-memory effect, and inducing internal stresses.

EXPERIMENTAL PROCEDURES

Powder metallurgy (P/M) was chosen as the method of fabrication for several reasons. Primarily, a quality composite can be fabricated at temperatures well below the melting temperature of the matrix. This step allows the constituent materials' composition to be preserved, leaving the shape-memory effect intact. During manufacture, the use of pressure aids in the densification of the composite, in comparison to a casting or deposition process. The P/M processing also yields a material that contains relatively fine grains, which enhance the physical properties. The sizes of these grains are in proportion to the powder size. Thus, finer powders will produce a finer grained P/M product. Also, P/M processing is less laborious and more cost-effective than many other methods for producing such a composite of the same quality.

In the present work, aluminum possesses a major volume content of the composite (90 volume percent [vol.%]). It was chosen as a matrix material because of its extensive commercial use, low density, and thus, marketability. To increase the strength, most composites that have an aluminum matrix are not made with pure aluminum, but with aluminum alloys. However, after producing several specimens that were ruined because of extensive diffusion that created intermetallics, the author adopted Al 1090 (99.9 wt.% aluminum) as a matrix material to avoid complications arising from the reaction of Ni-Ti with aluminum-alloying elements, such as copper. Simplifying the composition of the entire composite to three elements eliminated the problem of identifying phases and constituent elements for the initial work.

The basic fabrication procedure was as follows. Before pressing, the authors subjected the Ni-Ti powders to a process known as mechanical milling.[11,12] This process reduced the particle sizes from 40 μm to approximately 5 μm in diameter, thereby roughening the surface texture of the powders and enhancing the particle-to-matrix bonding interface. The powders were then subjected to a heat-treatment at 800°C. This step allowed the phase transformation associated with the shape-memory effect to be recovered from the mechanical-milling process, which leaves the material in a high free-energy, amorphous condition. This same heat-treatment also deposits a thin oxide layer on the surfaces of the Ni-Ti particles, which inhibits diffusion and allows the shape-memory effect to be preserved in the material through the hot-pressing procedure.

After processing, the Ni-Ti powders were combined with aluminum powders in a 10 vol.% proportion, and mixed in a rotating blender for four hours. The powder was removed from the blender and weighed into appropriate sized charges of about 110 g for pressing. A steel die was used for pressing, with inner surfaces lined by a carbon foil. The die was filled with the charge of powders and placed in the press. The pressure inside the press was reduced to 10^{-5} torr and a degassing technique[13–15] may have been employed. In degassing, the sample is brought to an elevated temperature, usually between 200°C and 400°C, and held for an extended amount of time, usually around two hours. This step allowed surface oxides and gas particles to escape from the powders. The sample then was quickly brought up to the desired pressing temperature and pressed for ten minutes using a hydraulic ram. After pressing, the heating elements were turned off and the chamber allowed to cool. An inert flushing gas, such as nitrogen, argon, or helium, may have been used to induce a greater heat flow away from the sample in cooling. A rapid cooling was necessary to protect the sample from extensive diffusion. After the temperature dropped below 300°C, the atmospheric pressure was equalized and the chamber opened. Note that above 300°C, the carbon heating elements may combust if exposed to an oxygen environment. The die was then removed, and the sample was pressed out of it using a Carver™ hydraulic press.

The densities of these samples were then tested and recorded, using a standard Archimedes immersion technique, in ethanol. Samples were then sliced into 1.4 mm thick pieces. These samples were cold rolled, some at room temperature, and some at the sub-zero, martensitic temperature, to enact the shape-memory effect, with ten percent deformation, resulting in pieces 1.27 mm thick.

The presence of the Ni-Ti alloys shape-memory related phase transformation was detected and characterized using a Perkin-Elmer™ Differential Scanning Calorimeter (DSC). Rolled samples were cut into pieces slightly over 38 mm in length. Testing coupons were then punched from these samples with appropriate tooling in a hydraulic press. These testing coupons were in accordance to ASTM standard E8-91.[16]

A servohydraulic Material Test System (MTS) ™ test frame, with a load capability of 100 kN, was used to conduct static tension loading tests and cyclic fatigue tests for several different samples of Ni-Ti reinforced aluminum matrix material as well as for samples of unreinforced materials. Selected samples were mounted in a shoulder-seating pair of specimen grips and secured with setscrews. The specimen/grip assembly was then clamped into the electrohydraulic grip system of the MTS machine. First, the upper grip was clamped and, then, with the specimen/grip assembly hanging freely, the lower one followed. The specimen/grip assembly is designed with universal joints at each end, causing it to become self-aligning. The load reading was set to zero, and the necessary load settings for the test program were entered into the testing software. The tension test was then conducted at a strain rate of 4.1 mm/s, the strain rate typically used for most tension tests. Force, strain, and displacement data were collected and stored on timed intervals. The data were later reconstructed into stress-strain curves.

For fatigue tests, the maximum applied stress versus fatigue life cycle (S-N) curves were developed at a load ratio, R, of 0.2. R = μmin / μmax, where μmin is the minimum stress experienced by the specimen during the cycle, and μmax is the maximum stress during the cycle. The load reading was set to zero, and the necessary load settings for the test program were entered into the testing software. A peak-to-peak compensator in the program was used to control the accuracy of the load limits. Fatigue tests were started at a frequency of 1 Hz to allow the compensator to quickly adjust. Then, the frequency was slowly increased to 50 Hz. This procedure ensured that each cycle experienced accurate values of μmin and μmax, and that the test produced reliable data. For higher stress level (e.g., fatigue life < 500 cycles) tests, the frequency was only increased to 10 Hz to allow more control and accuracy over the short length of the test. At least 10 fatigue tests were conducted for each material over a wide stress range, so that a fairly accurate representation of the data could be displayed in the S-N curves.

Following testing, the microstructure of the materials and the fracture surfaces were examined with a Hitachi 8500 scanning electron microscope (SEM).

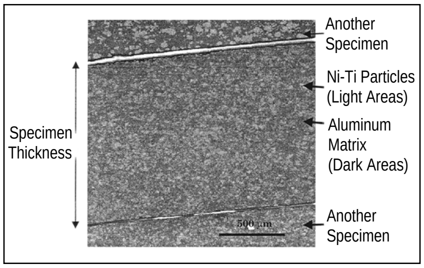

Figure 2. The SEM sample shows a Ni-Ti/Al metal-matrix composite specimen with a thickness of 0.127 cm. Mechanically milled Ni-Ti particle sizes are about 5 μm in diameter.

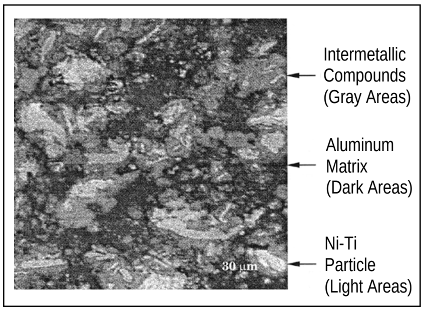

Figure 3. The SEM sample shows a Ni-Ti/Al metal-matrix composite. Mechanically milled Ni-Ti particle sizes are approximately 5 μm in diameter.

Table I. Applications of Shape-Memory Alloys in Science and Industry[3]

Aircraft and Space Exploration
- Antenna opening
- High-damping parts
- Hubble telescope
- Release mechanisms
- Triggering device for the separation of auxiliary fuel tanks for jet fighters

Arts, Toys, and Gadgets
- Memory box
- Model railways
- Sculptures
- Thermobile

Automobile Industry
- Cold-start performance
- Fuel injection
- Rattling noise reduction
- Ventilation and climate control

Composite Materials
- AMM (active modal modification)
- ASET (active strain energy tuning biased actuators)
- Reinforcement material for aluminum-matrix composites

Domestic Appliance Industry
- Automatic steam vent for cooking utensils
- Electric kettle switch
- Fire shield damper
- Lamp robot with memory metal drivers, controlled by means of speech recognition

Electronic Engineering
- Circuit breakers
- Connectors
- Gas decharge switch
- Wire connectors

Mechanical Engineering
- Actuator for micro-valve control
- Bearings with adjustable lubricating oil supply
- Microactuators
- Nuclear power plant applications
- Optical fiber splice
- Robot actuators
- Rock blasting
- Reusable pseudoelastic sealing rings for use in vacuum or corrosive atmosphere

- Precise and time-consuming production processes.

However, progress has been slow in the development and subsequent testing of this material. These difficulties have been successfully overcome by the authors.

RESULTS AND DISCUSSION

An examination of the composite microstructure is necessary to determine or verify physical characteristics such as the Ni-Ti vol.%, the presence or absence of diffusion or intermetallic reactions, particle-bonding characteristics, grain structure and size, porosity, locations of pores, and sources of crack initiation. The optical microscope and SEM have been used extensively as a guide towards the production and examination of the Ni-Ti/Al composite material.

The SEM results shown in Figure 2 display the mechanically milled 5 μm Ni-Ti powder in an Al 1090 aluminum matrix material. Figure 2 is a macroscopic cross-sectional view of the finished specimen, cut from a tension coupon blank. Figure 3 shows that the particles are fairly well distributed throughout the microstructure. However, quite a bit of diffusion between the Ni-Ti particles and aluminum matrix is evident, which leads to the formation of intermetallics shown in the gray areas. Fortunately, even though the sizes of the Ni-Ti phase have been reduced, the shape-memory effect remains, although not nearly as pronounced as it is in the condition as received from the manufacturer.

Phase-Transformation Characterization by Differential Scanning Calorimetry

After experimenting with several samples, Differential Scanning Calorimetry (DSC) tests showed that the phase transformation could be retained in the Ni-Ti powders through the milling process by way of the heat-treatment procedure. Figure 4 displays the DSC curves for the Ni-Ti powder in the as-received condition. Figure 5 depicts the DSC curves for the Ni-Ti powder after being mechanically milled and recrystallized through the heat-treatment procedure. The shifts in the transformation temperatures are probably due to the small amount of surface oxidation, which was hoped to occur during heat treatment. Since the free energy of titanium is much lower than nickel, the formation of a TiO_x compound is thermodynamically preferential over NiO_x.[17,18] This thin, titanium oxide layer was expected to act as the necessary barrier to prevent diffusion during hot-pressing. As a result, the Ni-Ti powders used for the composite materials retained their phase transformation throughout the hot pressing procedure

- **Medical Applications**
  - Implants
    - Adjustable dental implants
    - Porous pseudoelastic tissue
    - Pseudoelastic armature wire for the fixation of an artificial hip with automatic correction when bone necrosis occurs
    - Pseudoelastic dental arch wires
    - Pseudoscoliosis correction system
  - Medical Tools
    - Guide wires for catheters
    - Mounting device for intraocular lenses
    - Pseudoelastic catheter coils
    - Remote controlled closing system for small blood vessels, etc.
  - Tools for Prosthesis and the Disabled
    - Braille system
    - Intramedullary fixation mail
    - Revalidation equipment for rheumatism patients
- **Safety Technology**
  - Gas valves
  - Oil well valves
  - Overheating protection system
  - Removable and self-locking fire-break ceilings
  - Restorable heat sensitive elements
- **Sensors, Heating, and Ventilation Engineering**
  - Adjustments of flaps or slats
  - Air discharge flaps in air conditioning plants
  - Automatic ventilation
  - Solar actuator used for automatic adjustment of venetian blinds
  - Thermomarker

as well. Remarkably, the phase-transformation behavior, as indicated by a DSC analysis, had no appreciable change between that of the annealed powder and the composite material after pressing.[19] This trend indicates that the oxide layer on the particles has demonstrated a sufficient hindrance to diffusion.

The Influence of the Ni-Ti Reinforcement on Mechanical Properties

Several factors contribute to the observed mechanical properties of the material. One is the addition of the second-phase Ni-Ti particles, which stiffen the matrix and affect the modulus and strength. Another possible influence is the properties of the aluminum matrix. Also, the shape-memory action of the particles is expected to exert stresses and alter the mechanical properties of the material.

Figure 6 reveals the comparison of the stress-strain curves for the composite materials and aluminum matrix materials. The mechanical properties of the Ni-Ti-reinforced materials display a significant amount of strengthening over the unreinforced material, although the elongation properties are reduced. However, this behavior may be commonly observed in almost any reinforced composite material.

The numerical values for the mechanical properties are listed in Table II. This table shows that the composite material made with the mechanically milled powder, which has an Ni-Ti particle size of about 5 μm, drastically supplements the yield strength by 42% over the unreinforced material. Likewise, the ultimate strength is 54% greater than that of the unreinforced aluminum. Similarly, the composite material exemplifies a 34% magnification in the modulus, as compared to the unreinforced material.

The Influence of the Ni-Ti Reinforcement on the Fatigue Life

The peak stress during the fatigue test was plotted against the number of cycles endured by each sample, producing the S-N curves displayed in Figure 7. The composite material made with the mechanically milled Ni-Ti powders displays a remarkable amplification of both the peak-stress capability and the fatigue life, for the entire tested range of values. The S-N curve for the composite material converges with that of the matrix material at eight million cycles, with a corresponding stress level of 93 MPa. Again, this stress is just above the yield stress for the matrix material. For numbers of cycles greater than ten million, it is thought that the curves approach the

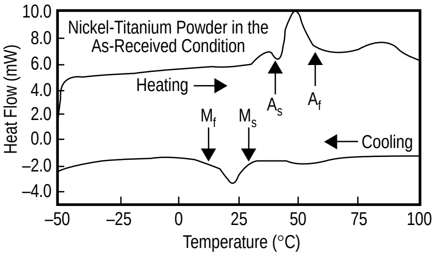

Figure 4. DSC curves for the Ni-Ti powder in the as-received condition from the manufacturer.

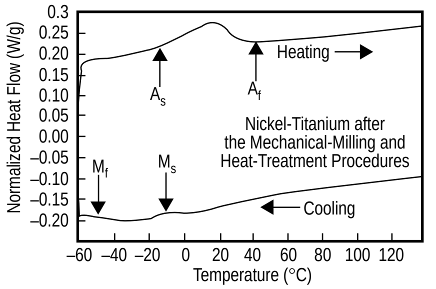

Figure 5. DSC curves for the Ni-Ti powder after being mechanically milled and heat treated.

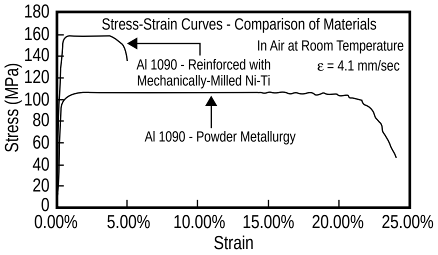

Figure 6. A comparison of the stress vs. strain curves for the Ni-Ti reinforced and unreinforced materials.

Table II. Physical and Mechanical Characteristics of the NiTi SMA Reinforced Composite Material, and the Unreinforced Matrix Material. *

Matrix Material	Al Powder Size (μm)	NiTi Powder Size (μm)	Hot-Pressing Temperature (°C)	Density (% th.)**	Deformation Temperature (°C)	σ_{yield} (MPa)	$\sigma_{ultimate}$ (MPa)	Young's Modulus (MPa)	Elongation (%)
Al 1090	20	5	550	99.94	–30	128	165	47,000	5
Al 1090	20	~	540	99.61	–30	90	107	35,000	24

*All samples were produced by hot-pressing constituent powders, contained within a 5.715 cm steel die, using 69 MPa pressure.
** Percentage of the theoretical density.

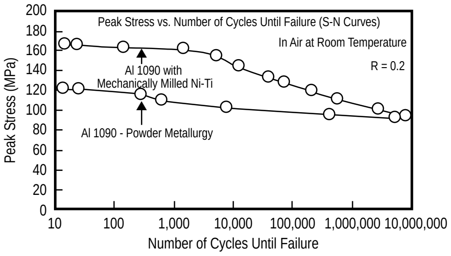

Figure 7. A comparison of the peak stress vs. number of cycles until failure (S-N Curves) for samples of Al 1090 produced by P/M processing, and the same material reinforced with mechanically milled Ni-Ti particles.

yield strength value of the matrix (90 MPa) asymptotically. For the higher stress levels, the composite materials have a decidedly longer life than the unreinforced material. Note especially at the peak stress of 120 MPa, where the matrix material can only withstand 50 cycles until failure, the composite material made with the mechanically milled Ni-Ti powders retains its integrity for 150,000 cycles.

As the SEM study shows, the overall cross-section of the samples generally is weakened from the continual void formation during the extended period of cyclic loading. As a result, the prime mode of crack propagation is largely matrix-dominated, due to void coalescence through the matrix material. However, as evidenced by the SEM study of the crack faces, the stresses that cause crack propagation vary at different load levels. At high stress levels, the primary stresses leading to failure are shear stresses, whereas at low stress levels normal stresses contribute most towards fracture. Therefore, much of the failure at high stress levels, as contrasted with the increase in the fatigue life, is considered to be intrinsically linked to stress fields interacting with strain barriers, as described by the Peach-Koehler relation.[21] Currently, it is difficult to quantitatively determine the exact mechanism by which the SMA affects crack growth. However, the increased fatigue life at higher stress levels is due possibly to a stress-induced phase transformation in the shape-memory particles. Additional experimentation is needed to acquire a deeper understanding of the relationship.

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