



On the transformation behaviour of NiTi particulate reinforced AA2124 composites

R.R. Thorat^a, D.D. Risanti^{b,*}, D. San Martín^c, G. Garces^d, P.E.J. Rivera Díaz del Castillo^a, S. van der Zwaag^a

^a Fundamentals of Advanced Materials Group, Faculty of Aerospace Engineering, Delft University of Technology, Kluyverweg 1, 2629 HS Delft, The Netherlands

^b Materials Innovation Institute, Mekelweg 2, 2628 CD Delft, The Netherlands

^c MATERIALIA Group, Department of Physical Metallurgy, National Center for Metallurgical Research (CENIM-CSIC), Av. Gregorio del Amo 8, 28040 Madrid, Spain

^d Department of Physical Metallurgy, National Center for Metallurgical Research (CENIM-CSIC), Av. Gregorio del Amo 8, 28040 Madrid, Spain

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ABSTRACT

The transformation behaviour of AA2124 alloys reinforced with NiTi particulates of 10 vol pct and 20 vol pct has been studied by differential scanning calorimetry (DSC), thermoelectric power (TEP) and internal friction (IF). Addition of NiTi particulates increases the damping capacity as well as the precipitation kinetics of AA2124 composites with respect to the base AA2124. Heat treatments performed to change the matrix condition do not alter the transformation significantly, other than homogenising the Al level in the particulates, which is present due to the preceding extrusion treatment. The presence of Al in NiTi stabilizes the R-phase transformation but the M_s remains stable.

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1. Introduction

Metal matrix composites (MMCs) are compound materials whose microstructure consists of a metallic alloy into which a particular reinforcing component is introduced. MMCs offer advantages in applications where low density, high strength and high stiffness are a primary concern. Among the various types of composites, the family of discontinuous MMCs containing particulates, whiskers, nodules and platelets, are favoured because they offer improvements to the mechanical properties of the monolithic alloys while remaining relatively easily deformable. The particulate reinforced MMCs are particularly attractive because they exhibit near isotropic properties compared to the continuously reinforced counterparts [1,2]. The availability of various types of reinforcements at competitive costs, the feasibility of mass production and high damping capacity [3,4] make this type of MMC more attractive. However, these materials may suffer from inhomogeneous distribution, size or shape of particulate, low ductility, inadequate fracture toughness and inferior fatigue crack growth performance compared to that of the matrix [5,6].

To optimize the mechanical and physical properties, in particular the damping conditions and damage tolerance of such particulate reinforced MMCs, one can utilize shape memory alloys as reinforcement. Shape memory alloys (SMAs) have received great attention because of their shape memory effect (SME) and many investigations are conducted on their basic performance and applications. To date SMA fibers have been added to soft and ductile metal or polymer matrices, e.g. [7–10], to improve the mechanical properties making use of the reversible transformation of SMA from martensite to austenite. The transformation under compression can result in stress in the matrix, which in turn enhances mechanical properties such as yield stress [9–12], fracture toughness [13,14], suppression of crack growth [15,16] and fatigue [11,17].

Two SMAs which generate large amounts of strain and are capable of generating a large force upon transformation back to the austenitic phase are NiTi alloys and Cu-based alloys. Copper-based SMAs are particularly interesting because of their low cost and relative ease of processing. However, copper alloys possess low strength compared to NiTi alloys and also exhibit degradation of the shape memory capacity when thermally cycled [18]. On the other hand, NiTi alloys tend to be more thermally stable and to have a lower density, higher yield and ultimate tensile strength; they are also more resistant to corrosion than Cu-based alloys [19].

The high temperature austenite phase of NiTi alloys has a B2 structure that transforms into B19 monoclinic martensite phase by cooling or applied stress. The transformation behaviour in pure

* Corresponding author at: Kluyverweg 1, 2629 HS Delft, The Netherlands.
Tel.: +31 15 278 8621; fax: +31 15 278 4472.

E-mail address: d.d.risanti@tudelft.nl (D.D. Risanti).

systems is known to be sensitive not only to compositional variations [20–26], heat treatment [27,28] and applied stress [29], but also to transformation cycles [26,30,58]. So far, the NiTi transformation behaviour has been studied using different tools, such as electrical resistivity [22,31], differential scanning calorimetry (DSC) [27,28], internal friction measurements [22,23,30,32,33], magnetic susceptibility [34] and thermoelectric power [31]. The transformation behaviour of NiTi embedded in a precipitation hardenable aluminium alloy has not been investigated yet, as earlier work on such systems was aimed at tailoring the mechanical properties [7–9,11,12,35,36] and processing route [12,36].

In the present work the transformation behaviour of NiTi particulates embedded in AA2124 matrix was studied. Particular emphasis has been placed on ascertaining the roles of the matrix microstructure and precipitate states produced by heat treatments, as the transformations may be sensitively affected by the surrounding matrix microstructure.

2. Experimental methods

2.1. Fabrication of NiTi SMA-AA2124 composite and heat treatments

The material used in the present experiments was a powder metallurgically processed AA2124 composite, reinforced with 10 vol.% and 20 vol.% NiTi particulates fabricated at the Centro Nacional de Investigaciones Metalúrgicas (CENIM). The nominal elemental composition of the atomized AA2124 powder with the average diameter of 32 μm from Alenco is given in Table 1. The atomized NiTi powder (Ni 55.7 wt.% and average diameter of 30 μm) from Nanoval GmbH & Co.KG (Germany) was used as the reinforcement.

Billets with a diameter of about 10 mm containing 0 vol.%, 10 vol.% and 20 vol.% NiTi particulates were produced by hot extrusion. During processing the air tight cans were kept at 773 K in furnace for 90 min before hot extrusion to soften the powders and improve their extrudability. The extrusion rate used was 0.4 mm/s with a starting pressure around 440 MPa. The extrusion temperature was maintained at 753 K.

To study the effect of the matrix microstructure conditions on the martensitic transformation behaviour of the embedded NiTi particulates, the composite samples were subjected to two different heat treatments: solutionising at 768 K for 1 h followed by water quench to obtain a soft matrix, hereafter termed as *SOL* and solutionising at 768 K for 1 h followed by water quench and subsequently aged for 12 h at 463 K to achieve an optimum precipitate amount and maximum hardness in the matrix, designated as *AGED*.

2.2. Characterisation techniques

The microstructure of the base AA2124 alloy and the composites were studied by means of light optical microscopy (LOM). For that purpose, the specimens were ground, polished and etched using Kellers reagent. The NiTi particulate's morphology, composition and its spatial distribution in the composite matrix were further analyzed by scanning electron microscopy (SEM) using a FEG-SEM JEOL-7500F equipped with EDX Thermo-Noran.

Thermal transformation effects were measured in a Sapphire-Cell Perkin Elmer DSC on all sample conditions. The as-extruded composite and NiTi bulk samples were cut by spark erosion in a cylindrical form with a weight of about 150 mg. The scans were carried out in an argon gas atmosphere with a heating/cooling rate of 2 K/min. The heat flow was recorded in a range of temperatures between 100 K and 350 K. To study the changes in the precipitation reactions in the aluminium matrix due to the presence of NiTi particulate, the scans were also performed between 300 K and 723 K with a heating rate of 10 K/min. Each DSC scan was repeated at least 2–3 times to confirm the reproducibility of the results.

The scanning thermoelectric power (TEP) measurements were undertaken with an Anatech Scanning TEP instrument. A description of this equipment can be found elsewhere [37]. Samples with dimensions 70 mm \times 2 mm \times 1 mm were employed.

Table 1
Composition of AA2124 powder.

Element	wt.%
Cu	3.93
Mg	1.41
Mn	0.64
Fe	0.0624
Si	0.21
Al	Balance

A temperature difference of 10 K between the two ends of the specimen was used in order to generate the Seebeck effect. The thermoelectric voltage of the specimen with respect to the aluminium reference was measured as a function of temperature during heating/cooling at 2 K/min in a range of temperatures between 100 K and 350 K.

Internal friction (IF) measurements were used to characterise the damping capacity of the composites. The measurement was performed using an inverted free torsion pendulum with a constant strain amplitude of 5×10^{-5} in all studied samples which dimensions were of 58 mm \times 4 mm with variable thickness. Different thickness results in different natural frequencies and thus the influence of frequency on the damping can be studied. The samples were cooled/heated in a He atmosphere between 100 K and 600 K at a rate of 2 K/min. At least two independent test runs were performed to check for full reproducibility.

3. Results

3.1. Materials

Fig. 1 shows a central cross-section of as-extruded composites. A number of particulate clusters are present and a significant amount of side-by-side particulate contact occurs within transverse planes. The scanning electron images in Fig. 1 show diffused interfacial products, which were identified with EDX as Al_3Ti (grey) and Al_3Ni (light grey) as indicated in detail in Fig. 1c. Those intermetallics layers were also observed in NiTi fiber reinforced AA6061 during processing at temperatures between 773 K and 873 K [10]. The NiTi particulate interfaces are smoother compared to the intermetallics layers indicating that Al atoms diffuse from the matrix into the particulates. The diffusion of Al lead to an Al-rich layer inside NiTi particle as indicated in Fig. 1c. The presence of Al in the interior of NiTi modifies the NiTi particulate composition to a lower Ni concentration due to Al substitutes Ni. *SOL* and *AGED* treatment results in homogenization of Al inside the NiTi particulates, as evidenced that the NiTiAl layers inside NiTi particulate disappear after those treatments. The average atomic Ni/Ti ratio of *SOL* and *AGED* as determined by EDX was found to be uniform over the particle interior. For the *SOL* and *AGED* state the Ni/Ti atomic ratio was 0.909 ± 0.02 . In addition, the as-received materials show constituents in the size of about 1–2 μm which were mainly found at the grain boundaries and dispersoids of about 0.3 μm within the grains. Only few pores were found particularly in the NiTi containing materials.

3.2. Differential scanning calorimetry study

3.2.1. Scanning between 300 K and 700 K

The DSC thermograms of the composites revealed a change in the precipitation kinetics of AA2124 as shown in Fig. 2. DSC thermograms of AA2124 shows the general features of this precipitate hardenable alloy [38–40]: an exotherm between 300 K and 423 K due to the formation of GPB zones (Peak 1); an endotherm between 423 K and approximately 518 K due to the dissolution of GPB zones (Peak 2); an exotherm between 518 K and 583 K due to the formation of S' (Al_2CuMg) precipitate (Peak 3) followed by a broad endothermic above 583 K due to the dissolution of S' precipitate (Peak 4). Overall, the presence of NiTi particulates does not principally change the precipitation sequence, but some aspects of the reactions are altered.

The *SOL* materials generally exhibit the expected prominent reaction peaks due to the solute supersaturation in the matrix. However, an additional dip (Af) slightly above 300 K was observed in NiTi containing materials, which is attributed to the austenitic transformation of NiTi particulates. Apart from that, the GPB zone formation peak (Peak 1) is still visible in the composites containing 10% and 20% with the smaller enthalpy compared to AA2124 indicating the activation energy of GPB zone formation has increased due to the presence of dislocations in the matrix which act as vacancies sink. Consequently, the dissolution of GPB zone is also smaller

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