



Properties of the pulsed electric current sintered Ni–Mn–Ga–Co–WC composites



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ABSTRACT

Two double dispersion composites were prepared by pulsed electric current sintering (PECS) from two powders on Ni–Mn–Ga–Co/WC–Co system. Composite 1 consists of 90 vol% WC–12Co and 10 vol% of non-modulated Ni–Mn–Ga–Co martensite and Composite 2 of 18 vol% of WC–12Co and 82 vol% of 10 M Ni–Mn–Ga–Co martensite. Damping capabilities of both composites were studied with DMA and cavitation erosion resistance tests. Both composites show promise as candidates as damping element materials when high hardness and wear resistance is required but Composite 2 had a higher damping capability. The high damping ability of composites are proposed to be derived from the combined effect of energy dissipation by twin boundary movement in the Ni–Mn–Ga matrix combined with the high hardness and wear resistance arising from the tungsten carbides.

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1. Introduction

Combining magnetic shape memory materials (MSMs) with other materials, such as polymers or ceramics, can lead to composites that have unique and useful properties such as tunable vibration damping, superelasticity and the magnetic shape memory effect [1,2]. Usually these active composites consist of MSM particles or structures, which are embedded in a suitable polymer matrix, which has a matching stiffness [3–5]. However, in applications where also high hardness, good tribological behavior and wear resistance are important the addition of ceramics is of interest due to their better mechanical properties. Previously such materials have been studied e.g., by producing coatings using high-velocity oxygen fuel and atmospheric plasma spraying from NiTi–TiC or NiTi–TiC/TiB₂ powders prepared by self-propagating high-temperature synthesis (SHS) [6,7]. Nevertheless, dense MMC composites can also be prepared by using the pulsed electric current

sintering (PECS) method in minutes [8].

Suitable powder for the PECS process can be produced in a variety of different ways but the easiest method is to crush ingots and ribbons mechanically [9–11]. Mechanical alloying has also been shown to be a viable method for the preparation of Ni–Mn–Ga magnetic shape memory alloys [12]. However, these methods have not so far been used together to manufacture Ni–Mn–Ga–Co ceramic composites. For this work a common (recycled) cermet WC–Co, was used as the ceramic addition for the studied Ni–Mn–Ga composite [13,14]. Adding of WC–12Co particulates into the Ni–Mn–Ga matrix produces so called double dispersion composites, which have earlier been studied e.g., in the context of wear resistant tools steels [15]. The goal of the research was to study the effect of ceramic addition to Ni–Mn–Ga alloys and so two different alloys were chosen; an alloy with non-modulated martensitic structure and an alloy with 10 M martensitic structure. Since previous investigation on the cavitation resistance, and the hence damping ability, of Ni–Mn–Ga alloys showed that the non-modulated structures have higher cavitation resistance than the 10 M structures [16], a higher WC–Co percentage was chosen for the NM alloy composite while a smaller volume fraction was

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chosen for the 10 M alloy to find out what effect the increasing ceramic content would have on the properties of the composite during DMA and cavitation experiments. In this paper we report PECS processing of Ni–Mn–Ga–Co/WC–Co composites, and their microstructure and properties.

2. Experimental

For the highest possible damping capability, single crystal Ni–Mn–Ga powder was used for the composites [5]. The MSM powder was produced from single crystal ingots by mechanical milling. Polycrystalline ingots (20–50 g) were manufactured in arc-furnace in argon atmosphere from 99.99% pure Ni, Mn and Ga and a Bridgman method was used to grow single crystals from the obtained ingots with compositions of Ni₅₀Mn₂₅Ga₂₅ (Alloy 1) and Ni₅₀Mn₃₀Ga₂₀ (Alloy 2). Then single crystals were annealed in argon atmosphere for 72 h at 1273 K for homogenization and 48 h at 1073 K for ordering. Crystals were ball-milled in a WC–Co pot using WC–Co balls to form the Ni–Mn–Ga + WC/Co composite powders 1 and 2 from the Alloys 1 and 2. The ball milled powder was then annealed in vacuum quartz ampoules at 1083 K for 4 h to remove residual stresses formed during ball milling. The particle size of the annealed composite powders was determined using Malvern Mastersizer 2000 and their phase structures were studied by X-ray diffraction (XRD) with Philips PW3830 CuK α diffractometer. Following [11] the powders were pulsed electric current sintered in nitrogen atmosphere at 1148 K under pressure of 50 MPa for 8 min. The diameter of the obtained finished Compacts 1 and 2 was 20 mm. Samples for the different experiments were cut from the compacts with a slow speed diamond saw, wet ground and the polished either with electro-polishing (25% HNO₃ ethanol, 253 K for 15 s) or with 20 nm colloidal silica solution.

Leica DMRX polarized microscope was used for optical studies and Sartorius CPA224S for measuring the density by Archimedes method. The microstructure and chemical composition analysis were studied with scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) on Tescan Mira 3 FEG-SEM equipped with Thermal Scientific UltraDry EDS detector. Zwick & Co Z323 hardness tester was used for the hardness (HV1) measurements. The phase transformations of the materials were determined both with the DSC (Linkam 600 at 5 K/min rate) and with low field *ac* magnetic susceptibility. The magnetic measurements, i.e. the *ac* magnetic susceptibility as well as the magnetization from 10 K to 400 K, were performed using the Quantum Design PPMS-14 system. The moduli and $\tan \delta$ were determined using the TA Instruments Q800 DMA instrument in the single cantilever mode. The sample for the DMA measurement was a cuboid with dimensions of 1.88 mm $\times$ 0.52 mm $\times$ 16.59 mm. Additionally, in order to evaluate if there is strain dependency of the damping phenomena, a DMA measurement was done for both composites at temperature of 299 K and the changes in $\tan \delta$, stress and strain were measured to see if the $\tan \delta$ was strain dependent.

Small cubes with dimensions of 3 mm $\times$ 3 mm $\times$ 4 mm were cut from the reference material steel 304 and the Compacts 1 and 2 for cavitation erosion tests. Samples were ground with grit 600 sandpapers. Misonix Sonicator XL2020 was used for the cavitation and due to the small size of the samples specially made brass sample holders were used during cavitation (Fig. 1a). In order to compare the results with the previous cavitation tests (Fig. 1b) [16] the samples were placed exactly 0.50 ± 0.01 mm from the 16 mm diameter titanium cavitation head in 1000 ml deionized water. Titanium cavitation head was changed always between the samples. Amplitude of 40% was used for all tests. This amplitude correlates with the peak-to-peak displacement of 76 μ m. Cavitation was continued until either maximum time of 55 h was reached or

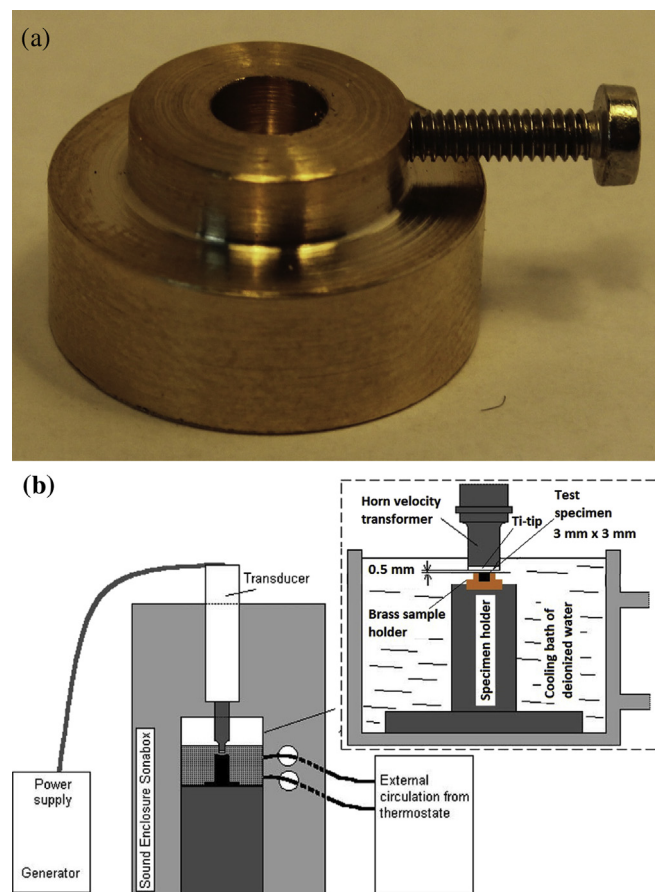


Fig. 1. (a) Brass sample holder for cavitation samples and (b) cavitation test set-up.

until cumulative mass loss reached 4 mg [16].

Before the cavitation experiment was started the water was sonicated for 30 min. After cavitation samples were cleaned with ethanol in an ultrasound cleaner and dried with hot-air blower. Sample weight was measured before cavitation and during cavitation using the Sartorius CPA224S. Weighting was carried out with intervals of 30 min until cumulative time of 39 h was reached after which the sampling time was increased first to 1 h and finally to 2 h. Stainless steel 304 samples were used both for calibration of the device and later as a reference material, while other reference values were gotten from literature [16,17]. Stainless steel 304 samples were used both for calibration of the device and later as a reference material, while other reference values were obtained from literature [16,17]. Steel 304 was chosen as a reference material because it is a well-known material with a known cavitation behavior. Stainless steel has also been used in the previous cavitation research as a reference material [16]. After cavitation experiments the surfaces morphology of the samples was also studied using Tescan Mira 3 FEG-SEM.

3. Results and discussion

XRD analyses of compacts (Fig. 2) showed Ni–Mn–Ga–Co-based martensite and WC–Co to be present. The martensite is non-modulated (NM) in the Compact 1 and five-layered modulated (10 M) in the Compact 2. The peak intensity comparison with the different components revealed that the amount of metallic phase in Compact 1 decreased during consolidation, while in Compact 2 the phase ratios stayed nearly the same. The SEM investigation of the

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