

Production of Ni-Mn-Ga foam

Izdelava porozne zlitine Ni-Mn-Ga

Iztok Naglič¹*, Kristina Žužek Rozman², Paul McGuinness², Luka Kelhar², Spomenka Kobe², Boštjan Markoli¹

¹University of Ljubljana, Faculty of Natural Sciences and Engineering, Department of Materials and Metallurgy, Aškerčeva cesta 12, 1000 Ljubljana, Slovenia

²Jožef Stefan Institute, Department for Nanostructured Materials, Jamova 39, 1000 Ljubljana, Slovenia

*Corresponding author. E-mail: iztok.naglic@omm.ntf.uni-lj.si

Abstract

Ni-Mn-Ga alloys presents the most promising materials among the functional metallic materials showing the magnetic field induced strain (MFIS) especially due to recent discovery of the effect of porosity in this material. This work presents the production of the Ni-Mn-Ga alloy foam via casting replication method using the sodium aluminate as a space holder for the later porosity and their characterization. Two different compositions of Ni-Mn-Ga alloys and space holders were produced. Space holders were prepared in one case of powder size range of 500 μm to 600 μm and in the other case of 63 μm to 100 μm and 315 μm to 500 μm powder sizes. Samples microstructure was characterized after the alloy infiltration in to a sintered space holder to determine alloy and sodium aluminate fraction.

Key words: magnetic shape memory, casting, Ni-Mn-Ga- alloy, porous alloy

Izvleček

Zlitine iz sistema Ni-Mn-Ga spadajo med bolj obetavne materiale iz skupine funkcionalnih kovinskih materialov. Ti materiali kažejo pojav spremembe dimenzij, induciranih z magnetnim poljem, ki je zanimiv predvsem v povezavi z nedavno odkritim vplivom poroznosti. V delu je predstavljen postopek izdelave zlitine Ni-Mn-Ga in infiltracije zlitine v porozno predformo iz natrijevega aluminata ter karakterizacija. Izdelali smo dve različni zlitini in predformi. Predforma je bila v enem primeru izdelana iz delcev velikosti med 500 μm in 600 μm , v drugem pa med 63 μm in 100 μm ter 315 μm in 500 μm . Po infiltraciji zlitine v porozno predformo natrijevega aluminata je potekala karakterizacija mikrostrukture, pri čemer sta bila določena deleža zlitine in natrijevega aluminata.

Ključne besede: magnetni oblikovni spomin, ulitek, zlitina Ni-Mn-Ga, porozna zlitina

Introduction

An important group of functional metallic materials are shape memory alloys. There are several ferromagnetic alloys which are capable of so called magnetic-field-induced strain (MFIS) like Dy, Fe-Pd alloy, Fe₃Pt and Ni-Mn-Ga alloys.^[1] These materials are capable of changing the shape or dimensions under the externally applied magnetic field.^[1, 2] The most promising material among these are Ni-Mn-Ga alloys. Monocrystalline Ni-Mn-Ga alloys show very large MFIS up to a 10 %. MFIS effect (strain) in these alloys is produced by moving of twin boundaries caused by internal stresses which are generated by magnetic anisotropy energy.^[3] These twin boundaries in this alloy form after the diffusionless martensitic type phase transformation from L2₁ ordered type structure.^[4] Fine grained non-textured polycrystalline alloys, on the other hand, shows nearly zero MFIS effect.^[1] Nearly zero MFIS effect in fine grain polycrystalline alloy is consequence of the fact that grain boundaries effectively suppress the motion of twin boundaries and that neighbouring grains in non-textured alloy have incompatible MFIS.^[1] MFIS in these alloys can be increased by increasing the grain size and introducing the texture. Recently it was also found and explained that large MFIS (2–9 %) in these polycrystalline alloys can be gained by the introduction of porosity smaller than the grain size.^[2, 3, 5, 6]

Main aim of this paper is to present the synthesis of the Ni-Mn-Ga alloy foam via casting replication method^[7] using the sodium aluminate as a space holder for the later porosity and their characterization.

Materials and methods

The Ni-Mn-Ga alloy foams were prepared by infiltration of liquid alloy in to a sintered sodium aluminate (NaAlO₂) space-holder. Sodium aluminate powders of the specific sizes were prepared from the commercial sodium aluminate (Al₂O₃·Na₂O or NaAlO₂) powder (purchased from Alfa Aesar). Commercial powder was first sintered at 1 550 °C for 3 h in air to produce the bulk material. Bulk material was

milled in vibrating mill for short time (couple of seconds) to produce the powder of appropriate size distribution. Powder was later sieved to extract the powders with sizes ranging between 63 μm to 100 μm, 100 μm to 200 μm, 315 μm to 500 μm, and 500 μm to 600 μm.

Space holder composed of two different sizes of the particles was prepared by dry mixing of powders with sizes of 63 μm to 100 μm and 315 μm to 500 μm. Mixed powders were poured in to an alumina crucible with diameter of 11 mm to height of approximately 5 mm and sintered at 1 550 °C for 3 h in air. The space holder specimen produced of monosized distribution powder was prepared by pouring the powder of 500 μm to 600 μm in to an alumina crucible with diameter of 11 mm to height approximately 5 mm. Space holder was in this case sintered at 1 580 °C for 3 h.

Ni-Mn-Ga alloy was synthesized from pure nickel (mass fractions $w = 99.83\%$), manganese (99.9 %) and gallium (99.999 %) using arc melting furnace (Compact Arc Melter MAM-1, Edmund Bühler GmbH). Pieces of the arc melted alloy were put on the top of the space holder within the alumina crucible and heated in a vacuum to a temperature of 1 250 °C and held at the temperature for about 30 min. Afterwards the Ar was introduced in to a furnace chamber to gain the pressure of 1 bar which forced the alloy in to a porous space holder. After the infiltration the furnace was cooled down to an ambient temperature.

Infiltrated samples were cold-mounted, ground and polished. Metallographic observation has been performed using light microscopy for the evaluation of the distribution of the sodium aluminate (porosity) and determination of the fraction of the sodium aluminate (porosity). The presence of twinning which is characteristic feature of the non-cubic phase at the ambient temperature was represented by polarized light microscopy. Light microscope ZEISS AXIO Imager.A1m combined with software for digital image processing Axio Vision was used in this work. Scanning electron microscope Jeol JSM 5610 equipped with energy dispersion spectrometer (EDS) Gresham scientific instrument model no.: Sirius 10/SUTW was used for determining compositions of synthesized alloys.

Results and discussion

Beside samples presented in this work more sintered space holder samples were prepared. For the successful infiltration the space holder must be adequately sintered, which means that particles in space holder are attached to each other and that there is still enough open porosity between them which enables successful infiltration. Based on previously mentioned samples it was found that appropriate conditions for sintering of space holder are between 1 550 °C and 1 580 °C for 3 h. Higher temperature limit stands for the space holder composed only of large particles while lower temperature stand for the space holder composed of large and small particles. This finding is also in the agreement with the fact that sintering temperature also depends on the particles size which means that smaller particles lead to a lower temperature of sintering.

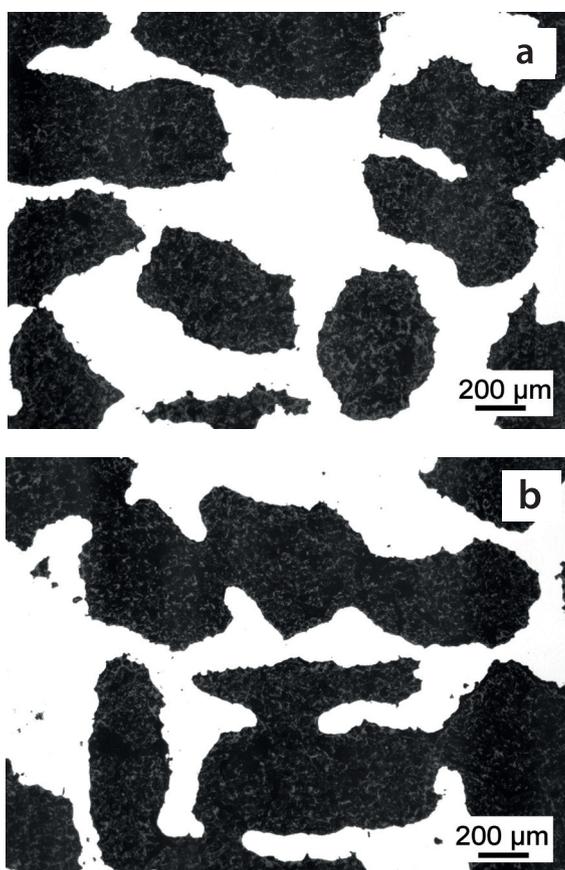


Figure 1: Microstructures of sample T23 prepared of the preform composed of 500 μm to 600 μm particles with a) higher and b) lower fraction of the alloy.

Typical microstructures of the sample T23 prepared of the preform composed of 500 μm to 600 μm particles are presented in Figure 1. Microstructures in Figure 1 show that sodium aluminate particles are rather evenly distributed with slightly different fraction in different regions of the sample. This was also confirmed by the determination of the fraction of the alloy presented in Table 1. As presented in Table 1 the average fraction of the alloy and sodium aluminate are 38 % and 62 % in sample T23.

Table 1: Fraction of alloy and sodium aluminate in the sample T23 prepared of the preform composed of 500 μm to 600 μm particles

	Fraction (%)	
	Sodium aluminate	Alloy
M1	58.7	41.3
M2	65.2	34.8
Average	62.0	38.0

Macrostructure of the sample T3 prepared of the space holder composed of the particles with sizes ranging between 63 μm to 100 μm and 315 μm to 500 μm is presented in Figure 2. Macrostructure in this figure shows that the central region of the sample was not completely infiltrated.

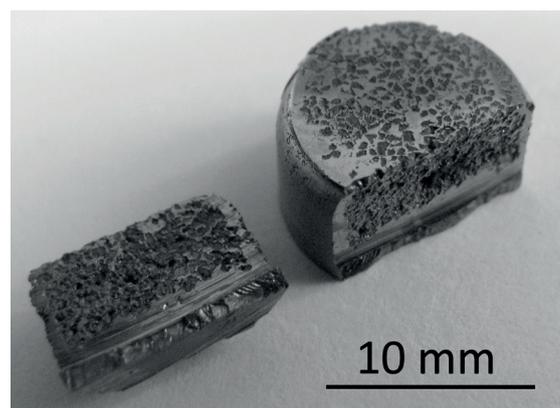


Figure 2: Macrostructure of the sample T3 prepared of the preform composed of the particles with sizes ranging between 63 μm to 100 μm and 315 μm to 500 μm .

Microstructures of the sample T3 are presented in Figure 3. It can be seen from the microstructures that the distribution of small sized particles is not even. Quantity of small sized particles is also too small to enclose the large

particles and it should be increased in further work. On the other hand, large particles are more evenly distributed. The exception is the central region of the sample where alloy did not completely filled the sample and we cannot judge, based on these figures, whether dark regions are only large particles or there are large particles with unfilled space. Locally denser space holder can lead to a locally lower fraction of alloy or it can even prevent further progression of alloy in to a preform. If progression of the alloy is the reason, it could be overcome by higher pressure of infiltration or smaller size of the sample. Uneven distribution of small sized particles is probably also the consequence of the way the powders were mixed (dry mixing) and poured in to an alumina crucible. Zhang et al. report that even distribution of the particles can be accomplished by the introduction of powders to crucible layer by layer in the acetone.^[5]

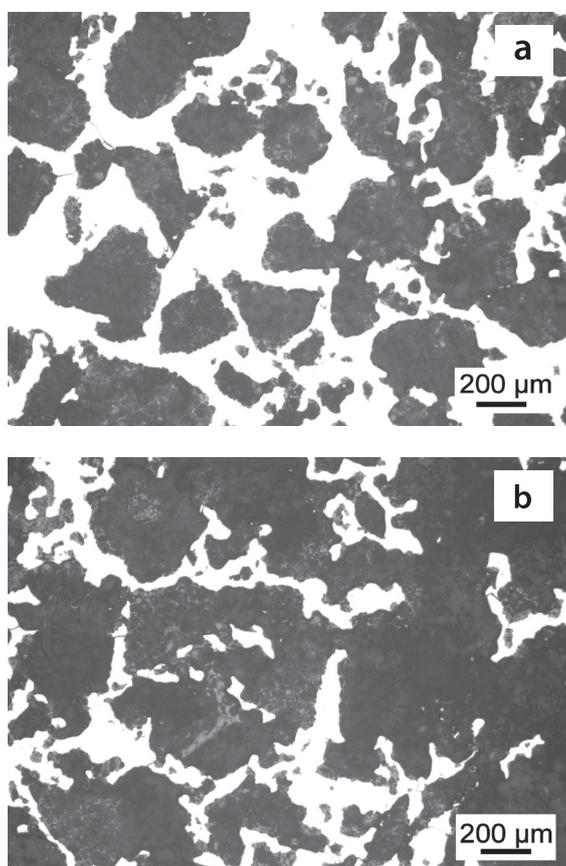


Figure 3: Microstructures of the sample T3 prepared of the space holder composed of the particles with sizes ranging between 63 µm to 100 µm and 315 µm to 500 µm. a) represents periphery of the sample and b) centre of the sample.

Fraction of the alloy and the sodium aluminate in the sample T3 prepared of the space holder composed of the particles with sizes ranging between 63 µm to 100 µm and 315 µm to 500 µm is presented in Table 2. Results show that there is a difference in alloy fraction between the central and periphery regions of the sample. As it is given in the Table 2 the average fraction of the alloy and sodium aluminate in this sample are 31.4 % and 68.6 %. Comparison of these results to results for sample T23 prepared of monosized space holder shows that in the case of monosized space holder fraction of the alloy is larger. This result is expected since the powders of several different sizes which are properly mixed in comparison to monosized possess higher green density.

Table 2: Fraction of the alloy and the sodium aluminate in the sample T3 prepared of the space holder composed of the particles with sizes ranging between 63 µm to 100 µm and 315 µm to 500 µm

	Fraction (%)	
	Sodium aluminate	Alloy
M1	64.4	35.6
M2	66.3	33.7
M3	70.1	29.9
M4	73.7	26.3
Average	68.6	31.4

Different compositions of alloys were used in these experiments. Compositions of two alloys determined by EDS after remelting under controlled conditions (10 K/min in an atmosphere of Ar gas) are presented in Table 3 and their microstructures are presented in Figure 4.

Table 3: Compositions of the alloys A and B determined by EDS in mole fractions (x/%)

	Alloy A	Alloy B
Ni	49.7	56.0
Mn	26.9	23.2
Ga	23.4	20.8

Ni-Mn-Ga alloys undergo diffusionless martensitic type of phase transformation which is necessary for the MFIS effect. Martensite start temperature in these alloys depends on the composition of the alloy.^[4, 8] It was assumed that the stability of β -Ni₂MnGa is controlled by

Hume-Rothery electron compound mechanism and it was shown that linear relationship exists between martensite start temperature M_s and electron/atom (e/a) concentration.^[4, 8] Using such a relationship presented by Schlagel et al.^[8] and compositions of alloys determined by EDS give us (e/a) concentration in the case of the alloy A of 7.56 and the martensite start temperature in the range between 200 K and 250 K. This temperature is below the ambient temperature. In the case of the alloy B (e/a) concentration and the martensite start temperature are 7.85 and approximately 450 K. Polarised light microscopy images presented in Figure 4 shows that twinning, which is characteristic feature of non-cubic martensitic phase, is observed only in the case of the alloy B (Figure 4b). This result is consistent with the previously accessed martensite start temperatures.

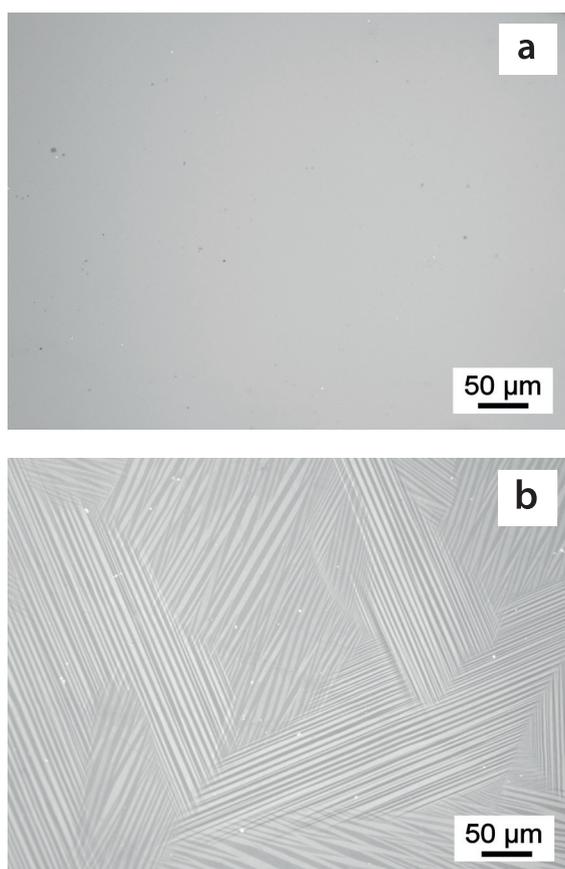


Figure 4: Polarized light microscopy images of a) alloy A and b) alloy B.

Conclusions

It was found, based on the prepared sodium aluminate space holders that the appropriate conditions for sintering of space holder are between 1 550 °C and 1 580 °C for 3 h. High temperature limit was found to stand more for the space holder composed of large particles while lower temperature stand for the space holder composed of large and small particles.

Two samples were prepared by infiltration of the alloy in to a space holder composed of only large particles (500 μm to 600 μm) and in to a space holder composed of large (315 μm to 500 μm) and small (63 μm to 100 μm) particles. Microstructural analysis of the sample composed of only large particles shows rather evenly distributed particles of sodium aluminate with slightly different fraction in different regions of the sample.

Distribution of small sized particles in sample composed of large and small particles is not even and the quantity of small sized particles is also too small. Large particles are more evenly distributed. The exception is the central region of the sample where the alloy did not completely filled the sample. It was found that the average fraction of alloy and sodium aluminate in sample composed only of large particles are 62 % and 38 % while in the case of sample composed of large and small particles are 68.6 % and 31.4 %.

Microstructures of both alloys are consistent with expected martensite start temperatures based on compositions.

Acknowledgments

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