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Correlation of microsegregation and variant distribution in directionally solidified Ni-Mn-Ga alloys

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ABSTRACT

In this work, the microsegregation and variant distribution in directionally solidified Ni-Mn-Ga alloys have been investigated. The results show that the martensitic variant distribution changed dramatically, as well as a decrease in microsegregation between dendrite core and interdendritic regions, after the directionally solidified samples underwent a high-temperature heat treatment. Further theoretical calculations reveal that the preferential martensitic configuration is accommodated with martensitic-transformation-induced stress in the Ni-Mn-Ga alloys with pronounced microsegregation. This finding is of significance as it highlights the role of microsegregation in variant distribution and provides a possible way to obtain martensite with a controllable variant distribution.

Ni-Mn-Ga alloys have attracted much attention owing to their excellent magnetic shape memory effect (MSME), with potential applications in sensors and actuators [1–4]. Crystal orientation and texture of constituent martensitic variants have a strong influence on the activation of the MSME [5]. For instance, a straight and coherent twin boundary between two adjacent variants can be easily activated during variant reorientation induced by a magnetic field. Moreover, a reduced number of martensitic variants are also beneficial for overall variant reorientation. Usually, the martensitic variant reorientation can be induced by mechanical training [6] and even the stress accompanied with a thermal field or magnetic field [7]. So far, some processing techniques, including heat treatments, compression, magnetic-field treatments or combined treatments [8–11], have been devoted to microstructure control and property optimization of this new class of magnetic shape memory materials. The above-mentioned researchers mainly focused on the effect of thermal-mechanical treatments on crystal structure and crystal orientation of martensitic variants in samples with a homogeneous composition, while little attention has been paid to the martensitic variant distribution confined by composition inhomogeneity, especially by microsegregation with regular features. This research is essential for better understanding the mechanism of the martensitic variant distribution confined by composition inhomogeneity and even γ precipitates, which may offer another promising route to fabricate martensite with a controllable variant distribution.

Hence, the determination of microsegregation during directional solidification and how it will affect a martensitic variant distribution are of practical importance for microstructural control and theoretical interest to gain insight into the control of the martensitic variant distribution. In the present study, Ni-Mn-Ga alloys were prepared by Bridgman directional solidification. The correlation of microsegregation and martensite variant distribution has been investigated. The obvious microsegregation between dendrite core and interdendritic regions directionally solidified can be reduced by following heat treatment. Meanwhile, the martensitic variant distribution changed dramatically with a decrease in microsegregation. A possible mechanism from the viewpoint of accommodation capacities of the twin variant distribution that is constrained by compositional inhomogeneity is also exhibited. The present research would bring about not only a comprehensive understanding to martensitic variant selection but also a possible way to obtain martensitic samples with controllable variants.

Ni-Mn-Ga alloys with nominal compositions of $Ni_{54}Mn_{24}Ga_{22}$ and $Ni_{48.5}Mn_{32.5}Ga_{19}$ were prepared by arc-melting using high-purity Ni (99.99 wt%), Mn (99.9 wt%), and Ga (99.99 wt%) as the raw materials. The alloys were re-melted four times and then suctioned into a quartz tube to obtain a cylindrical alloy of diameter 3 mm and length 150 mm. The alloy was enveloped in a high-purity corundum tube with an inner diameter of 3 mm and length of 200 mm for subsequent directional solidification. During the experiments, the samples in the

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corundum crucibles were re-melted and directionally solidified at a growth speed of 20 μm/s in the Bridgman apparatus by pulling the crucible assembly into the liquid metal (Ga-In-Sn) cylinder. A typical transverse section in the solid zone with stable growth was cut for microstructural characterization. For the microstructure and orientation observations, the sample surfaces were first mechanically polished, and then electrolytically polished with a solution of 20% HNO_3 in CH_3OH at 12 V for 20 s at room temperature.

Microstructural characterization was performed by scanning electron microscopy (SEM) using an instrument (Hitachi SU70) equipped with a Hikari high-speed electron backscatter diffraction (EBSD) camera. All EBSD data were analyzed by OIM analysis software (EDAX, USA). As is elucidated in our previous paper [12], the orientation imaging maps presented in this article were all obtained by auto-collection software, whereas the precise information for the reconstruction of the crystallographic configurations was obtained by manually indexing the Kikuchi patterns in view of EBSD resolution achieved by auto indexing and NM martensitic plates composed of alternately distributed fine lamellae with width of hundreds of nanometers. Moreover, a statistical analysis method known as the grid measurement technique was applied to capture the microsegregation [13,14]. This method generally involves composition measurements from a statistically significant number of individual measurement points, which are spaced within a measurement grid. The grid used in this work covered several dendrites in an area of approximately 400 × 400 μm^2. The live sampling time for each point was 30 s. The program TEAM was used to apply ZAF corrections and deduce the fractions of each alloying element at each measured location. The EDS data were ranked via rank sorting [14] to obtain the composition against the cumulative fraction of the measured data-points.

Fig. 1 shows the microstructural, compositional and orientational characteristics of the transverse section in the directionally solidified NM Ni-Mn-Ga alloys without and with heat treatments. It can be found that the morphology and variant distribution of martensite plate have a dramatic change after high-temperature heat treatment. However, this change cannot be found during the reverse martensitic transformation heating up to 473 K and cooling to temperature in a muffle furnace (see Fig. S1 in the Supporting information). This suggests that the preferential distribution of martensite plates in this study should not be induced by the thermal stress induced by temperature gradient during directional solidification. Moreover, the typical areas as marked by white dash lines in the BSE images were selected to measure the distribution of Ni, Mn, and Ga elements. The corresponding microsegregation profiles in Fig. 1 verifies that the microsegregation has been reduced by the high-temperature heat treatment.

In order to further study the evolution of martensite variant distribution, two typical amplified EBSD maps on the transverse section selected from directionally solidified NM Ni-Mn-Ga alloys without and with following high-temperature heat treatment, respectively. Compared with the variant colony that mainly consisted of long, straight

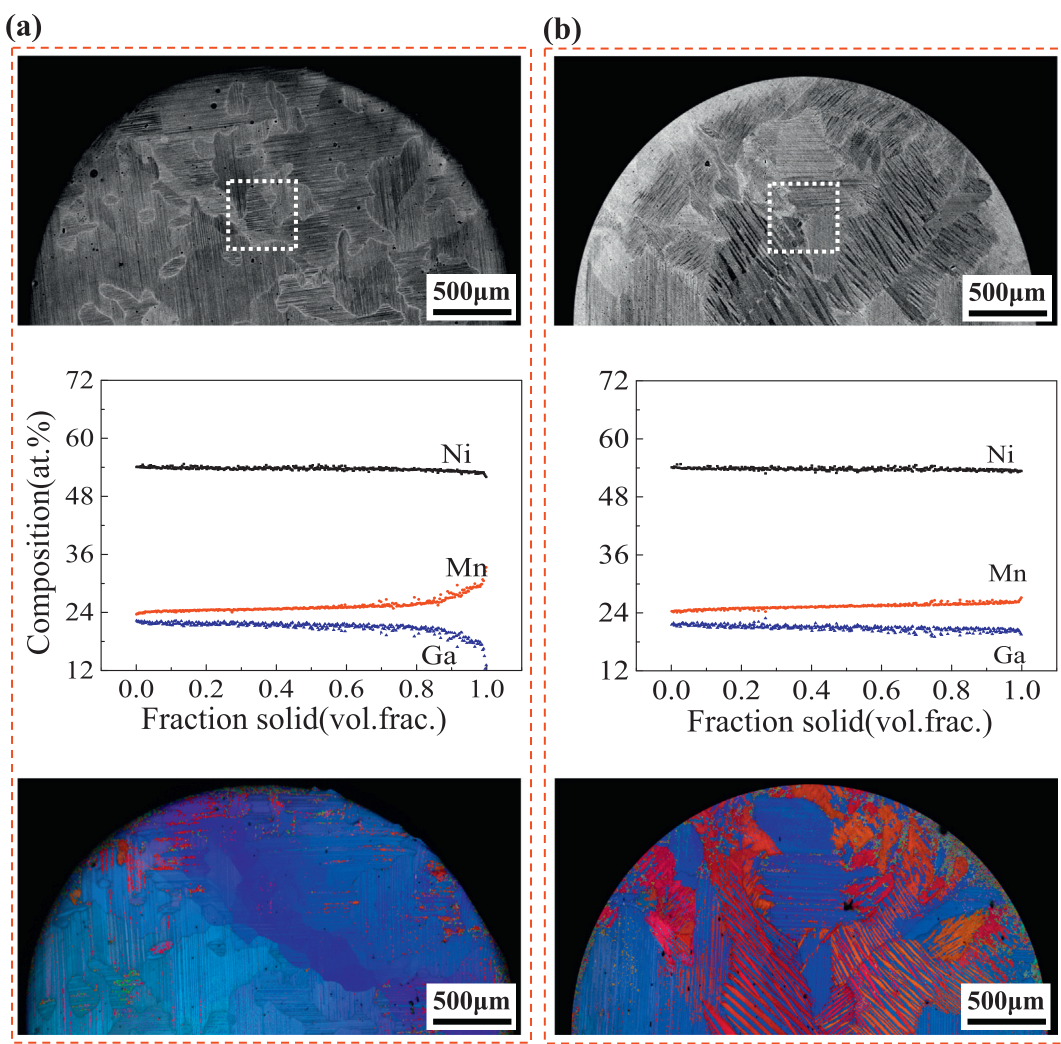

Fig. 1. BSE images (top) and corresponding microsegregation profiles of Ni, Mn and Ga elements (middle), and inverse pole figure (IPF, bottom) in the transverse section of directionally solidified $Ni_{54}Mn_{24}Ga_{22}$ alloys at a growth speed of 20 μm/s: (a) without heat treatment; and (b) heat treatment (HT) at 1273 K for 12 h for the same sample in a muffle furnace.