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MAGNETOCALORIC EFFECT IN Ni-Mn-Ga-Si ALLOY SUBJECTED TO PLASTIC DEFORMATION BY THE MULTIPLE ISOTHERMAL FORGING

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In Heusler alloys, in the region of martensitic and magnetic phase transformations, a magnetocaloric effect is observed, which allows considering these alloys perspective for applying as solid refrigerant in cooling units. However, these alloys possess degraded performance properties. This study presents results of the investigation of the magnetocaloric effect in Ni-Mn-Ga alloy in two structural states: the initial as-cast state and subjected to deformation processing by multiple isothermal forging at 953 K. It is shown that in the as-cast state microstructure of the alloy is represented by equiaxial grains with a size of several hundreds of microns. As a result of forging, a binary microstructure is formed, where initial coarse grains are surrounded with a layer of a fine-grained structure. The investigation of the temperature dependence of the thermal expansion shows that the martensitic transformation in the as-cast state alloy occurs in the temperature range from 194 K to 214 K. In the treated state the phase transformation in the alloy shifts to the low temperature range by 10 K. In both states the value of magnetocaloric effect in the region of the martensitic transformation equals 0.15 K in the magnetic field of 1.8 T, and in the region of the Curie point — to 0.9 K. The only difference of the treated state is a shift of the $\Delta T\,(T)$ dependence to the low temperature range by 10 K.

Keywords: *Heusler alloy, martensitic transformation, magnetocaloric effect, multiple isothermal forging.*

Introduction

Ni-Mn-Ga Heusler alloys display unique physical effects in the region of martensitic transformation occurring in them. The effects include ferromagnetic shape memory effect and magnetocaloric effect [1–8]. The effects in the polycrystalline and the single crystalline alloy samples of this system have already been sufficiently studied. Despite considerable values of functional effects, a problem of degraded performance properties

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stands in the way of practical application of these materials. During multiple cycles of martensitic transformations the alloys are subject to destruction [9; 10]. In order to solve this problem, different methods of thermo-mechanical treatment (TMT) can be applied to the alloys [11–13]. However, a considerable decline of the functional effect value may result from the treatment [14; 15]. As it has been shown earlier, the deformation treatment by forging and extrusion results in the formation of the thermal expansion anisotropy level in the initial as-cast state [16–18]. This suggests that in treated alloys it might be possible to acquire the ferromagnetic shape memory effect value, comparable to that in the as-cast state alloys. Thus, it is necessary to carry out a comparative analysis of the magnetocaloric effect value of the alloy in the as-cast and the treated state.

This study presents results of the investigation of the magnetocaloric effect in $Ni_{2.16}Mn_{0.79}Ga_{0.98}Si_{0.07}$ polycrystalline alloy in the initial as-cast state and after the TMT by multiple isothermal forging (MIF). The advantage of this method of deformation treatment consists in a higher density of dislocations, a high level of internal stresses and the required microstructure in the obtained bulk ingot of the material. It is possible to get samples of the necessary size from the obtained ingot.

1. Material and Methods

The alloy was obtained by argon arc melting from Ni, Mn and Ga elements of a high purity. An ingot, melted by this method, usually has a shape of a «tablet». Such ingot cannot be used for subsequent TMT, where a bulk sample, such as a cylinder, is required. Therefore, after melting the melt was poured into a quartz crucible, heated to approximately 970 K. As a result of the intensive crystallization of the melt, macropores were formed in the structure of the melt. In order to get a quality ingot, the alloy was additionally subjected to vacuum remelting. It is known that the vacuum circulation of the melts also contributes to the enhancement of the quality of the material. The remelting was carried out in a new quartz crucible of a larger diameter. During the remelting, silicon atoms diffused from the material of the crucible into the melt. The elemental composition analysis by the energy dispersive spectroscopy showed that the alloy had the following composition: $Ni_{2.16}Mn_{0.79}Ga_{0.98}Si_{0.07}$. According to the further investigation of the alloys, obtained by this method, the presence of the silicon does not have any significant effect on functional properties of the material, merely shifting the temperature of martensitic transformation to the low temperature range. The microstructure analysis by SEM and XRD methods does not display any additional phases, caused by the presence of silicon in the alloy composition.

After the removal of the shrink hole, an ingot in the shape of a cylinder with a diameter of 16 mm and a height of 13 mm was obtained. The forging was performed on the Schenck Trebel RMC 100 complex loading machine at the temperature of 953 K by sequent deformations of the sample with the upset for 15–35% at each transition. In between the transitions, the shrinking direction of the sample changed by 90 ° according to the A-B-C-B-C-B-C scheme, where A, B and C stand for three orthogonal directions. The true degree of deformation was $e = 1.9$. Resulting from the MIF, an ingot in the shape of an elongated parallelepiped was obtained. The last four of the upset transitions were performed with a rotation of the ingot along the A axis in order to form a texture in the metal, which is supposed to contribute to the enhancement of the ferromagnetic shape memory effect, for instance. An assessment of the influence of the texture on the magnetocaloric effect was intended.

The microstructure analysis was carried out by the electron backscatter diffraction (EBSD) on the Mira 3 LMH scanning electron microscope (Tescan). The survey was carried out at the room temperature. A section for the investigation was prepared by polishing on the sandpaper, the diamond paste and final electro polishing in the electrolyte (10% HCl + 90% Butanol).

The temperature of the martensitic transformation and the anisotropy of properties were investigated through recording a temperature dependence of the thermal expansion on an induction type dilatometer. For the investigation, samples with a size of $1\,\mathrm{mm} \times 1\,\mathrm{mm} \times 7\,\mathrm{mm}$ were cut. The direct measurement of the magnetocaloric effect was performed on a special installation [19; 20]. A chromel — constantan thermocouple made of 50-μm-thick wires was cemented to a $2\,\mathrm{mm} \times 2\,\mathrm{mm} \times 0.5\,\mathrm{mm}$ sample by the BF-2 glue. To minimize the influence of a magnetic field on the measured signal, the differential thermocouple wires were twisted bifilarly. To improve the thermal contact and to decrease the thermal inertia, the wire ends were flattened to a thickness of 3–5 μm. The thermocouple junction was fabricated by the electric welding. The measurement was performed through the use of two sources of cyclic magnetic fields. The first source was a controlled magnetic system with a magnetic field of 1.8 T; the frequency of change in the magnetic fields was 0.2 Hz during all the experiments. For the investigation, samples in the shape of plates with a size of $2\,\mathrm{mm} \times 2\,\mathrm{mm} \times 0.5\,\mathrm{mm}$ were cut. In order to indentify the magnetocaloric effect anisotropy, plates of the treated alloy were cut in the two orthogonal directions: parallel and perpendicular to the drawing axis.

2. Results and Discussion

2.1. Microstructure of the alloy in the initial as-cast and in the treated states

Fig. 1 demonstrates results of EBSD scanning of local areas on the surface of the sample in the as-cast and in the treated states. EBSD map of the microstructure area of the sample in the initial as-cast state is shown in Fig. 1, a.

A survey of the map was made at an area of $1\,\mathrm{mm} \times 1\,\mathrm{mm}$ with a scanning step of 10 μm. The colors correspond to the different crystallographic orientations (the legend is indicated in the top right corner of the figure). It can be seen that the equiaxial grains have a size from units to several hundreds of microns and different crystallographic orientation. The grain boundaries map of the same area is shown in Fig. 1, b. The misorientation of the adjacent scanning points from $2\,^\circ$ to $15\,^\circ$ is indicated in red (low-angle boundaries), and of those above 15° are in black (high-angle misorientations). The absence of low-angle misorientations in the grain body points to the absence of a substructure and internal stresses. This is caused by the slow crystallization of the melt during remelting and its slow cooling to the room temperature.

EBSD map of the alloy in the treated state is shown in Fig. 1, c. The sample for the investigation was cut in a way that the drawing axis is parallel to the section surface and is oriented horizontally in the figure. Scanning area was $2.25\,\mathrm{mm} \times 2.25\,\mathrm{mm}$ with a scanning step of 3 μm. As it can be seen, coarse grains are surrounded with a layer of a fine-grained structure, the thickness of which is only several grains. The general arrangement of the figure points to the presence of an insignificant metallographic texture. The grains elongated during drawing along its axis. The presence of a blurred contrast in the grain body points to the presence of a substructure, for defining of which a misorientation map was compiled (Fig. 1, d). As in the previous case, low-angle misorientations are indicted in red (2°–15°), high-angle misorientations are in black. It can be seen that there is a large number of low-angle boundaries in the grain body. This indicates the presence of a substructure and internal stresses, which were formed during forging. The presence of internal stresses in the microstructure has a positive effect on

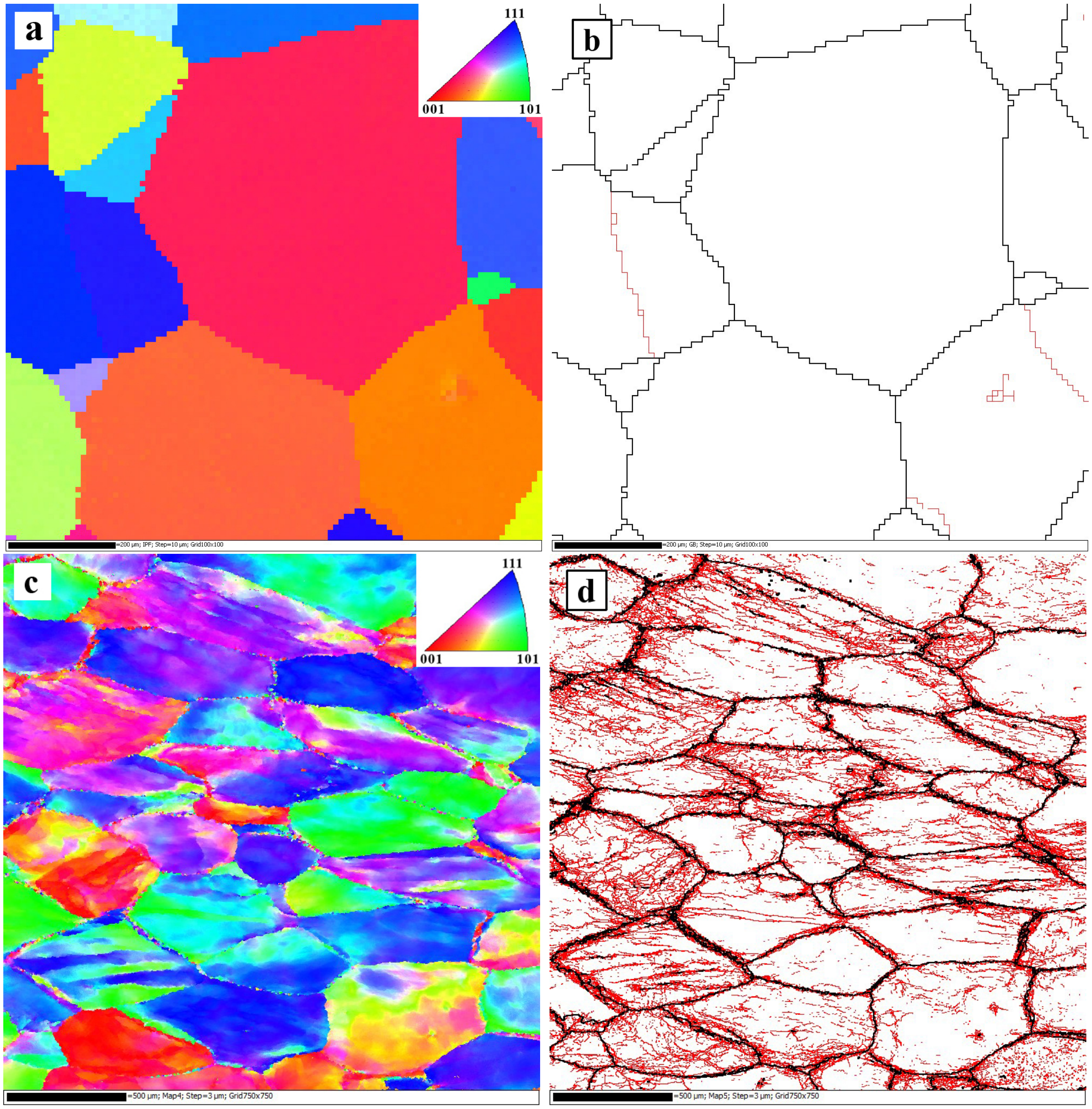

Fig. 1. EBSD mode microstructure maps of the alloy in the as-cast (a, b) and in the treated (c, d) states: a, c — orientation maps in the inverse pole figure mode, b, d — misorientation maps of low-angle boundaries (in red) and high-angle boundaries (in black)

the thermal expansion anisotropy in Heusler alloys of this system. It is necessary to define the influence on the value of the magnetocaloric effect.

2.2. Thermal expansion in the region of martensitic transformation

Fig. 2 demonstrates results of the temperature dependence of thermal expansion of the alloy in different states during heating and cooling of the samples within the temperature range from 173 K to 250 K.

The sample of the alloy in the as-cast state was cut across the radial component of the ingot. In such sample, in the region of the martensitic transformation, a stepwise length shrinking during the direct transformation is observed (Fig. 2, a). During the reverse transformation a stepwise elongation is observed. Phase transformation points have the following values: $M_S = 204$ K; $M_F = 194$ K; $A_S = 207$ K; $A_F = 214$ K. Notably, the contribution of the martensitic transformation to the change of the sample length equals approximately 0.03 %. The stepwise change of the sample length at the direct phase transformation is caused by a formation of a preferred orientation of the

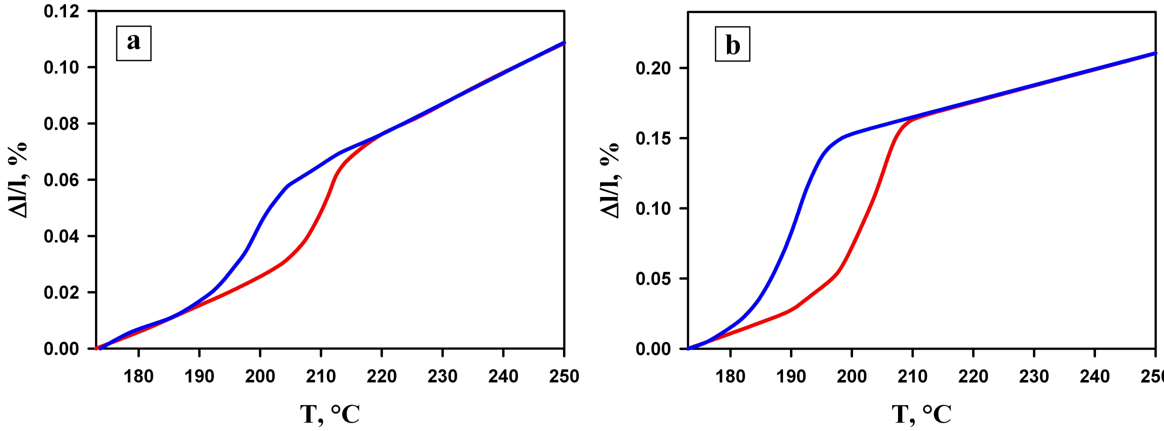

Fig. 2. The temperature dependence of thermal expansion of the sample of the alloy in the initial as-cast state (a) and of the sample after forging (b, across the drawing axis)

martensitic plates during the phase transformation. In this case, the insignificant value of the step is explained by the insignificant texture of the crystal growth, since during the vacuum remelting a very slow crystallization of the melt took place.

To investigate the alloy in the treated state the sample was cut with its long side across the drawing axis. In such sample during the direct martensitic transformation a stepwise shrinking of the length at the direct transformation and an elongation during the reverse transformation are observed. At the same time, the phase transformation points a shift to the low temperature range by 10 K. Unlike the as-cast state, the contribution to the length change during the martensitic transformation equals 0.13%. The growth of the stepwise length change a value indicates the formation of deformation texture in the material and increase of the fraction of martensitic plates with a preferred orientation. It should be noted that, according to the literature, the value of the step is close to that of the as-cast alloys, obtained by the regular argon arc melting.

2.3. Magnetocaloric effect in the initial as-cast and the treated states

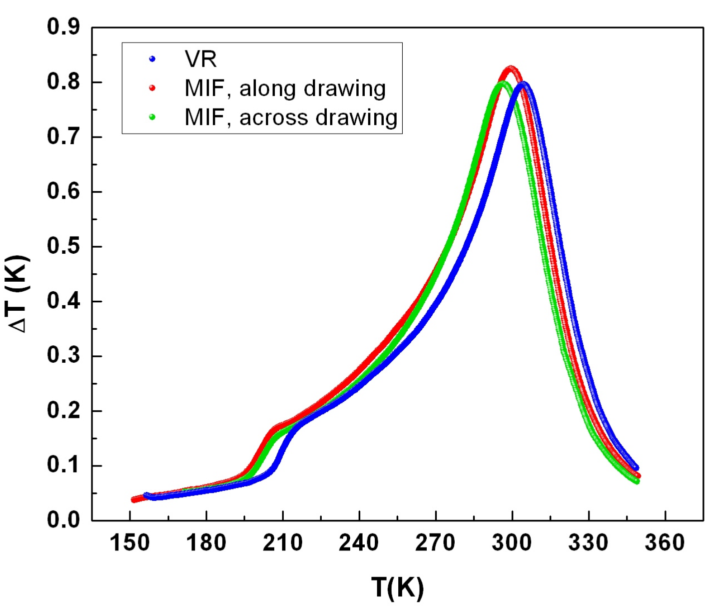

Fig. 3. Magnetocaloric effect in the alloy in the initial as-cast state (blue curve), in the state after forging on the sample, cut along (red curve) and across (green curve) the drawing axis

Fig. 3 demonstrates the investigation results of the magnetocaloric effect in the alloy in the initial as-cast and the treated states. The magnetocaloric effect was measured under the following protocol: the sample was cooled to the temperature below the martensitic transformation end point, and then an alternating magnetic field of 1.8 T was applied to it at a constant heating speed.

As it can be seen in the figure, the property of the $\Delta T(T)$ curves for the as-cast and the treated state is identical. In the region of the martensitic transformation a direct magnetocaloric effect of approximately 0.15 K is observed. In the Curie point region its value reaches 0.9 K. The difference between the curves of the alloy in the as-cast and in the treated state is that the $\Delta T\,(T)$ dependence shifts to the low temperature range by the value of approximately 10 K. This is caused by the shift of the martensitic transformation temperature, which was observed on the $\Delta l/l\,(T)$ curves. Thus, resulting from TMT by MIF, the value of the magnetocaloric effect does not decline and stays similar to the as-cast state. Furthermore, as it has been mentioned above, a binary microstructure, obtained by forging, is expected to contribute to the enhancement of performance properties of the material.

Conclusion

The study received results of the influence of the microstructure of $Ni_{2.16}Mn_{0.79}Ga_{0.98}Si_{0.07}$ alloy on the magnetocaloric effect in the region of the martensitic and magnetic phase transformations. It shows that due to multiple isothermal forging of the alloy at 953 K with a true degree of deformation $e = 1.9$ a binary microstructure is formed. In this microstructure initial coarse grains of approximately 100 μm are surrounded with a layer of a fine-grained structure. Such structure type is expected to enhance performance properties of Heusler alloys. Investigation of thermal expansion of the alloy in the region of martensitic transformation indicates that anisotropy of properties is formed in the material. Analysis of magnetocaloric effect in the alloy in different structural states shows that in the region of the Curie point its value reaches 0.9 K in the magnetic field of 1.8 T. In the region of martensitic transformation it equals 0.15 K. Moreover, a shift of the magnetocaloric effect value to the low temperature range is observed, which is explained by the shift of martensitic transformation itself in the alloy in the treated state. An important result is the absence of degradation of functional properties of the material, subjected to thermo-mechanical treatment, which, in its turn, is expected to enhance performance properties of the material.

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МАГНИТОКАЛОРИЧЕСКИЙ ЭФФЕКТ В СПЛАВЕ СИСТЕМЫ Ni-Mn-Ga-Si, ПОДВЕРГНУТОМ ПЛАСТИЧЕСКОЙ ДЕФОРМАЦИИ МЕТОДОМ ВСЕСТОРОННЕЙ ИЗОТЕРМИЧЕСКОЙ КОВКИ

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В сплавах Гейслера в области мартенситного и магнитного фазовых превращений наблюдается магнитокалорический эффект, что позволяет рассматривать их как перспективные материалы для применения в качестве твердотельного хладагента в охлаждающих устройствах. Однако данные сплавы обладают пониженными эксплуатационными свойствами. В работе представлены результаты исследования магнитокалорического эффекта в сплаве системы Ni-Mn-Ga в двух структурных состояниях: исходном литóм и подвергнутом деформационной обработке методом всесторонней изотермической ковки при температуре 953 К. Показано, что в литóм состоянии микроструктура сплава представляет собой равноосные зёрна размером несколько сотен микрон. В результате ковки формируется двухкомпонентная микроструктура, в которой крупные исходные зёрна окружены прослойкой мелкозернистой структуры. Исследование температурной зависимости термического расширения показывает, что мартенситное превращение в сплаве в литóм состоянии протекает в интервале температур от 194 до 214 К. В деформированном состоянии фазовое превращение в сплаве смещается в область отрицательных температур на 10 К. В обоих состояниях сплава в области мартенситного превращения величина магнитокалорического эффекта составляет 0,15 К в магнитном поле 1,8 Тл, а в области точки Кюри — 0,9 К. Отличие деформированного состояния заключается только в смещении зависимости $\Delta T\,(T)$ в область низких температур на 10 К.

Ключевые слова: *сплав Гейслера, мартенситное превращение, магнитокалорический эффект, всесторонняя изотермическая ковка.*

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Сведения об авторах

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