Magnetic properties and magnetostructural phase transitions in Ni$_{2+x}$Mn$_{1-x}$Ga shape memory alloys

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A systematic study of magnetic properties of Ni$_{2+x}$Mn$_{1-x}$Ga ($0 \leq x \leq 0.19$) Heusler alloys undergoing structural martensite-austenite transformations while in ferromagnetic state has been performed. From measurements of spontaneous magnetization, $M_s(T)$, jumps $\Delta M$ at structural phase transitions were determined. Virtual Curie temperatures of the martensite were estimated from the comparison of magnetization in martensitic and austenitic phases. Both saturation magnetic moments in ferromagnetic state and effective magnetic moments in paramagnetic state of Mn and Ni atoms were estimated and the influence of delocalization effects on magnetism in these alloys was discussed. The experimental results obtained show that the shift of martensitic transition temperature depends weakly on composition. The values of this shift are in good correspondence with Clapeyron-Clausius formalism taking into account the experimental data on latent heat at martensite-austenite transformations.

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I. INTRODUCTION

In Ni$_3$MnGa Heusler alloy, a structural transformation from cubic austenitic to tetragonal martensitic phase is observed upon cooling. The interest in the study of Ni$_3$MnGa-based alloys has mainly been conditioned by the fact that the martensitic phase in these alloys is ferromagnetic. The combination of ferromagnetic ordering and martensitic transformation allows realization of magnetically driven shape memory effect, which expands considerably the area of technical applications of this effect.

Despite a large number of experimental and theoretical studies, many fundamental aspects of Ni$_3$MnGa-based alloys are not clearly understood yet. For instance, magnetic properties of thoroughly studied Ni$_{2+x}$Mn$_{1-x}$Ga system were not sufficiently clarified. For these alloys, the compositional dependencies of Curie temperature $T_C$ and martensite-austenite transformation temperature $T_m$ were determined but the temperature and compositional dependencies of magnetization have been not investigated in details. In particular, no systematic study was performed on the jump of magnetization at martensitic transition, which determines the shift of $T_m$ under external magnetic field. Besides, the exchange interaction parameters have been not estimated for these alloys. All these factors are important to get a better insight into physical mechanisms, underlying the magnetically driven shape memory effect. This paper deals with a systematic study of magnetic properties of Ni$_{2+x}$Mn$_{1-x}$Ga ($0 \leq x \leq 0.19$) alloys.

II. CRYSTAL STRUCTURE AND MAGNETIC PROPERTIES OF Ni$_{2+x}$Mn$_{1-x}$Ga SYSTEM

The high-temperature austenitic phase of Ni$_{2+x}$Mn$_{1-x}$Ga Heusler alloys has a cubic structure of Fm3m space group. A structural transition to a modulated tetragonal ($c/a < 1$) phase is observed in these alloys on cooling. It is worth noting that the crystal structure and space group of the low-temperature phase is still a subject of controversy (see, for example, Refs. 1,2) which is aggravated by a compositional dependence of the crystal structure of martensite. Thus, for example, recent results of high-resolution neutron diffraction give ground to conclude that for the stoichiometric Ni$_2$MnGa composition the martensitic phase, being considered for a long time as a tetragonal, has indeed an orthorhombic symmetry of Pnma space group. The structural martensitic transformation in Ni$_3$MnGa-based Heusler alloys was described as driven by a band Jahn-Teller effect.

The martensitic transformation temperature $T_m$, which is about 200 K in stoichiometric Ni$_3$MnGa, linearly increases with $x$ in Ni$_{2+x}$Mn$_{1-x}$Ga alloys and reaches about 330 K at $x = 0.18 − 0.19$ (Ref. 3). The alloys with a higher Ni content were not studied so far. Note that different values of $T_m$ are given in literature, indicating probably the sensitivity of physical properties of these alloys to structural disorder and/or deviations from the nominal composition.

The Ni$_{2+x}$Mn$_{1-x}$Ga alloys are ferromagnetic at low
temperatures. The Curie temperature $T_C$ is about 370 K in stoichiometric composition ($x = 0$). $T_C$ approximately linearly decreases with increasing Ni content, so that for $x = 0.18 - 0.19$ Curie temperature merges with the martensitic transformation temperature $T_m$. Hence, the alloys with $x = 0.18 - 0.19$ experience a structural (martensitic) transition from paramagnetic austenite to ferromagnetic martensite. At the same time, the magnetic state of the alloys with a lower Ni content does not change during martensitic transformation and both austenitic and martensitic phases are ferromagnetic. The martensitic transformation, however, influences the magnetic parameters of these alloys and reveals itself in a sharp change of magnetic anisotropy and magnetization saturation.

The neutron diffraction measurements of stoichiometric composition$^{10}$ show that the magnetic moment is localized mainly on Mn atoms. The reported values of the Mn magnetic moment range from 3.8 to 4.2 $\mu_B$. The magnetic moment of Ni atoms is considerably smaller, about 0.2-0.4 $\mu_B$. The concentration dependence of magnetic moment in Ni$_{2+x}$Mn$_{1-x}$Ga alloys has been not reported. It is known only that magnetization saturation decreases with increasing $x$.

III. SAMPLES PREPARATION AND MEASUREMENTS

Polycrystalline samples of Ni$_{2+x}$Mn$_{1-x}$Ga alloys were prepared by a conventional arc-melting method in the atmosphere of spectroscopically pure argon gas. The samples were homogenized at 1050 K for 9 days with subsequent quenching in ice water. For the measurements of physical properties those samples were used whose weight loss during arc-melting was less than 0.2%. The measurements of magnetic properties were performed on samples with $x = 0$, 0.04, 0.08, 0.12, 0.16, and 0.19; some measurements were also done on the sample with $x = 0.02$.

The magnetization up to 5 T was measured in a temperature range 5 – 700 K by a SQUID magnetometer "Quantum Design"; it was also measured by a vibrating sample magnetometer (VSM) in magnetic fields up to 1.8 T. Additionally, measurements in pulsed magnetic fields up to 10 T were performed. Spontaneous magnetization $M_s$ at low temperatures was determined by linear extrapolation of $M(H)$ dependencies from high fields. $M_s$ in the vicinity of Curie temperature, where $M(H)$ dependencies are non-linear, was estimated by Belov-Arrott method for second-order magnetic phase transitions. Using this method, the Curie temperatures were determined for every alloys except the $x = 0.19$ sample, where the ferromagnetic-paramagnetic transition is a first-order phase transition. The paramagnetic susceptibility of the alloys was defined from $M(T)$ dependencies measured above $T_C$ up to 700 K in a magnetic field of 0.2 T.

The latent heat of martensitic transition was determined from differential scanning calorimetry, performed by a Pyris-1 DSC equipment.

IV. EXPERIMENTAL RESULTS

Temperature dependencies of spontaneous magnetization $M_s$ of the Ni$_{2+x}$Mn$_{1-x}$Ga alloys are shown in Fig. 1. It is seen that $M_s$ gradually decreases with increasing temperature and exhibits a pronounced change (smeared jump) when approaching a certain temperature $T_m$. This jump in magnetization is caused, as has been shown in numerous studies,$^{10,13,14}$ by a structural phase transition from martensite to austenite. As evident from these measurements, the austenitic phase is ferromagnetic above $T_m$, for $x < 0.19$, while in the $x = 0.19$ alloy the transformation from martensite to austenite is accompanied by a transition from ferromagnetic to paramagnetic state. The compositional dependence of the martensitic transformation temperature $T_m$ is shown in Fig. 2. This figure also shows the dependence of ferromagnetic ordering temperature $T_C$ on Ni content $x$. It is seen that both these dependencies are practically linear with $T_m$ increasing and $T_C$ decreasing with Ni content. These
FIG. 2: Compositional dependencies of martensitic transformation temperature $T_m$, Curie temperature $T_C$ and paramagnetic Curie temperature $\Theta$.

FIG. 3: Compositional dependencies of saturation magnetic moment $M_s(0)$ and effective magnetic moment $\mu_{\text{eff}}$ of the Ni$_{2+x}$Mn$_{1-x}$Ga alloys.

FIG. 4: Magnetization jump at the martensitic transition in magnetic fields of 3 T (dashed line) and 5 T (solid line). For compositions $0 \leq x \leq 0.16$ $M(T)$ dependencies were measured upon heating. For the composition $x = 0.19$ a temperature hysteresis loop of the magnetization observed at martensitic transition is shown. The inset shows temperature derivatives of magnetization for Ni$_2$MnGa measured in magnetic fields 3 and 5 T. Temperatures merge in a range of $x = 0.18 - 0.19$. The phase diagram of the Ni$_{2+x}$Mn$_{1-x}$Ga system obtained in the present study is in good agreement with previously found ones.

The magnetic moment of these alloys, $M_s(0)$, was obtained by extrapolation of $M_s(T)$ to 0 K. It was found that $M_s(0)$ approximately linearly decreases at substitution of Mn by Ni, as is shown in Fig. 3. The value of magnetic moment in the stoichiometric Ni$_2$MnGa appears to be close to those reported in others studies.

The $M_s(T)$ dependencies in Ni$_{2+x}$Mn$_{1-x}$Ga alloys, shown in Fig. 1, evidence that the change of spontaneous magnetization at martensite-austenite transformation increases with Ni content. A jump of magnetization at this transition is also observed in magnetic fields larger than the saturation field, as is shown in Fig. 4. The compositional dependencies of the magnitudes of magnetization jump $\Delta M$ measured in various magnetic fields are shown in Fig. 5.

As can be seen from Fig. 4, with increasing magnetic field the magnetization jumps shift to higher temperatures. This is due to the influence of magnetic field on martensitic transformation temperature. It follows from these measurements that the shift $\Delta T$ of $T_m$ under magnetic field increases weakly with Ni content (see Table 1).

The influence of a magnetic field on the martensite-austenite transition temperature was studied only for $x = 0$ (Ref. [14]) and $x = 0.18 - 0.19$ (Refs. [15, 16, 17]) com-
TABLE I: Theoretical and experimental values of the shift $\Delta T$ of the martensitic transition temperature $T_m$ in a magnetic field $\Delta H = 2$ T for $\text{Ni}_{2+x}\text{Mn}_{1-x}\text{Ga}$.

| $x$ | $Q$ (J/mol) | $T_m$ (K) | $\Delta M$ ($\mu_B$) ($\Delta H = 2$ T) | $\Delta T$ theory (K) | $\Delta T$ experiment (K) |
|-----|-------------|-----------|--------------------------------------|-----------------------|---------------------------|
| 0   | 270         | 201       | 0.1                                  | 0.82 $\pm$ 0.2        | 0.8 $\pm$ 0.5             |
| 0.04| 600         | 237       | 0.17                                 | 0.75 $\pm$ 0.2        | 0.95 $\pm$ 0.5            |
| 0.08| 910         | 265       | 0.28                                 | 0.92 $\pm$ 0.2        | 0.95 $\pm$ 0.5            |
| 0.12| 1250        | 294       | 0.41                                 | 1.07 $\pm$ 0.2        | 1.10 $\pm$ 0.5            |
| 0.16| 1710        | 315       | 0.62                                 | 1.28 $\pm$ 0.2        | 1.30 $\pm$ 0.5            |
| 0.19| 2260        | 342       | 0.96                                 | 1.62 $\pm$ 0.2        | 1.60 $\pm$ 0.5            |

positions. For the stoichiometric composition the shift of $T_m$ under magnetic field was estimated as $dT_m/dH \approx 0.2$ K/T. For the $x = 0.18$ and $x = 0.19$ compositions, $dT_m/dH \approx 1$ K/T was reported in Refs. 15,17, whereas in Ref. 16 this quantity was estimated as 3.5 K/T. It is worth noting that the shift of $T_m$ is determined with a significant error. This is caused mainly by the fact that the jump of magnetization at martensitic transformation is broad which makes difficult correct determination of $T_m$ temperature. Besides, martensitic transformation is a first-order structural phase transition and is characterized by a temperature hysteresis. Therefore, $T_m$ temperature can differ from the temperature at which the jump of magnetization occurs. The most correct method to determine $T_m$ is to determine this temperature as the average of the temperatures, at which magnetization jump is observed on cooling and heating, respectively. In the present study, $T_m$ was determined as a temperature of the magnetization jump while heating the sample. The temperature hysteresis loop was measured for the $x = 0.19$ sample. It was found (see Fig. 4) that $T_m$ determined at increasing temperature differs from $T_m$ estimated from averaging of measurements in hysteretic regime by 2–3 K. The width of the temperature hysteresis loop is approximately the same in different magnetic fields, so the additional error in determination of the shift of $T_m$ caused by a magnetic field does not exceed 0.3 K.

Temperature dependencies of reciprocal paramagnetic susceptibility of the $\text{Ni}_{2+x}\text{Mn}_{1-x}\text{Ga}$ system are shown in Fig. 6. In the temperature range studied, the susceptibility follows Curie-Weiss law. The compositional dependencies of paramagnetic Curie temperature $\Theta$ and effective magnetic moment $\mu_{\text{eff}}$ are shown in Figs. 2 and 3, respectively. Clearly, both these parameters decrease monotonously with increasing $x$. The paramagnetic susceptibility was measured earlier in stoichiometric $\text{Ni}_2\text{Mn} \text{Ga}$ alloy only12,14. The values of $\Theta$ and $\mu_{\text{eff}}$ obtained are somewhat larger than the reported previously. This difference can be due to the fact that the present measurements were performed in a wider temperature interval.

The compositional dependence of the latent heat $Q$ of the martensite-austenite phase transition is shown in Fig. 7. Evidently, $Q$ strongly increases with increasing $x$. These results are in good agreement with recently published ones.

V. DISCUSSION

Based on the compositional dependencies of saturation magnetic moment and effective magnetic moment of $\text{Ni}_{2+x}\text{Mn}_{1-x}\text{Ga}$ alloys (Fig. 3), the magnetic moments and effective magnetic moments of Mn and Ni atoms were calculated from the equations.

![FIG. 6: Temperature dependencies of paramagnetic susceptibility in $\text{Ni}_{2+x}\text{Mn}_{1-x}\text{Ga}$ alloys.](image)

![FIG. 7: The magnetization jump at martensitic transition in various magnetic fields as a function of Ni concentration in the $\text{Ni}_{2+x}\text{Mn}_{1-x}\text{Ga}$ alloys.](image)
TABLE II: Magnetic moments $\mu$ and effective magnetic moments $\mu_{\text{eff}}$ of Mn and Ni atoms.

|     | $\mu$ ($\mu_B$) | $\mu_{\text{eff}}$ ($\mu_B$) | $\mu_{\text{loc}}^{\text{Mn}}$ ($\mu_B$) | $\mu_{\text{loc}}^{\text{Ni}}$ ($\mu_B$) | $\mu_{\text{loc}}^{\text{Mn}} / \mu_{\text{eff}}$ |
|-----|-----------------|-------------------------------|----------------------------------------|----------------------------------------|----------------------------------------|
| Mn  | 2.99 ± 0.32     | 4.43 ± 0.13                  | 3.86 ± 0.14                            | 0.87 ± 0.11                            |
| Ni  | 0.43 ± 0.14     | 1.35 ± 0.18                  | 1.05 ± 0.21                            | 0.77 ± 0.10                            |

\begin{equation}
M_s(0) = (1-x)\mu_{\text{Mn}} + (2+x)\mu_{\text{Ni}} \quad (1)
\end{equation}

\begin{equation}
\mu_{\text{eff}} = \sqrt{(1-x)\mu_{\text{eff}}^{\text{Mn}} + (2+x)\mu_{\text{eff}}^{\text{Ni}}} \quad (2)
\end{equation}

The results of these calculations are presented in Table 2. The obtained values of the magnetic moments of the constituting atoms are in good accordance with the results of neutron diffraction and nuclear magnetic resonance studies for the stoichiometric composition. It was shown\textsuperscript{14,20} that in Ni$_2$MnGa the Mn magnetic moment is about 2.84 – 3.41$\mu_B$ and the Ni magnetic moment is about 0.3 – 0.41$\mu_B$.

Note that these calculations are based on assumptions that the magnetic moments of constituting atoms does not change with deviations from stoichiometry and that the Ni atoms possess similar moments in different crystallographic sites. In general this is not the case, because magnetism of Heusler alloys is described in a band model. It means that the values of magnetic moments depend on density of states at Fermi level and on the exchange splitting parameter, being therefore the concentration and structure dependent values. As has been noted in Ref.\textsuperscript{21} in Ni$_2$MnX Heusler alloys the distance between the atoms is sufficiently large so that direct overlap of electron orbitals is negligible and the delocalization effects are of secondary importance. Therefore, in the first approximation a localized moments model is applicable for the description of magnetic properties of these alloys. However, from the results of magnetic and nuclear magnetic resonance measurements of Ni$_2$MnGa\textsuperscript{20} it was concluded that in this alloy the Mn magnetic moments are mainly localized, while Ni magnetic moments are essentially delocalized.

The character of magnetism can be judged from the comparison of the magnetic moments of the constituting atoms and their effective magnetic moments (see Table 2).

In the model of localized magnetic moments for the spin-only state (orbital moment is quenched) the interrelation between effective magnetic moment and the moment in the magnetically ordered state is given by Wohlfarth-Rhodes equation

\begin{equation}
\mu_{\text{eff}}^{\text{loc}} = \sqrt{\mu(\mu + 2)} \quad (3)
\end{equation}

In the band model the value of $\mu_{\text{eff}}^{\text{loc}}$ calculated from Eqn. (3) should be smaller than the experimental value of the effective moment due to the influence of delocalization effects. The values of $\mu_{\text{eff}}^{\text{loc}}$ for the Mn and Ni subsystems are given in Table 2. As evident from these data, both Mn and Ni subsystems $\mu_{\text{eff}}$ and $\mu_{\text{eff}}^{\text{loc}}$ are close to each other, although in both cases $\mu_{\text{eff}}^{\text{loc}}$ is slightly smaller than $\mu_{\text{eff}}$. Within the experimental error of the measurements the $\mu_{\text{eff}}^{\text{loc}} / \mu_{\text{eff}}$ ratio is the same for both Mn and Ni subsystems. Thus, present experimental data do not suggest that the Ni subsystem is more delocalized than the Mn one.

It should be understood, however, that the magnetic moments in the magnetically ordered state were determined in the martensitic phase, whereas the effective magnetic moments were calculated from the paramagnetic susceptibility measured in the austenitic state. It makes no difference if magnetism is described in the localized model, because in this case the magnitude of magnetic moment depends weakly on the crystallographic environment. In the band model, magnetic moments depend on the degree of overlap of electron orbitals, which changes at structural transformation. Because of this, a possibility that the magnetic moments will change at structural transition must not be ruled out. The qualitative arguments given above are supported by the experimental data reported in Ref.\textsuperscript{20} which indicate that the magnetic moment of Mn is the same in austenitic and martensitic phases, whereas magnetic moment of Ni in austenitic phase is larger than that in the martensitic phase. The latter observation is conditioned by a higher density of states of Ni at the Fermi level in austenitic state than that in the martensitic state, as electronic structure calculations have revealed\textsuperscript{5}.

As evident from Fig. 2, Curie temperature of the
austenitic phase decreases at substitution of Mn for Ni. This is due to the fact that this substitution leads to an increase in the number of atoms with smaller magnetic moments. Similar tendency takes place presumably for a virtual Curie temperature of the martensitic phase. This follows from the observation that in the low-temperature martensitic phase magnetization of the Ni$_{2+x}$Mn$_{1-x}$Ga alloys with a higher $x$ decreases more rapidly with increasing temperature. The magnetization data shown in Fig. 1 allow estimation of virtual Curie temperature of the martensitic phase. Fig. 8 shows temperature dependencies of reduced spontaneous magnetization $m = M_s(T)/M_s(0)$ of the alloys as a function of reduced temperature $t = T/T_C$. It is seen that the magnetization of austenite phase and the magnetization of martensitic phase change with temperature in different way, whereas the reduced magnetizations of these phases are similar for different compositions. It can be assumed that the difference in $m(t)$ of martensite and austenite is due to the difference in their Curie temperatures. Comparing $m(t)$ dependencies of martensitic and austenitic phases, it is possible to reconstruct the virtual Curie temperature of martensitic phase, which is shown by the solid line in Fig. 8. It appears to be 17% higher than the Curie temperature of austenite state. This value is twice as large as that obtained from phenomenological Landau theory.

The larger value of Curie temperature of martensite as compared to Curie temperature of corresponding austenite is due to changes in interatomic distances and in overlap of electronic orbitals. As evident from the analysis of experimental data, this effect cannot be attributed solely to a change in the unit cell volume at martensitic transformation. Indeed, a study of the influence of hydrostatic pressure on Curie temperature $T_C$ and martensitic transformation temperature $T_m$ of stoichiometric Ni$_2$MnGa has shown that the exchange interaction of the austenite increases with decreasing unit cell volume. At the same time, it is known that the unit cell volume of martensite is larger than that of austenite. Therefore, it seems likely that the primary role in martensitic transformation in Ni$_2$MnGa Heusler alloys belongs to the crystal lattice distortions. Such a mechanism of the influence of a structural transition on exchange interaction in intermetallic compounds Gd$_5$(Si$_x$Ge$_{1-x}$)$_4$ was discussed recently in Ref. 23.

As evident from Fig. 5, the magnitude of magnetization jump $\Delta M$ at structural transition strongly increases with Ni content. This is caused by the fact that the increase of $x$ leads to the increase of $T_m$. Under these circumstances, the difference between magnetizations of martensite and austenite at $T_m$ increases as well. It is also seen from Fig. 5 that the magnetization jumps $\Delta M$ at $T_m$ diminishes at increasing magnetic field, which is the most pronounced at high $x$. The behavior of $\Delta M$ in the alloys with a small $x$ results from the fact that the martensitic transformation in these alloys occurs at temperatures far below Curie temperature $T_C$ and therefore the influence of a magnetic field on magnetization is weak. In the alloys with a large $x$, $T_m$ is close to $T_C$ of the austenitic phase and the external field strongly affects magnetization of this phase, whereas magnetization of the martensitic phase depends weakly on the magnetic field.

It has been already mentioned that the temperature of structural transition shifts to higher temperatures upon application of a magnetic field. Such behavior is governed by the influence of Zeeman energy, which stabilizes martensitic phase with a larger magnetization. Experimental data on the shift of $T_m$ are presented in Table 1. These results indicate that for the alloys studied the shift is rather small (1–2 K as the magnetic field changes for 2 T) and slightly enhances with increasing Ni content. The table also contains theoretical estimation of the shift of $T_m$ in magnetic field, derived from a thermodynamical Clapeyron-Clausius relation for first-order phase transitions:

$$\Delta T = \Delta M H T_m / Q.$$  

The agreement between experimental and theoretical values can be considered as satisfactory.

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