Magnetic and Magnetocaloric Properties of Spin-glass Material DyNi_{0.67}Si_{1.34}

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Abstract

Structural, magnetic and magnetocaloric properties of DyNi_{0.67}Si_{1.34} were investigated using x-ray powder diffraction, magnetic susceptibility and magnetization measurements. X-ray powder diffraction pattern shows that DyNi_{0.67}Si_{1.4} crystallizes in the AlB₂-type hexagonal structure (space group: *P6/mmm*, No.191, $a = b = 3.9873(9)$ Å, and $c = 3.9733(1)$ Å). The compound is a spin-glass with the freezing temperature $T_G = 6.2$ K. The ac magnetic susceptibility measurements confirm magnetic frustration in DyNi_{0.67}Si_{1.34}. The maximum value of the magnetic entropy change determined from M(H) data is -16.1 J/kg K at 10.5 K for a field change of 70 kOe.

Keywords: Rare earth intermetallics; Magnetic frustration; Magnetocaloric effect.

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I. INTRODUCTION

One of the most prominent features of many R-M-X systems (where R = rare earth element, M = transition metal, and X = *p*-element) is a large number of the ternary intermetallic compounds crystallizing in a variety of crystal structures. These ternary compounds often reveal intriguing and important physical properties, particularly magnetic. For example, Nd₂Fe₁₄B is the base compound for the broadly used family of permanent magnets ^[1-2], while LaFe_{13-x}Si_x-based alloys and their hydrides are considered as promising magnetic refrigeration materials ^[3-4].

In the ternary Dy-Ni-Si system, a total of 23 compounds (among them 20 with known crystal structures) have been reported ^[5-6]. The magnetic property studies showed that the magnetic ordering transition from the paramagnetic (PM) to ferromagnetic (FM) states occur at low temperatures in DyNi₄Si ($T_C=15.6$ K) ^[7], DyNi₂Si ($T_C=16$ K) and Dy₃Ni₈Si ($T_C=17$ K) ^[5]. However, in DyNi₄Si the net moment is not fully saturated even in 140 kOe applied magnetic field (at $T = 2$ K) suggesting potentially non-collinear magnetic structure due to strong magnetocrystalline anisotropy ^[7]. No clear conclusion regarding the value of the Ni moment in DyNi₂Si and Dy₃Ni₈Si was obtained, but the effective moments – 10.75 and 10.59 μ_B /Dy, respectively^[5] – are close to 10.65 μ_B expected for non-interacting Dy³⁺ ions, indicating that Ni 3d band is nearly completely filled. In other studied Dy-Ni-Si compounds the PM - antiferromagnetic (AFM) transitions were observed at different Néel temperatures (T_N). Usually, T_N s of these compounds are low except for the Dy₃NiSi₃ which has T_N of 140 K ^[8-14].

The magnetic properties of the AFM-ordered Dy-Ni-Si compounds are interesting and point to complex magnetism. For example, DyNiSi exhibits a square wave-modulated magnetic structure at 1.5 K, which at 3 K transforms into a sine wave-modulated magnetic structure stable up to the Néel point at 8.8 K ^[8]. DyNiSi₂ has a two-step magnetic ordering, where $T_N=24$ K is closely followed by a second transition at 22 K, and a complex magnetic structure with the *c*-axis as the easy

magnetization direction^[9]. DyNi₂Si₂ has a T_N of 5.6 K and an order-to-order phase transition at about 3.5 K, which is an unusual first-order transition from a commensurate square-wave-type modulated magnetic structure below T_N to a ground state incommensurate sine-wave type magnetic structure^[14]. Dy₂Ni₃Si₅ orders antiferromagnetically at 9.5 K^[12].

Therefore, quite a few compounds from the Dy-Ni-Si system show interesting magnetic behavior yet no spin-glass system has been reported so far. To the best of our knowledge, magnetic properties of Dy-Ni-Si alloys that crystallize in a simple AlB₂-type structure have not been examined to date. Here we report a detailed study of structural, magnetic and magnetocaloric properties of one of the AlB₂-type intermetallics – DyNi_{0.67}Si_{1.34}.

II. EXPERIMENT

Approximately 4 grams of polycrystalline DyNi_{0.67}Si_{1.34} material were prepared by using Dy obtained from the Materials Preparation Center of the Ames Laboratory (99.98 wt.% pure with respect to all other elements in the periodic table), and Ni (99.95 wt. %) and Si (99.999 wt. %) that both were obtained from Alfa Aesar. The components weighed in stoichiometric proportions were arc-melted on a water-cooled copper hearth under high purity argon atmosphere. The button was turned over and re-melted six times to ensure homogeneity. The sample was then wrapped in a Ta foil, sealed in a helium-filled quartz tube, and annealed at 870 K for 100 hours. Powder X-ray diffraction (XRD) was employed to perform the phase analysis and determine the lattice parameters (Philips X'Pert Pro diffractometer with Cu $K_{\alpha 1}$ radiation). The microstructure and EDS measurements were carried out on an FEI Teneo SEM equipped with an Oxford Instruments Aztec EDS system. The sample was embedded in epoxy and then ground and polished through 1 μ m diamond slurry. EDS analysis were carried out from one end to other end or center to edge of sample to check the concentration gradient throughout the sample. Magnetic measurements of bulk

polycrystalline sample were performed using a superconducting quantum interference device (SQUID) magnetometer (MPMS XL, Quantum Design, USA) in the temperature range from 2 to 300 K and in magnetic fields from 0 to 70 kOe. The ac magnetic susceptibility was measured in a zero bias dc field with 5 Oe ac driving field using 1, 10, 100, and 1000 Hz ac frequencies from 4 to 8 K.

III. RESULTS AND DISCUSSION

The AlB₂-type structure is stable over an extended range of concentrations with variable Ni and Si contents – Dy₃₄Ni₁₆₋₂₇Si₅₀₋₃₉ at 1070 K^[5,15]. In this work, we chose to study one composition close to the middle of the solid solution, i.e. DyNi_{0.67}Si_{1.34} (Dy_{33.3}Ni_{22.2}Si_{44.4}). The phase purity of the sample and its crystal structure were first verified by the Rietveld refinement of the powder XRD data, as shown in Fig.1. These data indicate that the DyNi_{0.67}Si_{1.34} phase has the AlB₂ hexagonal crystal structure (space group: *P6/mmm*, No.191) and its actual Ni/Si ratio is slightly rich in Ni compared to the starting composition. The lattice parameters are $a = b = 3.9873(9)$ Å and $c = 3.9733(6)$ Å. According to scanning electron microscopy and energy dispersive spectroscopy (EDS) analyses the sample contains a very minor amount of a second phase (DyNi₂Si₂), and the Ni/Si ratio of the main AlB₂-type phase corresponds to the nominal, i.e. Ni/Si = 0.34/0.66 as determined from EDS. A few vol. % of the impurity phase – DyNi₂Si₂, which is weakly magnetic (AFM) – does not affect magnetization data of DyNi_{0.67}Si_{1.34} presented below.

Fig. 1. The x-ray powder diffraction pattern of $\text{DyNi}_{0.67}\text{Si}_{1.34}$ sample.

The temperature dependences of magnetization measured under zero-field-cooled-warming (ZFC), field-cooled-cooling (FCC), and field-cooled-warming (FCW) protocols in magnetic field of 100 Oe are shown in Fig. 2. A distinct peak at 6.2 K is reminiscent of a transition to an AFM-like state upon cooling. A noticeable thermal hysteresis between ZFC and FC data is present, indicating a non-equilibrium state below 6.2 K potentially related to magnetic frustration, which can be expected considering trigonal-prismatic arrangement of the Dy atoms.

In the PM state, in the 6 - 300 K temperature range, the magnetic susceptibility obeys the Curie-Weiss law $\chi = C/(T - \theta_p)$ (inset in Fig. 2). The Weiss temperature (θ_p) is equal to -0.6 K pointing to a weakly magnetic ground state. The effective magnetic moment per Dy^{3+} ion is $10.73 \mu_B$, in good agreement with the calculated Dy^{3+} free ion value of $10.65 \mu_B$. Thus, we can assume that Ni is non-magnetic in $\text{DyNi}_{0.67}\text{Si}_{1.34}$, as it is commonly observed in numerous other R-Ni and R-Ni-X systems, e.g. in GdNi [16].

Fig. 2. The temperature dependence of the zero-field-cooled (ZFC) heating, field-cooled cooling (FCC), and field-cooled heating (FCW) magnetization of $\text{DyNi}_{0.67}\text{Si}_{1.34}$ in applied magnetic field of 100 Oe; the inset shows the inverse molar susceptibility fitted using the Curie-Weiss law.

In order to probe the ground state of $\text{DyNi}_{0.67}\text{Si}_{1.34}$ further, low temperature isothermal magnetization curves (M-H) were measured, which are shown in Fig. 3. The high-field magnetization is similar to a conventional ferromagnetic-like material, however the magnetization rise is slow, without clear domain-ordering process. Even at 70 kOe, the Dy moment is not saturated ($\mu_s \sim 6 \mu_B/\text{Dy}$ instead of the expected $gJ = 10 \mu_B$) indicating a non-collinear magnetic structure. Below 6 kOe, the 2 K magnetization is the lowest compared to 3, 4, and 5 K but at 70 kOe it becomes the largest. A weak but noticeable field-induced metamagnetic-like transition is observed at 2 and 3 K (inset to Fig. 3). The critical fields (H_c) of this transition at 2 and 3 K, obtained from the peak value of the dM/dH plots (not shown), are 2.5 and 1.5 kOe, respectively. These low critical fields indicate that if long-range AFM-like interactions are present in the $\text{DyNi}_{0.67}\text{Si}_{1.34}$, they are rather unstable. At and above 4 K, the metamagnetic-like transition is no longer seen. Therefore, it is difficult to assume a

specific type of magnetic order below T_N which further supports the hypothesis of the magnetically frustrated state in $\text{DyNi}_{0.67}\text{Si}_{1.34}$.

Fig. 3. The isothermal magnetization of $\text{DyNi}_{0.67}\text{Si}_{1.34}$ measured at 2-5 K up to 70 kOe magnetic field. The inset shows magnetic field dependence of magnetization in the 0-6 kOe range.

Figs. 4(a) and 4(b) show the temperature dependence of the ZFC magnetization measured in several applied magnetic fields (0.1 - 2 kOe and 2 – 15 kOe data are plotted separately for clarity). A broad maximum in the $M(T)$ curves is observed around 6.2 K in magnetic fields up to 6 kOe. However, these maxima are too broad for a typical long-range AFM ordering and the $M(H)$ data (Figure 3) do not support the AFM state. With increasing field to 10 kOe and above, the magnetization exhibits FM-like behavior although the transitions are very broad as well. The phase transition temperature defined as a minimum of dM/dT moves toward higher temperature, reaching 11 K at $H = 15$ kOe (Figure 4). This result shows that the ground state magnetism in $\text{DyNi}_{0.67}\text{Si}_{1.34}$ is not well defined; both the AFM and FM interactions exist in the magnetic structure of this alloy, which is characteristic for a spin-glass state.

Fig.4. The temperature dependence of ZFC magnetization between 2 and 40 K measured in applied magnetic fields of (a) 0.1-2.0 kOe and (b) 2-15 kOe. The temperature dependence of dM/dT curves derived from ZFC magnetization data measured in magnetic fields of (c) 0.1-2.0 kOe and (b) 2-15 kOe.

As shown in Fig. 2, a clear splitting occurs between zero-field and field-cooled data below 6.2 K indicating possible magnetic frustration. Ac magnetic susceptibility of $\text{DyNi}_{0.67}\text{Si}_{1.34}$ as a function of temperature was measured at frequencies ranging from 1 to 1000 Hz (Fig. 5). As mentioned above, for a typical AFM material the real component (χ') of the ac susceptibility is expected to show a sharp peak at T_N . However, broad peaks appearing in the real part confirm that $\text{DyNi}_{0.67}\text{Si}_{1.34}$ is not a typical antiferromagnet even at low fields. At the same time, the non-zero value of the imaginary (χ'') component indicates energy loss similar to the one observed in ErCo_4Ge_2 compound^[17]. The

peak amplitudes and temperatures of both real and imaginary components of ac magnetic susceptibility are sensitive to the frequency and the latter increases with increasing frequency. These observations underline the presence of a frustrated magnetic state, which is likely associated with the spin glass behavior. Because Ni and Si are randomly occupying the same $2d$ atomic site, the nearest neighbor environment of the magnetic Dy atoms changes from one unit cell to another, leading to magnetic frustration seen during the low field magnetization processes.

Fig. 5. The temperature dependence of (a) real (χ') and (b) imaginary (χ'') components of the ac susceptibility between 4 and 8 K at frequencies varying from 1 to 1000 Hz.

The full isothermal magnetization loop of $\text{DyNi}_{0.67}\text{Si}_{1.34}$ measured at 2 K as a function of applied magnetic field is shown in Fig. 6(a). Before the measurement, the sample was heated to the paramagnetic state and then cooled to the measurement temperature in zero field in order to ensure a demagnetized sample. Small field hysteresis was observed between the magnetization and demagnetization curves indicating that magnetic coercivity caused by magnetic frustration is present but very weak in this sample. As mentioned above the saturation moment derived from the $M(H)$ curve is about $18.4 \mu_B/\text{f.u.}$ or $6.13 \mu_B/\text{Dy}$ at 70 kOe for the $T = 2$ K (assuming non-magnetic Ni), which is much lower than the theoretical value of $\mu_s = 10 \mu_B/\text{Dy}$. Therefore, the magnetic structure

is not fully collinear even at 70 kOe, which is in general agreement with the complexity of magnetic structures reported for other Dy-Ni-Si compounds. Fig. 6(b) shows the magnetic field dependence of isothermal magnetization measured from 2 to 33 K.

Fig. 6. (a) Magnetization as a function of applied magnetic field from -70 to 70 kOe at 2 K. The 0-10 kOe field region is shown in the inset (highlighted by a dashed rectangle on the main plot); (b) The isothermal magnetization measured in applied magnetic fields from 0 to 70 kOe at temperatures ranging from 2 to 33 K. The inset shows the Arrott plots (see text) from 2 K to 18 K.

Some R-T-X compounds, such as HoNiSi ^[18], RNi_2Si_2 (R = Dy, Ho, Er) ^[19], HoCuSi ^[20], ErRu_2Si_2 ^[21], exhibit interesting first order field-induced AFM-FM metamagnetic transitions accompanied by large magnetocaloric effect. Usually, if the negative slope of the Arrott plot below T_N occurs in many curves, the field-induced AFM-FM metamagnetic transition is first order. On the contrary, if negative slope is observed for one temperature only or for a narrow range of temperatures the transition is second order. For example, the negative slope can be observed from 2 K to 8 K in the Arrott plots of $\text{HoNi}_{0.7}\text{Cu}_{0.3}\text{Al}$ alloy, which has first order field-induced AFM-FM metamagnetic transition. However, for $\text{HoNi}_{0.2}\text{Cu}_{0.8}\text{Al}$ alloy with the second order transition, the negative slope exists only at 2 K ^[22]. For better understanding of the magnetic transition in $\text{DyNi}_{0.67}\text{Si}_{1.34}$ alloy,

Arrott plots (M^2 vs. $\frac{H}{M}$) were derived from isothermal magnetization data (inset of Fig.6 (b)). The negative slope of the Arrott plot is observed only at 2 K confirming the occurrence of a second-order phase transition in DyNi_{0.67}Si_{1.34}.^[23]

Fig. 7(a) shows the isothermal magnetic entropy change ($-\Delta S_M$) of DyNi_{0.67}Si_{1.34} calculated from the isothermal magnetization data for several magnetic field changes (up to $\Delta H = 70$ kOe) using the Maxwell relation. Positive magnetic entropy change occurs below T_N in low fields where DyNi_{0.67}Si_{1.34} has a significant AFM component. The maximum value of $-\Delta S_M$ is 16.1 J/kg K for a field change of 70 kOe field at 10.5 K. It is comparable with the magnetocaloric effects reported for several other second-order materials in the same temperature region, such as ErRu₂Si₂ (17.6 J/kg K)^[21], Er₄NiCd (18.3 J/kg K)^[24], and ErCo₄Ge₂ (14.2 J/kg K)^[17]. The curve of $-\Delta S_M$ broadens asymmetrically towards high temperatures with increasing field.

Fig.7. Temperature dependences of the magnetic entropy change ($-\Delta S_M$) of DyNi_{0.67}Si_{1.34} for different applied magnetic field changes.

For SOPT materials, the relation between the magnetic entropy change $\Delta S_M(T)$ and the magnetic

field around T_C can be expressed as: $\Delta S_M|_{T=T_C} \propto H^n$ [25], while relative cooling power (RCP) obeys the scaling law and can be expressed as: $RCP \propto H^{1+\frac{1}{\delta}}$ [26], where n and δ are the critical exponents of the magnetic transition and depend on the magnetic state of the sample, and $RCP = -\Delta S_{Max} \times \delta T_{FWHM}$, where δT_{FWHM} is the full width at half-maximum of the magnetic entropy change $\Delta S_M(T)$ curve. There are four known models to explain SOPT, such as the mean-field model ($n = \frac{2}{3}$ and $\delta = 3$), 3D-Ising model (0.57 and 4.82), 3D-Heisenberg model (0.63 and 0.47) and the critical model (0.50 and 5.96). Fig. 8 shows the field dependence of experimental and fitted $-\Delta S_{Max}$ and RCP of $\text{DyNi}_{0.67}\text{Si}_{1.34}$ alloy. The fitted values of n and δ are 0.89 and 2.4, respectively, however the fitting is rather poor due to magnetic frustration and spin-glass state. Consequently, we were unable to determine a model with the clear fit to describe the phase transition of $\text{DyNi}_{0.67}\text{Si}_{1.34}$.

Fig. 8. (a) Field dependence of the maximum magnetic entropy change, (b) field dependence of the RCP. The exponential fitting (dotted line) used to determine the values of n and δ , respectively, has been performed as described in text.

IV. CONCLUSIONS

The magnetization study of $\text{DyNi}_{0.67}\text{Si}_{1.34}$ reveals the spin-glass state below 6.2 K. The Arrott plots

of $\text{DyNi}_{0.67}\text{Si}_{1.34}$ suggest the second order type of the transition. The frequency dependence of the real (χ') and imaginary (χ'') components of the ac magnetic susceptibility and the presence of magnetic frustration are in line with the random distribution of Ni/Si atoms in the crystal structure. For a magnetic field change of 0–70 kOe, the maximum values of ΔS_M is -16.1 J/kg K at 10.5 K.

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