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Intermartensitic Transformation in $Ni_{54.8}Mn_{23.2}Ga_{21.7}$ Ferromagnetic Shape Memory Alloy Nano Powder

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Abstract The Ni-Mn-Ga Ferromagnetic shape memory alloys are becoming an important element of sensor and actuator materials due to their large magnetic-field-induced strain and shape memory effect in recent years. The martensitic transformation temperature of the Heusler alloy system has been intensively studied. The $Ni_{54.8}Mn_{23.2}Ga_{21.7}$ irregular agglomerated nanoparticles were prepared by ball milling method, and characterized by x-ray diffraction, differential scanning calorimetry and transmission electron microscopy techniques. These particles reveal a disordered non modulated face centered tetragonal structure. For the first time, nanopowder has been found to undergo a sequence of temperature-induced intermartensitic transformation in addition to the martensitic transformation on cooling. It is believed that relaxation of internal stresses is the main issue in this case. The resistivity of the material has been measured on cooling from 450 K to 300 K. It is observed that the resistivity is maximum in the nano crystalline state and it decreases substantially with decrease of the temperature. Both amorphous and nanocrystalline material coexisted in the ball-milled sample, such a nanomagnetic structure is considered to be ideal for soft nanomagnetic material.

Keywords Ferromagnetic Shape Memory Alloy, Ni-Mn-Ga, Martensitic Transformation, X-Ray Diffraction, Vibrating Sample Magnetometer, TEM, Active Materials, Nanomaterials

1. Introduction

The unique properties of Ferromagnetic Shape Memory Alloys (FSMAs) make these materials, and in particular Ni-Mn-Ga attractive as actuator and sensor elements since materials exhibiting large displacements, fast response in time and good ductility for bio-medical applications [1, 2]. These qualities make FSMAs promising for applications such as underwater communications, structural morphing of unmanned aerial vehicles, acoustic attenuation and vibration control applications. Many of these applications require FSMA devices of compact size, high energy density, and broad frequency response. FSMAs are intermetallic compounds able to recover, in a continuous and reversible way, a predetermined shape during a heating/cooling cycle. From a microscopic point of view, this transformation consists in a transition from a crystallographic phase stable at low temperature, i.e. martensite, to a different crystallographic phase stable at high temperature, namely austenite. The critical temperatures at which transition occurs depend on the composition of the alloy, its thermo-mechanical history and the applied load. These critical temperatures indicate the onset and offset of the direct transition from austenite to martensite during cooling (Ms and Mf for martensite starting and finishing temperatures), and of the reverse transformation during which austenite is created from martensite (As and Af for austenite starting and finishing temperatures).

Close to stoichiometric Ni2MnGa Heusler-type, Ferromagnetic Shape Memory Alloys (FSMAs) have been extensively studied because of the large magnetic field induced strains. Ni-Mn-Ga undergoes a martensitic phase transition from high temperature austenitic cubic structure to a complex martensitic tetragonal structure on cooling. The formation of Inter Martensitic Transformations (IMTs) in the Ni2MnGa alloys is of special interest because the martensite crystal lattice is unstable in these alloys and is capable to induce intermartensitic transformations not only by the magnetic field [3] but also internal stress [4] and as a result of cooling [5]. Martensitic Transformation (MT) temperature is found to be modified by the occurrence of one or more intermartensitic transformations (IMTs) which can be observed during temperature variation or under mechanical stress [6-9]. MTs are accompanied by anomalies of the magnetic properties of the ferromagnets [10-11], it plays a vital role in physical metallurgy and shape memory phenomenon. Especially, sharp changes of the saturation magnetization and coercivity [12-13] were observed in the vicinity of the phase transition temperature. These effects are of interest in view of the possible applications such as

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magnetic storage devices.

The number of the martensite phases, their structures, and the sequence of their occurrence in the Ni-Mn-Ga system depending upon the composition and stress state [14-16]. Stress–temperature non-equilibrium phase diagrams were previously studied in Ni-Mn-Ga single crystals which did not show any spontaneous IMTs at zero external stress [17]. The IMTs are reported in the literature can be completely thermoelastic [4] or they can show a different sequence on cooling and heating [18]. As a general trend, the role of internal stresses in favouring the occurrence of IMTs is recognized [19-20]. The study of structural and physical property relationships has been always the center of material science. Nanotechnology is rapidly entering the world of active materials and taking them to the next level. Initial nanotechnology influenced, improvements to smart materials will be relatively simple changes to existing technologies. These new materials may incorporate nanosensors, nanocomputers and nanomachines into their structure. This will enable them to respond directly to their environment rather than make simple changes caused by the environment. Hence, nanoscale smart materials are used in the nano-scale engineering and system integration of existing materials to continuously develop better materials and better products.. Hence, more attentions have been shown to utilize these materials for practical applications.

The study of MT in Ni-Mn-Ga nano particles is a great challenge in many aspects and it is important to tune the MT above RT for practical applications. These new materials may incorporate nanosensors, nanocomputers and nanomachines into their structure and also these nanoparticles can be used to prepare the polymer composites for the applications of vibration damping and acoustic attenuation. Exploring nanostructured high surface- area materials for this purpose has recently attracted much interest. Our paper is presented in this context. The preparation of materials in nanocrystalline state significantly alters their physical properties. Though the phase transformation and crystal structural behavior of nanocrystalline Ni2MnGa particles have been studied to date [21, 22], a systematic investigation of martensitic transformation on ferromagnetic nanoparticles is still lacking. Martensitic transformations in alloys are known to be accompanied by changes in their physical properties. One of the properties that are most sensitive to these processes is the electrical resistivity. Therefore, in this work, we studied the temperature dependences of the resistivity of Ni54.8Mn23.2Ga21.7 powder. It is interesting to note that nanopowder has been found to undergo a sequence of temperature-induced IMT in addition to the MT for the first time. It is believed that relaxation of internal stresses is the main issue in this case. Besides, MT in nanosized particles is predominantly dependent on the strain energy and the particles size.

2. Experimental Procedure

High purity elements Nickel (99.99%), Manganese (99.8%) and gallium (99.99%) were used to prepare polycrystalline Ni-Mn-Ga alloy using conventional arc melting technique in argon atmosphere. To ensure better homogeneity of the samples, ingots were inverted and melted again and the process was repeated four times. The composition of the resultant powder was found to be $Ni_{54.8}Mn_{23.2}Ga_{21.7}$ using inductively coupled plasma optical emission spectroscopy (ICP-OES) technique. The ingot was mechanically crushed to the range of 1-5 mm in size followed by wet milling in a planetary ball to achieve nano sized particles. Ball mass-to-powder mass ratio of 10:1 was chosen to produce the alloy powders. The milling was performed with a rotation speed of 500 rpm in the presence of toluene. Finally, resultant powder was annealed at 973 K for 5 h to remove stress imparted during ball milling. The powder X-ray diffraction measurement has been carried out to study the crystal structures using Cu-Kα radiation at room temperature.

The powder samples allow for a texture free structure analysis in XRD pattern. Ni–Mn–Ga powder was produced by a grinding process and annealed at different temperatures such as 673 to 973 K for 5 h to relieve internal stresses which are imported during milling process. The optimum annealing temperature from this study was found to be 973 K, which revealed noticeable martensitic transformation. Hence, this optimized nano crystalline alloy was subjected to further characterization process. The standard four- probe method was used for ρ-T measurements in absence of magnetic field. Only cooling cycle has been recorded for this measurement. For ρ-T measurements, samples were first heated to 450 K and resistivity was measured as the temperature was slowly lowered up to 300 K. The transformation temperatures were measured in a Differential Scanning Calorimeter (PERKIN-ELMER) with cooling rates of 5 K min-1. The Vibrating Sample Magnetometer (VSM-5, TOEI industries) has been carried out to study the room temperature magnetic properties. The nanostructure of alloy is observed by using Transmission Electron Microscope.

3. Results and Discussions

3.1. Crystal Structure

XRD pattern of $Ni_{54.8}Mn_{23.2}Ga_{21.7}$ nano powder is shown in Figure 1. The reflection peaks (222)mart, (400)mart, and (004)mart of tetragonal structure is observed. This is corresponding to the typical non-modulated martensite structure (c/a < 1) [23-24]. The peak at $2\theta \sim 43^{\circ}$, which is related to the NM structure, is more pronounced in the XRD pattern. The crystal lattice parameters of the tetragonal martensite structure are a=b=0.6859 nm and c=0.6421 nm. Characteristic reflection peaks are appeared with broadening features indicating disordered crystal structure (see inset Figure 1) which is attributed to the high speed collision as a result in generation of defects [25]. This is common to all metallic system prepared by ball milling technique. The Ni2MnGa nano crystal display distorted fct structure, which has also been found by Wang et al [21]. The particle size of the sample was calculated using the Scherrer formula [26] and values of ε (strain) were obtained from different Bragg peaks. The average strain (ε) in the ball milled samples was calculated using the relation used in Ref. [4, 27]. The particle size is 72 nm and it is comparable with transmission electron microscopy measurement.

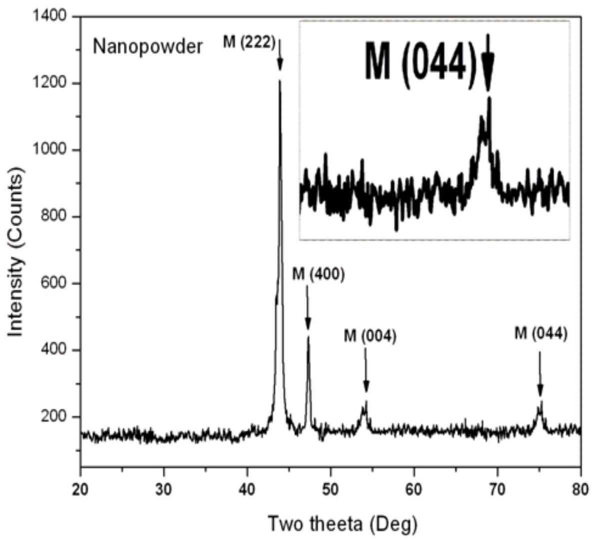

Figure 1. X-ray-diffraction pattern of the powder sample with size 72 nm after annealed at 973 K for 5 h.

In Ni_2MnGa, the martensitic transition was found to be thermoelastic and takes place at room temperature. This makes the material as a good candidate to be applied at room temperatures than current Ni–Mn–Ga actuators, which usually have a transition temperature of about 330-354 K. It was shown that mechanical crushing changes the structure of the martensite. U. Gaitzsch et al reported that a bulk sample is annealed to give a modulated structure; this structure is changed to NM by mechanically crushing the sample. The reason for the phase transformation upon crushing is the applied mechanical stress. So a stress induced intermartensitic transformation has happened here [28]. It is believed that relaxation of internal stresses is the main issue in this case [21]. We find that the internal strain is around 0.8% in the as milled sample that reduces to 0.17% after annealing at 973 K. Moreover diffraction peaks in Figure 1 indicates the presence of residual stress. Indeed residual stress induced tetragonal phase without any modulation has been observed earlier for Ni_2MnGa [14, 21-34].

3.2. Intermartensitic Transformation

The calorimetric measurement has adequate sensitivity to notice the occurrence of IMTs in Ni-Mn-Ga alloys [5, 17]. In Ni-Mn-Ga alloys, a sequence of martensitic and intermartensitic transformations on cooling was already studied in detail [5, 29] where the martensite structures involved in bulk alloys. DSC cooling curve is displayed for $Ni_{54.8}Mn_{23.2}Ga_{21.7}$ nanoparticles as shown in Fig. 2. It displays a sequence of MT and IMT transformations on cooling with temperature locating at 352 and 329 K respectively. IMT is the subsequent phase formed upon further cooling. The forward MT and IMT temperatures correspond to T_{M1} and T_{M2} respectively as marked by arrows and labeled in Figure 2. So far, several intermartensitic phases have been found, their structures and transformation temperatures depend on the levels of applied stress, temperature and the chemical composition [14,16, 18].The question arises about why the as-ground powder undergo completely different structural transformation paths during the martensitic transformation upon cooling because it concerns the condition of the internal stress. We found that the thermally induced intermartensitic transformation in this material is sensitive to the internal stress [4, 21, 28]. To the best of our knowledge, IMT for Ni_2MnGa nanoparticles do not exist in literatures. It is worth noting that temperature and internal stress induced IMT has been noticed in the nanocrystalline alloy. A small kink is observed near 428 K indicating curie transition (TC).

3.3. Electrical Resistivity

Figure 3, shows the temperature dependence of the resistivity for nanocryatalline $Ni_{54.8}Mn_{23.2}Ga_{21.7}$. The resistivity is maximum in the nanocrystalline state; this can be attributed to the small crystallite sizes, a high defect density, and disordering of the alloy. The resistivity decreases linearly with decrease of temperature. At higher temperatures, the resistivity decreases smoothly; however, near the Curie point (at 431 K) and transformation temperatures; the curve has a weak discontinuity in slope. MT and IMT are indicated by the two abrupt changes in the resistivity at the MT temperatures (T_{M1} and T_{M2}) of 350 and 332 K. At higher temperature in the range 450-400 K, resistivity remains virtually unchanged, although it decreases rapidly at lower temperatures. The character of the ρ (T) dependence is caused by the scattering of charge carriers on lattice defects (impurity atoms or atomic disorder), phonons (lattice vibrations) and by spin disorder. The phonon contribution increases with increasing temperature and is proportional to T at high temperature [34-38]. In magnetic materials, there is a large contribution to the resistivity due to spin disorder. The results of resistivity measurement can be compared with results obtained by DSC which confirm the intermartensitic transformation on cooling.

3.4. Hysteresis Loop

The coercivity is often seen as an important parameter if low losses are to be achieved and affected by most types of defects. This includes dislocations, grain boundaries, precipitates and non-magnetic particle distribution [25, 39-41]. This magnetic property is an important factor for the identification of soft magnetic behavior. The saturation magnetization (M_S) which is another important parameter from a magnetic point of view increases when particle size is decreased. This could be certainly attributed to the reduction in magneto crystalline anisotropy due to the grain refinement

It is well known that Ni-Mn-Ga alloy has easy and hard axis of magnetization, which depends on the crystallographic direction. Commonly c-axis is the easy axis of magnetization.

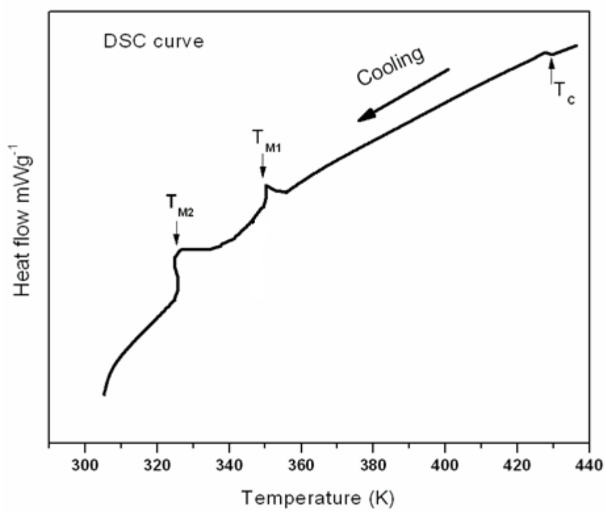

Figure 2. Shows a DSC cooling curve. $T_{M1}$and T_{M2} corresponds to MT and IMT respectively.

M–H measurements were carried out in the direction perpendicular to the magnetic field at room temperature to study the magnetic properties of the $Ni_{54.8}Mn_{23.2}Ga_{21.7}$ alloys using vibrating sample magnetometer as sown in Figure 4. Formation of hysteresis loop display that sample is ferromagnetic in nature. The small coercivity and saturation magnetization of $Ni_{54.8}Mn_{23.2}Ga_{21.7}$ sample at room temperature indicates that the nano crystal is magnetically soft. In common, some magnetic properties can be improved when the particle size is reduced to the nanoscale [26]. The ball milling technique has been carried out to reduce the particle size at nano scale which results in increase in the stress/strain. Hence, the coercivity and saturation magnetization is reduced in the nano crystalline sample. Both amorphous and nanocrystalline material coexisted in the ball-milled sample, such a nanomagnetic structure is considered to be ideal for soft nanomagnetic material. It has been observed that a coercive force (H_C) of 9.24 emu and saturation magnetization (M_S) of 6.97 emu from the hysteresis curve.

3.5. Bright Field Transmission Electron Microscopy Image

In order to perform Transmission Electron Microscopy observations, the particles of the ball-milled powder are spread over a copper grid with 400 µm mesh size with the carbon film. The samples placed in a FEI single tilt holder were investigated in a FEI Tecnai T20 microscope, which has a LaB6 gun, a super twin lens and was operated at 200 kV. For imaging, the standard bright field techniques were used. Figure 5, shows the transmission electron microscopy image of optimized nanopowder at room temperature. The size of the nano crystalline particles was calculated from the bright field image and found to be in between 64 and 78 nm. It shows the distribution of agglomerated particles. In this, the particles get cold welded together and form a clustered structure. It is believed that the compressive nature of the ball milling typically forms this type of structure [42-50].

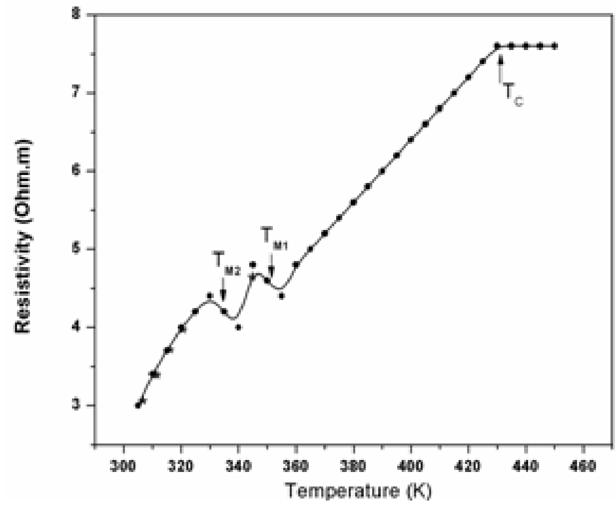

Figure 3. Temperature dependence of the electrical resistivity during cooling run.

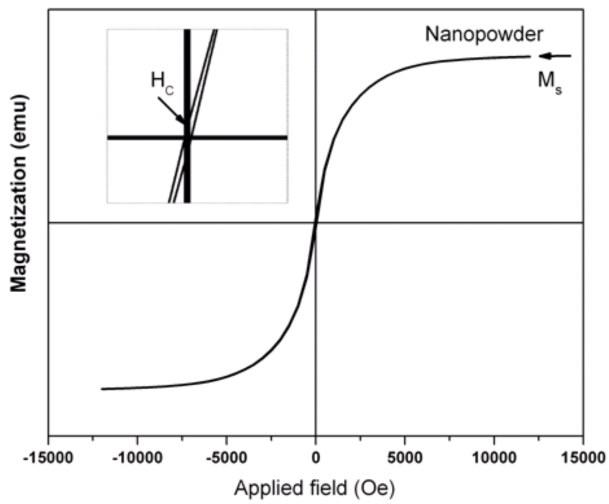

Figure 4. M–H curve of $Ni_{54.8}Mn_{23.2}Ga_{21.7}$ powder in the direction perpendicular to the magnetic field at room temperature. Inset Fig. shows the enlarged view of coercivity region.

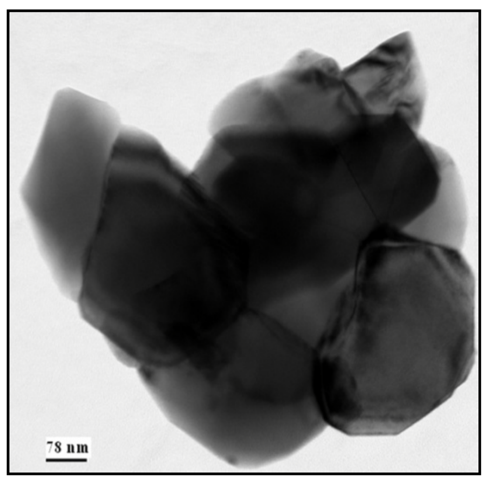

Figure 5. Bright field TEM image of $Ni_{54.8}Mn_{23.2}Ga_{21.7}$ nanopowder after annealed at 973 K for 5 h.

4. Conclusions

The transformation temperatures and electrical resistivities are analyzed. The $Ni_{54.8}Mn_{23.2}Ga_{21.7}$ powders are changed to non modulated martensite structure by mechanically crushing the sample at room temperature. Thermally induced intermartensitic transformation has been found by calorimetric and resistivity methods. These data stress the significant role of the internal stresses in the transformation sequence. Further accurate and specific measurements would be needed to elucidate the behavior of IMT and the particular crystal structures of the martensite. In absence of temperature dependent structural analysis on our sample, we are unable to comment on the structures of these two distinct martensitic phases present in this nanocrystal.

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